



Study of the Optical and Morphological Properties of Thin Cadmium Oxide Films Prepared Using Spin Coating Technology

N. A. Al-obaidi ⁽¹⁾  M. A. Al-Jubbori ^(2*)  A. E. Elzwam ⁽³⁾ 

^(1,2) Department of Physics, College of Education for Pure Science, University of Mosul, Mosul, Iraq
³ Physics Department, College of Sciences, Tripoli University, Libya

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Correspondence:

Mushtaq Abed Al-Jubbori

mushtaq_pht@uomosul.edu.iq

Abstract

The study aims to prepare thin CdO films with a thickness of 232 nm using the spin coating method. This involved depositing the cadmium acetate in 2-methoxyethanol on glass substrates. This thin layer was prepared at a rotation speed of 3500 rpm for 30 seconds, then dried at 250 °C for 10 minutes, and finally heat-treated at 350 °C for one hour. The optical properties of the prepared CdO thin films were studied using UV-Visible spectroscopy in the wavelength range of 190-1100 nm. The results indicated that the absorptivity was a function of wavelength and recorded an absorptivity peak in the range of 375-450 nm (short wavelength region), then gradually decreased with increasing wavelength and began to stabilize in the range of 850-950 nm. The optical constants were calculated, and the results confirmed that the behavior of the optical constants followed a pattern similar to that of the absorption behavior. The energy gap of the thin film was calculated using the Tauc equation to be 2.30 eV, and direct-type electronic transitions were observed. A scanning electron microscope (SEM) was used to study the surface morphology of the thin films. Microscopic images showed that the nanoparticles formed a semi-spherical (cauliflower-like) cluster structure with a porous surface during the preparation process, which had an average size of 38.43 nm, with an average crystallite size of 12.9 nm, and their transparency in the infrared region, making them suitable for multiple applications in materials science and optoelectronic devices such as sensors.

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1. Introduction

Thin film physics is one of the main specialisations in solid state physics and has become a separate discipline in its own right [1]. Thin films have revolutionised many industries and technological developments due to their unique properties and diverse applications. These nanolayers range in thickness from nanometres to micrometres. They have remarkable properties that make them invaluable in various fields such as optics, electronics, energy, and others [2].

Over the past 20 years, this period has been a catalyst for the development of modern growth techniques, especially thin films. The field of functional oxide materials has seen exceptional growth, with the discovery of new material systems that represent the state of the art [3]. Semiconducting compounds have attracted attention due to their electronic and optical properties and applications, such as high absorption coefficients in the visible and infrared regions of the solar spectrum and high efficiency [4].

Researchers have worked to discover new deposition techniques and improve known methods, and the physical properties of the films have varied according to the deposition method [5]. In this context, cadmium oxide nanoparticles have attracted considerable attention due to their distinctive electrical, optical, chemical and physical properties, leading to their use in advanced applications. [6]. One of the main properties in applications of CdO as a transparent conductive oxide (TCO) is its

high electron mobility [7]. Cadmium oxide is an n-type semiconductor with a direct band gap of (2.2-2.5) electron volts and an indirect band gap of approximately 2.0 electron volts [8]. It has high electrical conductivity and excellent optical transparency in the visible range [9]. These properties make cadmium oxide films suitable for various applications such as catalysts, sensors, and transparent electrodes in solar cells and flat panel displays [10].

Cadmium oxide films are prepared by several methods, including chemical vapour deposition, thermal decomposition spraying, sol-gel method, and DC magnetic spraying [11]. The spin coating process of the gel solution offers several advantages, including: simple technology, easy control of composition and formation of homogeneous films, and low cost compared to other techniques [12].

Spin coating plays a key role in coating substrates. The process involves placing a small amount of solution in the centre of the substrate and rotating it at high speeds. As a result of centrifugal force, the prepared solution spreads to the edges of the substrate until the desired film thickness is achieved. One of the most notable advantages of spin coating is its repeatability [13].

The most important factors affecting film thickness in the spin coating process are: rotation speed, rotation time, solution properties, and evaporation rate [14]. This work aims to prepare thin CdO films by spin coating and study their optical and morphological properties at an annealing temperature of 350°C.

2. EXPERIMENTAL DETAILS

2.1. Preparation of glass bases and their weighing before thin film deposition

Glass substrates with a thickness of 1 mm and dimensions of (1.5 × 2 cm) were used. The glass substrates were cleaned thoroughly, as cleaning is not just a routine process, but affects the quality of the prepared films and gives inaccurate results. The cleaning stage begins with distilled water to remove contaminants such as dust and impurities, then the substrates are immersed in acetone and distilled water and placed in an ultrasonic device for 20 minutes at a temperature of 30 degrees Celsius to remove grease and oils. The glass substrates are dried with special lens cleaning wipes, then with air using an electric dryer for two minutes to ensure that the surface is completely dry and free of moisture. After cleaning, the weights of the glass substrates are measured using a high-precision balance with a sensitivity of 10^{-4} g manufactured by JOANLAB, AB. As shown in the figure 1 The weights are recorded in a notebook. The thickness of the prepared membranes is calculated later using the gravimetric method.



Fig .(1) Photograph of the weighing device.

2.2 Preparation of the solution

The cadmium oxide solution was prepared using cadmium acetate dihydrate. Its chemical formula is $(\text{CH}_3\text{COO})_2\text{Cd}\cdot 2\text{H}_2\text{O}$ (molecular weight 266.53g/mol), a white powder of American origin, manufactured by the American company SCMA. A 0.3 M dissolve 0.79 g of cadmium acetate dihydrate is dissolved in 10 ml of 2-methoxyethanol for two hours, as shown in Figure 2. The mixture is stirred using a magnetic stirrer for the first 20 minutes at room temperature to ensure the homogeneity of the solution. The temperature of the solution is then gradually raised to 70 °C while continuously stirring for one hour and 10 minutes to obtain a colourless solution with continuous stirring. Then, the mono-ethanolamine stabiliser is gradually added in drops until the pH reaches 7, which is achieved when the fourth drop is added. Finally, the final solution is filtered using filter paper to ensure a pure solution and left for 24 hours before sedimentation.

