

# Preparation and Characterization of CdS and PbS Chalcogenides Compounds and n-CdS/p-PbS photodiode

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ARTICLE INFO	ABSTRACT				
Keywords	In this study, a chemical bath deposition method (CBD) is used to				
Chemical bath deposition	synthesize CdS nanocrystalline thin films onto FTO substrate using two				
method. CdS, PbS,	temperatures of 70 and 80 °C for 1h. PbS nanocrystalline thin films are				
photodiode	also prepared using CBD using temperature of 35 °C for 2h. XRD				
	measurements confirmed that the prepared CdS and PbS thin films are				
	polycrystalline with hexagonal phase for CdS and cubic for PbS.				
	Scanning Electron Microscope images show increasing particles size of				
	CdS thin films with increasing temperature of preparation and the films				
	became more adherent on the substrates without pinholes or vacancies.				
	Optical properties are investigated through UV-Visible Spectroscopy				
	and found decreasing in the transmittance of prepared thin films with				
	increasing temperature. Energy band gap calculated for CdS and PbS				
	nanocrystalline thin films found with values higher than bulk value				
	indicating to due to the quantum confinement effect.				
	Photoluminescence spectra of prepared CdS nanocrystalline thin films				
	appeared sharp emission beak at around 550 nm for all samples				
	indicating high crystallinity. Photoresponse measurements of the				
	fabricated photodetector showed a significant sensitivity to visible light				
	at zero applied voltage, indicating that the fabricated device is a self-				
	powered photodetector. The device was highly photosensitive of				
	16833, responsivity of 1.44 mAW <sup><math>-1</math></sup> , and a low rise/fall time of				
	0.284/0.298 s.				

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#### 1. Introduction

The scientific revolution of nanotechnology began with the arrival of the scanning tunneling microscope (STM) in the 1980s [1]. After the discovery of NTs in 1991 by the Japanese scientist Sumio Iijima, nanostructured materials have attracted great interest due to their optical, electrical, magnetic and other properties [2]. Many non-carbon structures have been synthesized during the past few years, and the study of the nanostructures of inorganic Chalcogenides has become one of the main topics in the nanoscale field. Chalcogenides refer to the compounds sulfides, selenides, and tellurides. Chalcogenides have been used in a wide range of applications such as optoelectronic devices and solar cells due to their excellent optical and electrical properties [3]. CdS is One of the important Chalcogenides semiconductors of the n-type belongs to group (II-VI), its crystal structure is either hexagonal, which is more stable at room temperature, or cubic. It has a direct energy gap of the order of (2.42ev) at room temperature. It is used as a permeable layer in the manufacture of solar cells with various materials of p-type, light-emitting diodes and photodetectors. It can be prepared using several methods such as Chemical deposition, Vacuum evaporation, Sputtering and other methods [4, 5], on the other hand, Lead sulfide is one of the IV-VI chlorine semiconductors with a narrow direct energy gap of about 0.41 eV at room temperature [6]. It has a relatively large excitation Bohr radius. This provides a strong quantitative confinement for electrons and holes, and then the energy gap can be controlled by controlling the particle size according to the effective mass model [7, 8]. This property makes it desirable for new applications such as sensors and solar cells [9]. The use of inexpensive methods for preparing high-quality nanocomposites has attracted the attention of researchers. Chemical bath deposition is one of the chemical methods used for this purpose. This method allows controlling growth factors such as sedimentation rate, film thickness, and crystal quality by changing the temperature, PH, and concentration of the precipitation solution. The most important features of this method are the strong adhesion of the membrane to the base, the possibility of preparing the membrane over a wide area and on different bases, and the possibility of preparing the membranes at low temperatures (usually less than 100 °C) [10,11].

# 2. Experimental

# 2.1 Preparation of nanocrystalline CdS thin film

The FTO substrates are first washed with water and a cleaning fluid to get rid of any stuck-on materials. The FTO substrates were kept in dilute HCL for 5 min and then cleaned using an ultrasonically with acetone, ethanol and propanol each for 10 minutes and then washed with deionized water, respectively. Then, substrates are dried using a convection oven at 150 °C for 15 minutes. The CdS thin films were deposited on the FTO substrate using the CBD chemical bath deposition method in alkaline aqueous solution. Cadmium acetate  $[Cd(CH_3COO)_2]$  and thiourea [(NH<sub>2</sub>)<sub>2</sub>CS] were used as a source of cadmium ions (Cd<sup>+2</sup>) and sulfur ions (S<sup>-2</sup>), respectively, and ammonium acetate (NH<sub>2</sub>CH<sub>3</sub>COO) was added as a regulator to control the reaction rate [12], and ammonia solution to adjust PH. The aqueous solution is prepared by dissolving 0.05 M of cadmium acetate and 0.5 M of ammonium acetate in 90 ml of deionized water, and dissolving 0.05 M of thiourea in 10 ml of deionized water and mixing well for 10 minutes at room temperature. The ammonia solution is added dropwise to gradually adjust the pH until it reaches 10. where the total volume of the reaction solution was 100 ml. The samples were placed vertically inside the beaker at two bath temperatures (70, 80 °C) at a fixed time of 60 min. When all the primary solutions are mixed, chemical reactions begin between cadmium ions and sulfur ions and the color of the solution turns yellow, indicating the formation of cadmium. After sedimentation, the substrates were removed from the solution and then ultrasonically rinsed with deionized water for 2-3 min to remove loosely bound CdS particles. Then the samples were washed with deionized water using ultrasonically and dried using hotplate at 50 °C for 5 minutes. Preparation steps of nanocrystalline CdS thin films using CBD are shown in Fig.1.



