Investigation and characterization of simple chemical method Synthesized CdO-NiO Nanocomposite.

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Abstract. The aim of this research is to study the effect of annealing on the physical properties of CdO:NiO thin film. In this research, cadmium oxide (CdO) and Nickel oxide (NiO) nanoparticles were synthesized by chemical method. The optical, structural and topographical properties of the synthesized nanoparticles were investigated by using UV-VIS measurement.

The plotted graphs show the optical characteristics of the films which varied with the wavelength and the photon energy, atomic force microscopy AFM, The structure of synthesized CdO and NiO thin films was analyzed by X-ray diffraction XRD which revealed that the CdO and NiO thin films are polycrystalline and have several peaks of cubic face structure. The crystallite size, dislocation density and microstrain of the thin films were calculated and listed. The same procedures are also calculated and included. CdO:NiO Nanofluorocarbons were synthesized by mixing% by weight (CdO and NiO).

Keywords: Cadmium oxide, Nickel oxide, XRD, Absorbance, Nanocomposite

1. Introduction

Nanotechnology is applied to various fields such as biological, physical, chemical, and engineering sciences where new techniques are developed to investigate and process single atoms and molecules. Among all nanoparticles, single-metal applications in diverse fields such as cosmetics, coating, electronics, packaging and biotechnology[1]. Cadmium oxide (CdO) is transparent conductive oxide (TCOs) material that possesses both high conductivity and high optical transparency (>80%) in the visible light of the electromagnetic spectrum[2]. CdO is a semiconductor type n with almost metallic conduction[3]. It has a gap in the direct energy range (for example) of 2.3 volts eV and indirect transformations at lower energies[4]. It has a wide range of applications such as solar cells, windows, flat panel display, photo transistors etc. It has been empirically confirmed that the structural, electrical and optical properties are very sensitive to film structure and sedimentation conditions[5,6]. these transparent connectors are used extensively in thin film solar cells[7] and optical electronic devices[8]. CdO films can be manufactured using various techniques such as sputtering[9], chemical vapor deposition (CVD)[10], thermal pyrolysis[11], thermal evaporation[12] sol gel[13] Nickel oxide (NiO) is the most selective oxide that has been extensively studied. It is an anti-chloride magnetic semiconductor of the NaCl type. It offers promising nominations for many applications such as solar thermal absorption[14], catalyst for O2 development[15], electrolysis of the image[16], and electrochromic[17] the chemical sensor[18] is artificial using various PV techniques such as thermal oxidation of nickel[19], chemical bath deposition[20], pyrolysis spray[21], sol-gel process [22]. Atomic layer[23], precipitation of a pulsed laser[24], organic chemical vapor deposition[25], beam molecular radial[26], and DC[27] RF magnetron and electrode[28]. The increase in surface area and the effect of quantitative detention have made nanomaterials quite different from their larger form in electrical and optical properties. Many of the NIRs single-dimensional structures, such as nanowires, nanotubes, and nano-belts, attracted much attention [29]. the chemical deposition method offers a simple, low cost, and rapid method of assembling the CdO and the new structure. Example. In this example we can see the footnotes after each author name and only 5 titles; for example, the sixth footnote may say, ‘The author
must be addressed by any correspondence.' In addition, contributions, funding, temporary titles, etc. can also be referred to as margins.

2. **Experimental works**
The preparation of the cadmium oxide film (CdO) in this study was done in two main stages:

   a- **Thin film deposition:**
   The cadmium component was selected in this study because it is a chemical element (Compound) with a high melting point of (1773)K. The deposition is done by using the Simple chemical method which consists of mixing several materials to obtain the required material and then deposition on glass bases. The simple chemical method is a distinctive, fast and inexpensive way of not being needed long time or complex equipment. The film or the preparation method was started by mixing cadmium nitrate Cd(NO\(_3\))\(_2\) (11.82g) with Sodium hydroxide NaOH (2g) with (50mil) of distilled water solution for each material prepared separately (i.e. 50mil of cadmium nitrate and 50mil Sodium hydroxide). The solution then is placed for a period 30 min in device the (HIT AND STIRR) (A device containing the heating properties and the movement of the prepared material to be homogeneous). Then the material is removed from the device and left for a certain period of time as the substance is down and the water solution is high and contains salts. This method (wash the material) is repeated three or four times to get rid of the salt as much as possible by a special syringe after completion of these stages are obtained on the final formulation of the solution, which is a semi-gelatin white material is required material (CdO), The required material is pulled from the top by a special syringe (YE3K061872:10-100ML) Put the material on the glass substrate by drop casting method. the method start with placing one drop by syringe in a certain amount on the glass sample and then heated to dry for (10min) Then put the second drop of the same quantity and method on the first drop on the glass sample directly and also heated in order to dry for (10min), after that we notice the color of shift thin film is white to light white indicating that we have a cadmium oxide film be sure from material by procedure checkup X-Ray diffraction.

