

Optical Characterization and Possible Solar Energy Applications of Improved Solution Grown Cobalt Oxide (CoO) Thin Films at 300K

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Thin films of cobalt oxide (CoO) were deposited on microscope glass slides using improved solution growth technique at 300K and pH values of 7, 9 and 11. Ethylenediamine-tetra acetate (EDTA), a complexing agent with pH opposite to that of deposition bath constitutions, was used to optimize the deposition conditions at different pH values to produce enhanced optical and solid state properties. X-ray diffractometry method was used to obtain the structural characterization. A single beam spectrophotometer was used to obtain spectral absorbance data within ultraviolet (UV), visible (VIS) and near infrared (NIR) regions. Other spectral optical and solid state properties were determined from absorbance data. Optical properties of the films produced at pH of 7 and 9 have low absorbance, low reflectance, high transmittance and refractive index of 1.21-1.95 in the visible region. Film produced at pH of 11 has relatively high absorbance, relatively high reflectance, relatively low transmittance and refractive index of 1.59 – 2.22. The band gap energy values of 2.75eV, 2.76eV and 2.48eV were obtained for films produced at pH of 7, 9 and 11 respectively. The optical and solid state properties of the deposited cobalt oxide (CoO) thin films are close to those earlier reported for transparent conducting oxide thin films. The films produced at pH of 7 and 9 with refractive index (n) of about 1.9 could be employed in antireflection coatings and solar control window coatings. Films produced at pH 11 with refractive index (n) much greater than 1.9 could be useful in poultry production, solar cells and anti dazzling coatings.

1. Introduction

The optical engineering properties of materials and thin films that are very important for solar energy applications include absorbance (A), transmittance (T) and reflectance (R). These properties characterize the interaction of a particular coating or material with incident radiation [11]. They are also related to intrinsic properties of materials such as refractive index (n), extinction coefficient (k), coefficient of absorption (α), optical conductivity, electrical conductivity, film thickness and bandgap [4, 26, 11, 3]. Thin film semiconductors especially the transparent conducting oxides (TCO) have many applications in optics and electronics as well as in the production of energy conversion devices such as flat panel displays (FPDS), transparent electrodes, smart windows, solar cells and polymer based electronic devices [17, 6, 16]. Some of the properties that enhance the applications of transparent conducting oxides (TCO) include high electron mobility, high electrical conductivity, weak absorbance and high transmittance towards higher wavelength in the visible region of electromagnetic spectrum [32].

The various methods used for deposition of thin films include spray pyrolysis [35], sol gel [12], sputtering [34], thermal evaporation [31], chemi-

cal vapour deposition [38] and chemical bath deposition [8, 16]. The high temperature deposition techniques ($>900^{\circ}\text{C}$) are not suitable for preparation of oxide thin films that are used as selective absorbing surfaces because they cause rapid oxidation ratio of the compound. The low temperature techniques ($<900^{\circ}\text{C}$), on the other hand, also show variations in the oxide ratio in spite of controlled growth in the thin film [16]. The solution growth technique (SGT) also known as chemical bath deposition (CBD) method was well employed in preparation of silver mirrors [28]. The method has been intensively studied for production of films that have comparable structural and optoelectric properties to those produced using other thin films deposition techniques. It is simple and cost effective in terms of equipment and chemical. It is now being adopted by some industries [2, 4, 9]. The method could be improved by controlled addition of another complexing agent with pH opposite to that of bath constitutions to enhance deposition conditions at different pH values [14, 15].

This paper reports the deposition of cobalt oxide (CoO) thin films using improved solution growth technique at 300K and different pH values. Optical and solid-state properties of the deposited CoO thin films were determined. Structural characterizations of the deposited films were obtained using x-ray diffractometry method.

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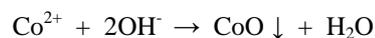
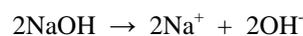
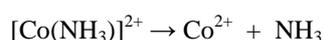
Possible applications of the deposited cobalt oxide (CoO) thin films are also discussed.