Figure1: Schematic of nanocrystalline CdS thin films prepared via CBD approach.

#### 2.2 Preparation of nanostructured PbS thin films

PbS particles were deposited on the surface of CdS films prepared on FTO substrates by CBD method to manufacture n-CdS/p-PbS photodetector. Briefly, the solution is prepared by dissolving 0.1M of lead nitrate Pb(NO<sub>3</sub>)<sub>2</sub> and 0.2M in of thiourea [SC(NH<sub>2</sub>)<sub>2</sub>] separately and mixing well for 10 minutes at room temperature and then mixing them together to produce a homogeneous solution. The PH of the solution was fixed at 11 by adding sodium hydroxide (NaOH), so that the total volume of the reaction solution was 100 ml. The samples were placed vertically inside the beaker at a temperature of 35°C for a settling time of two hours. After 10 minutes of mixing all the initial solutions, the color of the solution turns to a dark gray indicating the formation of PbS thin films. After sedimentation, the samples were removed from solution, ultrasonically rinsed with non-ionized water for 5 minutes, and dried using hotplate at 50 °C for 5 minutes. The steps for preparing PbS nanocomposite thin films using CBD are shown in Fig. 2.



Figure 2: Schematic of nanocrystalline PbS thin films prepared via CBD approach.

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#### 2.3 Fabrication of the Photodetector

The n-CdS/p-PbS photodetector is fabricated by dposition of PbS nanoparticles thin films on the surface of CdS films Preparated on FTO substrate by CBD method, then depositied of aluminum metal (Al) on the surface of PbS films by **a** thermal evaporation method. hen Connect the connecting wires with the FTO substrate and aluminum metal using Silver paste. FTO substrate was used as the electrode with dimensions of  $1.5 \times 1$ cm, and the effective area of the fabricated photodetector was 1cm<sup>2</sup>.



Figure 3: Schematic of fabrication CdS/PbS thin films photodetector

#### 3. Results and discussion

# 3.1 Structural analysis: X-ray diffraction

The structural properties of CdS and PbS films prepared by CBD method were studied using X-ray diffraction technique. From Fig.4, The X-ray diffraction pattern of CdS thin film prepared via the CBD exhibits two diffraction peaks (002) and (112) at the angle ( $2\theta$ =26.5) and ( $2\theta$ =51.5), respectively (JCPDS 80-0006), appear for films prepared on FTO substrate at temperatures (70, 80 °C) upon deposition of two layers. These peaks indicate that the prepared films are polycrystalline and have a hexagonal structure and this agrees with [13]. As we note an increase in the angle intensity ( $2\theta$  = 26.5) towards the (002) plane of the prepared films with increasing temperature [14]. On the other hand, all PbS thin films prepared via the same method on FTO substrate have diffraction peaks at the levels (111), (200), (220), (311), (222), (400), (331), (420), (422) corresponding to the diffraction angles  $2\theta$  (25.74, 29.83, 42.83, 50.77, 53.26, 62.36, 68.80, 70.80, 78.75), respectively, which indicate that the PbS films are polycrystalline and have a cubic structure according to (JCPDS card no. 05- 0592). the peaks are sharp, which indicates that the prepared films have good crystal structures, and this agrees with [15, 16]. from the results of XRD that the preferred growth direction of the resulting compound is along the level (200). The crystalline grain size and The lattice constants of the CdS and PbS nanostructured films were calculated and as shown in Table 1. The result of the ratio (c/a) was close to the typical ratio (1.623) for the hexagonal CdS compound and this result is consistent with (JCPDS 80-0006).



