Effect of Photoconductivity Precursor Volume on Structural, Physical, Electrical and Optical Properties of Thin Layers of Cadmium Oxide (CdO) Nanostructures Produced Using Spray Pyrolysis Technique

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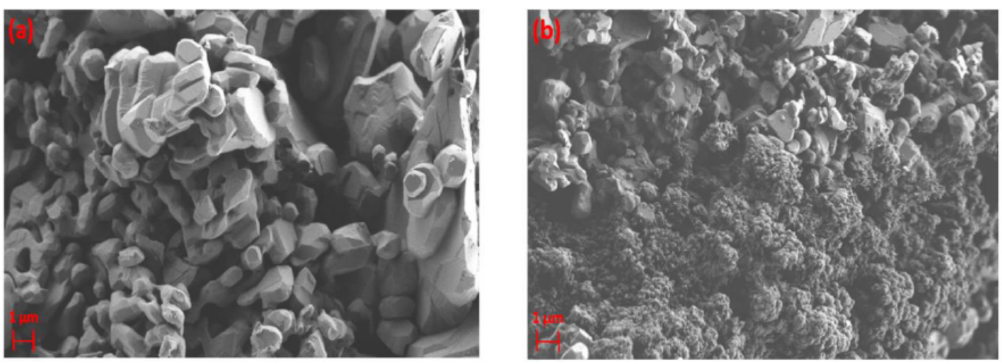
Abstract: Thin layers of Cadmium Oxide (CdO) are produced over glassy substrate by spray pyrolysis technique with precursor volumes of 50, 75 and 100 (ml). FESEM images of samples show the formation of nanometric structures and structural characterization of them resulted from XRD spectroscopy indicate the formation of cubic polycrystalline structure in growing layers with preferred direction of (111). Evaluating the optical properties of samples show that optical band gap of layers is reduced from 3.6 to 3.4 (eV) by increasing the precursor volume and the optical absorption coefficient of samples is in UV region at about 10^5 (cm^{-1}). Data analysis indicates that the produced sample in volume of 100 mL has the smallest penetration depth (smaller than 200 nm) in UV region. On the other hand, thin layers of Cadmium Oxide (CdO) with various volumes of Cadmium acetate solution (40, 50 and 70 ml) were deposited using spray pyrolysis technique over a glassy substrate. Samples were investigated using FESEM images, XRD and UV-Vis spectra as well as I-V characteristic. It was found that all samples were grew up with polycrystalline nanostructures along the preferred direction of (002). In addition, it was found that grew up sample in the volume of 50 (ml) are of optimum photoconductivity condition in visible range regarding optimum structural (largest crystallite size and lowest crystallite defect density) and optical (smallest band gap and highest light absorption) conditions.



SEM images of thin layers of Cadmium Oxide (CdO) nanostructures (a) before and (b) after using spray pyrolysis technique with 50000x zoom.

Keywords: Cadmium Oxide (CdO), Spray Pyrolysis Technique, Optical Properties, Penetration Depth, Photoconductivity, Nanostructures, Visible Light, Structural Properties, Physical Properties, Electrical Properties.

1. INTRODUCTION

Transparent, conductor oxide layers such as SnO_2, ZnO and ITO are widely used as transparent electrode, window covers and in opto-electronic pieces. However, thin layers of Cadmium Oxide (CdO) are mostly interested due to having semi-conductive property with conductivity type p and direct and wide band gap between 3.5 and 4 (eV) and as a promising material for electrochromic pieces, lasers, electrode manufacturing in p-n connections and layer type p for UV light detection [1-27]. Pure Cadmium Oxide (CdO) has specific resistivity of 10^3-10^{13} (Ω.cm) at room temperature and by approaching to elemental proportionality, material becomes more dielectric. This increase in resistivity is due to reduction of hole density

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that is expected in a pure and complete crystal with wide band gap [28-71].

The aim of the current study is synthesizing thin layers of Cadmium Oxide (CdO) by spray pyrolysis technique and investigating the effect of variation of precursor volume on optical and photoconductivity properties of the layers.

Cadmium Oxide (CdO) is one of the rare inherent semi-conductors of type P with a narrow band gap of about 1.2-2.1 (eV) which has a monoclinic structure with limited transparency in the region of visible light [72-104]. Thin layers of this material are frequently dark brown to black. This darkness is due to narrow band gap and direct transitions between bands [105-125]. This fact leads to high absorption of visible light and can be used in optical pieces such as solar cells. In addition, this material is considered due to abundance of raw material, non-toxicity, easy production and ability to change and optimizing its physical properties using various physical and chemical methods such as chemical vapor deposition [117-125], spray pyrolysis [118-123] and so on. This material is one of the important mineral oxides for applying in pieces such as solar cells, electrochromic pieces and gaseous sensors due to its availability, high absorption rate and low cost [104-107].

In the current research, cost effective spray pyrolysis technique was used to investigate photoconductivity of Cadmium Oxide (CdO) thin layers with various volumes of spray solution.

