

# Preparation and Characterization of Perfume Spray-Coated CdO Thin Films: Substrate Temperature Effect

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**ABSTRACT:** Cadmium oxide (CdO) thin films were produced in this study utilizing a spray pyrolysis approach using a perfume atomizer at varied substrate temperatures. The cubic crystal structure of the CdO was shown by XRD analysis. The size of the crystallites, dislocation density, and microstrain were determined and studied. All samples exhibit a sharp shift in transmission, indicating a straight transition and high crystallinity. In the wavelength range 400 nm - 800 nm, raising substrate temperature from 200 - 300 °C increases film transmission by up to 45 - 58%. The band gap  $E_g$  is calculated and found to be between 2.23 and 2.40 eV for the substrate temperature of 200 – 300°C. Scanning electron microscopy and energy-dispersive X-ray spectra were used to determine morphology and elemental composition, respectively. The photoluminescence spectra of the samples show violet to blue emission peaks centered around 439 nm. The films were found to have good optical properties, making them ideal for optoelectronic applications.

**KEYWORDS:** Thin films; Cadmium Oxide; X-ray diffraction; Thickness; Transmittance; Photoluminescence.

## INTRODUCTION

Because of the vast uses of thin-film devices in many disciplines such as optics, microelectronics, optoelectronic

space research, military, and medical science, car industry, and ornamentation, thin-film has now become a full-fledged

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profession. Many important semiconductor oxides have high optical characteristics. Cadmium oxide is one of them, and it has excellent optical characteristics (CdO). It has a wide range of uses due to its unique features and has been designated as a new type of transparent conductive film due to its remarkable performance in conduction and transparency [1]. Transparent metal oxide materials, such as cadmium oxide (CdO), are semiconductors with strong electrical conductivity and optical transparency that operate in the visible electromagnetic spectrum. CdO is an n-type semiconductor with metallic conductivity [2,3]. It is known that this is mostly owing to the presence of Cd interstitial or oxygen vacancies in the cubic lattice. Its direct band gap ranges from 2.2 to 2.6 eV, while its indirect band gap ranges from 1.3 to 2eV. Furthermore, CdO has a considerably high exciton binding energy in the range of 75 meV [4] crystal structure (FCC) with a lattice constant of  $a = 4.6483 \text{ \AA}$  [2] refractive index ( $n=2.75$ ), high density ( $8150 \text{ kg/m}^3$ ), low electrical resistivity, and strong transmission in the visible region [5]. As a result, it has a high potential for usage in a variety of applications such as solar cells, gas sensors, optoelectronic and photovoltaic applications [6-9].

CdO has piqued the scientific community's interest in recent decades due to distinguishing properties such as strong intrinsic mobility, huge carrier concentration even without external doping, great transparency, and so on [4]. The electrodeposition method [10], Pulsed Laser Deposition (PLD) [11], DC magnetron Sputtering [12], Chemical Vapor Deposition (CVD) [13], Chemical Bath Deposition (CBD) [14], sol-gel spin coating [15], Spray pyrolysis [16], thermal evaporation [17], sol-gel [18], reactive evaporation [19], and electrochemical method [20] were all used to prepare the CdO. The spray pyrolysis process offers several advantages, including its simplicity and low cost, the ability to prepare large-area films at air ambient temperatures, and the flexibility of compositional change to improve predicted physical qualities. The superior characteristics, superb controllability, high repeatability, and quick responsiveness are dependent on deposition factors such as spray rates, molar concentrations, substrate temperatures, deposition duration, and so on. The quality and physical qualities of the films are affected by process factors such as substrate temperature and starting solution molar concentration [20-23]. So far, several morphologies of CdO nanostructures have been described in the literature, including nanosheets [4], nanowires [24], nanotubes [25], nanoparticles [26], nanofilms [27],

nanoneedles and others. Many groups were prepared and reported about CdO thin films in various techniques. Mondal *et al.* prepared pure and Ni-doped CdO thin films by the CBD method [28]. Mahima Ranjan Das *et al.* have coated CdO thin films with the SILAR technique by the influence of cationic precursor molarity [29].