Figure 4: XRD pattern (a) CdS thin films with preparation temperature of 70° and 80° and (b) PbS thin films

# 3.2 Surface Morphology properties

The surface properties of CdS and Pbs thin films prepared on FTO substrate by CBD method were studied using scanning electron microscopy (FE-SEM). Fig. 5 shows the micrographs of CdS films prepared on FTO at 70 and 80°C, respectively. As seen from the micrographs the surface morphology of CdS films prepared at a temperature of 80 °C is spherical nanoparticles uniformly growing on the surface of the substrate, and the films are homogeneous and well adhered also without any holes or cracks due to the slow deposition rate and the ion by ion mechanism of membrane formation. We also note that the deposition temperature significantly affects the surface morphology of CdS films, as the microscopic images of the film prepared at

70 °C show some holes and voids, and with the increase in the deposition temperature, the density of the holes decreases significantly and the film prepared at 80 °C becomes dense and empty of holes and this is consistent with [17, 18]. Increasing the deposition temperature leads to an increase in the size of the particles and subsequently a decrease in the voids. Increasing the deposition temperature is an effective way to reduce the voids in CdS films, and the uniformity of the films can be controlled using parameters such as substrate difference, deposition time, the concentration of reactants, pH, and deposition temperature [19-23]. This type of morphology increases the interaction of light with the surface resulting in improved photosensitivity [19]. Using Image J program, the average diameter of CdS and PbS thin films prepared at different temperatures were calculated, as it was observed that the diameter increased with increasing temperature as shown in Fig 5.

Compound	T(°C)	a(nm)	c(nm)	c/a	G.S (nm)
CdS	70	0.4073	0.6651	1.6329	19.7
	80	0.4084	0.6669	1.6329	34.5
PbS	35	35			35

Table 1 Calculated structural parameters of CdS and PbS thin films deposited by CBD

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Figure 5: FE-SEM and diameter distribution of CdS prepared at temperature of (a)70 °C and (b) 80 °C and PbS thin films

### **3.3 Optical Properties**

UV-Vis spectrometry is utilized to observe the optical properties such as transmission, reflection, and optical band gap of films that provide useful information to analyze some features concerning the band structure of materials. The optical properties of nanostructured CdS thin films were studied using the absorbance and transmittance spectrum at room temperature for the wavelength range 400-800nm. From fig 6, we notice from the absorbance and transmittance spectrum of CdS thin films prepared on FTO substrate at different temperatures that the absorbance increases and the transmittance decreases with increasing deposition temperatures. The band gap energy of CdS thin films was calculated by extrapolating the straight line portion of the plot  $(\alpha h\nu)^2$  to the energy axis and was found to be 2.534 eV and 2.435 eV (70 and 80 °C). Found that the energy gap is decreased with increasing temperature which could be due to increasing the grain size with temperature [24]. The obtained optical band gap values were higher than the bulk value (2.42 eV at 515 nm). The blue-shifted absorption compared to the absorption of bulk CdS can be attributed to the excitonic absorbance due to the quantum confinement effect [25]. The absorption spectra declared in Fig.7a. Energy gap also calculated in the same manner used Tauc method as shown in Fig.7b with value of 1.6 eV. The obtained value of energy gap is agreed with those obtained by other researchers [26].



Figure 6: (a) The absorbance spectra (inserted with Tauc plot), and (b) optical transmittance of CdS thin film

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Figure7: (a) The absorbance spectrum of PbS nano thim films and (b) the Tauc plot of PbS nano thin films

#### 3.4 Photoluminescence (PL)

The quality of films influenced by the factors such as impurities, defect centers, and recombination of surface states can be analyzed by PL studies. Furthermore, information involving different energy states lies between valence and conduction bands that are responsible for radiative recombination due to cadmium and/or sulfur defects are also achieved by PL studies [27] Fig .4 shows The PL spectra at room temperature of CdS films prepared on FTO substrate at

70 and 80°C with a fixed deposition time of 60 min. Use an exciting wavelength at 450 nm for all samples. For both deposition temperatures, there was a sharp emission peak at 550nm for the prepared CdS films and this agrees with [28, 29]. It returns to the peak of the beam edge which is attributed to the inter-beam transport of charge carriers or the recombination of bound excitons [30]. We note that the intensity decreases when the temperature is increased for the prepared samples. We also observe a redshift of the PL photofluorescence peak for all samples prepared. The observed red shift cannot be attributed to the low degree of crystallinity of the nanoparticles. On the contrary, this red shift indicates that the perfect crystal structure of CdS nanoparticles leads to many surface defect states and this is in agreement with [31].