2. MATERIALS AND EXPERIMENTAL TECHNIQUES, METHODS AND WORK DETAILS

To prepare thin layers of Cadmium Oxide (CdO), Cadmium acetate powder was solved in deionized water and 0.15 (M) Cadmium acetate solution was prepared. Then, this solution was sprayed over glassy substrate in various volumes (40, 50, 70 ml)-corresponding to samples of V1, V2, V3-to prepare the considered layers. It is expected that in pyrolysis process, the following chemical reaction mechanism happens [108-115]:

$$Cd(CH_2COO)_2.H_2O+H_2O \rightarrow CdO+2CH_3COOH+H_2O$$

During each step, cleaned substrates were heated up to 440º C in spray device and then, solution was sprayed under air pressure (1.1 bar). In this process, distance of sprays from substrates was 35 (cm).

Structural analysis of samples was performed by X-Ray Diffraction device (XRD, Brucker AXS) with CuKα spectral line emission (1.5405 Å) and the surface morphology of samples were investigated by Scanning Electron Microscopy (FESEM Hitachi S.4160). Optical characteristics of layers were measured using passed and absorbed spectra by optical spectroscopy (Shimadzu UV-Vis 1800) in the range of 300-1100 (nm).

Thin layers of Cadmium Oxide (CdO) were synthesized over glass by spray pyrolysis technique. The precursor used for spraying was produced from Cadmium chloride hexahydrate powder with various volumes of two times de-ionized water. In the current research, spray distance from hot plate surface was 35 (cm), deposition rate was 10 (ml/min), precursor molar concentration was 0.1 (M), substrate temperature was 410º C and three samples of V1, V2 and V3 were produced by volume of 50, 75 and 100 (ml), respectively.

For structural characterization of samples, X-Ray Diffraction (XRD) was performed by D8 Advanced-Bruker XRD device dependent on spectral line CuKα with 0.15406 (nm) wavelength and 2θ between 10-70 degrees. To measure the transited and reflected spectra, UV-Vis.-Shimadzo-1800 spectrophotometer device was used in the range of 300-1100 (nm). Surface morphology of samples was determined by Field Emission Scanning Electron Microscope device (FESEM; Hitachi S-4160).

3. RESULTS AND DISCUSSIONS

3.1. Surface Morphology of Layers

Figure (**1**) shows the obtained images from samples V1, V2 and V3 in the scale of 500 (nm) from top (left side) and cross section (right side) for determining the thickness of samples. As can be observed, increasing the precursor volume of spray leads to more surface porosity of samples with larger gradation as well as larger thickness (220, 313 and 275 nm, respectively).

Figure (**2**) shows SEM images of samples in the scales of 5 microns and 500 (nm). Although the images for V1 and V3 samples show uniform surface along with some grains with 50 and 100 (nm), respectively, V2 sample is of porous surface along with woven fibers and mud-like particles that differentiate it from two other sample.