The following equation was used to calculate the weight to be melted [15]:

$$M = \frac{M_t}{M_{wt}} * \left(\frac{1000}{V} \right) \quad (1)$$

M: molar concentration, M_t : mass of cadmium acetate dihydrate, M_{wt} : molecular weight of the substance, V: volume of the solution.



Fig .(2) Photograph of the magnetic stirrer device.

2.3. Deposition process by the spin coating method

1. Placement the glass substrate

After cleaning, the glass substrates are placed horizontally and securely fixed inside the spin coating device, as shown in Figure 3.

2. Precipitation process

A small amount of the prepared solution was added to the centre of the glass substrate using a dropper.

3. Rotation phase

The substrate was rotated at a speed of 3,500 rpm for 30 seconds. Thanks to centrifugal force, the excess solution was expelled towards the edges of the substrate, while a thin layer remained on the surface of the substrate. Centrifugal force depends on the speed of rotation, resulting in an even distribution of the solution. The coating process was repeated 9 times to achieve a thickness of 232 nm.



Fig.(3) Photograph of the spin coating.

2.4 Drying Process

After the sedimentation process was complete, the glass substrates were placed on a hot plate at a temperature of 250 °C for 10 minutes, which led to the evaporation of the remaining solvent and the formation of a thin layer.

- **Electric Heater.**

To dry the substrates efficiently, the glass substrates are placed on a heating plate, as shown in Figure 4-a. Their temperature is controlled by a temperature controller connected to it, which is controlled manually.

- **Temperature control unit**

This device regulates the temperature and has a display showing two readings. The red reading indicates the laboratory temperature, while the green reading indicates the desired temperature. Adjust the temperature through a button below the screen, which automatically cuts off the power supply when the desired temperature for drying the membranes is reached, as shown in Figure 4-b.

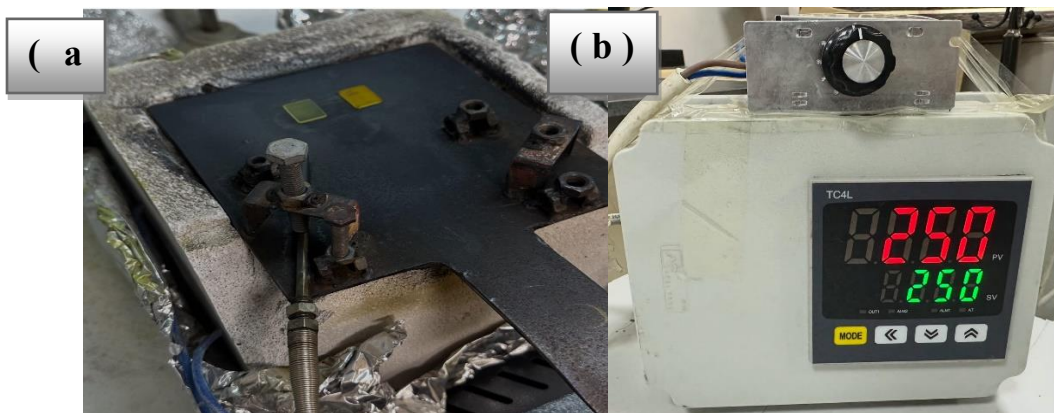


Fig.(4) a) Photograph of an electric heater, b) Photograph of the temperature controller

2.5 Heat treatment process

The substrates were placed in an oven at 350°C for one hour to improve the crystallisation of the thin film.

2.6 Measuring film thickness

After the heat treatment process was complete, the glass substrates were weighed again. The following equation was used to calculate the thickness using the gravimetric method [15]:

$$t = \frac{\Delta w}{\rho * A} = \frac{w_2 - w_1}{\rho * A} \quad (2)$$

(t) thickness of the prepared films, (Δw) weight difference before and after the spin coating process, (ρ) film material density (g/cm^3), (A) area (cm^2). The results showed that the thickness of the prepared films was approximately $232 \text{ nm} \pm 20$. Figure 5 below shows a schematic diagram of the spin coating system.

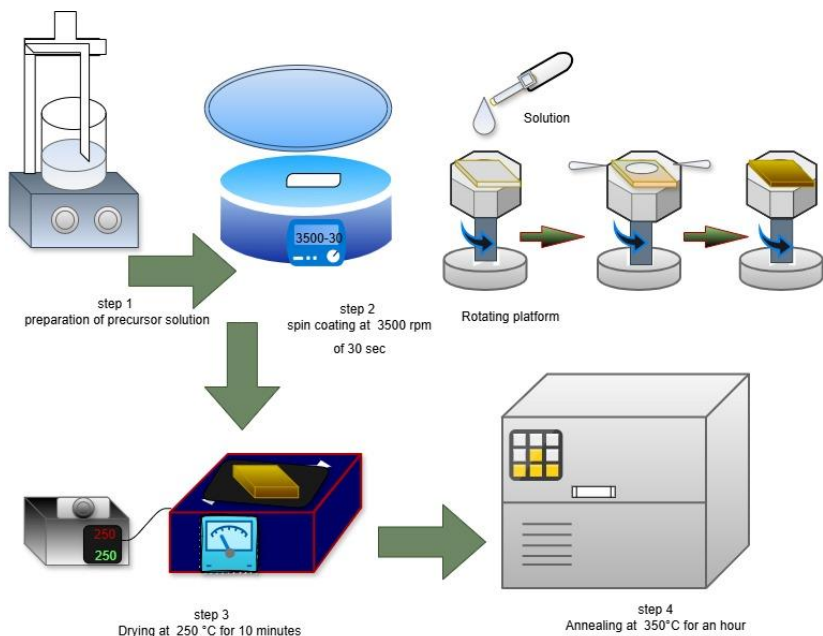


Fig .(5) Schematic diagram of the spin coating system.