Figure 7: PL spectra of the CdS thin films prepared with different temperature

#### **3.5 Photosensing properties**

The photosensing properties of the prepared n-CdS/p-PbS photodetector were studied by using light of intensity (3.5W/m<sup>2</sup>) at room temperature and by applying 0V bias voltage. These rays were beamed perpendicular to the photodetectors with a distance of 10cm. Response and sensitivity were calculated using equations:

$$R_{\lambda} = \frac{I_{ph}}{P_{in}} \tag{1}$$

$$S = \frac{I_{ph} - I_d}{I_d} 100\%$$
 (2)

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The rise and fall time were also calculated. The results obtained through the optical sensing properties of the n-CdS/p-PbS photodetector prepared from two layers of CdS at a temperature of 70 °C showed an increase in current from 0.003 to 0.098s when shining the light, and this explains the high sensitivity value which reached 3167 %, and the detector prepared at a temperature of 80 °C showed a light current of 0.508s and a high sensitivity of 16833% and by applying a bias voltage of 0V as shown in figure 8. The temperature of the CdS thin films has a clear effect on the sensitivity of the n-CdS/p-PbS optical detector, as we notice an increase in the sensitivity with increasing temperature.



Figure 8: Photoresponse of photodetectors under visible light

# 4. Conclusions

The XRD results exhibited that CdS thin film a hexagonal crystal structure with a preferential orientation along (002) with a crystallite size varied from 19.7 to 34.5 nm. From the FE-SEM analysis, uniform, smooth, dense thin films with less pinhole, and spherical grains are shown. The optical band gap was blue-shifted compared with the band gap value of bulk CdS. The PL results for all samples prepared CdS films showed a sharp emission peak in the green band 550 nm and its intensity decreased with increasing deposition temperature. Photodetectors prepared at 80 °C have a higher sensitivity those prepared at 70 °C up to 16833%.

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# تحضير وخصائص مركبات الجالكوجينات CdS و PbS و الثنائي n-CdS/p-PbS

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#### المستخلص

طريقة الترسيب بالحمام الكيمائي استعملت لتحضير اغشية المركب CdS النانوية التركيب على قواعد FTO باستعمال درجتي حرارة 70 و 80 سيلزية لساعة واحدة. اغشية المركب PbS النانوية التركيب أيضا حضرت بنفس الطريقة وبدرجة حرارة 25 سيليزي ولساعتين. فحوصات حيود الاشعة السينية اكدت ان الاغية المحضرة متعددة التبلور بتركيب سداسي للمركب CdS و مكعب للمركب PbS. صور المجهر الالكتروني الماسح أظهرت زيادة الحجم الحبيبي لاغشية المركب CdS الرقبة مع زيادة و مكعب للمركب PbS. صور المجهر الالكتروني الماسح أظهرت زيادة الحجم الحبيبي لاغشية المركب CdS الرقبة مع زيادة و مكعب للمركب PbS. صور المجهر الالكتروني الماسح أظهرت زيادة الحجم الحبيبي لاغشية المركب CdS الرقبة مع زيادة درجة حرارة التحضير وأصبحت الاغتية اكثر التصاقية بالقواعد واختفاء الثقوب او الفراغات. الخصائص البصرية تم فحصها من خلال امتصاص الضوء ووجد نقاصان بنفاذية الاغشية الرقبقة المحضرة بزيادة درجة الحرارة. فجوة الطاقة التي فحصها من خلال امتصاص الضوء ووجد نقاصان بنفاذية الاغشية المحضرة بنين دلالة على ظاهرة الحرارة. في الماسح المعني الاغتية الرقبقة المحضرة بزيادة درجة الحرارة. فكرى البصرية تم فحصها من خلال امتصاص الضوء ووجد نقاصان بنفاذية الاغشية الرقبقة المحضرة بزيادة درجة الحرارة. فحوة الطاقة التي الاغشية المركبين دلالة على ظاهرة الحصر الكمي. طيف فحسبت للاغشية الرقبقة المحضرة بزيادة مرحة الحرارة. فجوة الطاقة التي الانبعائية المحضرة بزيادة درجة الحرارة. فحوصات النورين المابعين الانبعائية الموينية لاغشية الرقبقة المحضرة وجد انها اكبر من فجوة الطاقة القباسية للمركبين دلالة على ظاهرة الحصر الكمي. طيف الانبعائية الضوئية الدفوئية المركبين دلالة على ظاهرة الحصر الكمي على الانبعائية الموئية المحضرة دلالة على كون الجهاز المصنع كاشف ذاتي القدرة. الاستجابة عالية الضوئية المركبي عند عدم تسليرا الموينية الكاشف المحضرة مالحضرة الموئية الكواشف المحضرة دلالة الموئية الكواشف المحضرة دلالة على كون الجهاز المصنع كاشف ذاتي القدرة. الاستجابة الموئية الكاشف المحضرة دلالة على كون الجهاز المصنع كاشف ذاتي القدرة. الاستجابة الموئية الكاشف المحضرة دلالة الكاشف المحضرة دلالة على كون الجهاز المصنع كاشف ذاتي القدرة. الاستجابة الموئية الكواشف المحضرة دلالة الكاشف المحضرة الكاشف المحضرة دلالة على كون الجهاز المصنع كاش