Comparative studies of Cobalt Oxide (CoO) and Copper Oxide (CuO) thin films deposited by improved electroless chemical bath technique at 320K

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Abstract

Semi conducting thin films of cobalt oxide (CoO) and copper oxide (CuO) were successfully deposited on glass slides at 320K and pH values of 7, 9 and 11 by electroless chemical bath deposition method. Ethylenediamine-tetra acetate (EDTA), another complexing agent, with pH oppose that of bath reagents was added to bath constitutions to enhance control and stabilize the deposition bath pH at different values suitable for film deposition. X-ray diffraction method was used to confirm the depositions. Electron micrographs of the films reveal uniform surface depositions. Absorbance (A) spectra data were measured by a single beam spectrophotometer at wavelength range 200nm to 900nm. Other optical and solid state properties were calculated from the data and compared with other deposited thin films. Average optical and solid state properties of CoO thin films include absorbance ranging from 0.051 to 0.212, transmittance 0.614 to 0.889, refractive index 1.64 to 2.43, electrical conductivity 0.43 to 0.58 (ohm-cm)$^{-1}$, film thickness 0.052 to 0.216μm and band gap 2.36 to 2.75 $\pm$0.05eV. For CuO thin films the absorbance ranges from 0.084 to 0.112, transmittance 0.646 to 0.824, refractive index 1.82 to 2.36, electrical conductivity 0.48 to 0.62 (ohm-cm)$^{-1}$, film thickness 0.003 to 0.051μm and band gap 1.75 to 2.45 $\pm$0.05eV. Films with refractive index lower than 1.9 could be used in antireflection coatings, eyeglass coating and solar thermal control coatings. Those with refractive index greater than 1.9 could be useful in construction of poultry houses and anti dazzling coatings. The films could be employed in solar cells.

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1.0 Introduction

Electroless chemical bath deposition (ECBD) method was predominantly employed in the field of commercial production of silver mirrors [24]. The technology is one of the intensively studied thin film deposition techniques for the preparation of various metal chalcogenide films [2, 4, 8, 16, 27, 28]. The deposition technique is simple, cost effective, convenient, reproducible and easy for coating good adhesive uniform films on large surfaces of both metallic and non-metallic substrates. The technique has been applied in producing emerging materials for solar cells, protective coating, photo thermal controls in buildings, energy efficient glazings including decorative and protective coatings, and is now being adopted by some industries [6, 9, 12, 16, 21]. The technology is based on the controlled release of cations and anions in an aqueous solution into which the substrates are immersed [4]. Properties of the films produced by electroless chemical bath deposition method depends on some complex and chemical processes of bath constitutions, formation of films and on heat treatment of the films. Any change in the

relative rates of complexation and precipitation in the bath, for example, has significant effect on the growth and structure of the films.

A problem associated with the electroless chemical bath method is the combinations of the various bath constitutions to obtain the pH for deposition. The combinations could be tedious and wasteful before obtaining the suitable deposition pH value. This could be improved by adding another complexing agent with pH opposing that of bath constituents to the bath reagents to enhance control and stabilize the deposition pH at different suitable values for film deposition.

We report the results for improved electroless chemical bath deposition of cobalt oxide (CoO) and copper oxide (CuO) thin films at 320K and pH of 7, 9 and 11. Ethylenediamine-tetra acetate (EDTA), with pH opposing that of bath constitutions was added to bath reagents to enhance control and stabilize the deposition pH at different values suitable for film growth. The optical and solid-state properties of the films where calculated and compared with other deposited films. Possible applications of the films where also discussed.

2.0 **Theory**

The absorbance (A) of a semi conducting film is the common logarithm of the reciprocal transmittance (*T)*, that is: $A = \log_{10}(T^{-1})$ (2.1)

From equation (2.1), the absorbance (*A*) and transmittance (*T*) are related [5] by:

$$T = 10^{-A} \quad (2.2)$$

The spectra reflectance (*R*), absorbance (*A)* and transmittance (*T*) have a relationship which allows for conservation of energy [13]: $A + T + R = 1$ (2.3)

At normal reflection, the reflectance (*R*) and the refractive index (*n*) of semi-conductors are related [10, 18] by: $n = (1 + R^{1/2}) / (1 - R^{1/2})$ (2.4)

The transmittance *(T*), coefficient of absorption (α) and distance (dμm) of the film traversed are related [5, 22] by: $T = \exp(-\alpha d)$ (2.5)

