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Synthesis of $NbSe_2$ Nanoparticles: An Insight into Their Structural, Morphological and Optical Characteristics †

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Abstract: $NbSe_2$ nanoparticles, the representative member of the VB TMDCs group, have garnered significant attention due to their unique structural and optical properties, which make them promising candidates for various applications in nanoelectronics and optoelectronics. In this paper, the authors report on the synthesis of $NbSe_2$ nanoparticles via the sonochemical method at ambient temperature with controlled size, well-defined crystal structure, and desirable optical properties. The investigation of the compositional and structural analysis revealed that the synthesized nanoparticles are well-defined, near stoichiometry, with a hexagonal crystal structure belonging to the space group $P63_{mmc}$. The other morphological and optical characteristics of the synthesized nanoparticles studied through scanning electron microscopy (SEM), transmission electron microscopy (TEM), atomic force microscopy (AFM), UV-VIS NIR spectroscopy, etc., were discussed here.

Keywords: TMDCs material; $NbSe_2$ nanoparticles; sonochemical method; XRD and optical properties

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1. Introduction

Nanoparticles, characterized by their myriad distinctive and tunable properties at the nanoscale, have evolved into a compelling focal point of research within the discipline of materials science. Materials in the nano range exhibit distinctive attributes that differentiate them from their macroscopic counterparts, which make them potential candidates for a diverse array of technological applications. Transition metal dichalcogenides (TMDs), a diverse group of nanomaterials, have garnered a lot of interest in recent times.

The TMD family member $NbSe_2$ has become well-known because of its fascinating electrical, optical, and mechanical characteristics. Niobium (Nb) and selenium (Se) atoms are placed in a hexagonal lattice arrangement to form the layered substance [1,2]. $NbSe_2$, which is two-dimensional, exhibits extraordinary quantum confinement characteristics that have a wide range of uses in optoelectronics, nanoelectronics, catalysis, and energy storage [3,4]. The peculiar qualities of $NbSe_2$, including its three-dimensionality, as well as electrical, optical, and catalytic properties, make it a versatile substance with a variety of uses. The potential for the utilization of $NbSe_2$ nanoparticles (NPs) in advanced technologies and devices is expected to expand with ongoing research.

The development of $NbSe_2$ NPs provides an opportunity to investigate and modify the material's characteristics at the nanoscale. Each technique affects the size, shape, and crystallinity of the nanoparticles and has certain advantages; $NbSe_2$ NPs have been developed employing a variety of synthesis methods, such as wet chemical method, hydrothermal processes, and sol–gel procedures, all of which exhibit certain disadvantages [5–9].

To fully grasp the promise of $NbSe_2$ NPs for practical applications, it is essential to comprehend their structural, morphological, and optical properties [10–12]. The crystal structure, phase purity, and crystallite size of the nanoparticles can be determined by structural characterization methods including X-ray diffraction (XRD). Researchers can see and measure the particle size, shape, and distribution with morphological analysis using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). To better understand the material's interaction with light and its potential in optoelectronic devices, it is also helpful to understand its optical properties, such as the bandgap and light absorption spectra. The aim of this study is to advance our knowledge of $NbSe_2$ at the nanoscale by offering insightful information about its synthesis, structural, morphological, and optical features.

2. Materials and Methods

2.1. Chemicals

Niobium chloride dihydrate ($NbCl_5 \cdot 2H_2O$) [minimum assay 99%, AlfaAesar, Haverhill, MA, USA], hydrochloric acid (HCl) [minimum assay 35%, HiMedia Laboratories Pvt. Ltd., Mumbai, India], tri-ethanol amine (TEA) [minimum assay 98%, Sisco Research Laboratories (SRL) Pvt. Ltd., Mumbai, India], sodium selenite (Na_2SeO_3) [minimum assay 98.5%, HiMedia Laboratories Pvt. Ltd., Mumbai, India] and Hydrazine Hydrate [minimum aasay 80%, Sisco Research Laboratories Pvt. Ltd., New Mumbai, India] were used for synthesis.