2. Experimental details

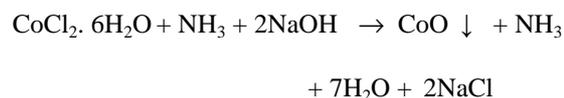
2.1 Film deposition

The reagents used for deposition of cobalt oxide thin films were freshly prepared 0.3M cobalt (II) chloride hexahydrate ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$) solution, 5.0M ammonia ($\text{NH}_3(\text{aq})$) solution as complexing agent and 2.5M sodium hydroxide (NaOH) solution. Different volumes of the reagents were measured into three different 50ml glass beakers as reaction baths labeled A, B and C. Distilled water was added into each reaction bath to raise the volume of solution to a required level. A controlled addition of ethylenediamine tetra acetate (EDTA), another complexing agent with pH oppose to that of bath constitutions, was used to enhance the deposition conditions at different pH values. The chemical bath reagents and deposition time were optimized to eliminate the incorporation of solid phase $\text{Co}(\text{OH})_2$ into the thin films. Reaction bath A at pH of 7 contained 8mls of 0.3M $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ solution, 3mls of 5.0M $\text{NH}_3(\text{aq})$ solution, 5mls of 2.5M NaOH solution, 24mls of H_2O and 36mls of 0.2M EDTA solution. Reaction bath B at pH of 9 contained 7mls of 0.3M $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ solution, 4mls of 0.5M $\text{NH}_3(\text{aq})$ solution, 6mls of 2.5M NaOH solution, 23mls of water and 21mls of 0.2M EDTA solution. Reaction baths C at pH of 11 contained 6mls of 0.3M $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ solution, 5mls of 0.5M $\text{NH}_3(\text{aq})$ solution, 7mls of 2.5M NaOH solution, 22mls of water and 17mls of 0.2M EDTA solution. Each reaction bath was stirred with a glass rod to ensure homogeneity of the solution before the pH values was noted. A cleaned microscope glass slide ($76 \times 26 \times 1\text{mm}^3$) was immersed vertically into each reaction bath and a plastic cover was used to shield impurities. The deposition took place at room temperature of 300K for 24 hours. The as-grown films, observed to be adherent on the substrates, were removed from reaction baths, rinsed with distilled water and dried in air. Prior to deposition, the microscope were cleaned by degreasing them with concentrated (trioxonitrate (v) acid (HNO_3) solution rinsed with distilled water and dried in air.

The deposition of cobalt oxide thin film results from dissociation reaction and controlled released of ions in solution according to equations



The overall equation for the chemical reaction is



2.2 Film characterization

Optical characterization of the deposited cobalt oxide (CoO) thin films was obtained using a single scanning spectrophotometer (Pharmacia LKB Biochrom 6040) in the wavelength range 200nm to 900nm from the ultraviolet (UV) through the visible (VIS) to near infrared (NIR) regions of the electromagnetic spectrum. An uncoated microscope glass slide was used as reference to standardize the results. X-ray diffractometry method was used to obtain the structural characterization of the films. Surface microstructure of the films were viewed using electron microscope at magnification of 100x.

3. Theory and calculations

The spectral absorbance (A) of the cobalt oxide (CoO) thin films obtained by direct measurement is related to the transmittance (T), which is the ratio of radiant power transmitted (I) by a body to the total radiant power incident (I_0) on the body, [13, 20, 26] by the expression

$$A = \log (1/T) \quad (1)$$

where $T = I/I_0$.

The relationship between the absorbance (A), transmittance (T), and reflectance (R) of a body which allows for conservation of energy [11] is

$$A + T + R = 1 \quad (2)$$

Reflectance (R) is related to optical constants, refractive index (n) and extinction coefficient (k), of semiconductors [19, 26] by:

$$R = (n-1)^2 + k^2 / (n+1)^2 + k^2 \quad (3a)$$

For a weak absorbing film $k^2 \ll (n-1)^2$ and eqn. (3a) reduces to the relation

$$R = (n-1)^2 / (n+1)^2 \quad (3b)$$

and refractive index, from eqn. (3b), is given by:

$$R = (1+R^{1/2}) / (1-R^{1/2}) \quad (3c)$$

The transmittance (T) is related to coefficient of absorption (α) and distance ($d\mu\text{m}$) transversed in the film [5, 26, 13] by the expression:

$$T = \exp(-\alpha d) \quad (4a)$$

and for a unit distance,

$$\alpha = \ln(1/T) \times 10^6 \text{ m}^{-1} \quad (4b)$$

Coefficient of absorption is also related to extinction coefficient (k) through the expression [26]

