



REGULAR ARTICLE

Optical and Structural Properties of ZnSe Nanoparticles

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(Received 13 August 2025; revised manuscript received 23 October 2025; published online 30 October 2025)

Zinc Selenide nanoparticles have been synthesis on glass substrate by chemical bath deposition. The optical properties were investigated and the annealing temperature at 250°C for one hour on the transmittance spectra and the energy bad gap were observed, transmittance was gradually decreased and band gap was increased. The samples were measured by FESEM, XRD pattern and EDX. FESEM at various magnification powers (15 000, 30 000, 60 000 and 120 000 X) ZnSe films have been viewed. ZnSe nanoparticles were distributed with different sizes (26.61 – 41.50 nm) and they were taken the form of nanosheets. ZnSe prepared samples were analyzed by XRD and they were exhibited polycrystalline structure; the results were showed that the samples have been mixture of cubic and hexagonal structure. After annealing at 250°C, we observed that the intensity of planes is low, a small shift in the diffraction angles and average crystal size was decreased. From elemental compositional of ZnSe films by EDX, the distinctive peak was due Zn element at 1 KeV and the appearance other elements such as Se, C, N, O, Si, S and Ca with different percentage.

Keywords: ZnSe, Nanoparticles, Structure, Optical, XRD.

DOI: [10.21272/jnep.17\(5\).05012](https://doi.org/10.21272/jnep.17(5).05012)

PACS numbers: 61.46.Df, 78.67.Bf

1. INTRODUCTION

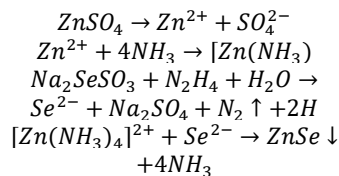
Zinc selenide is the II-VI family semiconductor [1]. It has several interesting features such as chemically inert, high shock resistance, non-hygroscopic, high transmittance to infrared [2, 3]. ZnSe thin films were basic material in the optical electronic devices such as lenses output couplers, transistors, lasers, solar cells, etc. and use in solid state lighting such as light emitting diodes and industrial application [4-5]. These films were characterized by owning a wide energy gap 2.7 eV at room temperature [6] and a large binary energy 21 MeV [7, 8]. ZnSe has been existed in two structural patterns: cubic and hexagonal [9]. There are several techniques for chemically deposition of thin films such as molecular beam epitaxy [10], Vacuum evaporation [11], electro deposition [12], chemical bath deposition [13-14], chemical vapor deposition [15].

Chemical bath deposition is an appropriate method for thin films because it can be a low cost and simplicity [16]. In this work we prepared ZnSe nanoparticles on glass substrates by CBD at room temperature and study the optical (transmittance & band gap) before and after annealing at 250°C in 1 hour and the structural properties of ZnSe thin films by X-ray diffraction, scanning electron microscopy and energy dispersive X-ray have been investigated.

2. EXPERIMENTAL DETAILS

ZnSe nanoparticles have been synthesis on glass substrates CBD at room temperature. They cleaning using ethanol, deionized water and acetone because removed any contamination from substrate's surface and washed again with distilled water for three minutes after that by wash with an acetone solution for three minute using ultrasonic wave followed by awash again with distilled water for tow minute and finally dried with an air dryer. ZnSe films are prepared by mixing 0.5 M selenium in 10 ml of distilled water with 0.5 M of sodium sulfide. Then we boil the mixture for three hours. The last solution used of the element zinc selenide. Then the mix this solution with 0.5 M of zinc sulphate in 10 ml of distilled water with 0.5 ml ammonia and 1 ml hydrazine hydrate and it is placed over magnetic stirrer for 1 min. then we put the samples inside the solution for 24 hours. After that we gently lift them out of the solution and leave them for an hour to ensure they dry. Finally, the samples were placed inside an evacuated glass tube and put in an electric oven at 250°C for one hour.

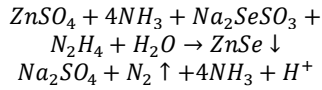
The chemical reaction is given by [17]



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Simplified overall equation for a chemical reaction can be given by:



3. RESULTS AND DISCUSSIONS

The variation in the transmittance spectra for wavelengths 300 – 1000 nm were shown in Fig. 1 (a, b). It appears with convergence of edges and a clear decrease at the edge of the range from 320 – 400 nm in the ultraviolet region. The highest transmittance for ZnSe films was recorded at 86.8% and we noticed that it gradually decreases to reach 80.7% in the infrared region with the annealing temperature at 250°C for 1 hour due to partial evaporation of Se or formation of new defects such as the presence of oxygen or moisture. This result is agreement to [18]. It is worth noting that electronic transitions occur in the visible region and are direct transitions.