3. RESULTS AND DISCUSSIONS

3.1. The Optical absorption analysis

To analyze the optical properties of thin cadmium oxide (CdO) films prepared by spin coating and annealed at 350°C , their optical response was evaluated based on UV-Vis spectroscopy (Shimadzu UV-1800). Measurements were performed at a scanning rate of $400 \text{ nm}/\text{min}$, and absorption spectra were recorded within the spectral range ($190\text{-}1100 \text{ nm}$).

3.1.1 Absorbance

The absorptivity values of cadmium oxide (CdO) membranes were determined for wavelengths within the range of $350\text{-}950 \text{ nm}$. There are three regions in the absorption spectrum. The first, primary absorption (the main absorption edge), corresponds to short wavelengths where the energy of the photons is sufficient to excite electrons from the valence band to the conduction band. The second, the weak absorption tail, corresponds to longer wavelengths. The third, between the two regions, follows the Urbach rule [16]. The figure shows the behavior of the absorption spectrum as a function of wavelength, where absorption increases as the wavelength decreases. Absorption peaks in the $375\text{-}450 \text{ nm}$ range (Ultraviolet/visible radiation) [17]. This increase is attributed to the initiation of transitions between electronic bands at the primary absorption edge. [18]. Then we observe a sharp and continuous decrease, which begins to stabilise in the range of $850\text{-}950 \text{ nm}$ (infrared region). This decrease in absorption is attributed to the fact that the energy of the photons is less than the energy gap value, making them unable to excite electrons and transfer them from the valence band to the conduction band [19]. These techniques are very suitable for the use of prepared thin films in photovoltaic cell applications.

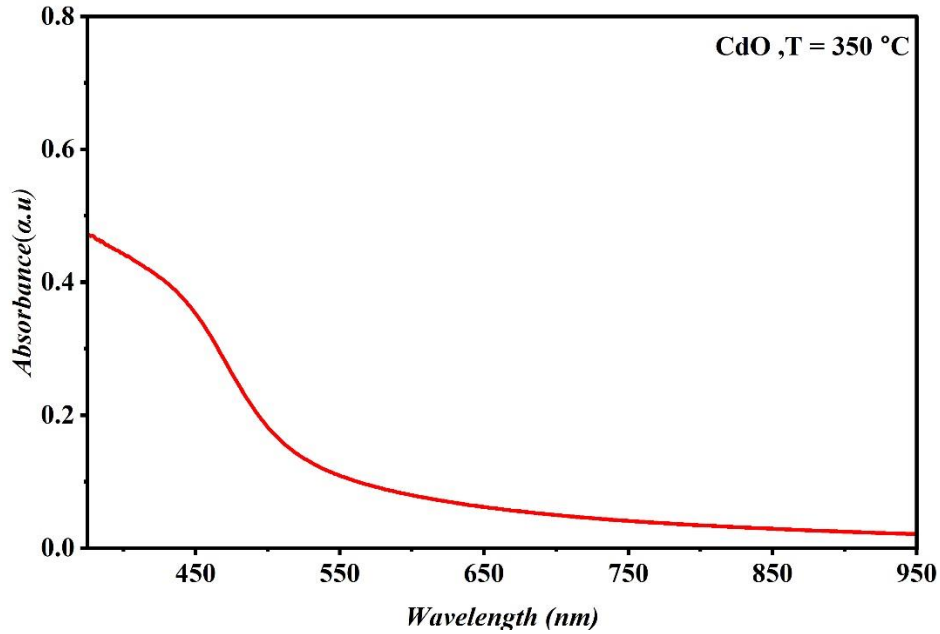


Fig.(6) Absorption spectrum as a function of wavelength for cadmium oxide (CdO) thin film

3.1.2 Absorption coefficient

The results shown in the figure illustrate the behavior of the absorption coefficient as a function of wavelength. The data show that the absorption coefficient increases as the wavelength decreases. The absorption coefficient reaches its maximum in the range of 375–450 nm, then gradually decreases to reach a stable phase in the range of 850–950 nm (infrared region). Absorption coefficient values of 10^4 cm^{-1} are observed, confirming the occurrence of direct electronic transitions between the valence band and the conduction band [20]. The absorption coefficient is the measured decrease in radiation intensity [1], and can be determined using the following equation [21]:

$$\alpha = 2.303 \frac{A}{t} \quad (3)$$

Where A represents absorptivity, and t represents thickness, and the thickness in our study was 232 nm.