From equation. (2.5), for a unit distance traversed:

$$\alpha = \text{In}\,(T^{-1}) \times 10^{6} m^{-1} \quad (2.6)$$

The coefficient of absorption (α) is also related to extinction coefficient, *k* and wavelength of radiation, λ [30] by: $\alpha = 4\pi k/\lambda$ (2.7)

According to Pankove [22] and Cho et al, [3] the complex dielectric constant is:

$$\in_c = (n + ik)^2 \quad (2.8)$$

where the real dielectric constant is: $\in_r = n^2 - k^2$ (2.9)

and the imaginary dielectric constant: $\in_i = 2nk$ (2.10)

The optical conductivity (σ_o) of the film is given by: $\sigma_o = \alpha nc/4\pi$ (2.11)

where *c* is the velocity of light. The electrical conductivity σ_e of the film [7] is giving by:

$$\sigma_e = 2\lambda\,\sigma_o/\alpha. \quad (2.12)$$

Near the absorption edge, the dependence of absorption coefficient (α) on photon energy ($h\nu$) [4, 18] is giving by: $\alpha = A(h\nu - E_g)^{\gamma}$ $^{\Psi}$ (2.13)

where E_g is the bandgap, $\Psi\gamma = 1/2$ for allowed direct transition and $\Psi\gamma = 3/2$ for forbidden direct transition. Value of $\Psi\gamma = 2$ for allowed indirect transition and $\Psi\gamma = 3$ for forbidden indirect transition. The constant $A = 3.38 \times 10^7\ n^{-1}\ (m_e/m_o)^{1/2}\ E_g/h\nu$ where m_e is the effective electron mass and m_o is the free electron mass for direct allowed transition. The plots of α^2against $h\nu$ is a straight line with a deviation in the region of absorption edge. Extrapolation to the point where $\alpha^2 = 0$ gives the energy gap. The thickness (*t*) of the films based on the optical method for transmittance (T) of light through a weak absorbing film on non-absorbing substrate [11, 22] is giving by:

$$t = \text{In}\,[(1 - R)^2/T] / \alpha \quad (2.14)$$

3.0 **Experimental details**

The deposition of cobalt oxide (CoO) and copper oxide (CuO) thin films on glass slides was successfully carried out using chemical bath method at temperature of 320K and pH of 7, 9 and 11.

The glass slides were cleaned by degreasing them in concentrated nitric acid (HNO_3) for 3 days, thoroughly washed in detergent solution, rinsed in distilled water and dried in air. The cleaned glass surfaces provided nucleation centres for growth, good adhesion and uniform deposition of the films. Reaction baths were 50ml glass beakers containing different molar solutions and volumes of deposition bath constituents. The bath constituents for the deposition of cobalt oxide (CoO) thin films were cobalt chloride – 6 – water ($CoCl_2.6H_2O$) as source of (Co^{2+}), sodium hydroxide (NaOH) as source of hydroxyl ions (OH^-), ammonia (NH_3) as complexing agent while ethylenediaminetretra acetate (EDTA) (another complexing agent) was used to vary pH values of the deposition solutions. Distilled water was added to raise the volume of the bath solutions to a certain level. For the deposition of copper oxide (CuO) thin films, copper chloride -2 - water ($CuCl_2.2H_2O$) was used to replace the cobalt chloride – 6 – water ($CoCl_2.6H_2O$) in a similar reaction bath. Details of bath constituents for the preparation of the metal oxide (YO) thin films are shown in table 1.The symbol Y represent Co and Cu in the deposition of CoO and CuO respectively. The complexing agent ammonia (NH_3) formed complex, ions with Y^{2b+}. It slowly released Y^{2b+}, ensured ion by ion condensation of Y^{2b+} and O^{2-}, controlled the growth rate of the deposited thin films and eliminated spontaneous precipitation of the chemical reagents in the bath. The solution baths were stirred with a glass rod and their initial pH values noted. The baths were placed in a hot water bath that was maintained at a steady temperature of 320K by a Stuart magnetic stirrer hot plate. A cleaned glass slide was suspended in each reaction bath for 3 hours. After deposition time, the coated glass slides were rinsed with distilled water and dried in air. Pretest runs were carried out to determine the optimum deposition parameters such as deposition time, pH and volumes of bath constituents. The most probable reaction equation for the deposition of CoO thin films is

$$CoCl_2.6H_2O + NH_3 + 2NaOH \rightarrow CoO\downarrow + NH_3 + 7H_2O + 2NaCl$$

The basic reaction equation for deposition of CuO thin films is

$$CuCl_2.2H_2O + NH_3 + 2NaOH \rightarrow CuO\downarrow + NH_3 + H_2O + 2NaCl$$

Table 1: Bath constituents for preparation of CoO and CuO thin films.