In this work, CdO thin films with varied substrate temperatures were produced on a glass substrate using perfume atomizer spray pyrolysis were reported. There are several benefits to using a perfume atomizer rather than a typical spray setup such as reproducibility, excellent operating characteristics with simplicity of use, absence of a gas phase need, precise atomization, improved hydrophilicity among sprayed tiny particles, and almost little loss of the precursor to the surroundings. The structural, morphological, elemental composition and optical characteristics of the produced films were investigated.

## EXPERIMENTAL SECTION

CdO films were deposited on glass substrates using spray pyrolysis using a perfume atomizer [30]. In the first phase of the preparation technique, 0.2 M cadmium chloride ( $\text{CdCl}_2$ ) was used as the starting material. The glass substrates (microscopic plane glass slides of area  $26 \times 76 \text{ mm}$  with  $1 \text{ mm} - 1.2 \text{ mm}$ ) were cleaned in a hot, near-boiling chromic acid solution before being washed with distilled water. To remove any pollution that had adhered to the substrates, an ultrasonicator with acetone and distilled water was used to clean them. Using a perfume atomizer, the solution was sprayed onto the cleaned glass substrates at temperatures ranging from  $200^\circ\text{C}$  to  $225^\circ\text{C}$ ,  $250^\circ\text{C}$  to  $275^\circ\text{C}$ , and  $300^\circ\text{C}$ .

The film thickness is measured using a stylus profilometer (SJ-301, Mitutoyo). Philips X Pert PRO X-ray diffraction system ( $\text{Cu-K}\alpha$  radiation;  $\lambda = 1.54056 \text{ \AA}$ ) is used to record the X-ray diffraction (XRD) pattern. The Surface morphology of the coated films is analyzed using a scanning electron microscope (SEM). The optical transmittance spectrum is recorded using a UV-Vis spectrometer (Shimadzu UV-1601). The room temperature photoluminescence (PL) spectrum is recorded using a (Shimadzu-5301) spectrofluorometer.

## RESULT AND DISCUSSION

### Surface morphology and compositional analysis

Fig. 1 (a-f) depicts the surface morphology of CdO thin films deposited with various substrate temperatures.

