

## Preparation and Characterization of CdS Thin Films at Varying Molarities Using Chemical Spray Pyrolysis



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### ABSTRACT

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*CdS thin films, Chemical Spray Pyrolysis (CSP), Group II-VI, CdS structural characteristics*

In this study, thin films of Cadmium Sulfide (CdS) were prepared on glass substrates at a temperature of 350°C using the Chemical Spray Pyrolysis (CSP) technique. Cadmium chloride and thiourea were used as sulfur sources. The structural properties and surface morphology of the CdS thin films were characterized using X-ray diffraction (XRD) and atomic force microscopy (AFM). The XRD analysis revealed that all the prepared films exhibited a polycrystalline hexagonal structure, with multiple peaks and angles, and a preferred orientation along the (002) direction. The XRD results also indicated that an increase in the solution concentration led to a significant increase in intensity and a slight increase in the crystallite size of the CdS film. AFM analysis was performed to investigate the surface structure of the films. The results showed that as the molarity increased, the surface roughness, grain size, and root mean square (RMS) roughness increased. This can be attributed to the increased molar concentration, which resulted in a slight increase in droplet size. However, despite these surface roughness variations, the prepared films can still be used as windows in Optical applications and Gas Sensors. The optical properties, including transmittance, absorbance, and band gap energy, were determined by measuring the absorbance spectrum using a UV-Visible Spectrophotometer within the wavelength range of 300 nm to 1100 nm. The CdS films exhibited transmittance values ranging from approximately 76% to 35%. The band gap energy of the films ranged between 1.85 eV and 2.38 eV. It was observed that as the concentration increased, the transmittance decreased and the absorbance spectrum increased, indicating a narrower band gap energy. Our findings suggest that controlling the molarity in the Chemical Spray Pyrolysis method can influence the structural and optical properties of CdS thin films, potentially impacting their use in Optical applications and Gas Sensors. So, the optimal molar concentration for the preparation of the CdS film was found to be 0.03 M for both cadmium chloride and thiourea. This is achieved through the results of textural and optical properties.

## 1. INTRODUCTION

Cadmium Sulfide (CdS) compound is a semiconductor material classified as a chalcogenide due to its sulfur (S) content. It belongs to Group II-VI of the periodic table. (CdS) exists in the form of yellow-colored crystals and is widely used in various applications, including solar cells [1], practical applications, optical sensors, and other fields [2]. (CdS) is considered an n-type semiconductor, but it can be converted into p-type thin films by doping with impurities such as copper (Cu) or indium (In) atoms [3]. (CdS) has a direct band gap energy of approximately 2.4 eV at a temperature of 300 K [4]. CdS thin films are polycrystalline and exhibit a crystalline structure of either zincblende or hexagonal type, as shown in Figure 1. These films possess suitable electrical properties for solar cell applications [5]. Numerous experiments have shown that CdS is an ideal compound when used as a window layer in thin-film solar cells, such as those based on CuInGaSe or CdTe [6]. Its cut-off wavelength occurs at approximately 520 nm in the visible spectrum, indicating that the film absorbs

light at wavelengths shorter than 520 nm, while longer wavelengths are transmitted through the film [7]. (CdS) thin films can be prepared using various methods, including thermal evaporation under vacuum [8], Chemical Spray Pyrolysis [9, 10], chemical bath deposition, and others [11, 12]. In our study, we relied on the Chemical Spray Pyrolysis technique for preparing CdS films due to its low cost, ease of use, ability to produce large-area films with good adhesion and uniformity. Additionally, this method allows for the mixing of two or more materials with different melting points [13]. Many researchers have studied and prepared CdS membranes using various preparation methods and other parameters. We will mention some of them in upcoming paragraph.