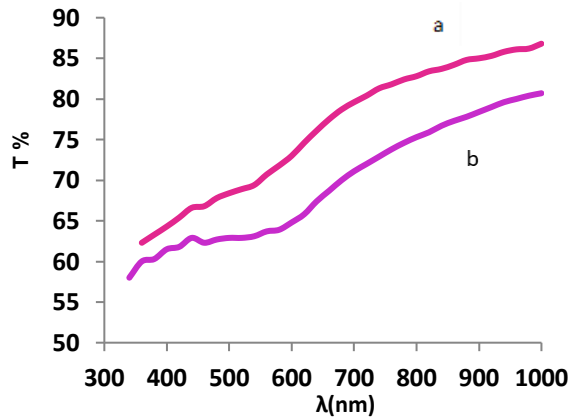


Fig. 1 – Transmittance spectra variation with wavelength (a) before annealing (b) after annealing

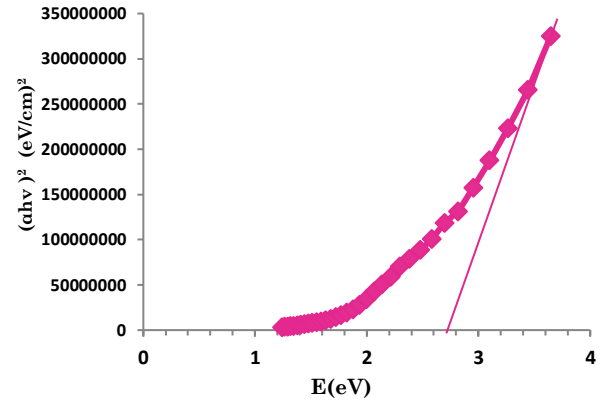
Band gap energy was calculated from the $(ah\nu)^2$ versus of photon energy as demonstrated in Fig. 2 (a, b) [19, 20, 21]. The energy gap 2.7 eV corresponding to the bulk E_g of ZnSe [22], the E_g values increased from 2.7 – 2.8 eV at annealing temp. 250°C. An increment in E_g may be due to the decrease in the defect and the crystallite sizes observed in XRD results and after annealing, the crystallinity has been improved which it was also agree with XRD analysis. The equation that relates the absorption coefficient to photon energy is given by [23].

$$ah\nu = A(h\nu - E_g)^{1/2} \quad (1)$$

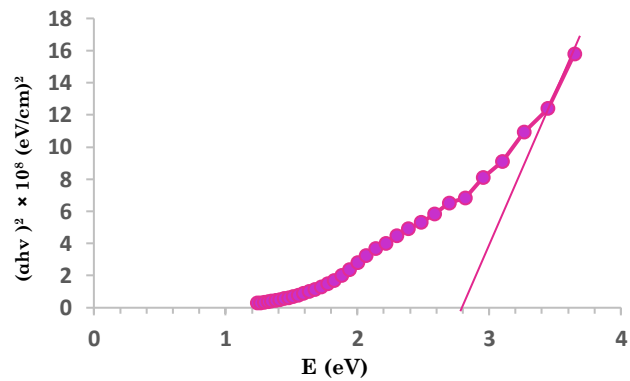
where h – Plank constant, ν – frequency, A – constant

In the FESEM images of the ZnSe films with different magnification powers (15 000 X, 30 000 X, 60 000X and 120 000 X) as shown in Fig. 3. It was observed that the nanoparticles covered all the surface, suspended on the substrates and densely distributed with various sizes. The shape of these particles is a rough disc, the upper part of

which contains interlocking cells ends with long thin tips. The images at (60 000 & 120 000 X), it seems that the particles were taken the form of two-dimensional nanosheets with sizes ranging from 26.61 to 41.50 nm.



a



b

Fig. 2 – $(ah\nu)^2$ vs. photon energy

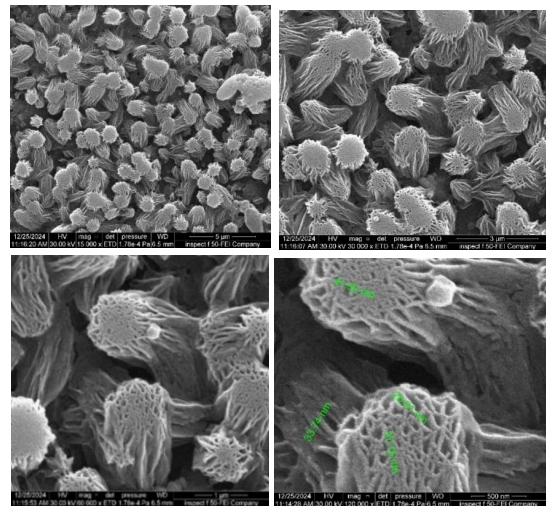


Fig. 3 – FESEM image for ZnSe nanoparticles syntheses at different magnification powers