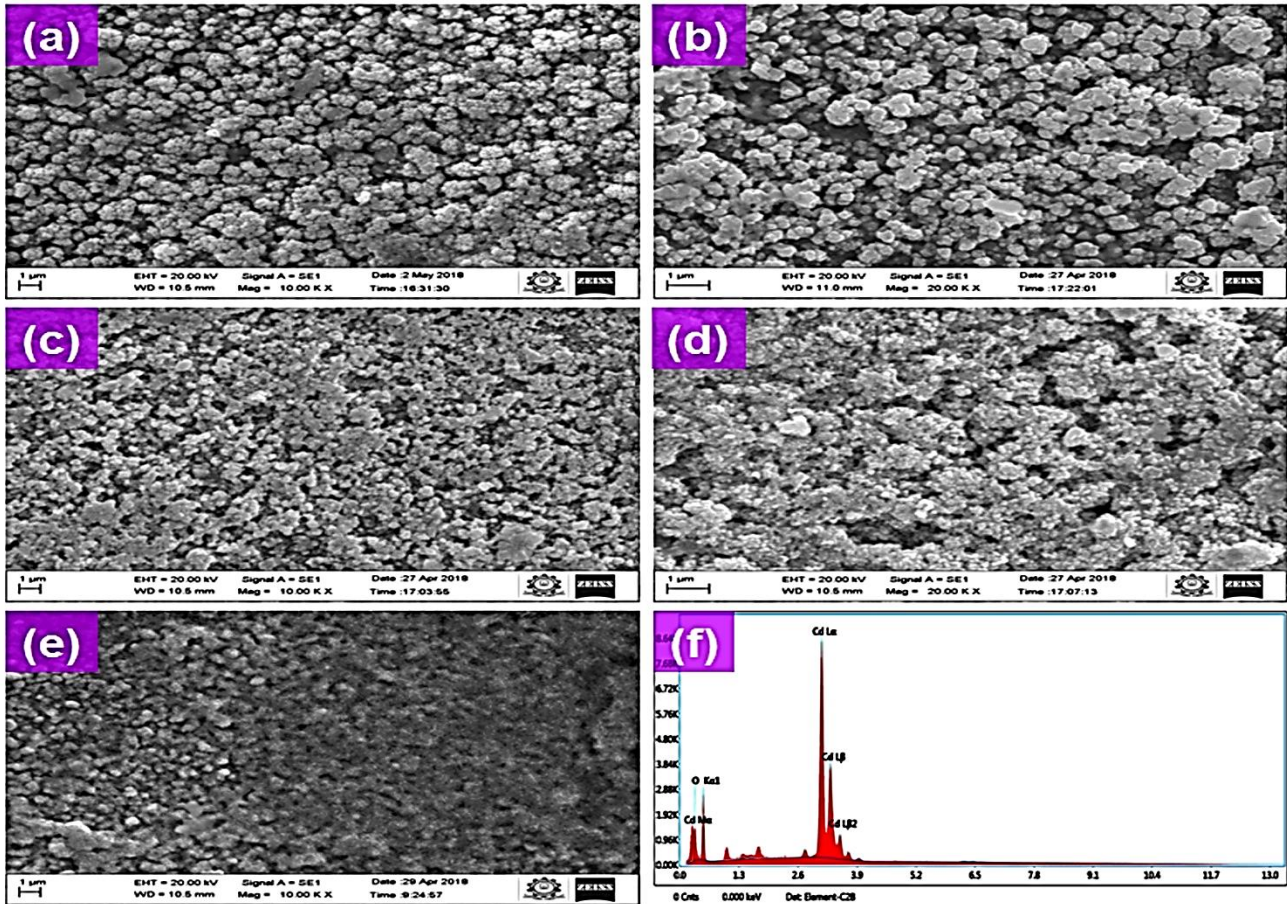


Fig. 1: SEM image of CdO thin film at substrate temperature (a) 200°C, (b) 225°C (c) 250°C, (d) 275°C, (e) 300°C, and (f) EDAX spectrum

The micrographs of CdO thin films produced on glass substrates at 200 °C demonstrate evenly dispersed spherical grains and homogeneous surface morphology free of fractures and pinholes. Spherical grains with well-defined borders and almost equal sizes were observed distributed across the surface, demonstrating the film's increased crystallinity. Thin films formed on glass substrates exhibit grain homogeneity, which may be ascribed to consistent nucleation on the substrate's surface [31]. As the substrate temperature rises, the particles seem to be closely packed and are organized into tiny clusters by agglomerating with surrounding particles. With rising substrate temperature, the surface microstructure and particle size change. The variation in atomic radius and electro-negativity of the dopant ions, which affects the thermodynamically stable growth process, may be responsible for this microstructural modification [32]. At 300°C, the particles are decreased and porous, with some holes.

The EDAX spectra of a typical 0.2 M CdO thin film formed at 300°C substrate temperatures are shown

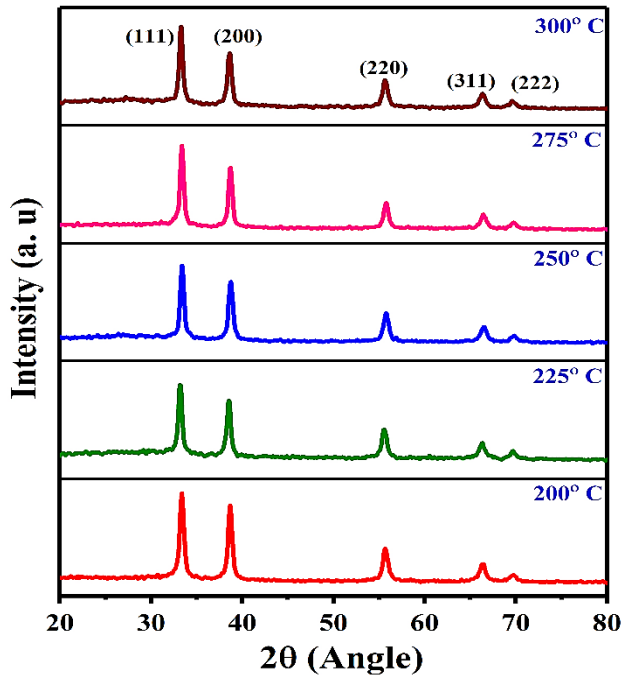
in Fig. 1-e. The existence of Cd and O elements in deposited CdO film is verified by EDAX spectra, and their atomic percentages are 25.05 percent and 74.95 percent, respectively. EDAX analysis reveals the existence of Cd and O elements without any additional impurity, indicating the thin film's purity [33]. The results show that there is a significant proportion of oxygen present.