The two studies were conducted by Pandya and Raval [14]. By preparing CdS films on glass bases at a temperature of 200°C using deposited by chemical technique with different molar concentration. Structural tests showed a multiple crystal structure with a cubic shape and the preferred dominant direction (111) with a crystal size ranging between (1.5 - 2) nm with increasing concentration. Molar also showed that

optical tests have a direct energy gap between (2.4-2.7) eV and decrease with increasing concentration. Researchers also studied it by studying and preparing CdS films on glass substrates at various temperatures using a thermochemical decomposition method. The synthetic results (XRD) showed that the films have a polycrystalline structure and are hexagonal in shape, with the dominant (002) direction, regardless of about the substrate temperature. As for visual tests, they confirmed that the membranes prepared at different temperatures have a direct gap between (2.44 - 2.58) eV. The best membrane obtained was at a temperature of 300°C [15].

The purpose of the study is to obtain the best CdS film for use as a window layer in optical and sensing applications in future projects. This is done by changing the molarity of the cadmium chloride and thiourea solutions while keeping the other experimental parameters constant, such as temperature, spraying rate, spraying pause time, and the height of the spraying device.

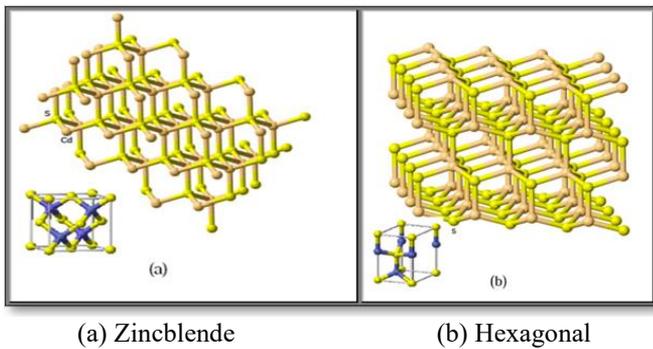


Figure 1. Crystal structure of CdS [5]

## 2. MATERIALS AND METHODS

### 2.1 Glass substrate preparation

Glass slides of standard quality with a Chinese origin (Glass Slide Microscopic REF 217102) were utilized in this study. The slides were approximately cut into square pieces with dimensions of 2.5cm×2.5cm×1mm. These glass pieces were employed for the purpose of depositing thin films on them. Prior to deposition, the slides underwent the following cleaning procedures:

1. Cleaning the slides with water and detergent powder, then placed in an ultrasonic cleaner for 10 minutes.
2. The cleaned slides are immersed in a glass beaker filled with distilled water and subjected to ultrasonic cleaning for 10 minutes, followed by drying.
3. The slides are submerged in a glass beaker filled with 99.99% purity ethanol, and again placed in an ultrasonic cleaner for 10 minutes.
4. Afterward, the slides are dried using hot air and a specialized drying paper, making them ready for use in the Chemical Spray Pyrolysis (CSP) system.

### 2.2 Preparation of spray solutions

Spray solutions with varying concentrations are prepared by dissolving cadmium chloride salts with thiourea supplied by Chemical Ltd, Poole, England, with a purity of 99.6%, in distilled water of specific purity as listed in Table 1. Each solution is prepared according to the following equation [16].

$$W_t = M_{wt} \times M_t \times \frac{V}{1000} \quad (1)$$

where:

$W_t$ : Weight required to be dissolved (g).

$M_t$ : Molar Concentration (M).

$M_{wt}$ : Molecular Weight (g/mol).

V: Distilled water Volume (ml).

Table 1. Percentage of Salts used in preparation of (CdS) thin films membrane per molar concentration

The Molar Concentration (M)	Net Weight (CH <sub>4</sub> N <sub>2</sub> S) (g)	Net Weight (CdCl <sub>2</sub> .H <sub>2</sub> O) (g)	Water Used for Dissolving Salt (ml)
0.03	0.23	0.604	100
0.05	0.38	1.0066	100
0.07	0.53	1.41	100

After preparing the solutions, then put it on the magnetic stirrer for 25 minutes to ensure the homogeneity of the solution. equal volumes of cadmium chloride and thiourea solutions with a molar concentration of 0.03 were taken to prepare a (CdS) membrane. A quantity of 60 ml of the solution was sprayed onto glass substrates placed on an electric heater at a temperature of 350°C. This is because at this temperature good films were obtained in terms of crystallization and crystal growth. Nozzle height (30 ± 2cm), with a duration of time (25 s), spray rate (5s), stop time (20s), and air pressure rate (3 bar).