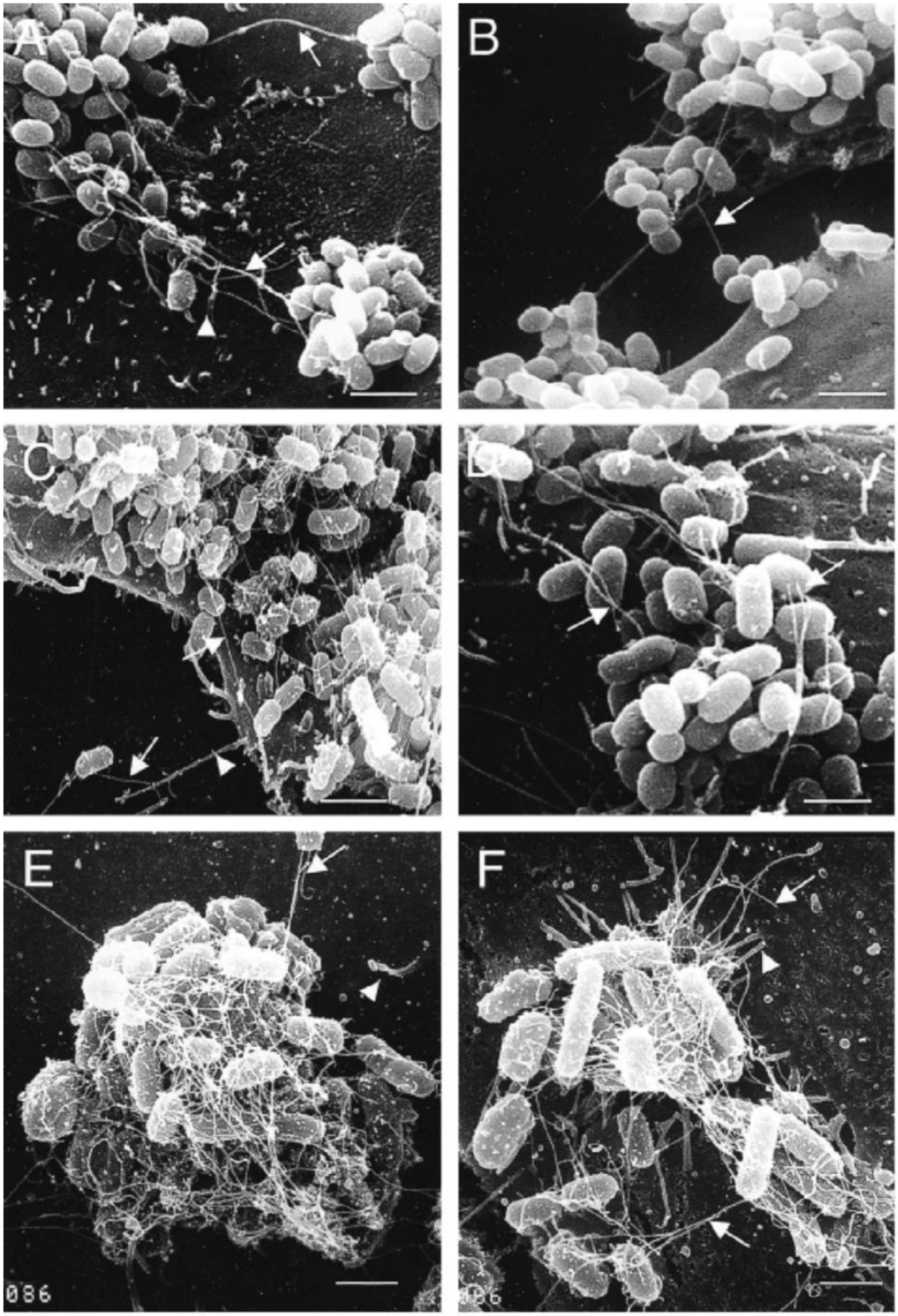

Figure 1: FESEM images of samples V1, V2 and V3.