#### X-Ray Diffraction (XRD) analysis

The X-ray diffraction pattern of CdO thin films produced for various substrate temperatures is shown in Fig. 2. It demonstrates that the films are highly orientated along the planes (111), (200), (220), (311), and (222) and their corresponding  $2\theta$  values are 33°, 39°, 55°, 66° and 69° respectively. All of the diffraction peak positions agreed well with JCPDS card no. 78-0653. The existence of diffraction peaks indicates that the material is polycrystalline with a face-centered cubic crystal structure. *Salunkhe et al.* and *Bari et al.* [34,35] obtained similar findings using the spray approach. The intensity

**Table 1: The estimated values of crystalline size, strain, dislocation density, and, lattice constants for CdO thin films for different substrate temperatures.**

Substrate Temperature °C	Lattice constant 'a' (Å)	Film Thickness (nm)	Crystallite size (nm)	Dislocation density ( $\times 10^{15}$ lines.m <sup>-2</sup> )	Strain ( $\times 10^{-3}$ )	Number of crystallites ( $\times 10^{16}$ m <sup>-2</sup> )	Band gap (eV)
200	4.646	980	21	2.2534	5.77	10.48	2.23
225	4.650	920	24	1.7241	5.01	6.58	2.32
250	4.651	850	25	1.7292	5.01	6.08	2.34
275	4.659	820	33	0.8000	3.59	2.13	2.37
300	4.677	800	34	0.8795	3.58	2.08	2.40

**Fig. 2: XRD pattern of CdO thin films.**

the (111) plane was observed to rise as the substrate temperature rose. This suggests that more Cd<sup>2+</sup> ions were incorporated into the film during the spray pyrolysis technique. However, when the substrate temperature increased, the strength of the peaks increased, indicating improved crystallinity and grain size [36,37].

The crystallite size of all deposited films is estimated using the well-known Scherrer's formula [38].

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad (1)$$

Where  $D$  is the crystallite size,  $\lambda$  is the wavelength of the X-ray used ( $\text{CuK}\alpha = 1.5406 \text{ \AA}$ );  $\beta$  – is full width at half maximum intensity and  $\theta$  is the peak position.

Table 1 summarises the structural characteristics estimated for the cubic phase of the films. The grain size ( $D$ ) was determined using Debye Scherrer's formula and was found to vary between 21 and 34 nm for films with varying

substrate temperatures. The size of the crystallites grows as the substrate temperature rises, showing that crystallization improves as the growth temperature rises. The diffusion of sputtering species is improved as grain size increases with rising substrate temperature. When the temperature is increased, the thickness of the prepared film is decreased because of that the atoms on the surface of the film can move faster to look for the lowest energy sites and from the low energy structure at relatively high temperatures as well as this might be because film growth is dominating at low substrate temperatures, but formed film evaporation at higher substrate temperatures is a significant component driving the decrease in film thickness.

As indicated in Table (1), the predicted crystallite size values are in the nano range. The size of the crystallites grows as the substrate temperature rises. This might be owing to the tendency of smaller crystallites to agglomerate to generate bigger crystallites. The defects like strain ( $\epsilon$ ) and dislocation density ( $\delta$ ) for the CdO were evaluated using the relations [39-41]

$$\epsilon = \frac{\beta \cot \theta}{4} \quad (2)$$

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

And the number of crystallites ( $n_c$ ) as

$$n_c = \frac{t}{D^3} \quad (4)$$

where  $t$  is the thickness of the film as indicated in Table 1, all of the films had a low micro-strain value, which supports better crystallinity. The dislocation density and strain decrease as the grain size rises, indicating that the crystallinity improves. This augmentation is due to the enhancement of clusters and the elimination of flaws produced during film deposition.