   b- **Heat Treatment or annealing**
   After obtaining the cadmium oxide film the heat treatment stage is performed which is intended exposure of the film to temperature in order to improve the properties of this film or to know the effect of heat on the treatments properties where the treatment is carried out in a special thermal oven (Victoreen) It reaches a temperature of (1000 K). In this study, two different temperatures (at room temperature and 200°C) were chosen for annealing the films deposited on glass. It was Insert the sample into the oven for a full hour and switch-off for another hour before leaving the oven to avoid occurrence cracks thus examining their effect on properties this film, and then exited from the oven for the necessary tests.

3. **Results and discussion**
The XRD diffraction patterns of synthesized CdO and NiO nanoparticles films and deposited on glass substrate are shown in Figure 1. XRD patterns for CdO film which presented in Figure 1., have peaks of FCC CdO corresponding to (111), (200), (210), (211), (220), (311) and (222) planes which have been compared with standard X-ray diffraction data file (JCPDS file No. 039-1221), (JCPDS file No. 005-0640) [30]. In the present investigation, the films exhibit a preferential orientation along the (111) and (200) diffraction planes which were grown CdO thin films on glass substrate by the chemical method.
Figure 1. X-ray diffraction of CdO films before and after annealing at 200°C.

Figure 1. shows the x-ray diffraction of all CdO films prepared by increasing the temperature of the annealing all the roots of the material appeared and the appearance of a new phase CdO₂ as a result of additional oxygen atom and the oxidation of the material after the process of annealing. The mean crystallites size D was determined using the following Debye–Scherer formula (XRD line broadening) [31] and listed in Table 1., where λ is the wavelength of x-ray, θ is the diffraction angle and β is the FWHM. The sharp XRD peaks indicate that the particles were of polycrystalline structure, and that the nanostructure grew with a random orientation. The micro strain (ε) and the dislocation density (δ) can be calculated by using the following relations[32], see Table 1.

\[ D = \frac{k \lambda}{\beta \cos \theta} \]  \hspace{1cm} (1)

\[ \varepsilon = \frac{\beta \cos \theta}{4} \]  \hspace{1cm} (2)

\[ \delta = \frac{1}{D^2} \]  \hspace{1cm} (3)

where, k is the shape factor, taken as 0.94
Table 1. shows summary of X-ray characterization.

| Sample                  | 2θ (deg) | β (deg) | hkl | D (nm) | DIS | Strain |
|-------------------------|----------|--------|-----|--------|-----|--------|
| As-Prepare              | 32.89    | 0.17   | 111 | 50.62  | 3.9 | 0.71   |
| 200°C after-annealing   | 38.41    | 0.18   | 200 | 48.57  | 4.23| 0.74   |
|                        | 29.24    | 0.15   | 111 | 56.86  | 3.09| 0.63   |
|                        | 32.9     | 0.16   | 111 | 53.79  | 3.45| 0.67   |
|                        | 33.67    | 0.17   | 200 | 50.73  | 3.88| 0.71   |
|                        | 37.8     | 0.16   | 210 | 54.54  | 3.36| 0.66   |
|                        | 38.43    | 0.14   | 200 | 62.45  | 2.56| 0.57   |
|                        | 41.6     | 0.14   | 211 | 63.09  | 2.51| 0.57   |
|                        | 47.92    | 0.16   | 220 | 56.48  | 3.13| 0.64   |
|                        | 57.87    | 0.17   | 311 | 55.54  | 3.24| 0.65   |
|                        | 60.59    | 0.16   | 222 | 59.82  | 2.79| 0.60   |

Figure 2. shows a 3-D AFM image of the high-density CdO film deposited on glass layer in a packing way. The surface of the thin membrane contains homogenous granules carved vertically on a spherical shape, and good coarse grains of CdO nanoparticles within the scanning area (2x2)μm. Using a special program (4.62 imager), the average grain size was about 83.73 nm.

Table 2. rate of roughness of the surface of the CdO films before and after the process of annealing.

| CdO thin films   | Average diameter (nm) | Roughness average (nm) | RMS (nm) |
|------------------|-----------------------|------------------------|----------|
| As-Prepared      | 68.41                 | 1.14                   | 1.34     |
| After-Annealing  | 83.73                 | 0.661                  | 0.762    |
Figure 2. AFM scan images of the CdO films prepared before and after annealing process.

Figure 3a. shows the absorbance spectra of CdO NPs prepared by chemical method is taken by Cary 100 Conc plus UV-VIS Spectro-photometer (300 – 900) nm. It is very important because it provides the details related with the optical band. The absorbance characteristics can consider a useful tool to analyze nanomaterials. It is clearly seen that the absorbance is decreasing around 20% at wavelength 350 nm then decreased sharply to 80% at wavelength 900 nm with increased wavelength.

