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A New Approach in Chemical Bath Deposition of Cadmium Selenium Thin Films

Metehan Önal and Barış Altiokka*

This study aims to produce cadmium selenium (CdSe) thin films with a hexagonal structure using the chemical bath deposition (CBD) method. In this study, 0.075 g of cadmium chloride ($CdCl_2$) is used as a Cd source, 0.06 g of etilendiamin tetra acetic acid [(EDTA), ($C_{10}H_{16}N_2O_8$)] as a complexing agent, and 0.1 g of selenourea [$CSe(NH_2)_2$] as a selenium source. Ammonia (NH_3) is employed to adjust the pH value of the solutions and varying amounts of Na_2SO_3 (from 0.1 to 1.6 g) are used as a reducing agent. This chemical combination has been used for the first time to produce CdSe thin films. X-ray diffraction (XRD) results confirm that CdSe thin films exhibit a hexagonal structure without requiring annealing. The energy band gap values calculated via absorption graphs range from 1.76 to 1.91 eV. The surface morphologies are examined using scanning electron microscope (SEM) images. SEM images show that there are no voids, cracks, or pinholes. The software named ImageJ is used to determine surface roughness, showing range from 6 to 8 nm. The photographs of the samples show that some films adhere homogeneously to the surfaces of substrates, depending on the amount of Na_2SO_3 used.

1. Introduction

CdSe belongs to the II–VI group and has wide applications in optoelectronic and photovoltaic devices. These applications include light-emitting diodes, photo detectors, gas sensors, gamma-ray detectors, thin film transistors, laser diodes, and solar cells.[1–5] Among these applications, CdSe is widely used in solar cells and photodetector.[6,7] Because of their acceptable band gap and photosensitive nature, CdSe thin films are commonly used as solar energy conversion materials.[8] The reason for this intense interest in CdSe thin films is the fact that the transparency of the deposited films varies between 85% and 98% at a wavelength of $\lambda = 550$ nm, has n-type conductivity, high electron affinity, and a bulk sample of CdSe has a direct band gap of 1.74 eV.[4,9,10] Depending on the deposition conditions, it can be produced in either hexagonal or cubic structures. Additionally, it can be converted from a metastable cubic structure to a more stable hexagonal structure through annealing.[7,8]

M. Önal, B. Altiokka
Vocational School
Bilecik Seyh Edebali University
Bilecik 11210, Turkey
E-mail: metehan.onal@bilecik.edu.tr

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Researchers have obtained CdSe films using various techniques such as metal organic chemical vapour deposition (MOCVD), magnetron sputtering, molecular beam epitaxy, dip coating, laser ablation technique, electro deposition, spray pyrolysis methods and sol–gel spin coating, and chemical bath deposition method (CBD).[5,9–16] CBD is a preferred method because it allows easy control of many variables such as solution concentration, pH value and temperature, precipitation time and stirring ratio, and its application is simple and does not require complex equipment.[3,17,18]

The literature reveals that researchers have produced CdSe thin films under a wide variety of conditions. Two of these studies were carried out to obtain hexagonal films. The first study investigated the effect of the concentration of the precursor solutions, while the second examined the effects of different pH values. In these studies, the deposition process was carried out at 80 °C for 4 h and at 50 °C for 3 h, respectively.[1,3] In some studies where cubic films were obtained, deposition periods of 2 h at 70 °C, 4 h at 60 °C, and finally 18 h at room temperature, 40 °C, and 50 °C were reported.[4,19–22] In another study, researchers deposited CdSe thin films using the CBD method using cadmium acetate, ammonia, and freshly prepared sodium selenosulfate (Na_2SeSO_3) solution. The Na_2SeSO_3 solution was prepared by mixing selenium powder with anhydrous. In this study, glass substrates were kept vertically in the chemical bath for 12 h for deposition.[23] The other studies reported that the solution was prepared by mixing for 1–6 h at solution temperatures of 60–90 °C.[20,24–27] As in the aforementioned studies, the studies in the literature that successfully produce CdSe thin films have some disadvantages, such as long solution preparation and deposition times and high solution temperatures. Time and energy consumption are critical parameters in the production stages. Relatively high temperatures, prolonged solution preparation and deposition times pose significant challenges. On the contrary, the advantage of our work is the fact that long deposition durations and high solution temperatures are not required. In our study, $CdCl_2$, $CSe(NH_2)_2$, NH_3, EDTA, and Na_2SO_3 were used together for the first time. The chemical reactions for the formation of CdSe are given below for Equation (2),[28] (3) and (5),[29] (4) and (6),[30] (8).[31]