## 3. RESULTS AND DISCUSSION

### 3.1 X-ray diffraction

Figure 2 illustrates the X-ray diffraction (XRD) results of the (CdS) film at molarities of 0.03, 0.05, and 0.07 M. It can be observed that there are diffraction peaks corresponding to crystallographic planes, indicating a polycrystalline structure. The observed diffraction peaks correspond to crystal planes (100), (002), (101), (110), and (112) with diffraction angles of 24.7°, 26.5°, 28.14°, 44.3°, and 51.7°, respectively.

The dominant preferred orientation is along the (002) plane, indicating a hexagonal crystal structure. These results are consistent with the International Card JCPDS:77-2306. It was also found that increasing the concentration of the solution led to a significant increase in intensity and a slight increase in the crystallite size of the prepared (CdS) film.

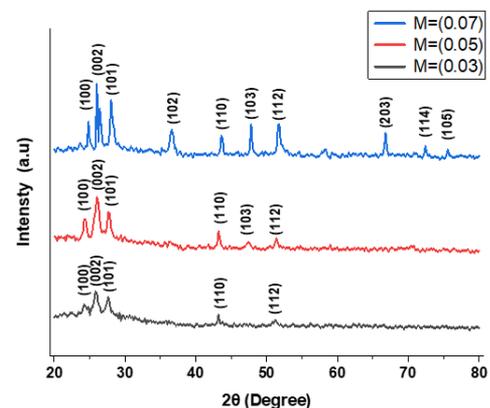


Figure 2. XRD spectrographs for CdS thin film at molarities of (0.03, 0.05, 0.07) M

The best homogeneity was achieved at a concentration of 0.03 M. Furthermore, increasing the concentration to 0.07 M resulted in the appearance of low peaks corresponding to crystal planes (102), (103), (203), (114), and (105) at angles of 36.9°, 47.811°, 66.793°, 72.586° and 75.615° respectively.

The reason for the clarity of the diffraction peaks with increasing molar concentration is due to the increase in the proportion of the sulfur ion (S<sup>2-</sup>) combining with the cadmium ion (Cd<sup>2+</sup>) in the formation of a compound (CdS). The reason may be attributed to the transformation of the crystal structure from its hexagonal form to a cubic form, and this is consistent with the study of researchers [17].

Table 2 presents the crystallographic planes, diffraction

angles, and crystalline size calculated using the Debye-Scherrer equation [18], for molarities M (0.03, 0.05, 0.07).

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

where:

D: Crystal size.

$\beta$ : Full Width Half Maximum (FWHM) in (rad).

K: Scherer's constant (0.94).

$\lambda$ : X-ray wavelength (Å°).

$\theta$ : Angle of Diffraction (Bragg's Angle).

**Table 2.** Results of XRD for CdS thin film at molarities of (0.03, 0.05, 0.07) M

Sample	Molarity (M)	(hkl) Hexag	2 $\theta$ deg	$\theta$ deg	d nm	FWHM rad	$\beta$ deg	D nm	D <sub>av</sub> nm
CdS	0.03	(100)	24.2	12.1	0.37	1.144	0.086	7	8.3
		(002)	25.8	12.9	0.35	0.886	0.015	10	
		(101)	.276	13.8	0.32	1.074	0.019	8	
CdS	0.05	(100)	24.3	12.2	0.37	0.721	0.013	12	11.3
		(002)	26.0	13	0.32	0.829	0.014	10	
		(101)	27.6	13.8	0.32	0.696	0.012	12	
CdS	0.07	(100)	.249	12.4	0.36	0.683	0.012	13	17.3
		(002)	.264	13.2	0.34	0.619	0.011	14	
		(101)	.282	14.1	0.32	0.358	0.006	25	