2.2. Synthesis of $NbSe_2$ Nanoparticles by Sonochemical Method

Firstly, 1 M of $NbCl_5 \cdot 2H_2O$ was weighed and 5 mL of 50% diluted HCl was added to the beaker. The beaker was placed in an ultrasonic wave generator and exposed to ultrasound waves for 15 min under continuous stirring. While continuing ultrasonic treatment and stirring, 2.79 mL of TEA was added to the transparent solution obtained from step 1. Then, the separately prepared solution was added dropwise to the sonochemically treated solution while maintaining ultrasound exposure. Next, already prepared 2 M of Na_2SeO_3 was dissolved in 10 mL of de-ionized water and 4 mL of hydrazine hydrate was added to the solution in continuous manner. The total volume of the solution was adjusted to 50 mL by adding double distilled water under continuous ultrasonic treatment. The solution was allowed to undergo sonochemical treatment for an additional duration of 1 h, till it exhibited a dark brown coloration, which is indicative of the formation of nanoparticles. After completion of the sonochemical treatment, the dark brown solution was isolated and allowed to cool down to room temperature. To obtain the $NbSe_2$ Nps, the dark brown precipitates that had accumulated at the bottom of the beaker were filtered using Whatman filter paper (Grade 5, Sigma-Aldrich, Banglore, India). Following multiple washing steps, they were left to dry for 8 h at atmospheric temperature, thereby resulting in the production of dark brown $NbSe_2$ nanoparticles. The synthesized $NbSe_2$ nanoparticles were preserved in an appropriate air tight container at ambient conditions.

2.3. Characterizations

Stoichiometric elemental composition (EDS) and surface morphology of the synthesized $NbSe_2$ NPs were characterized using scanning electron microscopy (SEM) attached with Nova Nano SEM-450, FEL, Ltd. (SICART, Vallabh Vidyanagar, India). The unit crystal structure is determined by XRD using Rigaku Ultima IV Powder X-ray diffractometer (SICART, Vallabh Vidyanagar, India) with Cu-K_α radiation. The surface topography of synthesized $NbSe_2$ NPs was observed using high resolution electron microscopy (HRTEM) carried out with Thermo Scientific Talos F200i S/TEM (CSMCRI, Bhavnagar, India). The optical properties of synthesized $NbSe_2$ NPs were identified by Lambda 19 Perkin Elmer, UV-VIS NIR spectroscopy (SICART, Vallabh Vidyanagar, India).

3. Results and Discussion

3.1. Compositional and Structural Analysis

3.1.1. Compositional Analysis

The synthesized $NbSe_2$ NPs can be qualitatively analyzed using the non-destructive EDS approach. The elemental analysis helps to identify any contaminants present in he nanoparticles and determine the phase purity of $NbSe_2$ NPs. High phase purity is necessary for consistent and repeatable outcomes in a variety of applications. Here, the elemental analysis of synthesized $NbSe_2$ NPs were analyzed without any elemental restriction. The EDS spectrum of synthesized $NbSe_2$ NPs is shown in Figure 1, which displays the intensity of X-rays emitted by the sample at various energy levels. Based on the diffraction peak profile, all of the observable peaks of Nb-Se are clearly visible and are close to stoichiometric composition.

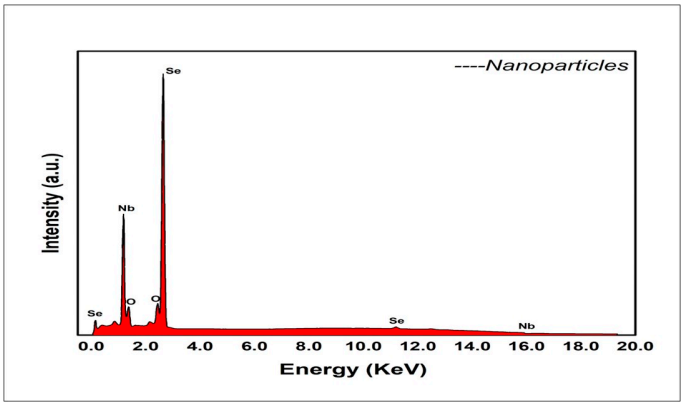


Figure 1. The EDS spectrum of $NbSe_2$ NPs synthesized via sonochemical method at room temperature.

3.1.2. Structural Analysis

The X-ray diffraction profile of sonochemically synthesized $NbSe_2$ NPs X-ray diffraction in the 2θ range of 10° to 80° is shown in Figure 2. The results showed that the hexagonal structure and high degree of crystallinity belonged to the $P6_{3/mmc}$ group. The determined lattice parameter was a = b = 3.446 Å and c = 12.55 Å, and the diffraction peak profiles of all the peaks were well matched with the standard JCPDS No. 01-072-0864.