$$CdCl_2 \rightarrow Cd^{2+} + 2Cl^- \quad (1)$$

$$Cd^{2+} + EDTA \rightarrow [Cd - EDTA]^{2+} \quad (2)$$

$$NH_3 + H_2O \rightarrow NH_4 + OH^- \quad (3)$$

$$Na_2SO_3 + H_2O \rightarrow Na_2SO_4 + 2H^+ + 2e^- \quad (4)$$

Na_2SO_3 is a reducing reagent. It was added to prevent the oxidation of selenourea.

$$CSe(NH_2)_2 + OH^- \rightarrow HSe^- + CN_2H_2 + H_2O \quad (5)$$

$$HSe^- + OH^- \rightarrow H_2O + Se^{2-} \quad (6)$$

$$[Cd - EDTA]^{2+} + 4NH_3 \rightarrow Cd(NH_3)_4{}^{2+} + EDTA \quad (7)$$

$$Cd(NH_3)_4{}^{2+} + Se^{2-} \rightarrow CdSe + 4NH_3 \quad (8)$$

It has been reported that CdSe films obtained at room temperature have a cubic structure.[4] However, in our study, hexagonal CdSe films could be obtained at room temperature. This result is significant because the hexagonal CdSe is more stable than its cubic counterpart.[2,11] In addition, since the films were produced at room temperature, they provided significant energy savings. Furthermore, the photograph of the samples indicated that some films adhered to the substrate homogeneously.

2. Experimental Section

CBD was chosen to obtain CdSe thin films. First, the glass substrates were immersed in acetone and then rinsed with deionized water. The bath container was washed with a 30% HCl solution and rinsed with deionized water. The glass substrate was immersed in 100 mL of deionized water. The solutions were mixed using a magnetic heater and stirrer at 600 rpm. The basic principle for obtaining thin films of semiconductors is to add metal source, complexing agent, pH regulator, and nonmetal source to deionized water. Based on this information, the final solution was obtained using the following chemicals. It was dissolved by adding 0.075 g $CdCl_2$ and 0.06 g EDTA to the deionized water, respectively. NH_3 was added using a dropper until the pH of the current solution reached 10. After adjusting the pH of the solution, Na_2SO_3 and 0.1 g $CSe(NH_2)_2$ were added to complete the final solution. The amount of Na_2SO_3 was chosen to be 0.1, 0.4, 0.8, 1.2, and 1.6 g. The samples were named from Set1 to Set5 based on the amount of Na_2SO_3 0.1, 0.4, 0.8, 1.2, and 1.6 g, respectively. It may be recommended to add a reducing agent such as Na_2SO_3 to prevent potential oxidation of the Se atom of Selena urea in solution.[32] The studies in the literature report that using Na_2SO_3 to produce thin films with CBD reduces the reaction rate. Besides, it has been reported that the reaction rate affects the adhesion quality.[33–35] All experiments were completed within 20 min after all chemicals were added. The solution temperature was measured as 20 °C. The glass substrates, removed from the bath container, were rinsed with pressurized deionized water for residue removal and then air-dried at room temperature. Experimental Section are given in **Table 1**.

The gravimetric method was used to determine film thicknesses. The optical, structural, and morphological characteristics of the films were analyzed using A&E lab with a single beam UV-Vis spectrometer, a PANalytical Empyrean XRD, and a Zeiss SUPRA 40VP SEM, respectively.

Table 1. Experimental parameters.