**Table 3.** Structural parameters for CdS thin film at molarities of (0.03, 0.05, 0.07) M

Sample	Molarity (M)	t nm	D nm	$\beta$ deg	$\theta$ deg	( $\epsilon$ )	N <sub>o</sub> nm <sup>-2</sup>	N <sub>L</sub>	$\delta$ nm <sup>-2</sup>
CdS	0.03	400	7	0.085	12.1	0.021	1.2	57.1	0.02
			10	0.015	12.9	0.004	0.4	40	0.01
			8	0.019	13.8	0.005	0.8	50	0.015
CdS	0.05	400	12	0.013	12.2	0.003	0.23	33.3	0.007
			10	0.014	13	0.004	0.4	40	0.01
			12	0.012	13.8	0.003	0.23	33.3	0.007
CdS	0.07	400	13	0.012	12.4	0.003	0.18	30.8	0.006
			14	0.011	13.2	0.0026	0.145	28.6	0.005
			25	0.006	14.1	0.0015	0.098	16	0.002

**Table 4.** AFM data for CdS at molarities of (0.03, 0.05, 0.07) M

Sample	Molarity (M)	Ave Diameter (nm)	RMS Roughness (nm)	Ave Roughness (nm)
CdS	0.03	55.65	10.46	7.771
CdS	0.05	92.64	34.02	28.07
CdS	0.07	170.8	48.64	41.21

Table 3 illustrates the structural parameters calculated from the following equations:

- **Dislocation density ( $\delta$ ) [19]:**

$$\delta = \frac{1}{D^2} \quad (3)$$

$\delta$ : Dislocation density in (mm<sup>-2</sup>).

- **Number of film layers (N<sub>L</sub>) [20]:**

$$N_L = \frac{t}{D} \quad (4)$$

where:

t: Film thickness (mm).

- **Number of crystals (N<sub>o</sub>) [21]:**

$$N_o = \frac{t}{D^3} \quad (5)$$

- **Micro strain ( $\epsilon$ ) [19]:**

$$\epsilon = \frac{\beta \cos\theta}{4} \quad (6)$$

where:

$\beta$ : Full Width Half Maximum (FWHM).

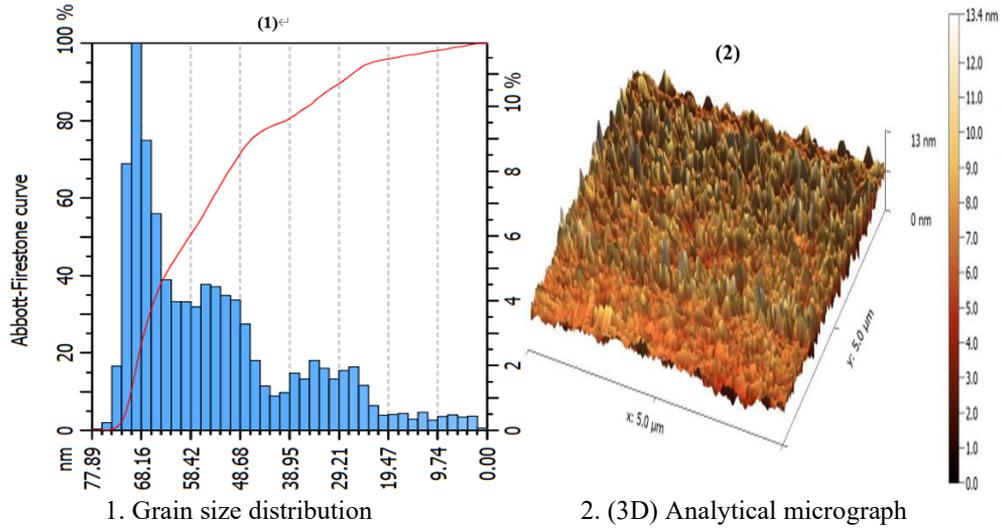
$\theta$ : Angle of Diffraction (Bragg's Angle).