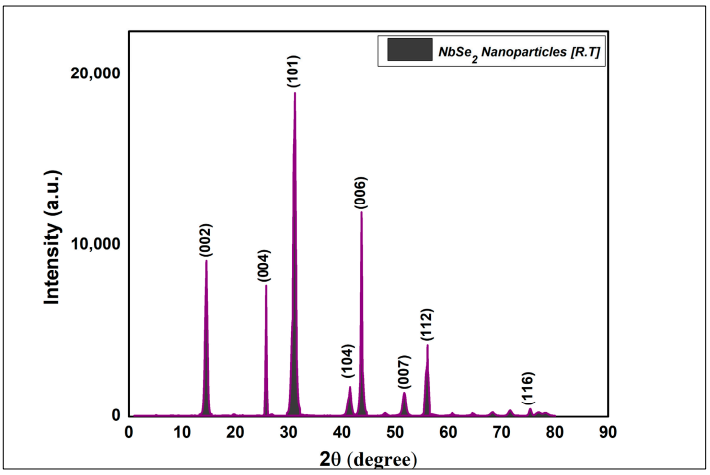


Figure 2. The XRD profile of $NbSe_2$ NPs synthesized via sonochemical method at room temperature.

This non-destructive technique reveals important details about the nanoparticle's atomic configuration and helps with the determination of the crystal structure, lattice parameters, and phase purity. The foundation of XRD is Bragg's law, which describes how a crystal lattice bends X-rays. Crystallite size and lattice parameters are crucial structural

characteristics of $NbSe_2$ NPs that significantly influence their properties and performance in various applications. This interference occurs only when the Bragg condition is met, which is obtained using [13]

$$n\lambda = 2d \sin\theta \quad (1)$$

where n is the order of the diffraction peak, λ is the wavelength of the X-rays, d is the interplanar spacing of the crystal lattice, and θ is the diffraction angle. The Scherrer equation is commonly used to estimate the crystallite size (D) using the full-width at half-maximum (FWHM) of the diffraction peak [14],

$$D = K \cdot \lambda / \beta \cdot \cos\theta \quad (2)$$

where K is the shape factor (typically taken as 0.89 for spherical nanoparticles), λ is the wavelength of X-rays, β is the FWHM of the diffraction peak, and θ is the diffraction angle. The average crystallite size from XRD analysis was found to be 15.12 nm.

The comprehensive structural characterization based on the analysis of crystallite dimensions and lattice attributes has augmented our comprehension of the correlations between the synthesis parameters, structural attributes, and properties of $NbSe_2$ NPs.

3.2. Morphological and Topographical Analysis

3.2.1. Scanning Electron Microscopy (SEM)

For morphological investigation and further comprehension of the physical properties, such as size, shape, and dispersion of $NbSe_2$ NPs, high-resolution scanning electron microscopy (SEM) was employed. Figure 3 depicts the surface morphology of synthesized $NbSe_2$ NPs, which displays the smooth surface and spherical nanoparticles. Additionally, it can be seen that the particles have a compact texture, without any pinholes or cracks and appear to be tightly packed.

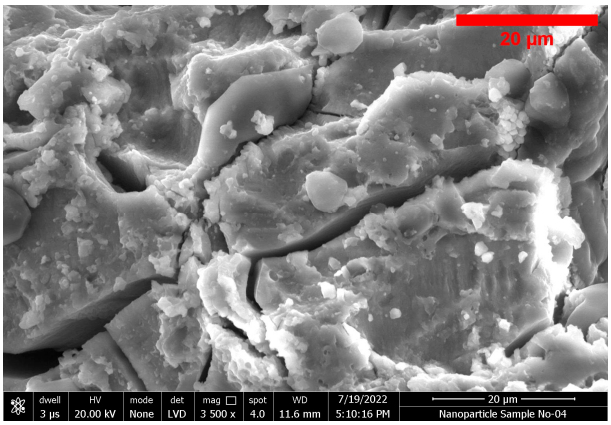
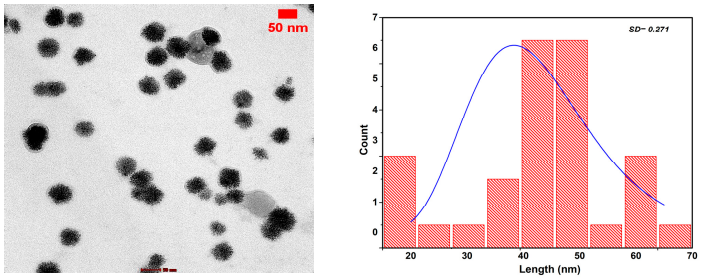


Figure 3. The surface morphology SEM image of NPs synthesized via the sonochemical method at room temperature.