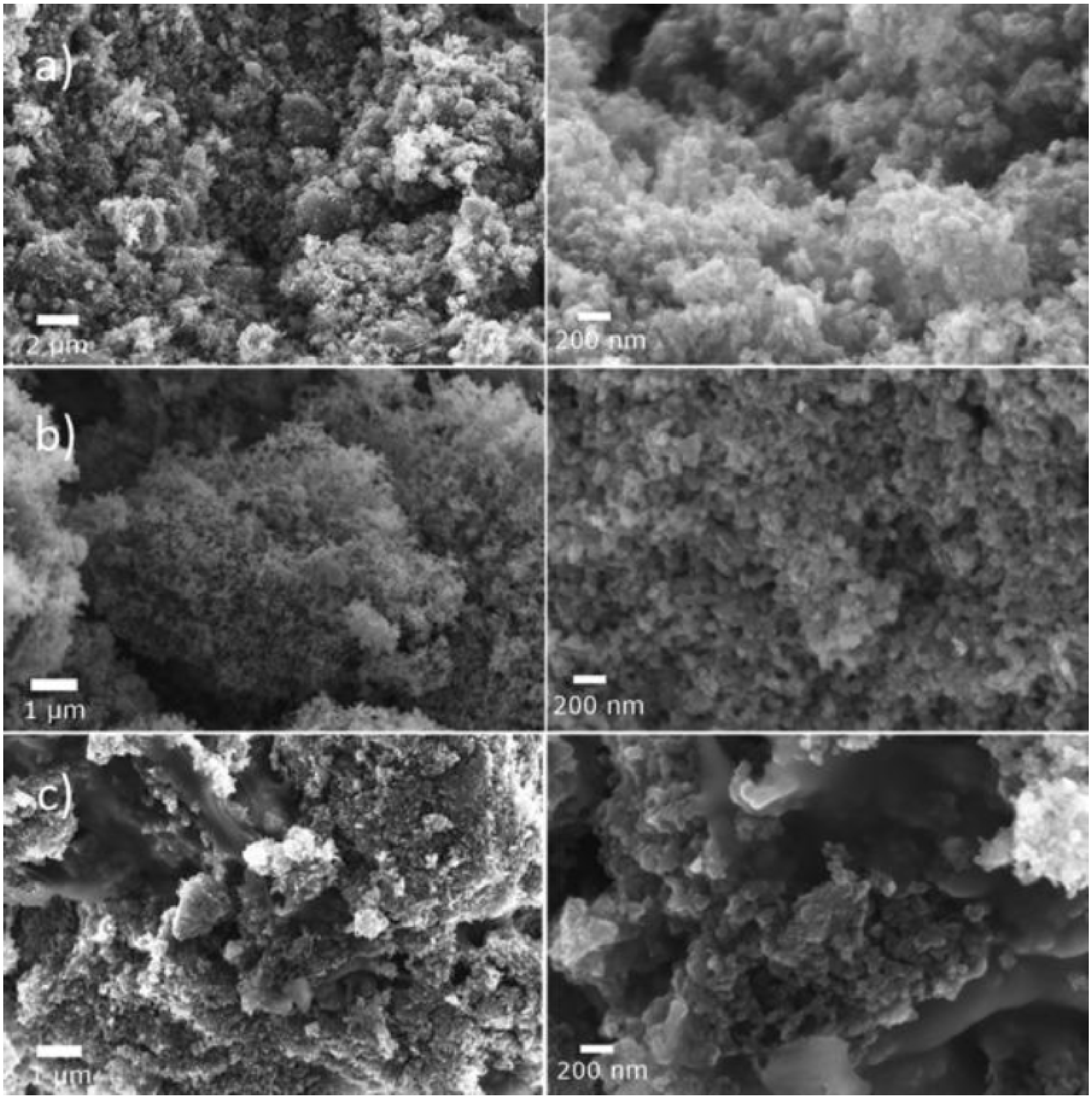

Figure 2: SEM image of thin layers of Cadmium Oxide (CdO) for samples prepared in various volumes.

3.2. Structural Properties

Figure (**3**) shows XRD spectra for grew up samples with various volumes. The obtained results show that all three samples only have been grew up with a cubic polycrystalline phase of Cadmium Oxide (CdO) in the preferred direction of (111).

Table (**1**) summarizes the detailed data related to XRD spectra for various samples, in addition to Full Width at Half Maximum (FWHM). The results indicate that by increasing the volume, spectral width of peak (111) is reduced, which can be due to variation of crystalline defects because of thickness variation during growing up [116-123].

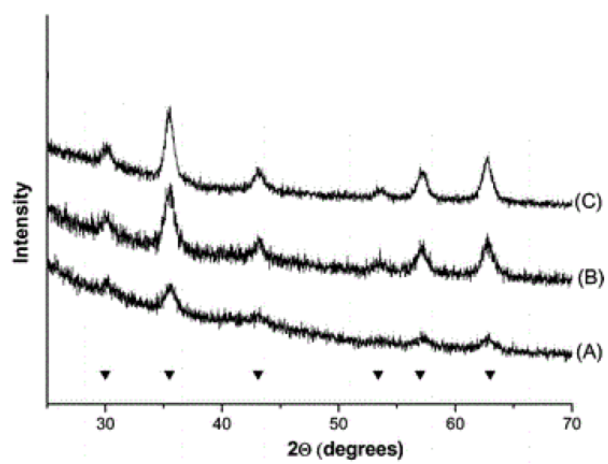

Figure 3: XRD spectrums for investigated Cadmium Oxide (CdO) samples.

Table 1: XRD Spectrums Data for Various Samples and their Analyses using Scherrer Equation Related to Peak (111)

D (nm)	2θ (Degree)	FWHM (Degree)	Sample
21.23	41.27	0.59	V1
21.56	41.63	0.55	V2
21.97	41.77	0.51	V3

The fourth column in the table shows the size of crystallites in the grew up samples which can be calculated by Scherrer equation:

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

where, λ is X-Ray wavelength, β is full width at half maximum and θ is Bragg's angle of diffraction.

Regarding the results listed for crystallite size in Table (**1**), it can be seen that the crystallite size is increase by increase in the volume of sample. These variations affect the optical properties of samples as shown in the following sections [124, 125].

XRD spectrums of samples are shown in Figure (**4**). Diffraction curves of samples indicate that they are of polycrystalline structure with monoclinic structure and principal planes of (002) and (111) located at angles of 35.56° and 38.74°. The results indicate that V2 sample with solution volume of 50 ml is of weaker peaks at directions of (202) and (020) at angles of 48.86° and 53.85°, respectively. The presence of these peaks along with relative intensity of the major peaks indicate that crystalline structure improves compared to other samples.

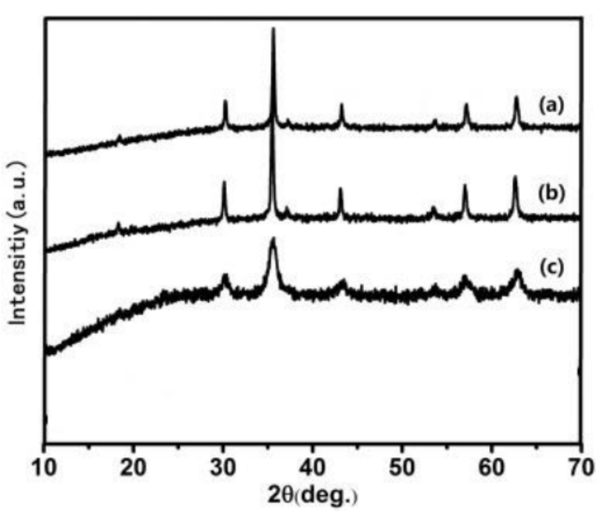

Figure 4: XRD spectrums of Cadmium Oxide (CdO) layers with various volumes of solution.