### 3.2 Atomic Force Microscope (AFM)

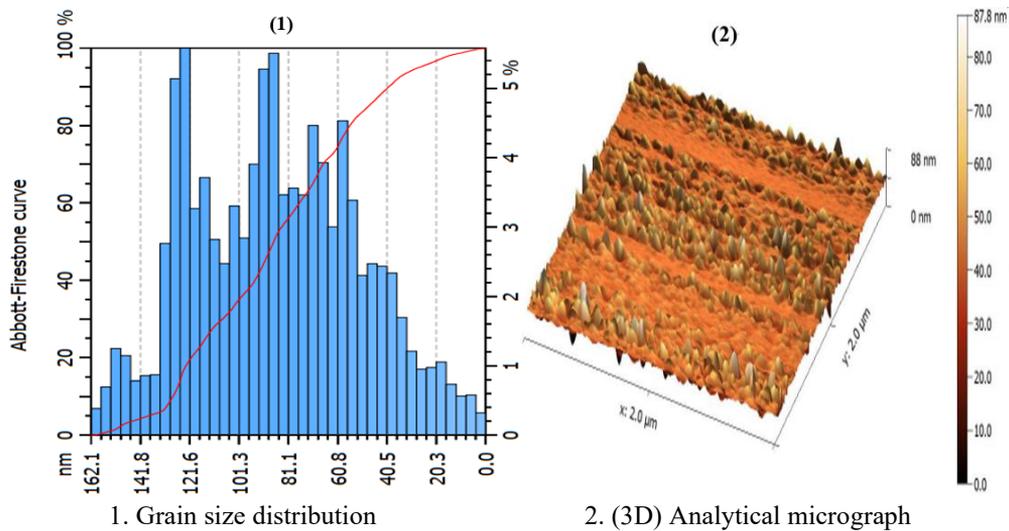
Figure 3 Illustrates three-dimensional (3D) atomic force micrographs and the grain size distribution of the (CdS) film prepared at a temperature of 350°C using the Chemical Spray Pyrolysis (CSP) technique with different molarities of 0.03, 0.05, 0.07 M. It is evident that as the molarity increases, the surface roughness, grain size, and root mean square (RMS) value also increase. This can be attributed to the higher molar concentration, which slightly increases the droplet size. However, despite this, the prepared films can be used as

windows in optical applications and gas sensors due to their high uniformity and smooth surface, which reduces reflectivity and enhances transmittance. The best film was

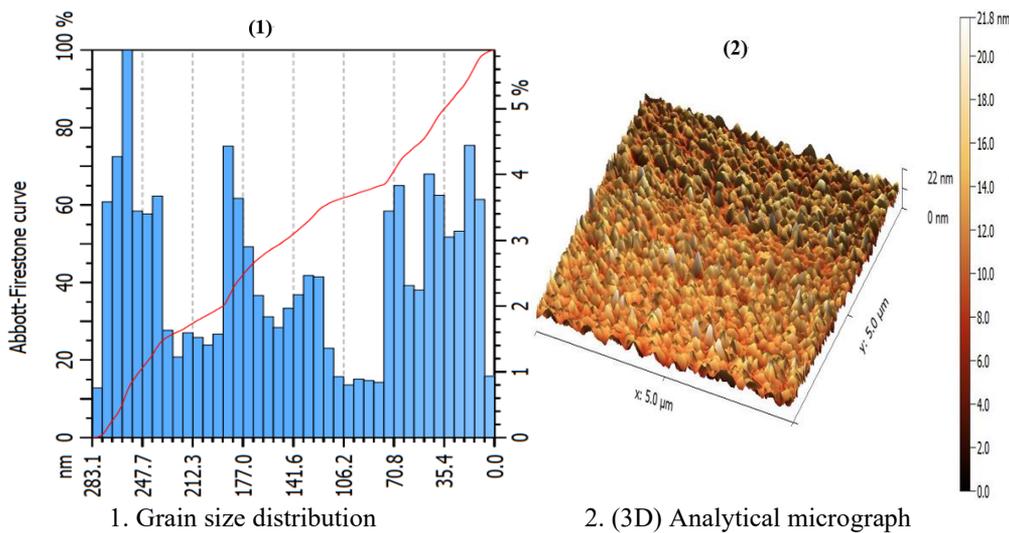
obtained at a molarity of 0.03M This study is consistent with the researchers [22]. Table 4 presents the detailed AFM examination results for the (CdS) film as mentioned above.