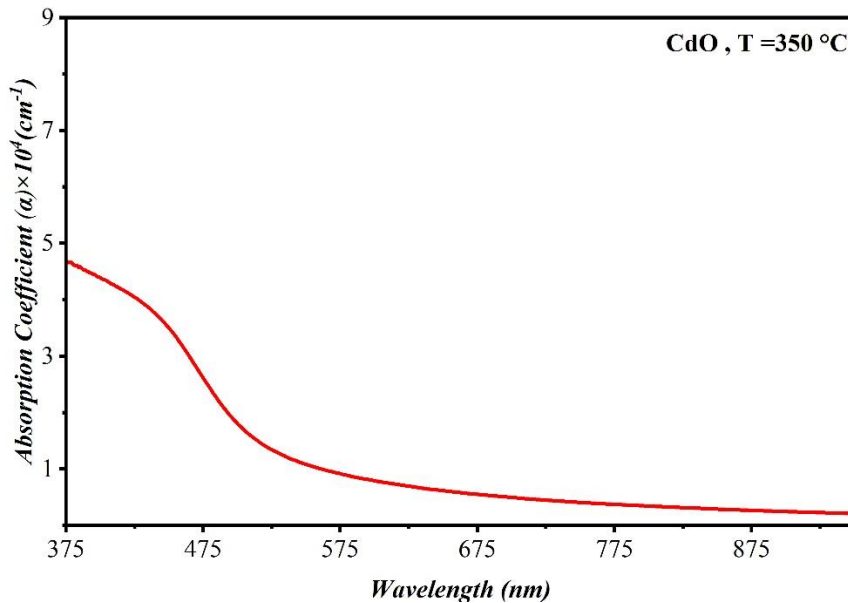


Fig.(7) Absorption coefficient of CdO thin films as a function of wavelength.

3.1.3 Energy gap

The crystal system plays a key role in the energy gap values of the prepared thin films [22]. The energy gap is defined as the minimum amount of energy required for an electron to move from the top of the valence band to the bottom of the conduction band [1]. The determination of the optical energy gap (E_g) of cadmium oxide (CdO) films is based on the relationship between the absorption coefficient (α) and the incident photon energy ($h\nu$) according to the equation Tauc below [23]

$$(\alpha \cdot h\nu)^{1/2} = k(h\nu - E_g^{opt}) \quad (4)$$

From the graph of $(\alpha h\nu)^2$ versus $(h\nu)$ in the figure, the values of the optical energy gap were determined by taking the shadow of the curve intersecting the horizontal axis at the point $(\alpha h\nu)^2 = 0$, where the results showed that the energy gap values for cadmium oxide films prepared at an treatment temperature of 350 °C are equal to 2.30 electron volts. This is consistent with the reported range of previous results [8].

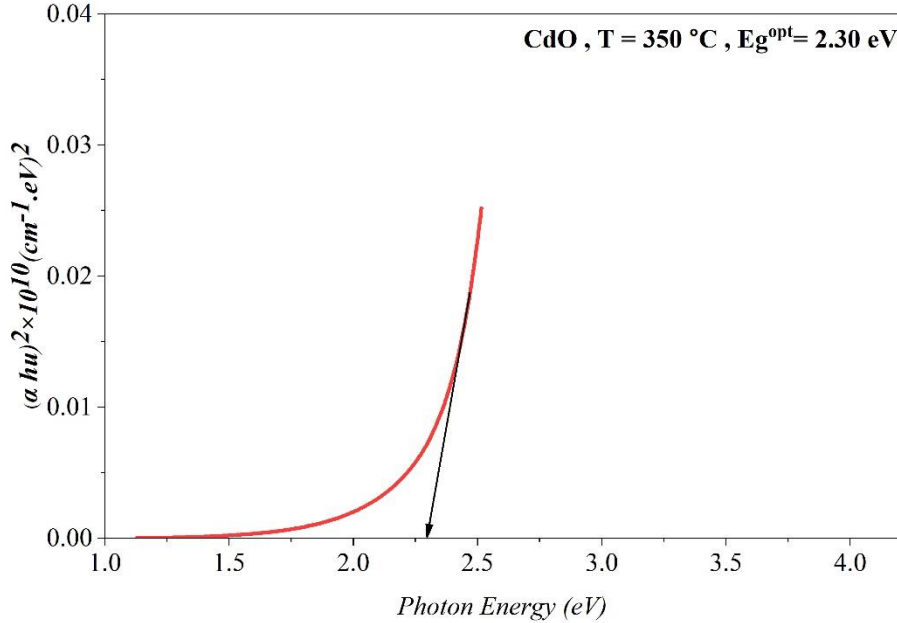


Fig.(8) Evaluate the optical energy gap of CdO as a function of photon energy.

3.1.4 Refractive index

The refractive index plays a pivotal role in the search for optical materials. It is considered a critical factor in the design of spectroscopic dispersion devices and in optical communications. The most influential factor on the refractive index is the density of voids [24]. The value of the refractive index is determined by the following relationship [25]:

$$n = \sqrt{\frac{4R}{(R-1)^2} - K^2} - \left(\frac{R+1}{R-1}\right) \quad (5)$$

The curve shows the relationship between the refractive index (n) and wavelength (λ) of thin cadmium oxide (CdO) films treated at a temperature of 350°C. The figure indicates that the refractive index reaches its highest value in the blue/violet spectral region (450 nm), then begins to decrease with increasing wavelength. The decrease in probability can be attributed to two interpretations: the effect of voids causing a reduction in density [24], and the effect of abnormal scattering, where the film shows abnormal scattering in the range of 450-750 nm, where n decreases with decreasing photon energy (i.e., with increasing wavelength) [9].