The cubic phase lattice constant 'a' of CdO films was found via the following relationships

$$\frac{1}{d^2} = \left\{ \frac{h^2 + k^2 + l^2}{a^2} \right\} \quad (5)$$

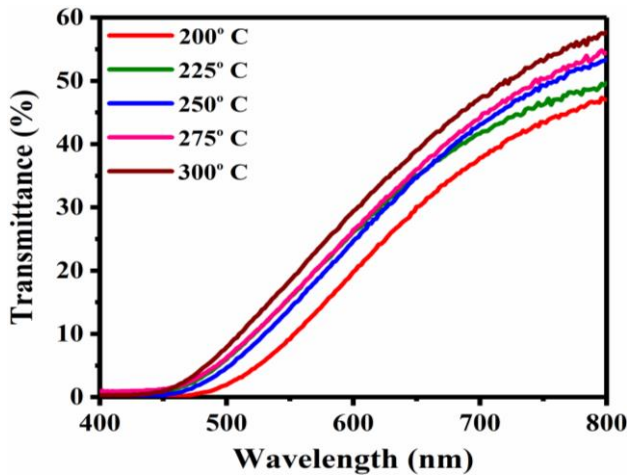


Fig. 3: UV transmittance spectrum of CdO thin films.

Where,  $(hkl)$  and  $d$  have their usual meanings. The lattice constant ( $a$ ) of CdO thin films is found in the range of  $4.646 \text{ \AA} - 4.677 \text{ \AA}$  and its values well agree with standard JCPDS data.

#### Optical studies

The optical characteristics of CdO thin films were investigated using a UV-Vis Spectrophotometer in the 400-800 nm wavelength range. Fig. 3 depicts the transmittance spectra of CdO thin films formed at various substrate temperatures. All of the produced films had a high transmittance in the visible range, indicating a straight transition and excellent crystallinity [42]. The transmittance of the films rose with increasing substrate temperature, with a transmittance of 45-58 percent. This increase in transmittance with increasing substrate temperature is caused by changes in the flaws, internal strain, and crystallite characteristics of the CdO thin films. The increasing transmittance in the visible range is consistent with Velusamy *et al.* [43].

The absorption coefficient is an essential metric for measuring a material's ability to absorb light, and it may be computed using the relationship from the transmittance spectrum (6)

$$\alpha = \left(\frac{1}{t}\right) \ln \left(\frac{1}{T}\right) \quad (6)$$

where  $t$  is the thickness of the deposited film and  $T$  is its transmittance. The absorption coefficient is connected to the energy band gap in solid band theory and may be stated using Tauc's equation.

$$\alpha h\nu = A (h\nu - E_g)^n \quad (7)$$

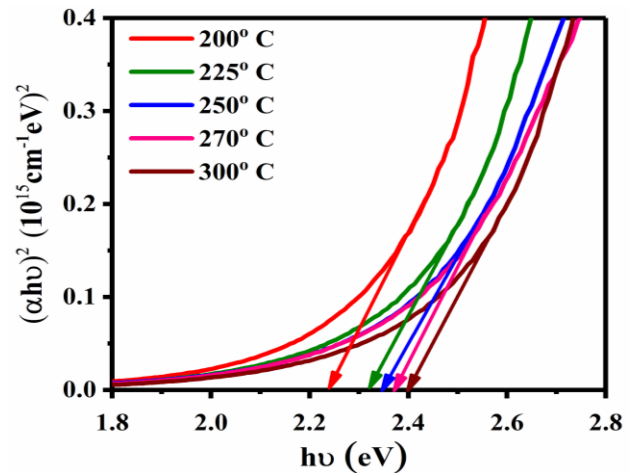


Fig. 4: Tauc plot of CdO thin films.

Where  $A$  is the characteristic constant,  $h$  is Planck's constant,  $\nu$  is the incoming photon frequency,  $E_g$  is the material's bandgap, and the exponent term  $n$  depends on the kind of transition. For crystalline semiconductors,  $n$  has values of  $1/2$ ,  $2$ ,  $3/2$ , and  $3$ , corresponding to allow direct, allow indirect, prohibited direct, and forbidden indirect transitions. Because CdO has a straight bandgap, the Tauc plot may be displayed between the photon energy  $h\nu$  on the X-axis and  $(\alpha h\nu)^2$  on the Y-axis, as illustrated in Fig. (4). The optical band gap energy for the films is obtained by extrapolating the linear part to a zero-absorption coefficient ( $\alpha=0$ ).