**Figure 3a.** Results of AFM for CdS film at Molarit of (0.03) M



**Figure 3b.** Results of AFM for CdS film at Molarit of (0.05) M



**Figure 3c.** Results of AFM for CdS film at Molarit of (0.07) M

### 3.3 Optical characteristics

The optical properties of the prepared (CdS) films were studied using a UV-Vis spectrophotometer to measure the transmittance and absorbance spectra as a function of wavelength within the range of 300-1100 nm. From these spectra, various optical properties such as the absorption coefficient and energy gap can be calculated.

Figure 4a shows the transmittance spectrum of (CdS). The relationship between transmittance (T) as a function of wavelength ( $\lambda$ ) was plotted. The transmittance was around 76% at a wavelength of 520 nm in the visible spectrum. With an increase in molar concentration, the transmittance gradually decreases to approximately 35% at the same wavelength due to the increase in film thickness.

Figure 4b illustrates the relationship between absorbance (A) as a function of wavelength ( $\lambda$ ). The absorbance was around 25% at a wavelength of 520 nm. With an increase in molar concentration, the absorbance gradually increases to approximately 36% due to the higher molar concentration.

Figure 4c presents the values of energy gaps ( $E_g$ ) for different concentrations 0.03, 0.05, 0.07 M. By using Tauc plot relation Eq. (7) [23].

$$\alpha h\nu = B_0(h\nu - E_g)^{\frac{1}{2}} \quad (7)$$

where:

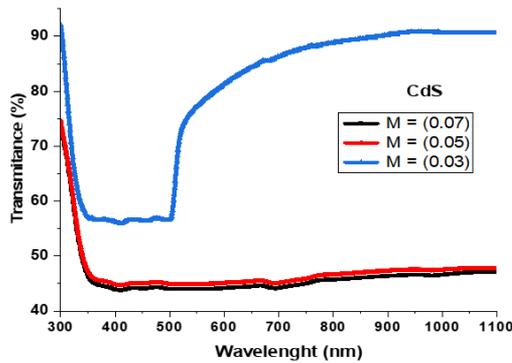
$\alpha$ : The absorption constant.

$h$ : Plank's constant.

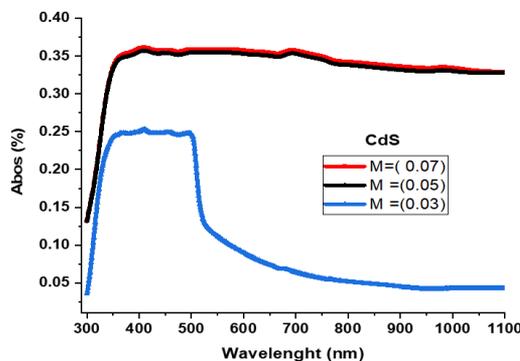
$B_0$ : Constant.

$\nu$ : The frequency of photon.

$E_g$ : Enrge gap.