$$\alpha = 4\pi k / \lambda \quad (5a)$$

and from eqn. (5a),

$$k = \lambda \alpha / 4\pi \quad (5b)$$

where λ is wavelength of radiation

The complex dielectric constant (ϵ_c) is related to refractive index (n) and extinction coefficient (k) [26, 3] through expression:

$$\epsilon_c = (n+ik)^2 = \epsilon_r + \epsilon_i \quad (6a)$$

where real dielectric constant

$$\epsilon_r = n^2 - k^2 \quad (6b)$$

and imaginary dielectric constant

$$\epsilon_i = 2nk \quad (6c)$$

The optical conductivity (σ_{op}), a measure of frequency response of material when irradiated with light, is related to index of refraction (n) and speed of light c [26] by

$$\sigma_{op} = \alpha n c / 4\pi \quad (7)$$

Electrical conductivity and optical conductivity of the film are related [10] by expression

$$\sigma_e = 2\lambda \sigma_o / \alpha \quad (8)$$

The optical expression for the thickness (t) of a weak absorbing film on non absorbing substrate [26, 7] is

$$T = (1-R)^2 \exp(-\alpha t) \quad (9a)$$

and from eqn. (6a), the film thickness (t) is given by:

$$t = \ln[(1-R)^2 / T] / \alpha \quad (9b)$$

In semiconductors, direct and indirect transitions can be obtained from energy dependence of absorption coefficient near fundamental absorption edge. The expression for direct transition [26, 4, 22] is:

$$\alpha = B (h\nu - E_g)^n \quad (10)$$

where E_g is band gap, $h\nu$ is photon energy, $n = 1/2$ for allowed direct transitions and $n = 3/2$ for forbidden direct transitions. The plot of α^2 against $h\nu$ when extrapolated cuts $h\nu$ axis at the point where α^2 is zero (0) gives the value of direct band gap.

4. Results and discussions

Absorbance (A) spectra of the deposited cobalt oxide (CoO) thin films at pH of 7, 9 and 11 are shown in fig. 1. The spectral absorbance is high for wavelength range 200 – 300 nm in ultraviolet (UV) region but decrease rapidly to low values in wavelength range of 350 – 750nm in visible (VIS) region and 800 – 900 nm in near infrared (NIR) region of electromagnetic spectrum. For films produced at pH of 7, absorbance decreases within 0.498 – 0.111 in UV, 0.007 – 0.096 in VIS and 0.036 – 0.063 in NIR regions. For films produced at pH of 9, absorbance decreases within 0.101 – 0.656 in UV, 0.006 – 0.098 in VIS and 0.019 – 0.080 in NIR regions. For films produced at pH of 11, values of absorbance (A) decreased from 0.127 – 0.592 in UV, 0.043 – 0.152 in VIS and 0.077 – 0.130 in NIR regions. The transmittance (T) - reflectance (R) spectra of the CoO thin films are shown in Fig. 2. The transmittance (T) spectra increases rapidly in UV but slowly into VIS and NIR regions. Transmittance (T) of film produced at pH of 7, increases rapidly from 0.318 – 0.774 in UV, but varies within 0.802 – 0.984 in VIS and 0.865 – 0.920 in NIR regions. Transmittance (T) of film produced at pH of 9, increases rapidly from 0.221 – 0.792 in UV, but varies within 0.798 – 0.986 in VIS and 0.832 – 0.957 in NIR regions. Transmittance (T) of film produced at pH of 11,