As the substrate temperature climbed from  $200^\circ\text{C}$  to  $300^\circ\text{C}$ , the band gap values ranged from 2.23 eV to 2.40 eV. The growing substrate temperature process lowers from the secondary levels and the structural flaws that lead to the contract tails region, resulting in a rise in the optical energy gap values [44]. Desai *et al* observed that the optical band gap for sprayed cadmium oxide thin film is in the range of 2.23 to 2.46 eV [45].

#### Photoluminescence studies

The photoluminescence spectra of CdO thin films produced at varied substrate temperatures are shown in Fig. 5. It is possible to discern emission maxima at 359, 376, 411, 439, 459, 494, 519, 544, and 572 nm. The sharp emission peak at 359 nm is attributed to UV emission caused by free exciton recombination [46]. The violet emission peak at 411 nm might be attributable to point defects in CdO caused by Cd or Vo, or to excitons linked to a donor-level impurity present in CdO thin film [32], whereas the peak at 439 nm could be caused by a singly ionized oxygen vacancy [46].

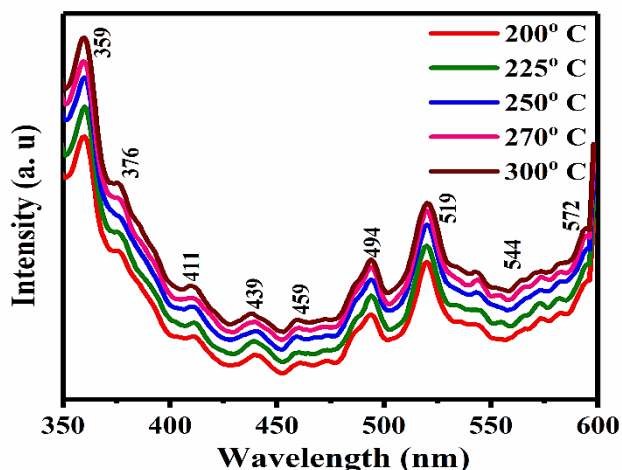


Fig. 5: Room temperature PL spectrum of CdO thin films

A high-intensity peak at 459 nm (blue region) corresponds to the NBE of CdO thin films, which is caused by the radioactive recombination of electrons and hole from the conduction and valence bands [47]. The peak at 494 nm is attributed to excitonic transitions, which are size-dependent and excitation wavelength-independent in a limited wavelength range [47]. The emission peaks at 493 nm are caused by the electron transition between cadmium interstitials (Cdi) and cadmium vacancy (Vcd) states. The emission peak at 519 nm is caused by a less size-dependent surface defect state and deep trap emission owing to intrinsic structural defects and impurities [47, 49]. Oxygen vacancy-related peaks were also found at 544 and 572 nm [47].

## CONCLUSIONS

The spray pyrolysis procedure was used to efficiently create cadmium oxide thin films on glass substrates at various substrate temperatures. To confirm the growth and phase purity of the made films, an XRD investigation was done, and the findings indicated that the polycrystalline growth of the films with a cubic structure has a favored orientation along the (1 1 1) plane. Using the XRD data, the crystalline size, dislocation density, and microstrain values were calculated. The crystallite size of the films increases as the substrate temperature rises, but other properties decrease. A straight transition and strong crystallinity are indicated by the UV examinations, which show a significant shift in transmission in all of the samples. By increasing the substrate temperature from 200 to 300 °C, film transmission in the 400–800 nm wavelength region rises by up to 45–58%. When the substrate temperature is between 200°C and 300°C, the band gap Eg is computed

and determined to be between 2.23 and 2.40 eV. The modulated shape and composition of the Cd and O elements, respectively, were confirmed by energy-dispersive X-ray spectroscopy and scanning electron microscopy. Violet to blue emission peaks with centers at 439 nm is visible in the samples' photoluminescence spectra. The films were discovered to have excellent optical characteristics, which made them perfect for optoelectronic applications.

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