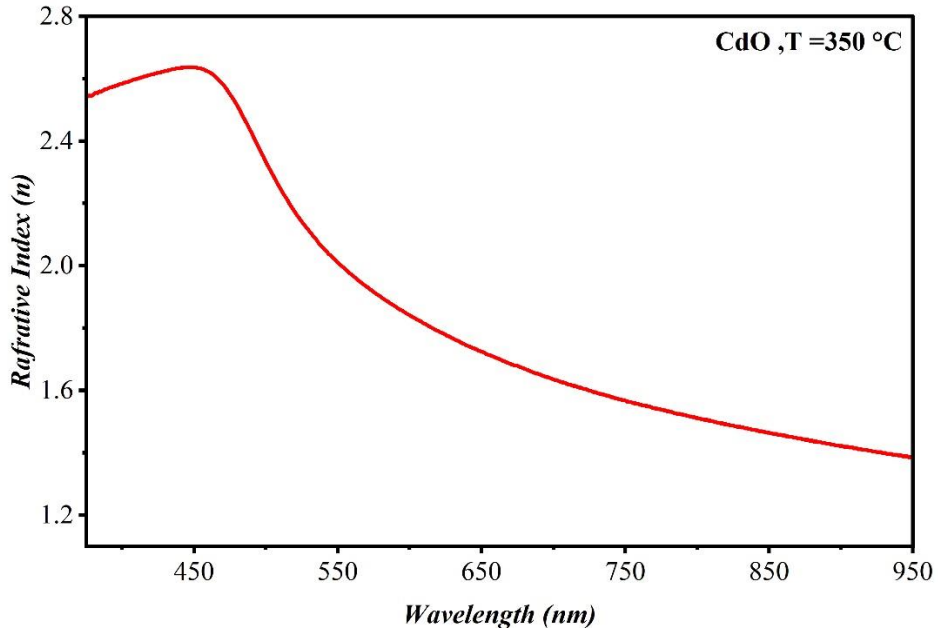


Fig.(9) Refractive index function of wavelength for CdO thin films.

3.1.5 extinction coefficient

The curve shows the relationship between the extinction coefficient of thin cadmium oxide (CdO) films prepared by spin coating and treated at 350 °C and wavelength. The figure shows that the extinction coefficient has its highest value (short wavelength region) in the range of 375-450 nm, then begins to decrease with increasing wavelength. This decrease is attributed to absorptivity [26], and then stabilises approximately in the range of 850-950 nm (this indicates that the material exhibits transparent behaviour in the infrared region). The extinction coefficient (k) is defined as a measure of the decrease in light intensity when passing through a material, according to the following equation [27]:

$$K = \frac{\alpha \lambda}{4\pi} \tag{6}$$

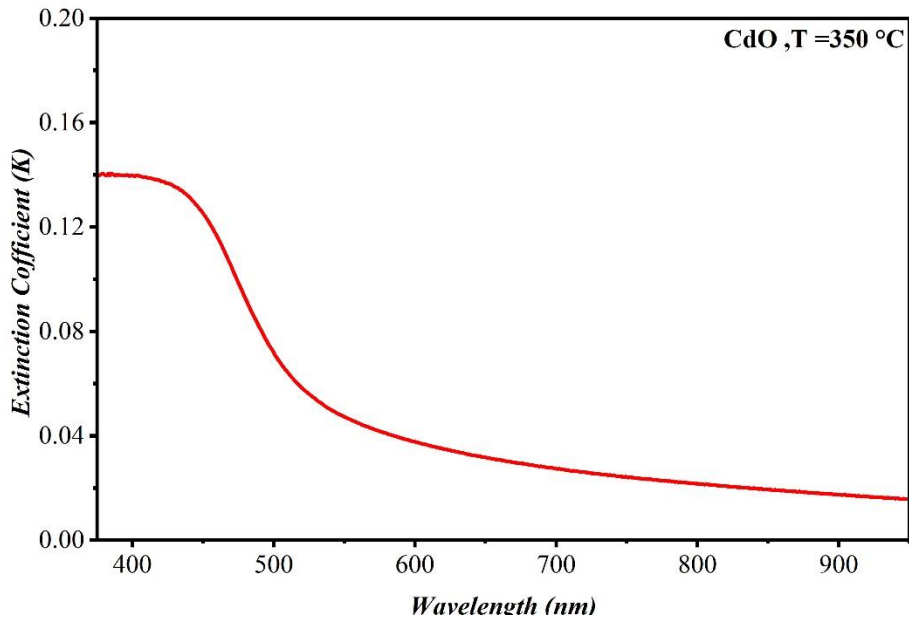


Fig.(10) extinction coefficient as a function of wavelength for CdO thin films.

3.1.6 Real dielectric constants

The complex permittivity consists of two components, real and imaginary, resulting from the interaction of light with the charges of the material, leading to its polarisation. The permittivity can be calculated from the following relationships [28]:

$$\epsilon_r = n^2 - K^2 \tag{7}$$

$$\epsilon_i = 2nK \tag{8}$$

The curve shows the relationship between the real part of the electrical insulation constant and the wavelength (λ) of thin cadmium oxide (CdO) films prepared by spin coating and heat-treated at 350 °C. The figure indicates that the real part of the dielectric constant reaches its highest value in the blue/violet spectral region (450 nm), then begins to decrease with increasing wavelength, and finally stabilises within the range of 850-950 nm (infrared region).

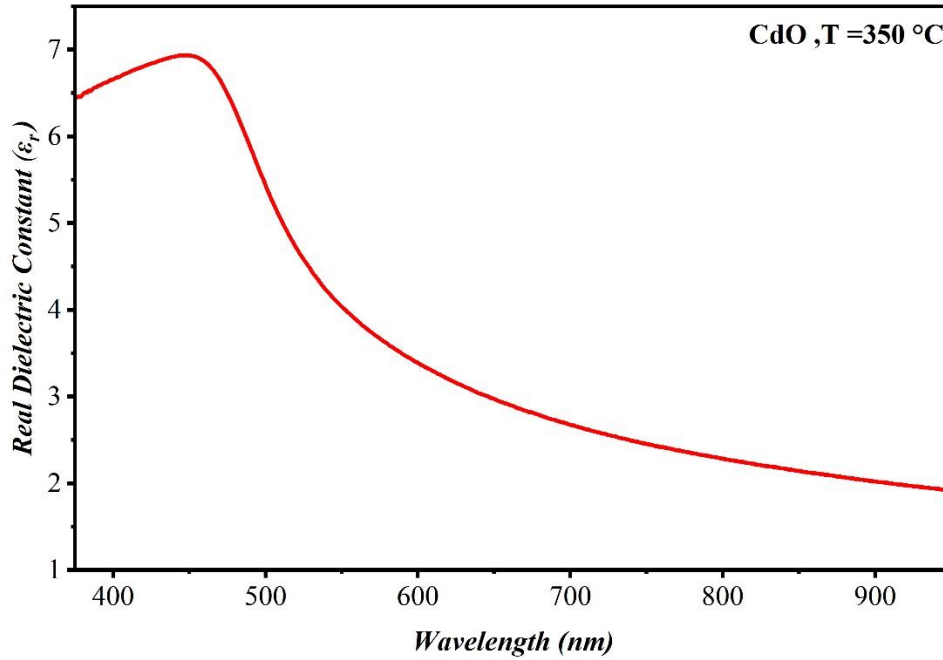


Fig.(11) Real dielectric constants as a function of wavelength for CdO thin films.