3.2.2. Transmission Electron Microscopy (TEM)

In order to study the topographical and internal makeup of synthesized $NbSe_2$ NPs, high-resolution transmission electron microscopy (TEM) was utilized. Figure 4a,b shows the topographic image and particle size distribution curve of $NbSe_2$ NPs, respectively. Figure 4a demonstrates that the nanoparticles are crystalline and have a spherical shape with an average particle size of 42.62 nm. Figure 4b displays the particle size distribution curve of $NbSe_2$ NPs, with a standard deviation of 0.271.

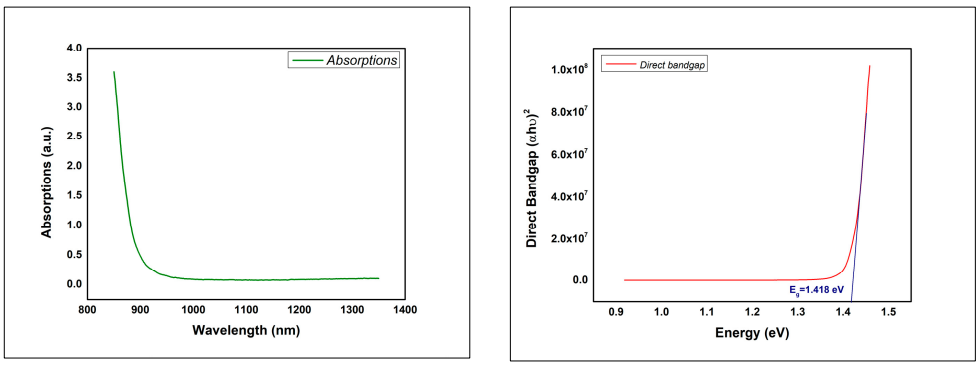


(**a**) (**b**)

Figure 4. (**a**,**b**) shows the TEM image and particle size distribution curve of $NbSe_2$ NPs synthesized via the sonochemical method at room temperature.

3.3. Optical Analysis

UV-Vis spectroscopy is frequently used to characterize the optical properties of synthesized $NbSe_2$ NPs, where the intensity of the absorption is displayed as a function of wavelength. It offers insights into the bandgap and absorption of the nanoparticles. Here, for the absorption spectra analysis, the sample was ultrasonically dispersed in acetone and recorded in the spectral wavelength of 800 to 1400 nm, as shown in Figure 5a.



(**a**) (**b**)

Figure 5. (**a**,**b**) shows the absorption and optical direct bandgap of $NbSe_2$ NPs synthesized via the sonochemical method at room temperature.

The absorption spectra in Figure 5a show strong absorption in the spectral wavelength range of 850–950 nm. The absorption edges for synthesized $NbSe_2$ NPs are seen at 910 nm. The bandgap of the synthesized $NbSe_2$ NPs has a direct bearing on this range. The intercept of the linear section of the plot with the energy axis is used to calculate the bandgap (E_g). Figure 5b shows the optical energy bandgap to be 1.418 eV for $NbSe_2$ NPs as calculated from the absorption spectrum using the near band edge absorption relation. Tauc plot analysis is one approach that is frequently used to find the bandgap. The absorption coefficient (α) as a function of the photon energy (hv) on a linear scale or $(hv)^2$ on a logarithmic scale is shown to create the Tauc graphic. The bandgap energy of 1.418 eV places the material in a favorable range for applications in photodetectors and solar cells. In the case of photodetectors, the material's bandgap falls within the visible spectrum, making it sensitive to a broad range of incident light. For solar cells, the 1.418 eV bandgap is well-suited for harvesting sunlight, as it corresponds to the energy range of visible light.

4. Conclusions

In essence, this study advanced the understanding of sonochemically synthesized $NbSe_2$ NPs at room temperature, providing valuable insights into their synthesis, structure, morphology, and optical properties. The analysis revealed a high phase purity of $NbSe_2$ NPs, thereby laying the foundation for consistent performance in various applications.

Structural investigations, including XRD analysis, elucidated the crystallographic arrangement of $NbSe_2$ NPs. The hexagonal structure with well-matched diffraction peak profiles underscored the nanoparticles' high degree of crystallinity. Morphological analysis of the SEM and TEM images showed that the nanoparticles were smooth, had a spherical morphology and a compact arrangement, thereby emphasizing their potential for diverse applications. The optical analysis shed light on the nanoparticles' absorption capabilities within the spectral wavelength. The characterization of the material with a bandgap of 1.418 eV opens up exciting possibilities for its application in photodetectors and solar cells. The material's position within the visible spectrum range, coupled with its electronic properties, makes it a promising candidate for optoelectronic devices. Further research is warranted to fully exploit its potential for these applications.

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Conflicts of Interest: The authors declares that they have no conflict of interest.

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