Exp.	$CdCl_2$ [g]	EDTA [g]	Selenourea [g]	Na_2SO_3 [g]	Deposition time [min]
Set1	0.075	0.06	0.1	0.1	20
Set2	0.075	0.06	0.1	0.4	20
Set3	0.075	0.06	0.1	0.8	20
Set4	0.075	0.06	0.1	1.2	20
Set5	0.075	0.06	0.1	1.6	20

3. Result and Discussion

3.1. Structural Analysis of CdSe Films

The gravimetric method described in Equation (9) was preferred to determine the thickness of CdSe films. The film thicknesses were calculated as follows: 200 nm for set1 and set2, 300 nm for set3, and 250 nm for set4 and set5. It can be said that the film thicknesses vary depending on the amount of Na_2SO_3 used. The use of Na_2SO_3 improved adhesion of the films to the glass substrates because Na_2SO_3 is a reducing reagent. It was added to prevent the oxidation of selenourea. In addition, in the absence of Na_2SO_3, no film adhered to the glass surface. Even when 0.1 g Na_2SO_3 was used, a film that may be described as amorphous was obtained.

$$t = \frac{m}{\rho A} \quad (9)$$

where t is the film thickness, m is the mass of the film, ρ is the density, and A is the surface area. The bulk sample CdSe density value is 5.81 g cm^{-3}.[36]

Figure 1 illustrates the X-ray diffraction (XRD) pattern of CdSe, which varies based on the quantity of Na_2SO_3. The 2θ scan range of 20°–70° was used. The XRD patterns of the samples were compared with the peaks on the ASTM card numbered 00-008-0459. While the peak of the film obtained in set1 is 42.0°, the peaks of the film obtained in set2 are related to 23.9° and 42.2°. It is seen that the peaks of the films obtained in set3, set4, and set5 are associated with 24.0°, 25.2°, and 42.0°. The peaks seen in Figure 1 belong to planes (100), (002), and (110). At the same time, Figure 1 shows that all films are formed in a hexagonal structure. This result is important because the unstable phase of CdSe is the cubic structure[2,11] and when the literature review was made, no study was found showing that hexagonal films can be obtained at 20 °C for 20 min of deposition time.

The Debye–Scherrer equation used to calculate the average crystallite sizes of the obtained films is given in Equation (10).[37–41]

$$D = \frac{K\lambda}{\beta \cos\theta} \quad (10)$$

where D symbolizes crystallite size, λ symbolizes the wavelength of X-ray radiation (1.54056 Å), θ symbolizes the

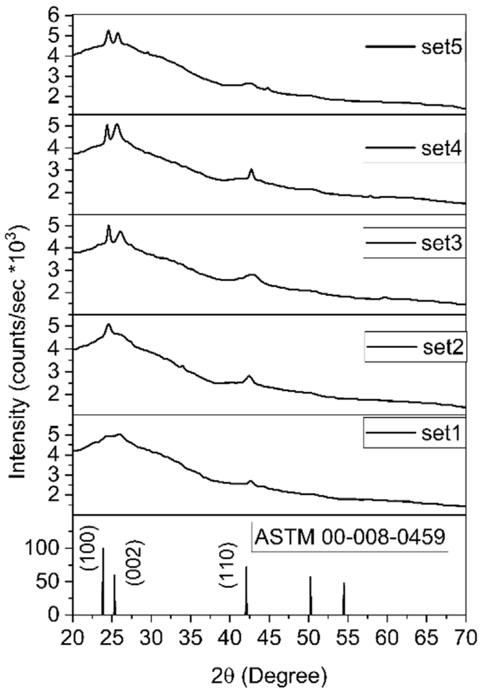

Figure 1. XRD pattern of CdSe formed depending on the amount of Na_2SO_3.

Bragg diffraction angle, and β symbolizes the full width at half maximum value (FWHM), which is calculated via origin pro software. Calculated average crystallite sizes are given in **Table 2**. Table 2 shows that crystallite sizes vary between 7 and 20 nm.

Dislocations are crystal defects related to the misalignment of the lattice in one part of the crystal with respect to another part. Unlike vacancies and interstitial atoms, dislocations are not equilibrium defects.[42] The dislocation densities of the films are calculated by Equation (11) and the results are given in Table 2.

$$\delta = \frac{1}{(D)^2} \tag{11}$$

where,[43] δ gives the dislocation density. The dislocation densities were calculated between 2.5 and 20.4 $(\text{line m}^{-2}) \times 10^{15}$. A low value of dislocation density is an indication that thin films are more compact and good.[44]

Table 2. Calculated crystallite size and band gaps of CdSe thin films.