increases rapidly from 0.256 – 0.746 in UV, but varies within 0.705 – 0.906 in VIS and 0.741 – 0.838 in NIR regions. The corresponding reflectance (R) spectra of the CoO thin films decreases rapidly in UV and then gradually in VIS and NIR regions. Reflectance (R) of film produced at pH of 7, decreased rapidly from 0.115 – 0.203 in UV, 0.009 – 0.102 in VIS and 0.044 – 0.072 in NIR regions. Reflectance (R) of film produced at pH of 9, decreased rapidly from 0.106 – 0.180 in UV, but varies within 0.008 – 0.104 in VIS and 0.024 – 0.088 in NIR regions. Reflectance (R) of film produced at pH of 11, decreased rapidly from 0.127 – 0.202 in UV, but varies within 0.064 – 0.143 in VIS and 0.085 – 0.127 in NIR regions. The variation of refractive index (n) with photon energy (h ν) is displayed in Fig. 3. The refractive index decreases rapidly with photon energy 4.14 – 6.21 eV in UV, but decreases slowly within photon energy range 1.65 – 3.55 eV in VIS and 1.38 – 1.55 eV in NIR regions. Values of refractive index (n) for film produced at pH of 7, decreased rapidly from 2.02 – 2.64 in UV, but varies within 1.21 – 1.94 in VIS and 1.53 – 1.73 in NIR regions. For films produced at pH of 9, values of n decreased rapidly from 1.97 – 2.47 in UV, but varies within 1.20 – 1.95 in VIS and 1.36 – 1.85 in NIR regions. Refractive index (n) for film produced at pH of 11, decreased rapidly from 2.10 – 2.64 in UV, but varies within 1.59 – 2.22 in VIS and 1.82 – 2.12 in NIR regions. The films produced at pH of 7 and 9 with low absorbance (A), high transmittance (T), low reflectance (R) and low refractive index (n) in the VIS and NIR regions (390 – 900 nm) of electromagnetic spectrum could find useful applications in antireflection coatings for transparent covers of solar thermal devices and solar control glass window coatings [21]. Oxide coatings with refractive index of about 1.9 could be employed in antireflection coatings to increase transmittance of glass from 0.91 to 0.96 and reduce the reflectance of photovoltaics from 0.36 – 0.04 [29, 1, 11]. The film produced at pH of 11 with relatively high absorbance (A), relatively low transmittance (T), relatively high reflectance (R) and relatively high refractive index (n) in the UV, through VIS to NIR regions could be employed in the construction of poultry houses to allow enough infrared radiation to warm the young chicks (a day to five (5) weeks old) which have little or no insulating feather covers to protect them from adverse effects of cold weather while at the same time protecting the chicks from ultraviolet radiation. This could also reduce the cost of energy consumption through the use of stoves, electric