Initial Bath pH	0.3M $YCl_2.b(2H_2O)^r$ Vol. (ml)	5.0M NH_3 Vol. (ml)	2.5M NaOH Vol. (ml)	H_2O Vol (ml)	0.2M EDTA Vol (ml)
7	8	3	5	20	36
9	7	4	6	20	21
11	6	5	7	20	17

where Y = Co, b = 3, r = 1 for $CoCl_2.6H_2O$; Y = Cu, b = 1, r = 1 for $CuCl_2.2H_2O$

The absorbance (A) spectra data of the deposited thin films where obtained using a computerized single beam spectrophotometer (Pharmacia LKB Biochrom 4060) in the ultraviolet -visible - near infrared (UV - VIS - NIR) regions at wavelength range of 200nm to 900nm. The reference and coated glass slides were mounted on a rotating holder at the reference and sample compartments respectively and scanned to obtain the absorbance spectra data. Other optical and solid state properties of the deposited thin films were obtained from the absorbance (A) spectra data by calculations based on the theoretical analysis. Structural characterization of the films was determined with x- ray diffraction technique using Diano cooperation x-ray diffractometry (model XRD 2100 E*) and copper target (CuKα) with wavelength 1.540502 Å**,** current 30mA and voltage 45 kV. The surface microstructure of the films was viewed using electron microscope at magnification x 100.

4.0 Results and analysis

The absorbance spectra data obtain by direct measurement for the deposited cobalt oxide (CoO) and copper oxide (CuO) thin films are displayed in Figures 1 and 2 respectively. The films have high absorbance for wavelengths lower than 300nm and low absorbance for wavelength range 350 – 850nm. Figure 1 shows that cobalt oxide (CoO) thin film produced at pH of 7 has negative absorbance at wavelength range of 350nm to 400nm and at 800nm with a minimum of –0.083 at 800nm. The film produced at pH of 9 has negative absorbance of –0.169 at 350nm. Films produced at pH of 11 have positive absorbance spectra. Similarly, Figure 2 shows that the spectra absorbance of copper oxide thin films have positive absorbance spectra except at pH value of 12 where the absorbance is –0.010 at a wavelength of 350nm. The absorbance spectra of both CoO and CuO produced at pH of 7 are low. Spetra transmittance (T) and reflectance (R) of cobalt oxide and copper oxide thin films calculated from equations (2.2) and (2.3) are shown in Figures 3 and 4 respectively. Both films have low transmittance for wavelengths lower than 300nm and high transmittance for wavelength range 300nm to 850nm. Film with negative absorbance has transmittance greater than 1.00 while those with positive absorbance have transmittance lower than 1.00. For cobalt oxide thin films, in Figure 3, the transmittance varies from 0.159 to 0.416 for wavelength lower than 300nm and from 0.685 to 1.211 for wavelength range 350 to 850nm. Film produced at pH of 7 has the highest transmittance of 1.211 at 800nm. For copper oxide thin films in figure 4, the transmittance varies from 0.257 to 0.449 for wavelength lowers than 300nm and from 0.771 to 1.023 for wavelength range 300nm to 850nm. The films produced at pH of 11 have the highest transmittance of 1.023 at 350nm. The spectra reflectance (R) of CoO thin films in Figure 3 exhibited high values of 0.042 to 0.203 for wavelengths lower than 300nm and low values from -0.128 to 0.151 for wavelength range 300nm to 850nm. The films with negative absorbance also have negative reflectance. Similarly, the spectra reflectance (R) of CuO thin films in fig 4 have high values of 0.141 to 0.203 for wavelengths lower than 300nm and low values of –0.013 to 0.118 for wavelength range from 350nm to 850nm. Films produced at pH of 11 have negative reflectance of –0.013 at 350nm. The high transmittance and low reflectance properties exhibited by cobalt oxide and copper oxide thin films produced at pH of 7 could be employed in antireflection coatings for solar thermal devices and eye glass coatings to reduce solar reflectance increase the transmittance and improve their efficiencies. The high transmittance and low reflectance properties of both CoO and CuO thin films produced at pH of 9 and 11 respectively in the visible region are desirable characteristics for ideal solar control glazing to avoid glare problems [17] and could be employed in solar thermal control coatings. The thin films of cobalt oxide (CoO) and copper oxide (CuO) thin films produced at pH of 11 and 9 with relatively lower transmittance and higher reflectance could be employed in construction of poultry houses to allow enough infrared radiation to warm the very young chicks during the day. This could reduce the cost of energy consumption through the use of stoves, heaters, electric bulbs etc and the hazards associated with them while at the same time protecting the chicks from ultraviolet radiation. The application of solar energy as a source of heat in chick brooding is environmentally acceptable and promotes sustainable development [20]. Solar energy technologies are also applicable to egg incubation and to the drying of chicken manure [19]. These films could also be used for photocells [21], solar cells [14], anti dazzling coating in car windscreens and driving mirrors to reduce the dazzling effects of light at night. Refractive index (n) was calculated from equation (2.4). The variation of refractive index (n) with photon energy for cobalt oxide (CoO) and copper oxide (CuO) thin films is shown in Figures 5 and 6 respectively. Both films exhibited high refractive index (n) for photon energies higher than 4.14 eV and low refractive index (n) values for photon energy range 1.46eV to 4.14eV. Values of refractive index of CoO thin films vary from 1.52 to 2.64 for photon energy higher than 4.14eV and from 1.17 to 2.27 for photon energy range 1.46eV to 4.14eV. In the case of copper oxide (CuO) thin films in figure 6, values of refractive index vary from 2.20 to 2.64 for photon energy higher than 4.14 eV and from 1.11 to 2.03 for photon energy range 1.46eV to 4.14eV. The thin films of cobalt oxide and copper oxide produced at pH of 7 have low values of refractive index. These films could find useful applications in antireflection coatings in agreement with the results of Brinker and Harringston [1] and Petit and Brinker [25]. Such films with refractive index