3.1.7 Imaginary dielectric constants

The curve shows the relationship between the imaginary part of the electrical insulation constant and the wavelength (λ) for thin cadmium oxide films prepared by spin coating and heat-treated at 350 °C. The figure shows that the imaginary part of the dielectric constant reaches its highest value in the blue/violet spectral region (430 nm) and then begins to decrease with increasing wavelength.

3.1.8 Optical conductivity

The interaction of light with matter leads to an increase in the number of charge carriers, known as optical conductivity. This phenomenon is one of the basic tools for studying electronic states in materials. It depends on the refractive index and the absorption coefficient. and its value is determined according to the following equation [29]:

$$\sigma_{opt.} = \frac{\alpha n c}{4\pi} \tag{9}$$

Figure 7 illustrates the optical conductivity behaviour of thin cadmium oxide (CdO) films treated at a temperature of 350 °C. Conductivity begins to increase with decreasing wavelength (increasing photon energy). This increase in conductivity is attributed to the excitation of charge carriers from the valence band to the conduction band [9]. The maximum conductivity value is at 375 within the ultraviolet range and the beginning of the visible range.

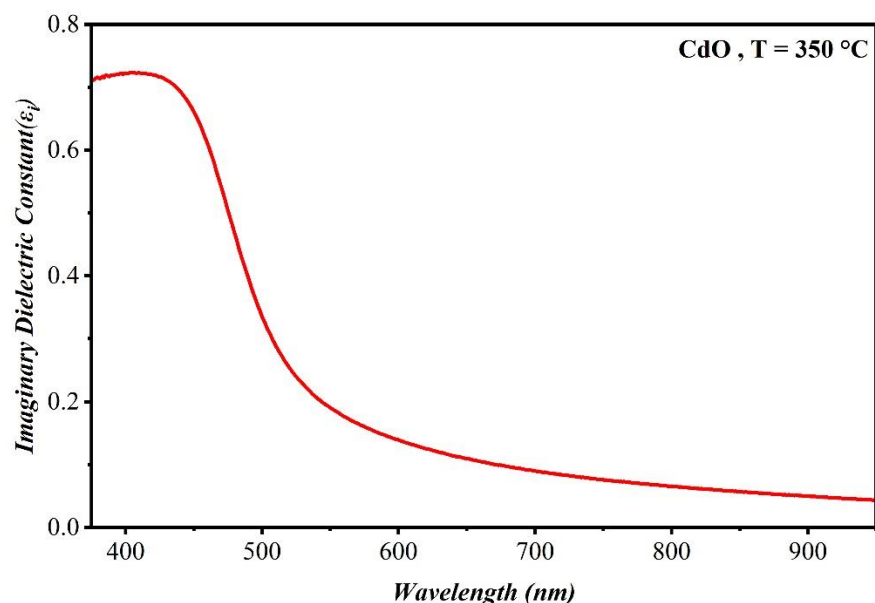


Fig.(12) Imaginary dielectric constants as a function of wavelength for CdO thin films.

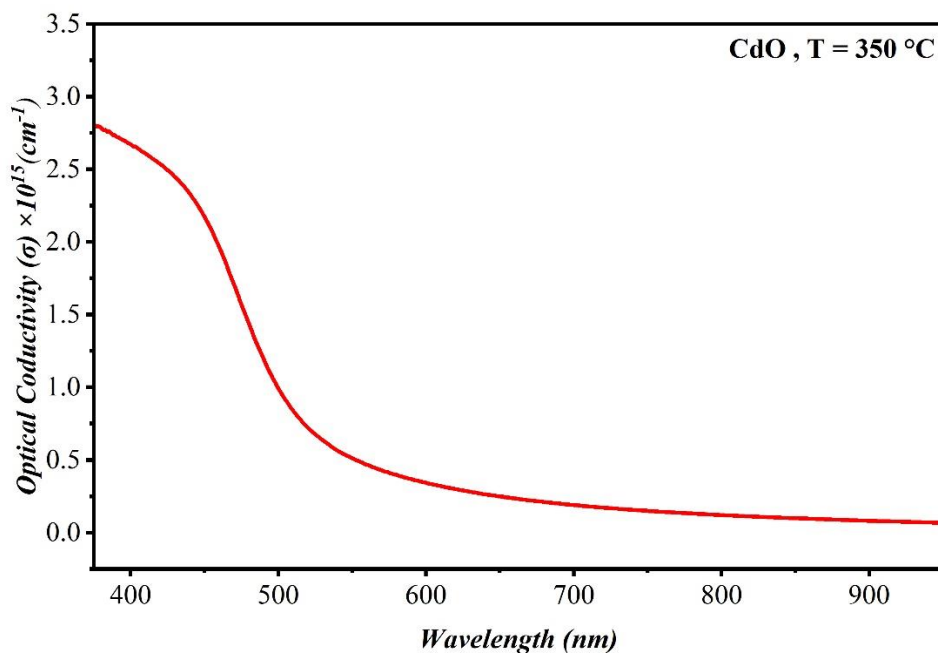


Fig.(13) Optical conductivity as a function of wavelength for CdO thin films.