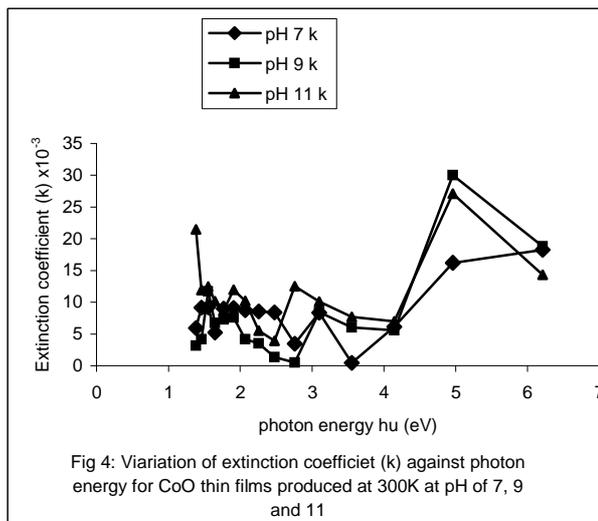
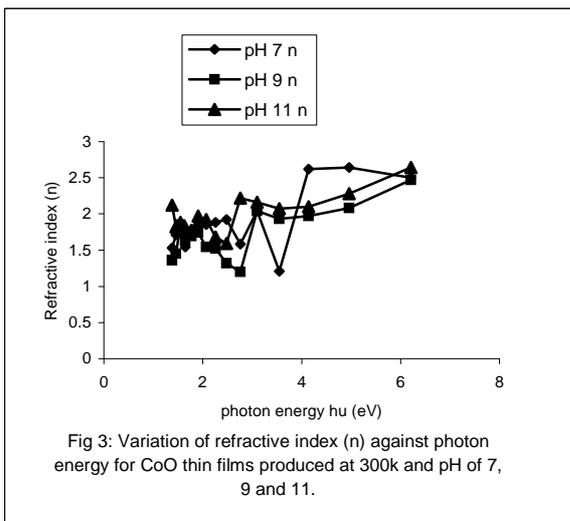
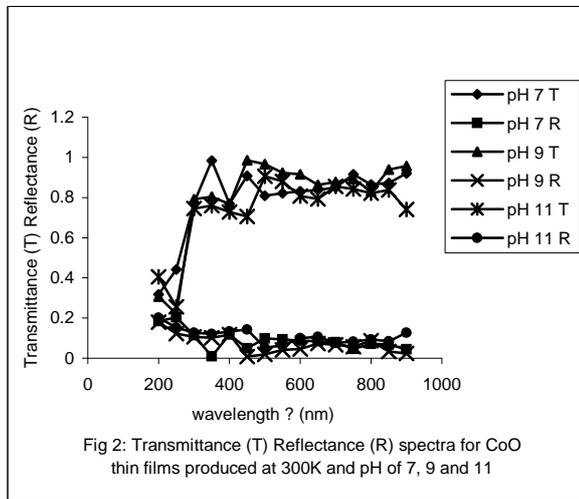
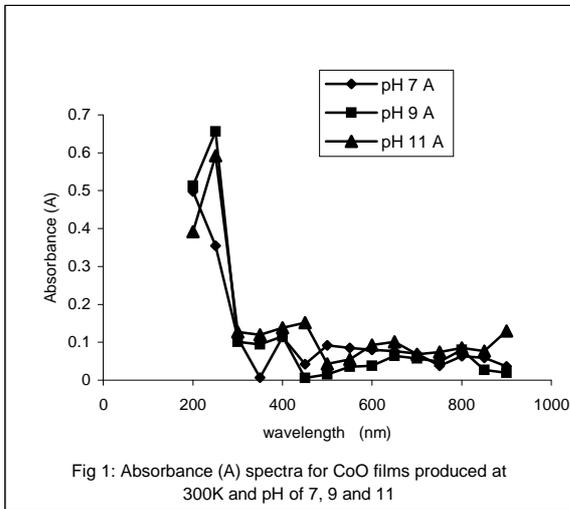
bulbs and heaters as well as the hazard associated with them. The application of solar energy as a source of heat in chick brooding is environment friendly and promotes sustainable development [24]. This film could also be employed in solar energy technologies for egg incubation and drying of chicken manure [23], photocells [25], solar cells [18] and in anti-dazzling coatings for cars windscreens and driving mirrors. Fig. 4 shows that the extinction coefficient (k) decreases rapidly with photon energy (h ν) ranging from 6.21 – 4.14 eV in UV but slowly through 3.35 – 1.65 eV in VIS to 1.55 – 1.38 eV in NIR regions. For CoO film produced at pH of 7, the values of k varies within (6.12 – 18.22) $\times 10^{-3}$ in UV, (0.45 – 9.14) $\times 10^{-3}$ in VIS and (5.94 – 9.23) $\times 10^{-3}$ in NIR regions. For film produced at pH of 9, the values varies within (5.57 – 18.79) $\times 10^{-3}$ in UV, (0.5 – 7.62) $\times 10^{-3}$ in VIS and (3.15 – 11.71) $\times 10^{-3}$ in NIR regions. For film produced at pH of 11, the values varies within (6.99 – 27.11) $\times 10^{-3}$ in UV, (3.93 – 12.52) $\times 10^{-3}$ in VIS and (11.95 – 21.47) $\times 10^{-3}$ in NIR regions. The low magnitude of 10^{-3} for k shows that the deposited CoO thin film is a semiconductor. Plot of coefficient of absorption (α) with photon energy h ν show that the values of α rapidly decrease with photon energy from UV but slowly through VIS to NIR regions. For films produced at pH of 7, values of α vary within (0.256 – 1.145) $\times 10^6 \text{m}^{-1}$ in UV, (0.016 – 0.221) $\times 10^6 \text{m}^{-1}$ in VIS and (0.083 – 0.145) $\times 10^6 \text{m}^{-1}$ in NIR regions. For films produced at pH of 9, values of α varies within (0.233 – 1.510) $\times 10^6 \text{m}^{-1}$ in UV, (0.014 – 0.226) $\times 10^6 \text{m}^{-1}$ in VIS and (0.044 – 0.184) $\times 10^6 \text{m}^{-1}$ in NIR regions. For films produced at pH of 11, values of α vary within (0.293 – 1.363) $\times 10^6 \text{m}^{-1}$ in UV, (0.099 – 0.350) $\times 10^6 \text{m}^{-1}$ in VIS and (0.177 – 0.300) $\times 10^6 \text{m}^{-1}$ in NIR regions. The high magnitude of 10^6m^{-1} of α is within semiconductor thin film α values of range $10^6 - 10^7 \text{m}^{-1}$ suitable for polycrystalline thin film solar cells [11]. Plots of variation of real dielectric constant (ϵ_r) with photon energy (h ν) are shown in Fig. 6. For films produced at pH of 7, values of ϵ_r decreases within 4.09 – 6.99 in UV, 1.46 – 3.72 in VIS and 2.34 – 3.01 in NIR regions. For films produced at pH of 9, values of ϵ_r decreases within 3.88 – 6.12 in UV, 1.43 – 3.03 in VIS and 1.86 – 3.40 in NIR regions. For films produced at pH of 11, values of ϵ_r decreases within 4.43 – 6.95 in UV, 2.51 – 4.65 in VIS and 3.32 – 4.49 in NIR regions. Fig. 7 shows the variation of imaginary dielectric constant (ϵ_i) with photon energy (h ν). The film produced at pH of 7 has ϵ_i decreasing within (24.71 – 91.12) $\times 10^{-3}$ in UV, (1.02 – 33.46)