lower than 1.9 could be employed to antireflect photovoltaic from 0.36 to 0.04 and improve the transmittance of glass from 0.91 to 0.96.

The coefficient of absorption (α) was calculated from equation (2.6). The variation of coefficient of absorption (α) with photon energy for the cobalt oxide (CoO) and copper oxide (CuO) thin films are shown in figures 7 and 8 respectively. The magnitude of the coefficient of absorption $\alpha = 10^6$ m^{-1} is within the α range 10^6 to 10^7 m^{-1} for semi-conductor thin films suitable for polycrystalline thin film solar cell [15]. The equation (2.13), coefficient of absorption method, was used to determine the band gap. The method is the simplest and perhaps the most direct method of determining the band gap of semiconductors. Values of band gap for the deposited cobalt oxide (CoO) thin films vary from 2.30 to 2.75 $\pm$0.05eV. For the copper oxide (CuO) thin films, the band gap varies from 1.75 to 2.45 $\pm$0.05eV. These values compare well with 2.3 to 2.7*eV* for CuO films and 2.00*eV* for Cu_2O films [4] and could be used in solar cells. The optical conductivity (σ_o) of the films was obtained from equation (2.11). Both films have good photo response with average optical conductivity (σ_o) of 10^{13} s^{-1}. The magnitude of average electrical conductivities obtained from equation (2.12) for both thin films is 10^{-1} (ohm-cm) $^{-1}$ within the electrical conductivity range 10^{-12} to 10^2 (ohm – cm)$^{-1}$ for semiconductors [23, 26, 29]. The film thickness was calculated from equation (2.14). Values of film thickness for CoO thin films vary from 0.052 to 0.216µm. For CuO thin films, they vary from 0.003 to 0.180µm. Other properties determined include extinction coefficient (*k*) from equation (2.7), real dielectric constant ($\in_r$) from equation (2.9) and imaginary dielectric constant ($\in_i$) from equation (2.10). Average optical and solid-state properties at wavelength of 550nm for cobalt oxide and copper oxide thin films are shown in tables 2 and 3 respectively. The x-ray diffraction patterns of the uncoated glass and deposited films on glass are shown in Figure 9 and Figure 11 for cobalt oxide (CoO) and copper oxide (CuO) thin films respectively. The diffraction patterns reveal diffraction peaks at some 2θ values. Electronmicrographs of the films at magnification of x100 shown in Figure 10 and Figure 12 for cobalt oxide (CoO) and copper oxide (CuO) respectively reveal their uniform surface deposition.