3.2. Field emission scanning electron microscopy (FE-SEM) results

The scanning electron microscope (SEM) is a suitable technique for studying morphology (external surface), distribution and particle shape. It is well known that surface structure has a clear effect on optical and electrical properties and is an essential element for applications in thin film technology [30]. From observing surface images of CdO membranes prepared with a magnification of 2.50kx, the particle distribution is not random but organised in clusters and arranged in clusters that are somewhat interconnected and more widely spaced, with particle sizes ranging from small to medium and varying density patterns [31]. The structure of some traces of empty sites (porosity containing holes) is visible [18], as is the nature of the particles, which

are composed of a number of irregular and mostly spherical shapes. A statistical distribution diagram of particle sizes was drawn using Image software. The diameters of the particles were measured [32], and the average diameter was approximately 38.43 nm.

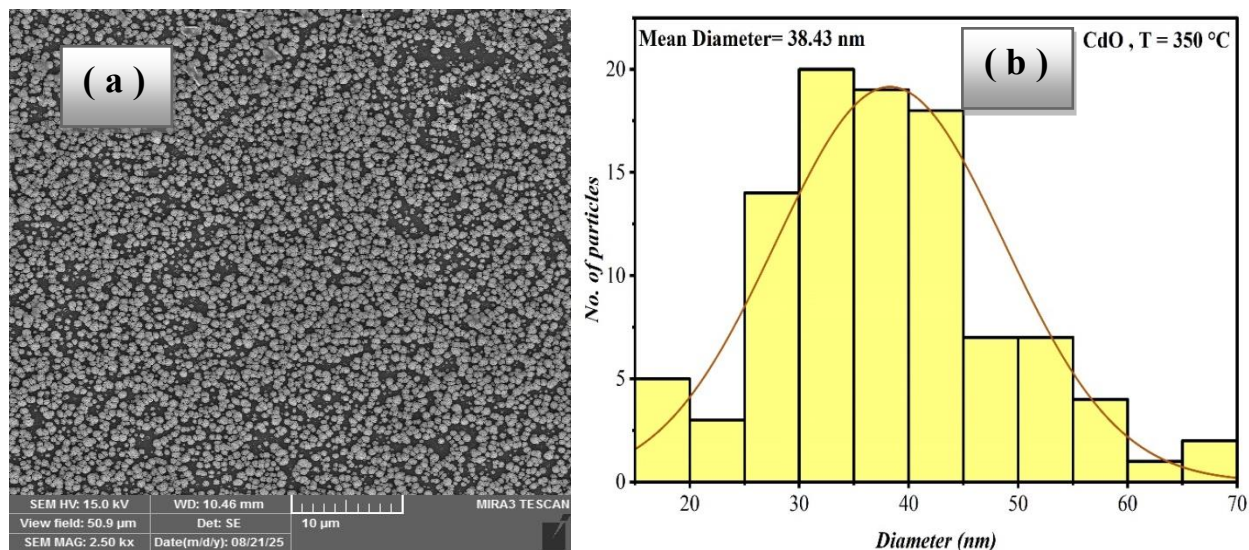


Fig.(14) a) Scanning electron microscope images of CdO membrane, b) Diameter distribution diagram

3.3. X-ray Diffraction (XRD) Results

The cadmium oxide films prepared were characterized by X-ray diffraction, and the results showed that the films had a cubic crystal structure [33]. By analysing the diffraction pattern, diffraction peaks appeared at angles of 2θ of (33.32°), (38.63°), (55.56°), (66.25°), and (69.57°), and the corresponding crystal planes (111), (020), (022), (131), and (222), as shown in Figure (15) and Table (1). The highest intensity was recorded at the (111) plane, which is the preferred direction of crystal growth, and the peaks were characterized by their narrow and sharp shape, indicating the high quality of the prepared films. The average crystal size was also calculated, and when comparing the pattern with the standard cards, it was found that there was a good match.

Table (1). X-ray diffraction (XRD) results of cadmium oxide (CdO).

T=232 nm			
Exp. 2Th. (deg.)	FWHM (deg.)	h k l	Crystallite Size (nm)
33.32	0.6927	1 1 1	11.9710975
38.63	0.6927	0 2 0	12.15260186
55.56	0.6927	0 2 2	12.96260713
66.25	0.6927	1 3 1	13.69415437
69.57	0.6927	2 2 2	13.96396555
Average Crystallite Size (nm)			12.94888528

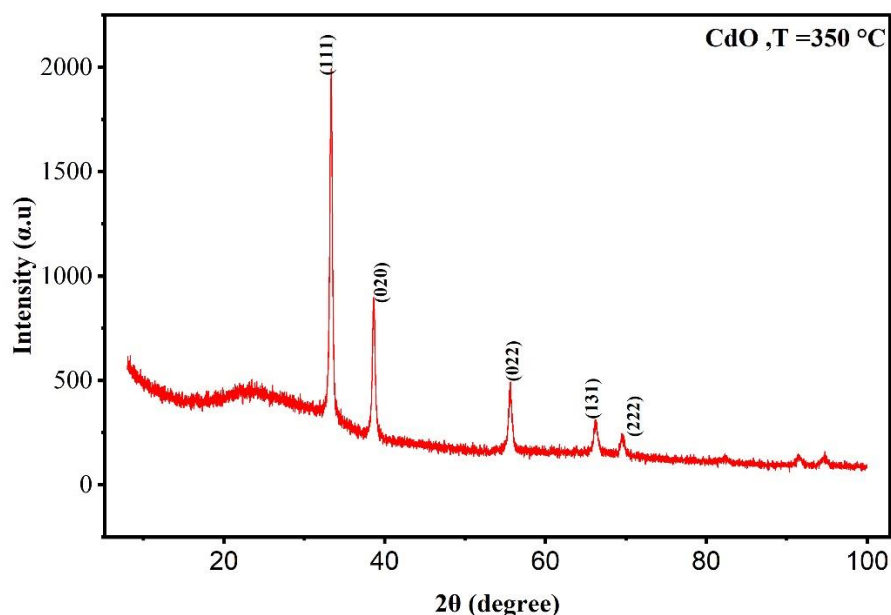


Fig. (15) X-ray diffraction (XRD) of cadmium oxide film.