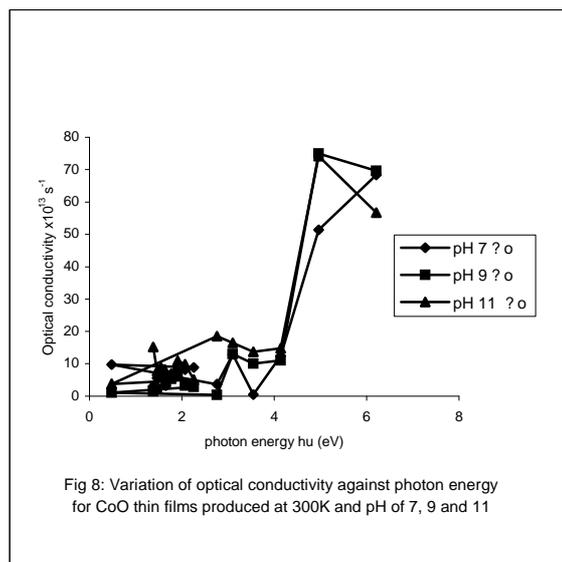
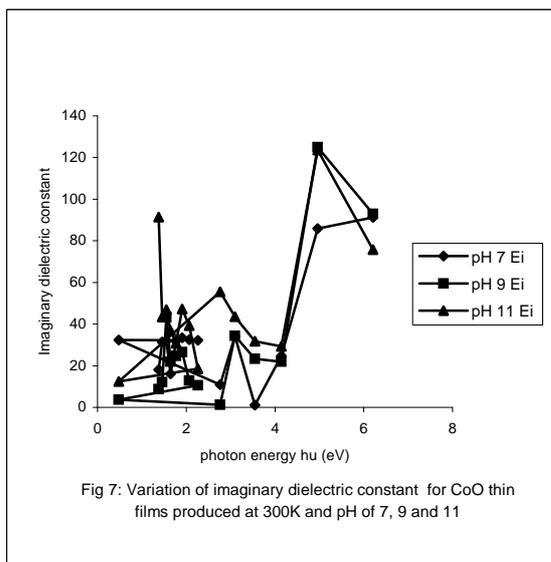
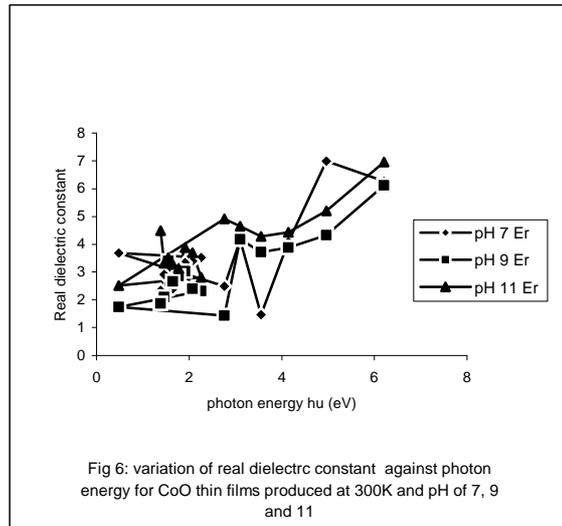
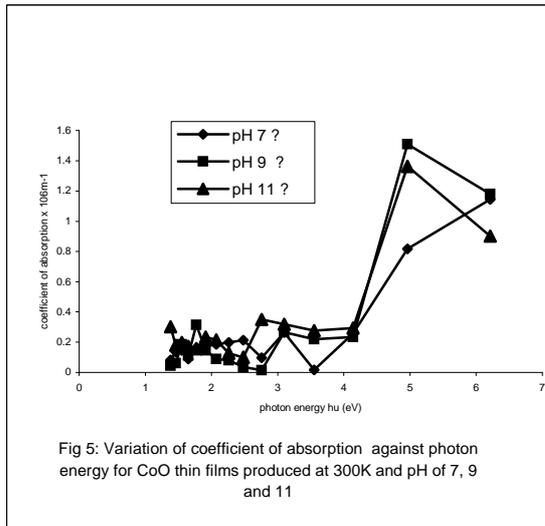
$\times 10^{-3}$ in VIS and $(18.18 - 31.94) \times 10^{-3}$ in NIR regions. Values of ϵ_i for film produced at pH of 9 decreases within $(21.93 - 124.93) \times 10^{-3}$ in UV, $(1.21 - 27.86) \times 10^{-3}$ in VIS and $(8.69 - 43.32) \times 10^{-3}$ in NIR regions. Values of ϵ_i for film produced at pH of 11 decreases within $(29.38 - 123.38) \times 10^{-3}$ in UV, $(12.49 - 55.58) \times 10^{-3}$ in VIS and $(43.51 - 91.03) \times 10^{-3}$ in NIR regions. The variation of optical conductivity (σ_o) with photon energy ($h\nu$) is displayed in Fig. 8. Values of optical conductivity σ_o of film produced at pH of 7 decreases within $(1.24 - 6.83) \times 10^{13} \text{ s}^{-1}$ in UV, $(0.05 - 1.02) \times 10^{13} \text{ s}^{-1}$ in VIS and $(0.03 - 0.50) \times 10^{13} \text{ s}^{-1}$ in NIR regions. For films produced at pH of 9, σ_o decreases within $(1.10 - 7.50) \times 10^{13} \text{ s}^{-1}$ in UV, $(0.040 - 1.05) \times 10^{13} \text{ s}^{-1}$ in VIS and $(0.15 - 0.81) \times 10^{13} \text{ s}^{-1}$ in NIR regions. The films produced at pH of 11 has σ_o decreasing within $(1.47 - 7.42) \times 10^{13} \text{ s}^{-1}$ in UV, $(0.38 - 1.85) \times 10^{13} \text{ s}^{-1}$ in VIS and $(0.77 - 1.58) \times 10^{13} \text{ s}^{-1}$ in NIR regions. The magnitude of 10^{13} s^{-1} of σ_o shows that the deposited CoO thin films has high photo response when irradiated with light. The average electrical conductivity (σ_e) of the films based on optical method is $0.53 \text{ (ohm - cm)}^{-1}$, $0.55 \text{ (ohm - cm)}^{-1}$ and $0.56 \text{ (ohm - cm)}^{-1}$ for films produced at pH of 7, 9 and 11 respectively. The values are within electrical conductivity range of $10^{-12} - 10^2 \text{ (ohm - cm)}^{-1}$ for semiconductors [27, 30] and [36]. The average films thickness (t) obtained by optical method is $0.062\mu\text{m}$, $0.072\mu\text{m}$ and $0.099\mu\text{m}$ for films produced at pH of 7, 9 and 11 respectively. Values of band gap energy of the films obtained using direct allowed transition of absorption coefficient method are $2.75 \pm 0.05 \text{ eV}$, $2.76 \pm 0.05 \text{ eV}$ and $2.48 \pm 0.05 \text{ eV}$ for films produced at pH of 7, 9 and 11 respectively. These