Exp.	D [nm] (100)	D [nm] (002)	D [nm] (110)	D [nm] Average	Dislocation density [line m^{-2} × 10^{15}]	Band gap [eV]
Set1	–	–	7	7	20.4	1.80
Set2	21	–	11	16	3.9	1.91
Set3	20	11	7	13	5.9	1.80
Set4	27	14	19	20	2.5	1.83
Set5	16	17	8	14	5.1	1.76

3.2. Optical Properties of the CdSe Films

The optical analysis of CdSe thin films was carried out using absorbance measurements at wavelengths from 800 to 500 nm with a UV–Vis spectrometer. The absorbance plots and transmittance plots are given in **Figure 2** and **3**, respectively.

When Figure 2 is examined, it is seen that the absorbance value of the film obtained in set3 is the highest compared to the other films, while the absorbance value of the film obtained in set2 is the lowest. Similarly, when Figure 3 is examined, it is seen that the transmittance values of the films at 700 nm wavelength vary between 60% and 80%. The thickness of the sample named set3 with the highest absorbance value is 300 nm, while the thickness of the sample named set2 with the lowest absorbance value is 200 nm. These results are also consistent with the calculated film thicknesses. Literature reports suggest that

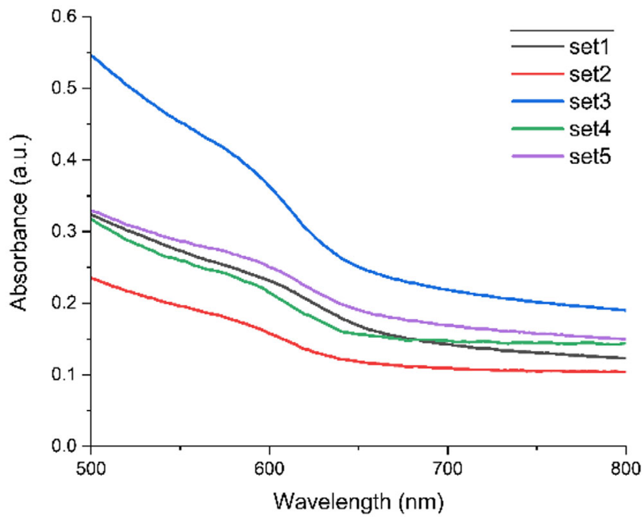

Figure 2. Absorbance measurements of CdSe thin films.

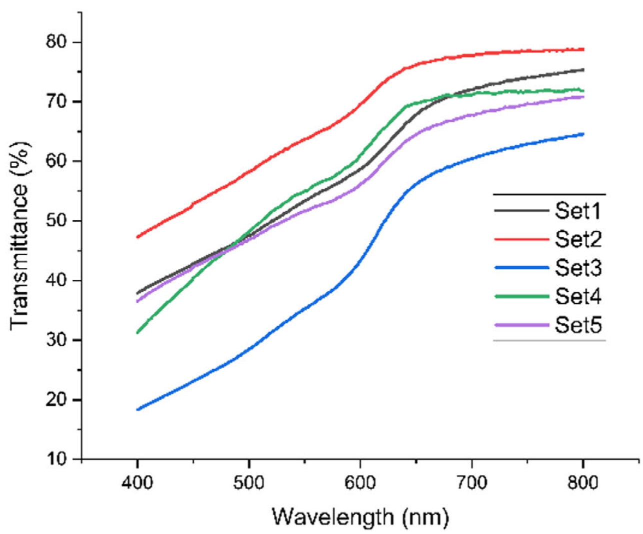

Figure 3. Transmittance spectra of CdSe thin films.

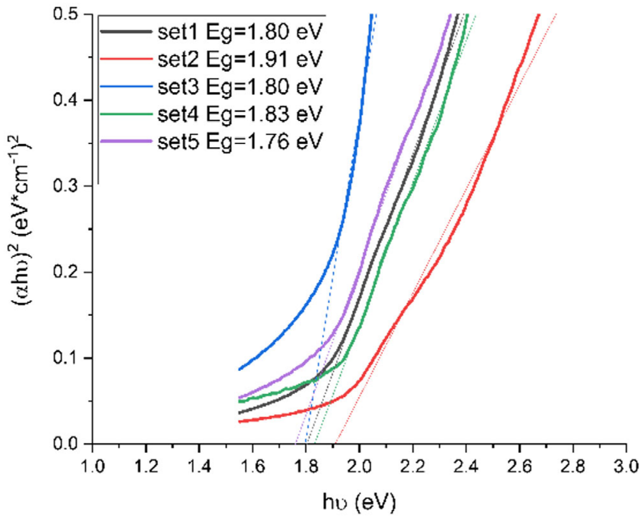

Figure 4. Energy band gaps of CdSe thin films depending on the amount of Na_2SO_3.

optical transmittance decreases and crystallization improves as the film thickness increases.[20] It has also been reported in the literature that the band gap of CdSe is tunable and can provide an optical response from the infrared region to the ultraviolet region.[45] It is considered that by optimizing the amount of Na_2SO_3 used, the film thickness, and thus the optical properties of the films such as absorbance, transmittance, and band gap, may be adjusted.