Table 2: Comparison of thickness, Optical bandgap, refractive index, and XRD parameters ($2\theta^\circ$ and hkl) of CdO thin films with recent literature.

Deposition Method	Thickness (nm)	Bandgap (eV)	Refractive Index (n) at 550 nm	Reference
Spin Coating	232 ± 10	3.30	2.0	Present work
Spray Pyrolysis	150	2.26	1.46	Ahmed et al. (2018) [34]
PLD	---	2.30	2.1	Hassan (2014) [35]
Dip Coating	140 ± 3	2.35	2	Ziabari et al. (2012) [36]
Sputtering	200	2.52	---	Şakar et al. (2021) [37]

The growth of CdO nanoparticles prepared at 350 °C proceeds through uniform film formation, thermal decomposition-induced nucleation, and controlled grain growth. The moderate annealing temperature promotes high nucleation density and limited grain growth, resulting in well-crystallized CdO nanoparticles with nanoscale dimensions and improved structural quality. These characteristics make the prepared CdO films suitable for optoelectronic and gas-sensing applications.

4. Conclusions

In this study, the spin coating method was used, which is a physicochemical process for producing thin cadmium oxide at an annealing temperature of 350°C. It is inexpensive, easy to prepare, fast, repeatable, and the thickness of the membrane can be changed by controlling the spin speed. The results of ultraviolet and visible spectral analysis showed that the absorption peak is within the range of 450-375 nm, and all optical constants (absorption coefficient, refractive index, extinction coefficient, real and imaginary parts of the dielectric constant, and absorptivity) decrease with increasing wavelength and transparency in the infrared region, making them suitable for applications such as sensors. The energy gap value of the prepared films was found to be 2.30 eV, and scanning electron microscope images revealed that the cadmium oxide nanoparticles had a semi-spherical cluster structure with a porous surface and an average particle size of 38.34 nm. According to XRD measurements, the average crystal size is 12.9 nm, confirming the appearance of cubic cadmium oxide.

5. Declarations

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

5.1 Ethics approval and consent to participate

Not applicable.

5.2 Consent for publication

I agree

5.3 Availability of Data and Materials

Data will be provided upon receiving a valid request.

5.4 Conflicts of interest

The authors declare that there is no conflict of interest

5.5 Funding

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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دراسة الخصائص البصرية والمورفولوجية للأغشية أكسيد الكاديوم الرقيقة المحضرة باستخدام تقنية الطلاء الدوراني

ندى أنور شاكر⁽¹⁾، مشتاق عبد داود الجبوري^(2*)، عياد الهادي الزوام⁽³⁾

^(1,2) قسم الفيزياء، كلية التربية للعلوم الصرفة، جامعة الموصل، الموصل، العراق
⁽³⁾ قسم الفيزياء، كلية العلوم، جامعة طرابلس، ليبيا

المستخلص

هدفت الدراسة هي تحضير أغشية CdO رقيقة بسمك 232 باستخدام طريقة الطلاء الدوراني. والتي تضمنت ترسيب أسيتات الكاديوم في 2-ميثوكسي إيثانول على ركائز زجاجية، تم تحضير هذه الطبقة الرقيقة بسرعة دوران بلغت 3500 rpm لمدة 30 ثانية، ثم تم تجفيفها عند درجة حرارة 250 °C لمدة 10 دقائق، وأخيراً تمت معالجتها حرارياً عند 350 °C لمدة ساعة واحدة. تمت دراسة الخصائص البصرية لطبقات CdO الرقيقة المحضرة باستخدام التحليل الطيفي فوق البنفسجي والمرئي (UV-Visible Spectroscopy) في نطاق الطول الموجي 190-1100 nm. أشارت النتائج إلى أن الامتصاصية كانت دالة للطول الموجي وسجلت ذروة امتصاصية في نطاق 375-450 nm (منطقة الطول الموجي القصير)، ثم انخفضت تدريجياً مع زيادة الطول الموجي وبدأت في الاستقرار في نطاق 850-950 nm. تم حساب الثوابت البصرية (معامل الامتصاص، معامل الانكسار، معامل الخمود، التوصيلية، الأجزاء الحقيقية والتخيلية من ثابت العزل الكهربائي)، وأكدت النتائج أن سلوك الثوابت البصرية يتبع نمطاً مشابهاً لسلوك الامتصاص. تم حساب فجوة الطاقة للفيلم الرقيق باستخدام معادلة Tauc بلغت 2.30 eV، ولوحظت انتقالات إلكترونية من النوع المباشر. وتم استخدام مجهر إلكتروني ماسح (SEM) لدراسة شكل سطح الأغشية الرقيقة. أظهرت الصور الميكروسكوبية أن الجسيمات النانوية شكلت خلال عملية التحضير بنية عنقودية شبه كروية (مشابهة للقرنبيط) ذات سطح مسامي. التي كان متوسط حجمها 38.43 nm ومتوسط حجم البلورة قدره 12.9 nm. الخاصية الشفافية في منطقة الأشعة تحت الحمراء مما يمكنها ان تكون مناسبة لتطبيقات متعددة في علم المواد والاجهزة الالكترونية الضوئية مثل اجهزة الاستشعار.