values compare well with band gap values of 2.2 – 2.4 eV for CdO thin films [16], 2.3 – 2.7 eV for CdO thin films [4], 2.49 – 2.88 eV for SnO_2 films [33] for transparent conducting oxide (TCO) thin films used in solar cells.

5. Conclusion

Cobalt oxide (CoO) thin films were produced using improve solution growth technique at pH values of 7, 9 and 11. Addition of ethylenediamine-tetra acetate (EDTA), a complexing agent with pH oppose to that of deposition bath constitutions, was used to enhance the deposition at different pH values. Films produced at pH of 7 and 9, have low absorbance (A), high transmittance (T) low reflectance (R) and low refractive index (n). Film produced at pH of 11, has relatively high absorbance (A), relatively low transmittance (T), relatively high reflectance (R) and relatively high refractive index (n). The optical properties and band gap of the deposited cobalt oxide (CoO) thin films are close to those earlier reported for transparent conducting oxide thin films. Hence, this deposition technique can be improved by using another complexing agent, with pH oppose to that of bath constitutions, to optimize the deposition conditions and produce enhance optical and solid state properties. The cobalt oxide (CoO) thin films produced at pH of 7 and 9 with refractive index (n) of about 1.9 would be employed in antireflection coatings and solar control glass window coatings. Films produced at pH 11 with refractive index (n) much greater than 1.9 could be employed in poultry production, solar cells and anti dazzling coatings.