For more accurate investigation of structural properties, crystallite size (D), dislocation density (δ) and crystalline strains (ε) are calculated [111-117]:

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

$$\varepsilon = \frac{\lambda}{D\sin\theta} - \frac{\beta}{\tan\theta} \quad (3)$$

where, β is half width at full maximum, D is crystallite size, θ is Brug angle and λ is X-Ray wavelength. Results of these calculations are listed in Table (**2**).

Table 2: Calculated Structural Properties for the Preferred Peak (002)

$\varepsilon(\times 10^{-3})$	$\delta(\times 10^{-2}\,nm^{-2})$	$D(nm)$	Sample
2.69	0.597	17.49	V1
2.31	0.323	19.93	V2
3.81	0.499	15.37	V2

3.3. Optical Properties

Figure (**5**) shows optical transition and reflection spectra for layers as a function of incident wavelength. According to experimental data, it can be seen that transition spectrums of layers in visible region is reduced from 55% to 30% by increasing the precursor volume. This reduction can be mainly due to increasing the thickness of investigated layers. The results of reflection spectrums of layers indicate that sample V3 with the highest surface porosity is of lowest reflection in visible region (about 1.5%), as expected

In order to determine direct band gap (Eg) of samples, the following equation can be used:

$$(ah\nu)^2 = A(h\nu - E_g) \quad (4)$$

By illustrating $(ahv)^2$ in terms of hv and determining intercept on the horizontal axis for zero optical absorption (a=0) in high energy region, band gap can

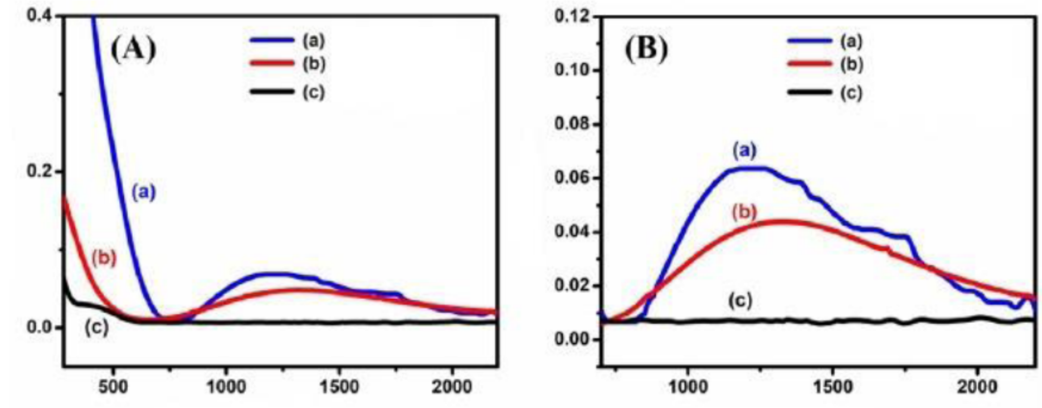

Figure 5: Optical (**a**) transition and (**b**) reflection spectra for samples.

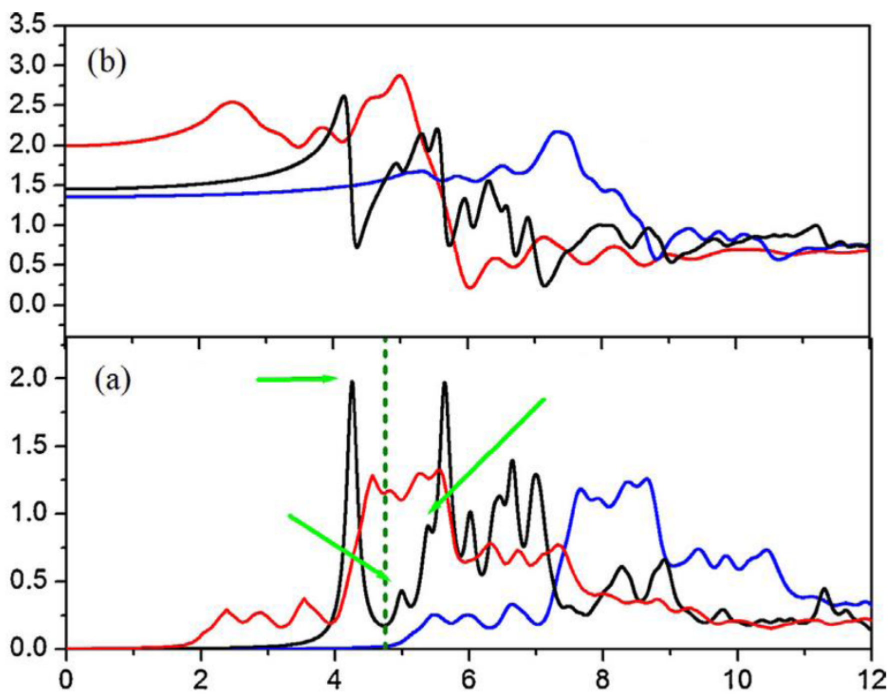

Figure 6: Direct optical (**a**) absorption spectrums and (**b**) band gap for the investigated samples.