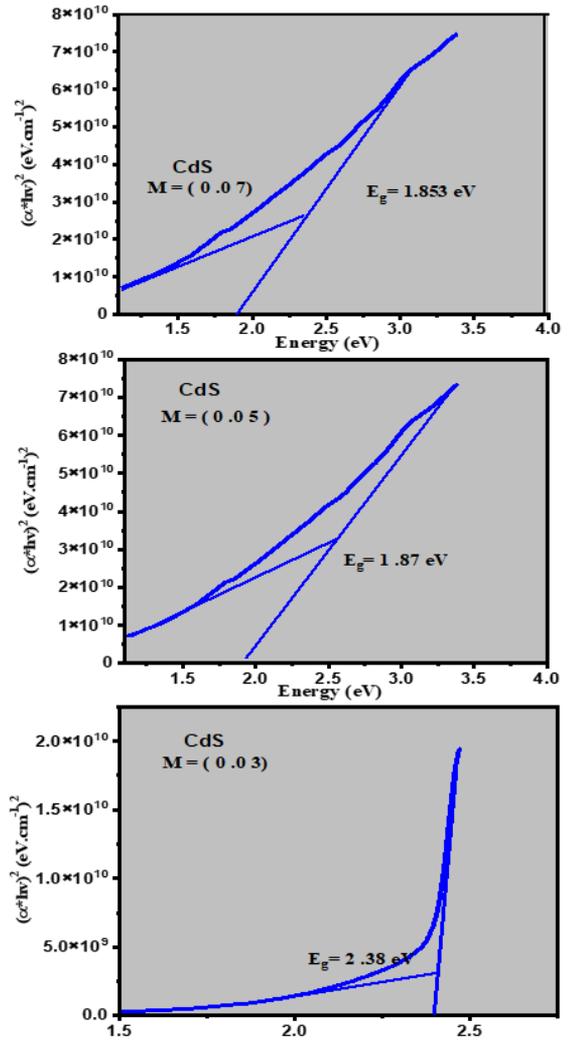


**Figure 4a.** Transmittance spectra for (CdS) film at molarities of (0.03, 0.05, 0.07) M



**Figure 4b.** Absorbance spectra for (CdS) film at molarities of (0.03, 0.05, 0.07) M

The results indicate that the films have energy gaps ranging from 1.853 eV to 2.38 eV, and an increase in molar concentration leads to a decrease in the energy gap due to the increase in solution concentration, which in turn increases the film thickness. Therefore, it is preferable to use the prepared (CdS) film with a concentration of 0.03 M as a window layer in in optical applications and gas sensors. This study is consistent with the researchers [24].



**Figure 4c.** Energy gap for (CdS) film at molarities of (0.03, 0.05, 0.07) M

### 4. CONCLUSIONS

From the current study of (CdS) films prepared using the Chemical Spray Pyrolysis method (CSP) on glass substrates at a temperature of 350°C and at different molar concentrations. it was observed that the concentrations 0.03, 0.05, 0.07 M of both cadmium chloride and thiourea were taken in equal molar ratios to prepare (CdS).

The X-ray diffraction (XRD) results revealed that the films are polycrystalline with a hexagonal phase and possess crystalline planes (100), (002), (101), (110), (112), corresponding to diffraction angles of 24.7°, 26.5°, 28.14°, 44.3°, and 51.7°, respectively. The predominant preferred orientation was found at (002). Various structural characteristic parameters were calculated for each molar concentration. As for the atomic force microscopy (AFM)

results, it was observed that with an increase in molar concentration, the surface roughness and grain size rate (RMS) also increased. This can be attributed to the slight increase in droplet size due to the higher molar concentration.

Regarding the optical properties, the transmittance, absorbance spectra, and energy gap showed that an increase in concentration leads to a decrease in transmittance and energy gap and an increase in absorbance spectrum.

Based on these examinations, it was concluded that the best-prepared (CdS) film in terms of structural and optical results is achieved at a molar concentration of (0.03 M). This film can be used as a window layer in solar cells due to its high homogeneity and smooth surface, which leads to reduced reflectivity, increased transmittance, and energy gap.

Our findings provide crucial insights into the optimization of CdS thin films as window layers in Solar Cells or in Optical applications and Gas Sensors, highlighting the promise of a 0.03 M concentration for achieving high performance.

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