The energy band gap values presented in **Figure 4** and Table 2 were estimated using Tauc plots. The equation used to estimate the energy band gap values from Tauc graphs is given in Equation (12).

$$(\alpha h\nu) = B(h\nu - E_g)^n \tag{12}$$

where $h\nu$ is the photon energy, for allowed direct transitions $n = 1/2$, E_g is the energy band gap, and B is a constant.[46,47] The band gap is estimated from the point where the $(\alpha h\nu)^2$ graph intersects the $h\nu$ axis ($h\nu = 0$). As seen in Figure 4 and Table 2, the energy band gap values of CdSe thin films vary between 1.76 and 1.91 eV. In the literature, band gap of the CdSe vary between 1.74 and 2.15 eV.[1,11,48]

3.3. SEM Analysis of the CdSe Films

Figure 5 and **6** show SEM images obtained at magnifications of 500× and 40 000×, respectively. When Figure 5 and 6 are examined, it is seen that all films adhere homogeneously to the glass substrate surface, with no cracks, holes, or voids on the film surfaces. It was observed that CdSe did not adhere to the glass surface when Na_2SO_3 was not used, indicating that the use of Na_2SO_3 facilitates CdSe adhesion to glass surfaces. However, it is understood from the SEM images that the use of Na_2SO_3 does not cause a significant difference on the surface of the films.

Data obtained from SEM images were analyzed using ImageJ software. The reliability of the studies calculating surface roughness analyzing with ImageJ software has been validated

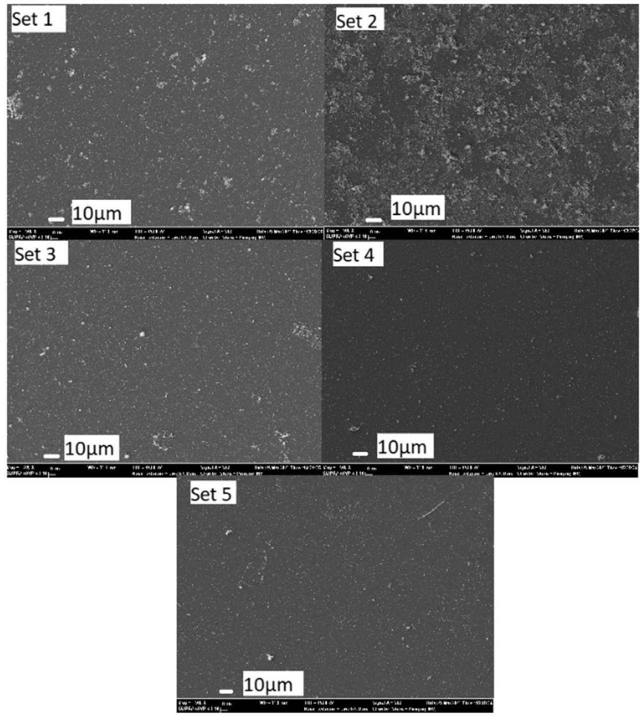

Figure 5. SEM images of CdSe thin films at 500× magnification.