be obtained. Figures (**6.a**) and (**6.b**) show experimental data related to absorption spectrums of layers and the results related to this analysis (Eq. (4)) and variations of band gap in the samples, respectively. The appendix image in Figure (**6.b**) indicates the variation of optical band gap of samples against precursor volume. As can be observed, band gap is reduced from 3.6 to 3.4 (eV) as precursor volume is increased. This reduction can be attributed to reduction of effectiveness of quantum limitation in nanostructures of these layers, regarding the increase in crystallite size (which is in good agreement with FESEM images of samples).

One of the most important parameters for determining the optical properties of material is penetration depth of light into the layer that is inverse of absorption coefficient (α) of material. In order to determine this quality, the following equation can be used [118-123]:

$$\alpha = 2.3026\frac{a}{t} \tag{5}$$

where, a is optical absorption of layer and t is layer thickness.

Figure (**7.a**) shows theoretical curves related to absorption coefficient of these layers obtained from Eq. (5). The results indicate that all three samples are of high absorption coefficient (~ 10^5 cm^{-1}) in energies

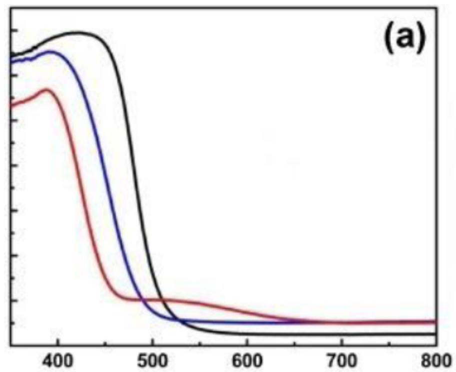

Figure 7: (**a**) absorption coefficient and (**b**) penetration depth in terms of wavelength.

higher than ~ 3.5 ± 0.1 (eV) in UV light region. This absorption edge is in good agreement with optical band gap values of samples.

Figure (**7.b**) shows the variation of penetration depth ($\delta = 1/\alpha$) in room temperature as a function of incident photon energy. As can be seen, in UV light region (wavelengths lower than ~ 400 nm), penetration depth of layers is lower than 200 (nm). It is expected that the main part of considered photons absorb by the layer regarding the layer thickness. This issue is of great importance in manufacturing the optical sensors in considered wavelengths. Regarding the obtained results from comparison of samples, sample V3 is of lowest penetration depth in UV light region.

Figure (**8**) shows optical transmittance spectrums of the under studied layers. It can be seen that in visible region of 400-700 (nm), V2 sample and V3 sample are of the lowest and highest transmittance, respectively. These variations may be largely due to relative

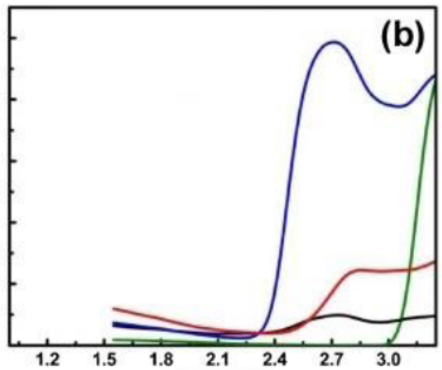

Figure 8: Transmittance optical spectrums of Cadmium Oxide (CdO) thin layers grew up in various volumes.

electrical conductivity of layers (Section 4.4) which is effective is relative amount of metal-like and or insulator-like of layers.

According to the reported results, Cadmium Oxide (CdO) layers are acted as a semiconductor with direct transition between bands so that during these transitions, absorption coefficient is a function of incident photon energy [118-125]. Figure (**9**) shows the variations of absorption spectra of layers against wavelength.

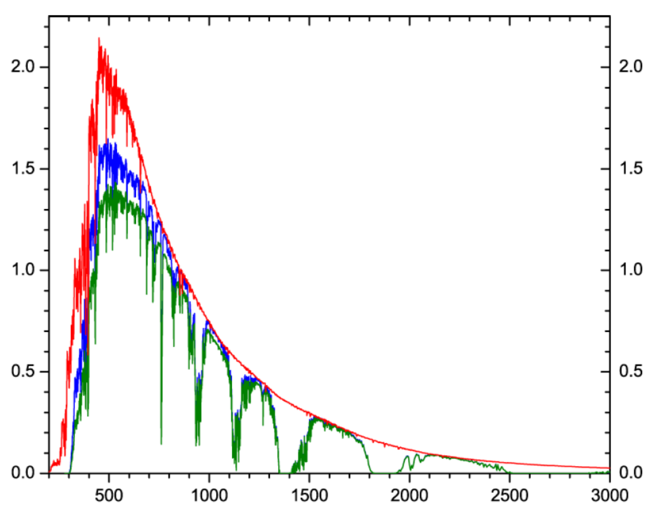

Figure 9: Absorption spectrums of under studied samples in terms of wavelength.