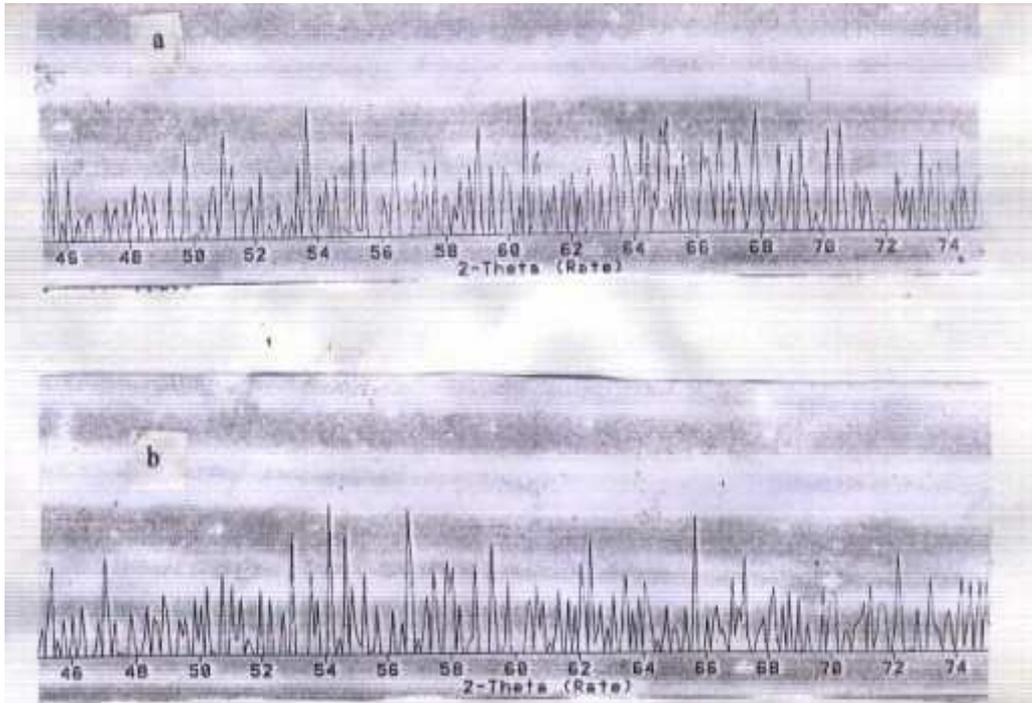


Fig. 9: X-ray diffraction results for (a) uncoated glass slide and (b) CoO thin films produced at 300K

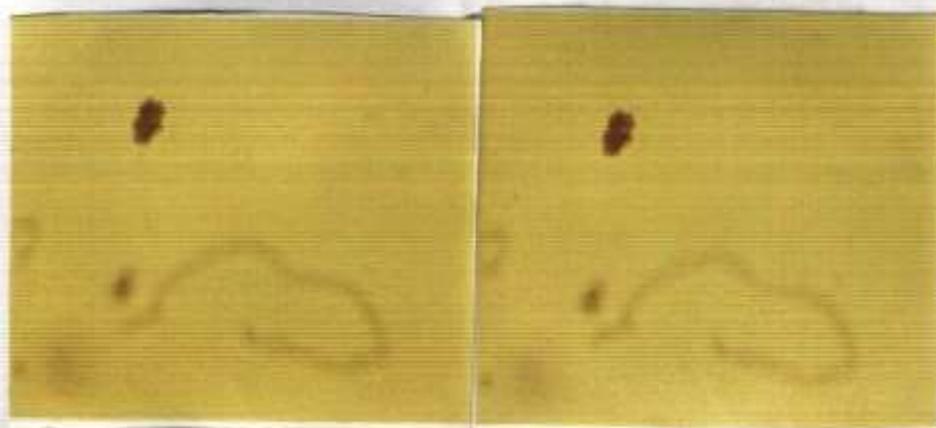


Fig. 10: Electron micrograph of CoO thin films produced at 300K

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