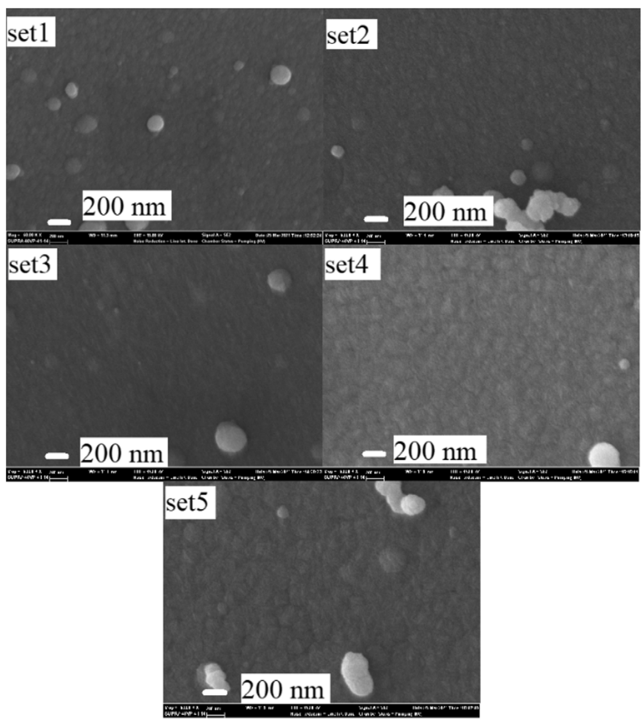

Figure 6. SEM images of CdSe thin films at 40 k× magnification.

in reputable journals in the literature.[49–51] The calculated surface roughness values of the films are given in **Table 3** with the surface roughness graphs given in **Figure 7**. In Figure 7, the blue

Table 3. Surface roughness values calculated with imageJ software.

Experiments	Na_2SO_3 [g]	Ra [nm]	Rq [nm]
Set1	0.1	7	10
Set2	0.4	7	10
Set3	0.8	6	8
Set4	1.2	8	11
Set5	1.6	8	10

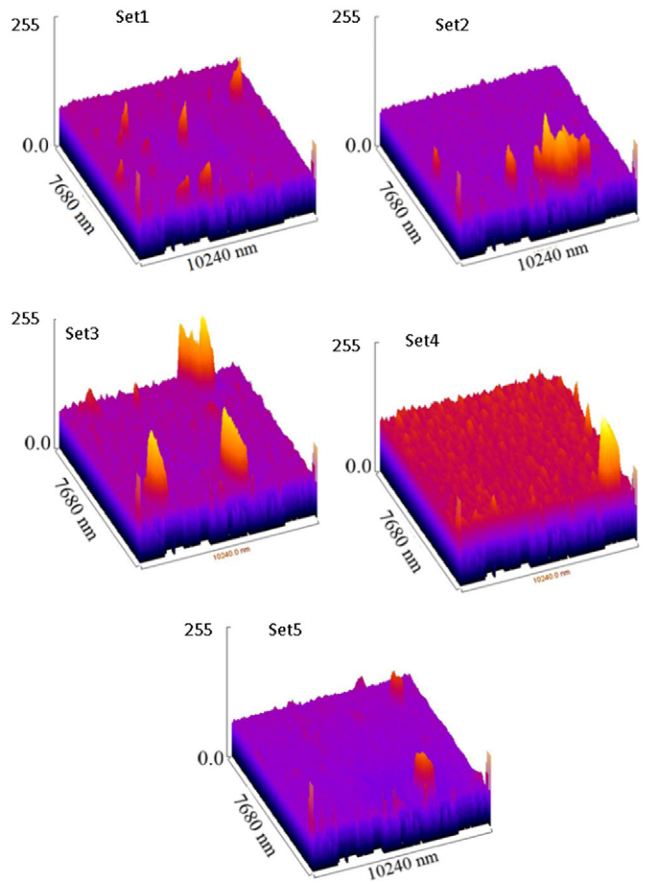

Figure 7. Surface roughness plots of CdSe thin films.

areas represent the sample surface while the orange areas indicate protrusions on the surface.

As seen in Table 3, average (Ra) surface roughness values were calculated in the range of 6–8 nm, and root mean square (rms) (Rq) surface roughness values were calculated in the range of 8–11 nm. The optical transmittance values of thin films deposited by the CBD method are closely related to the morphology of the film. Excessive particle growth on the film surfaces causes scattering losses, which can reduce the transmittance of the film.[25] As the grain size increases, the light-scattering losses also increase resulting in the observed decrease in the transmittance spectra.[52] These surface roughness results we obtained are consistent with a study reporting an average surface roughness of 5 nm, and indicate that such surface morphology features enhance performance by providing good interfaces with other layers in heterostructured devices.[1] The lowest value we obtained, 6 nm average surface roughness, was of the set3 sample in which 0.8 g Na_2SO_3 was used. On the contrary, other studies in the literature have reported surface roughness values ranging from 30 nm to 70 nm, from 10.6 to 78.8 nm, and from 53 to 74 nm.[44,53,54] In our study, surface roughness values averaged 7 nm. In Figure 7, the surface color is homogeneous. There are very few orange protrusions. This is an indicator of the smoothness of the surface. The smoothness of the films can be seen in Figure 7.