The XRD analysis of ZnSe nanoparticles are shown in Fig. 4 and listed in Table 1. The samples were exhibiting polycrystalline behavior, it seems that the peaks were sharper that can be indexed by the intense peak (111) and other planes (200), (220), (222) and (400) of the cubic structure which they were corresponding to the diffraction angles 27.39°, 30.18°, 43.93°, 56.12° and 65.54° respectively according to JCPDS of ZnSe, the results have been agreement [16]. We also observed (100), (101), (102) & (103) due to the hexagonal structure.

After annealing the samples at 250°C for 1 hour, all peaks become lower intensity and the appearance another peak of diffraction 74.58° at (420) plane. A small shift in the diffraction angles occurs, the reason for this is that during the process of twin crystal formation, a small displace occurs between the adjacent multiple planes. Moreover, the treatment of thermal decreased the defect and increased the crystallinity of the ZnSe nanoparticles. Average crystal size was decreased from 9.20 to 8.96 nm after annealing, which was calculated by the Scherrer equation (2) [24-25].

$$D = \frac{K\lambda}{\beta \cos \theta} \quad (2)$$

where λ – wavelength equal 1.54 (Å), β – width at half maximum and θ – Bragg's diffraction angle.

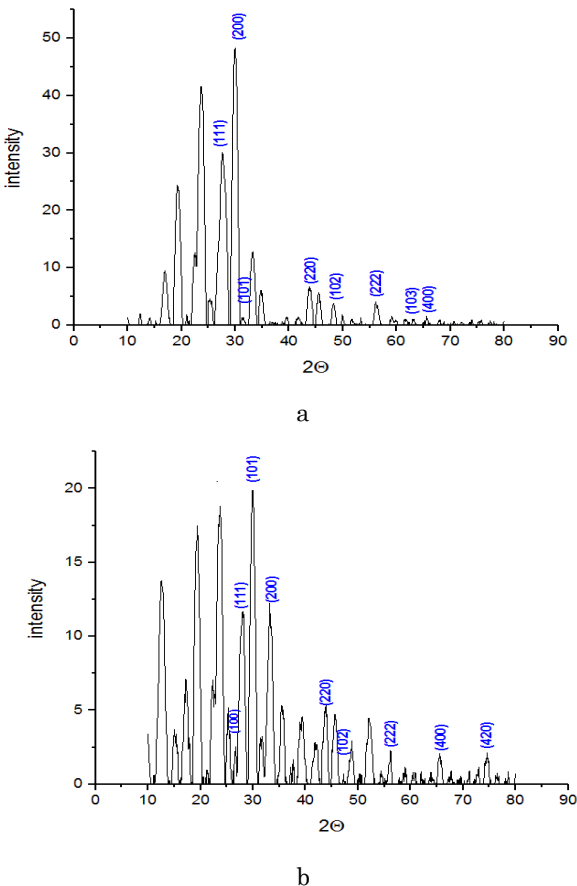


Fig. 4 – XRD pattern for ZnSe (a) synthesized (b) annealed

Table 1 – Results XRD for prepared and annealed ZnSe nanoparticles

Samples	(hkl)	2θ (deg.)	d(Å)	FWHM	D/nm	Average crystal size/nm
Before annealing	(111)	27.39398	3.28847	0.38670	3.6900	9.2090
	(200)	30.18467	2.83857	0.09330	15.3863	
	(101)	31.39534	2.83857	0.10000	14.4074	
	(220)	43.93296	2.06435	0.50400	2.9653	
	(102)	48.30369	1.90697	0.12000	12.6644	
	(222)	56.12175	1.63100	0.20000	7.8571	
	(103)	62.91381	1.47489	0.18670	8.7030	
	(400)	65.54035	1.42476	0.17330	7.9986	
After annealing	(100)	26.64630	3.35523	0.07330	19.4389	8.9654
	(111)	27.9274	3.19457	0.52000	5.71310	
	(101)	30.02051	2.95603	0.17330	8.2894	
	(200)	31.5800	2.84649	0.21330	6.7543	
	(220)	43.9429	2.06919	0.32000	4.6713	
	(102)	47.25718	1.91834	0.00000	0.0000	
	(222)	58.74829	1.56806	0.20000	7.9527	
	(400)	69.5417	1.35105	0.12000	14.0625	
	(420)	74.5896	1.27056	0.36000	4.8410	