Figure 3b. shows that graph between $(\alpha h\nu)^2$ versus photon energy $(h\nu)$ gives the value of direct band gap. The extrapolation of the straight line to $(\alpha h\nu)^2 = 0$, gives the value of band gap. From the UV spectra shows the absorbance decreases with increasing wavelength and the energy gap increase from 2.3 eV to 2.65 eV from bulk to the thin film via quantum size effect.
Either Preparation of NiO thin film was used Nickel nitrate Ni(NO3)2 and Sodium hydroxide NaOH, processed from AG (CH947-BUCHS), Nickel nitrate is a powder-green substance with a molecular weight of 290.81 g/mole. whereas the preparation method of NiO thin film is similar to the preparation method of CdO thin film. XRD patterns for NiO film which presented in Figure 4, showed at different temperatures annealing (room temperature, 200°C, 400°C) It has a polycrystalline structure with (FCC), note from figure 4, when preparing at room temperature appearance of crystalline levels at the orientation planes (110) and (200), either in the case of temperature (200)°C note from Figure 4 appearance of crystalline levels at the orientation planes (110) and (200), either in the case of temperature (400)°C note from Figure 4, indicates the appearance of different crystalline levels (110), (111), (200), (220), (311) and (222) when increasing the annealing temperature of (400)°C and planes which have been compared with standard X-ray diffraction data file (JCPDS file No. 047-1049)[30]. Note that the annealing at 400°C degrees increased the crystallization or improved crystallization indicated by XRD the levels mentioned above. The peaks appear at high intensity than at the annealing temperature of (200)°C This is due to the increased temperature of the annealing has led to the

![Figure 3a](image-url)  
**Figure 3a.** Absorbance Spectra of CdO thin film
development (growth) of crystalline film (NiO), it was also found that the intensity of the peaks is greater than it is at a temperature (400°C). In the present investigation, the films exhibit a preferential orientation along the (200) diffraction plane which were grown NiO thin films on glass substrate by the chemical method.

**Figure 4.** X-ray diffraction of NiO thin film at (a) as-prepared (b) after annealing at (200°C,400°C) for 2h.

**Table 3.** shows the results of X-ray diffraction of the dominant peaks of prepared NiO films.

| Samples (NiO)Thin films | 2theta (deg) | beta (deg) | hkl   | D  (nm) | DIS  | STRAIN |
|-------------------------|-------------|------------|-------|---------|------|--------|
| 400°C                   | 37.27       | 1.12       | (111) | 7.779   | 165.24| 4.652  |
|                         | 43.32       | 1.19       | (200) | 7.466   | 179.36| 4.846  |
|                         | 62.88       | 1.31       | (220) | 7.377   | 183.74| 4.905  |
|                         | 75.27       | 1.32       | (311) | 7.914   | 159.62| 4.572  |
|                         | 79.32       | 1.18       | (222) | 9.110   | 120.46| 3.972  |

**Table 4.** Surface roughness and grain size of NiO films before and after annealing treatment.

| NiO thin film   | Avg-diameter (nm) | RMS (nm) | Roughness (nm) |
|-----------------|-------------------|----------|----------------|
| As-prepared     | 87                | 1.84     | 1.6            |
| As-Annealing    | 90.31             | 1.14     | 0.984          |
Figure 5. AFM images of NiO films.

Figure 6a. shows the absorbance spectra of NiO NPs prepared by chemical method. The absorbance characteristics can consider a useful tool to analyze nanomaterials. It is clearly seen that the absorbance is decreasing sharply below ~ 350 nm with increased wavelength.

Figure 6b. shows the relation between \((\alpha hv)^2\) versus photon energy \((hv)\) gives the value of direct band gap. The extrapolation of the straight line to \((\alpha hv)^2 = 0\), gives the value of band gap. From the UV spectra shows the absorbance decreases with increasing wavelength and the energy gap increase from 3.6 eV to 3.9 eV from bulk to the thin film via quantum size effect.
Figure 6a,b. Absorbance Spectra of NiO thin film and (ahv)2 versus photon energy of NiO thin film.

Table 5. Values of energy gap of nickel oxide films at different temperatures before and after annealing.

| Samples          | Direct Allowed Transition Eg (eV) |
|------------------|-----------------------------------|
| As-prepared      | 3.6                               |
| As-annealing     | 3.9                               |

After the process of preparation of films CdO, NiO methods explained earlier. The second part of the work is the process of composite films that were prepared in certain proportions, It allows us to composite different percentages of the prepared films, but for time and cost where specific ratios were selected where we determined the ratio of film CdO to different ratios of NiO film, after that, each mixture was taken separately and deposited on slices of glass in the way the drop casting was previously described, after that, the necessary tests were conducted for the prepared samples that show the structural, optical and other characteristics.