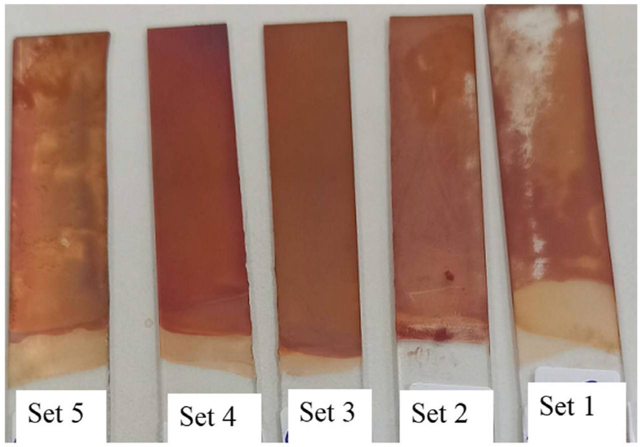

Figure 8. Image of CdSe films.

3.4. Visual Analysis of the CdSe Films

The top view of the produced films is given in **Figure 8**. This figure reveals the difference in the adhesion quality of the films to the glass substrate surface, which is not apparent in the SEM images. After the deposition process, the films were rinsed with pressurized deionized water, which caused some areas of the films in set1, set2, and set5 to be removed from the glass substrates. On the contrary, in the samples named set3 and set4, it can be seen that the films adhered very well to the glass substrate, completely covering the entire surface without visible defects. This observation suggests that the optimal amount of Na_2SO_3 results in homogeneous film adhesion, indicating a strong relationship between the amount of Na_2SO_3 used and the film adhesion quality.

4. Conclusions

In this study, CdSe thin films were produced with CBD. As a novel approach in CdSe production, the chemicals $CdCl_2$, EDTA, NH_3, Na_2SO_3, and $CSe(NH_2)_2$ were used together for the first time. This combination of five chemicals for CdSe thin film production has not been previously reported in the literature. The aim of this study was to simplify the production process of CdSe thin films, which can also be used in solar cells, and to investigate the optimization of production conditions in a shorter time and at lower temperatures. It was calculated that the thickness of the obtained films varied between 200 and 300 nm. XRD patterns showed that CdSe thin films with a hexagonal structure, which is more stable, can be produced at room temperature and without requiring annealing. Optical measurements showed that the transmittance values of the films varied between 60% and 80%. High optical transmittance values are important for films used in the window layer of solar cells. Energy band gap values, estimated using Tauc plots, showed that the energy band gaps of the films varied between 1.76 and 1.91 eV. When examining the SEM images of the films taken at 500× and 40 k× magnification, no gaps, cracks, or pinholes were observed on the film surfaces. The absence of cracks, voids, and pinholes on the film surfaces is an indication that the films we obtain are suitable for use in solar cells and photovoltaic applications. The absence of cracks, voids, and pinholes on the film surfaces is an indication that the films we obtain are suitable for use in solar cells and photovoltaic applications. Since defects on the film surfaces result in short circuits in the junction area. However, visual analysis revealed that the films adhered homogeneously to the surface only when 0.8 g to 1.2 g of Na_2SO_3 was used. This is significant because CdSe films with good adhesion to glass substrates at room temperature can be obtained without long solution preparation and deposition times. Surface roughness plots and calculations obtained using ImageJ software showed that very smooth and flat films were obtained. Low surface roughness makes the films suitable for solar cells. Since it reduces photon scattering on the surfaces of the films. In our future studies, we will try to obtain better quality films by changing variables such as solution concentrations, temperature, etc.

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Conflict of Interest

The authors declare no conflict of interest.

Author Contributions

Both authors contributed to the study conception and design. Material preparation, data collection, and analysis were performed by **Metehan Önal** and **Barış Altiokka**: The first draft of the manuscript was written by **Metehan Önal** and **Barış Altiokka**: Both authors read and approved the final manuscript.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Keywords

cadmium selenium, chemical bath deposition, Na_2So_3, thin films

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