The result of the elemental compositional analysis of ZnSe nanoparticles has been showed the presence of six main peaks, three for zinc located at the energies (1, 8.5 and 9.5 KeV). The distinctive peak with high intensities due to zinc element and three peaks for elemental Se located at 1.2 ,11.1 and 12.5 KeV. Also, we noticed that the appearance of a number of elements with distinctive peaks such as C, N, O, Si, S and Ca [26] as shown in Fig. 5. The relative analysis was depicted in Table 2.

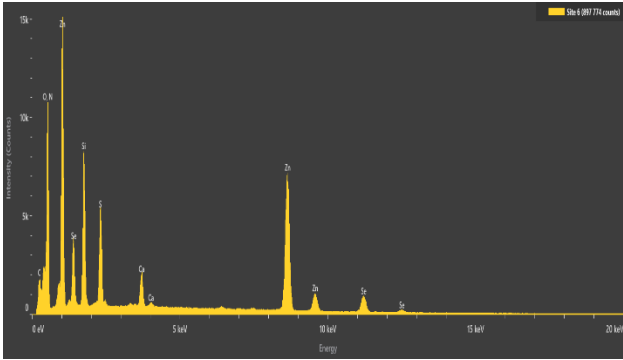


Fig. 5 – EDXA of ZnSe nanoparticles prepared by CBD

Table 2 – Elemental atomic and weight percentage of ZnSe nanoparticles by EDX analyses

Element	Atomic %	Atomic % Error	Weight %	Weight % Error
C	18.6	0.3	11.0	0.2
N	17.8	0.4	12.3	0.2
O	47.1	0.3	37.0	0.3
Si	5.0	0.0	6.9	0.0
S	2.6	0.0	4.1	0.0
Ca	0.7	0.0	1.4	0.0
Zn	6.7	0.0	21.6	0.1
Se	1.5	0.0	5.7	0.1

4. CONCLUSION

A direct energy gap value of ZnSe nanoparticles prepared by CBD method were found 2.7 eV which it was equal to the value of bulk band gap ZnSe. While the E_g value for the samples annealed at temperature 250°C at one hour has been increase to 2.8 eV. The transmittance spectra for ZnSe were reached about 87% in infrared region. From FESEM images of the ZnSe nanoparticles show the appearance of two-dimensional nanosheets stacked together with nanoscale sizes (26.61 – 41.50 nm). The synthesized

samples of ZnSe were occurred polycrystalline structure with mixture of cubic and hexagonal by using XRD pattern. At annealing temperature 250°C, the intensity of all planes is low and appearance other plane at (420), also the average crystal size has the maximum 9.20 nm and it decreases to 8.96 nm. The spectrum of elemental compositional analysis of ZnSe was showed that the element Zn appears with the highest intensity and is centered at energies 1 and 8.5 KeV.

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Оптичні та структурні властивості наночастинок ZnSe

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Наночастинки селеніду цинку були синтезовані на скляній підкладці шляхом хімічного осадження у ванні. Було досліджено оптичні властивості та температуру відпалу при 250°C протягом однієї години на спектрі пропускання та енергетичну погану щільну, пропускання поступово зменшувалося, а ширина забороненої зони збільшувалась. Зразки вимірювали за допомогою FESEM, XRD та EDX. Були переглянуті плівки ZnSe FESEM при різних ступенях збільшення (15 000, 30 000, 60 000 і 120 000 X). Наночастинки ZnSe були розподілені з різними розмірами (26,61 – 41,50 nm) і отримали форму нанолістів. Зразки, отримані з ZnSe, аналізували XRD, і вони демонстрували полікристалічну структуру; Результати показали, що зразки були сумішшю кубічної та гексагональної структур. Після відпалу при 250°C ми спостерігали, що інтенсивність площин низька, невеликий зсув у кутах дифракції та середній розмір кристала зменшився. З елементного складу плівок ZnSe за допомогою EDX характерний пік був зумовлений елементом Zn при 1 KeV і появою інших елементів, таких як Se, C, N, O, Si, S і Ca з різним процентним вмістом.

Ключові слова: ZnSe, Тонкі плівки, Наночастинки, Структура, Оптика, XRD.