Since Cadmium Oxide (CdO) is a semiconductor with direct transitions between bands, to determine optical band gap of samples, $(ahv)^2$ is drawn against hv and data is extrapolated in linear region of high energy with horizontal axis as a=0. Figure (**10**) shows this curve in order to determine direct optical band gap and the attached figure shows the results obtained from this analysis related to band gap amounts. The results indicate that the sample with largest crystallite size (V2) has the smallest band gap (1.74 eV) and the sample with smallest crystallite size (V3) has the largest band gap (2.01 eV) which can be a reason for happening a quantum limitation in these samples.

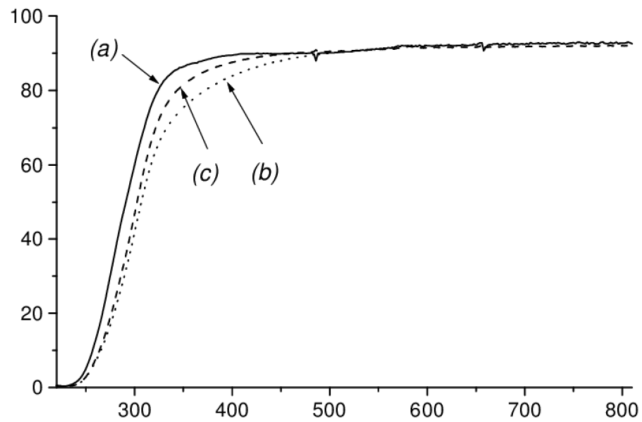

Figure 10: Analysis of optical data as a function of photon energy. The attached figure shows band gap of layers.

3.4. Electrical Properties

Figure (**11**) shows current-voltage curve of these samples. The results indicate that sample V2 has the highest electrical conductivity (metal-like property) while sample V3 has the lowest one (isolator-like property). This is in good agreement with optical transition behavior of layers.

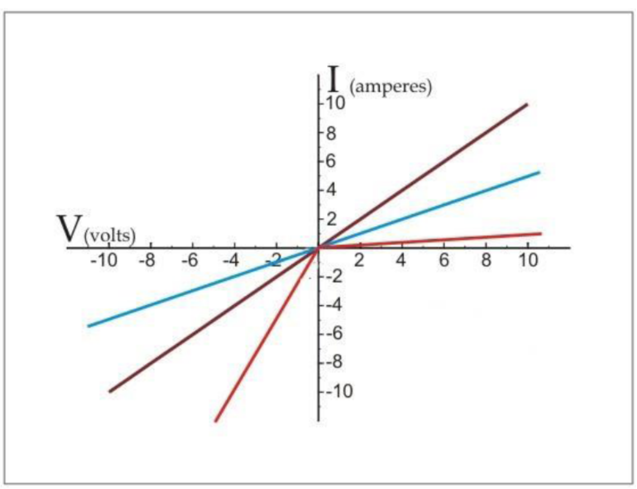

Figure 11: Current-voltage curve for samples grew up in darkness.

3.5. Photoconductivity Properties

To investigate photoconductivity of samples, the under studied samples were placed under visible light emission (halogen lamp). Figure (**12**) shows current-voltage curve of samples under light. As can be observed, all three samples are reacted to the light and after emission, more electrical current passed through samples. This is an expected event due to producing electron - hole pairs in layers as a result of optical photon emission in hv>E_g. In order to compare optical sensitivity of these samples, the passed electrical current through samples in voltage of 3 V in darkness and under visible light emission are shown in Figure (**13**). As can be seen, sample V2 is of highest relative change of electrical current (I_{Light}/I_{Dark} = 11) and sample V3 is of lowest one (=3). These variations may be due to the effect of various factors such as optical absorption, band gap, crystallite size and crystalline obliquity in the investigated layer.

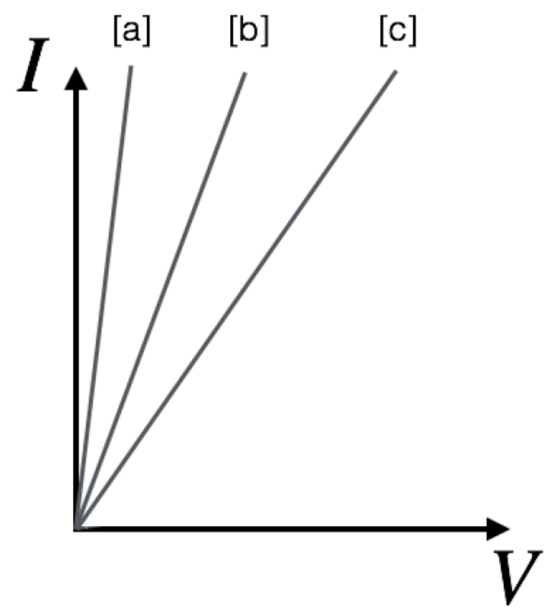

Figure 12: Current-voltage curve for samples subjected to visible light.