The XRD diffraction patterns of synthesized composite nanoparticles films deposited on glass are shown in figure 7. The XRD patterns of composite contain eleven main peaks at diffraction angles : 24.23°, 25.72°, 26.64°, 29.53°, 30.48°, 33.04°, 38.56°, 41.68°, 43.97°, 48.06°, 57.51° corresponding to (002), (101), (002), (111), (---)(----) its meaning does not have values in the international standard). (111),
(200), (200), (220) and (311) planes. which were match well the standard peaks (JCPDS NO. 00-005-0640, 00-047-1049, 00-008-0382, 00-016-0687, 00-018-1235, 00-038-0903).

![Image](image-url)

**Figure 7.** XRD pattern of nanoparticles films prepared in composite method with different percentages.

| 2θ (deg) | hkl Plane | d observed (Å) | d STEM (Å) | FWHM (deg) | D (nm) | D × 10^14 lines.m^-2 | η × 10^4 lines^2.m^-4 |
|----------|-----------|----------------|------------|-------------|--------|---------------------|----------------------|
| 24.23    | (002)     | 3.66           | 3.66       | 0.15        | 56.27  | 3.15                | 6.43                 |
| 25.72    | (101)     | 3.46           | 3.4        | 0.13        | 65.12  | 2.35                | 5.55                 |
| 26.64    | (002)     | 3.34           | 3.32       | 0.14        | 60.58  | 2.72                | 5.97                 |
| 29.53    | (111)     | 3.02           | 3.06       | 0.12        | 71.13  | 1.97                | 5.08                 |
| 30.48    | (---)     | 2.93           | 2.89       | 0.15        | 57.03  | 3.07                | 6.34                 |
| 33.04    | (111)     | 2.7            | 2.71       | 0.13        | 66.23  | 2.27                | 5.46                 |
| 38.56    | (200)     | 2.33           | 2.34       | 0.16        | 54.66  | 3.34                | 6.61                 |
| 41.68    | (---)     | 2.16           | 2.16       | 0.14        | 63.10  | 2.51                | 5.73                 |
| 43.97    | (200)     | 2.05           | 2.08       | 0.08        | 111.31 | 0.8                 | 3.25                 |
| 48.06    | (220)     | 1.89           | 1.87       | 0.11        | 82.20  | 1.47                | 4.4                  |
| 57.51    | (311)     | 1.6            | 1.6        | 0.11        | 85.74  | 1.36                | 4.22                 |

The 3D AFM images and granularity accumulation distribution chart of CdO and NiO films NPs synthesized with various percentages of the prepared films (1:0.25,0.5,0.75,1) are shown in Figure 8.
Figure 8. 2D,3D AFM image and Granularity accumulation distribution chart of CdO:NiO composite thin films prepared.
The thin film composite is a spherical compound with good distribution capacity, homogenous grains and vertical alignment. Using a special program, RMS root values for surface rate, roughness and average grain size were calculated and recorded in table 7.

| Sample (CdO:NiO) | Avg. Diameter | Roughness Avg. (nm) | RMS (nm) |
|------------------|---------------|---------------------|----------|
| 1: 0.25)         | 55.03         | 1.11                | 1.3      |
| 1: 0.5)          | 51.07         | 1.14                | 1.32     |
| 1: 0.75)         | 95.92         | 1.52                | 2.93     |
| 1: 1)            | 85.18         | 0.914               | 1.06     |

The absorbance spectrum as shown in figure 9a. of the composite process thin films, we observe a gradual decrease in absorption by increasing the wavelength (i.e. decrease around 20% at wavelength 350 nm then decreased sharply to 80% at wavelength 900 nm). from the UV spectra shows the absorbance decreased with increasing wavelength and The energy band gap of composite process is estimated by plotting the square of (αhυ) versus (hυ). as shown in figure 9b. The values of optical band gap of composite NPs are ranged from 2.2 to 2.7 eV almost. We observe a decrease in the energy gap due to the overlap between the energy gaps of the prepared films where approaching the valence band of the film with the conduction band of another film.
Figure 9. a,b. Absorbance Spectra and $(ahv)^2$ versus photon energy gap of prepared thin films after composite process.
Conclusions
This work has presented CdO and NiO thin films were prepared by chemical method (simple, cost, and quick method for the synthesis of CdO and NiO nanostructure). Note that the effect of annealing led to improved crystallization and increased energy gap. Either in the case of composite where observe the values of optical band gap of composite NPs are ranged from 2.2 to 2.7 eV. We observe a decrease in the energy gap due to the overlap between the energy gaps of the prepared films where approaching the valence band of the film with the conduction band of another film.

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