4. CONCLUSION

Cadmium Oxide (CdO) layers were deposited by spray pyrolysis technique in various volumes of 50, 75 and 100 (ml) over glass. FESEM images indicate that surface morphology of samples depend on variations of precursor volume spray and XRD spectrums indicates the polycrystalline structure of layers in preferred direction (111). Data analysis indicates that optimum crystallite size is for sample V3. Band gap of samples is between 3.4-3.6 (eV) so that sample V3 is of lowest band gap among other samples. Absorption coefficient in UV light region for all layers is in the order of 10^5 (cm^{-1}) and sample V3 has the lowest penetration depth in this region.

The thin layers of Cadmium Oxide (CdO) nanostructures were deposited using spray pyrolysis technique with various volumes of spray solution over a

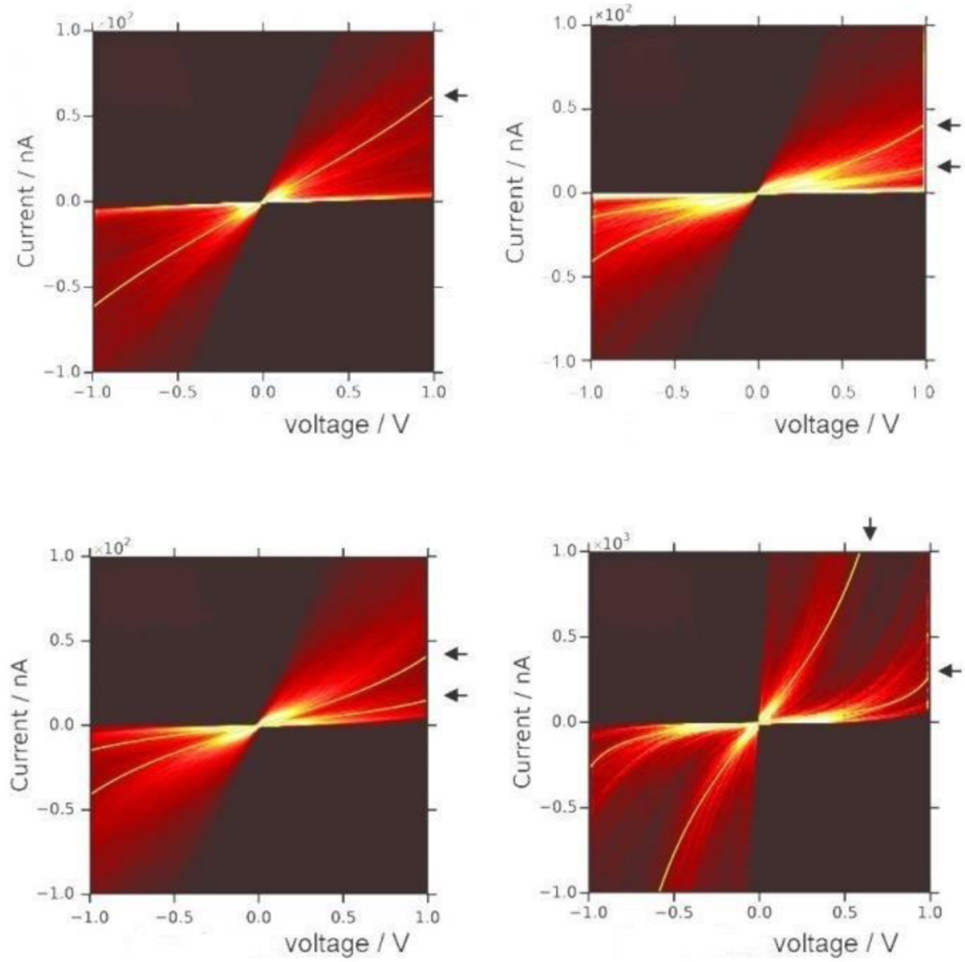

Figure 13: Passed electrical current through investigated samples.

glassy substrate. FESEM images indicate that surface morphology of samples are dependent on the variations of solution volume and XRD spectrums of layers indicate that polycrystalline structures are grew up in preferred direction of (002). Data analysis indicate that at solution volume of 50 (ml), crystallite size and crystallite defect densities are optimum and photoconductivity properties are improved. In visible light region, layers are of low optical transition and of optical band gap between 1.74-2.01 (eV) so that sample V2 has the lowest band gap among all samples. The obtained results indicate that band gap variations in these samples are controlled by crystallite size and under the effect of happening a quantum limitation. Photoconductivity results indicate that sample V2 is of highest optical sensitivity to visible light.

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