Table 2: Average optical and solid-state properties for cobalt oxide thin films

PH	T	n	k x10^{-2}	α x 10^6 (m^{-1})	$\in_r$	σ_o x 10^{13} (s^{-1})	σ_e (ohm-cm)$^{-1}$	t (µm)	E_g $\pm$ 0.05 (eV)
7	0.899	1.64	0.52	0.118	2.71	0.46	0.43	0.052	2.75
9	0.787	1.98	1.05	0.240	3.94	1.13	0.52	0.083	2.36
11	0.614	2.43	1.94	0.488	5.92	2.57	0.58	0.216	2.63

Table 3: Average optical and solid-state properties for copper oxide thin films

pH	t	n	k x10^{-2}	α x 10^6 (m^{-1})	$\in_r$	σ_o x 10^{13} (s^{-1})	σ_e (ohm-cm) $^{-1}$	t (µm)	E_g $\pm$0.05 (eV)
7	0.824	1.82	0.85	0.194	3.30	0.84	0.43	0.003	2.35
9	0.646	2.36	1.91	0.437	5.58	2.46	0.62	0.180	2.45
11	0.773	2.03	1.13	0.257	4.11	1.25	0.53	0.051	1.75

5.0 Conclusion

Semi conducting thin films of cobalt oxide (CoO) and copper oxide (CuO) with improved growth characteristics were successfully deposited on glass slides by improved electroless chemical bath technique at 320K and pH values of 7, 9 and 11. Ethylenediamine-tetra acetate (EDTA), a complexing agent, with pH oppose that of deposition bath constituents was added to bath constitutions to enhance control and stabilize the deposition pH at different values suitable for film deposition. A single beam

spectrophotometer (Pharmacia LKB Bichrom 4060) was used to obtain the spectra absorbance data. Other optical and solid-state properties of the films such as transmittance, refractive index, extinction coefficient, optical conductivity, electrical conductivity, coefficient of absorption, energy gap, etc. were calculated. The films with refractive index lower than 1.9 could be used in antireflection coatings, eyeglass coatings, and solar thermal control coatings. Those with refractive index greater than 1.9 could be useful in poultry production, antidazzling coatings and solar cells.

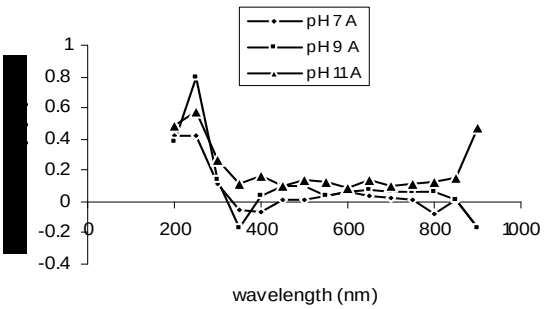


Figure 1: Absorbance (A) spectra for CoO thin films produced at 320k and pH of 7, 9 and 11

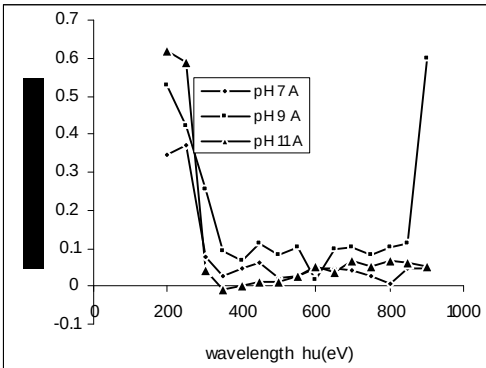


Figure 2: Absorbance (A) spectra for CuO thin films produced at 320k and pH of 7, 9 and 11.

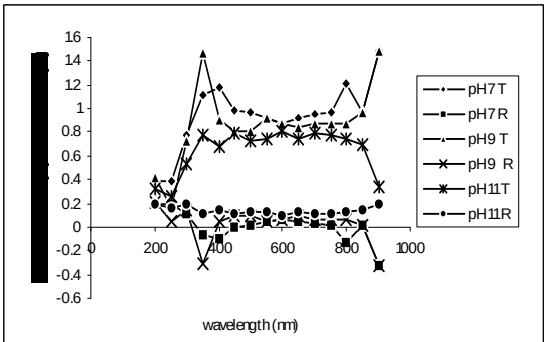


Figure 3: Transmittance (T)-Refectance (R) for CoO thin films produced at 320k and pH of 7, 9 and 11

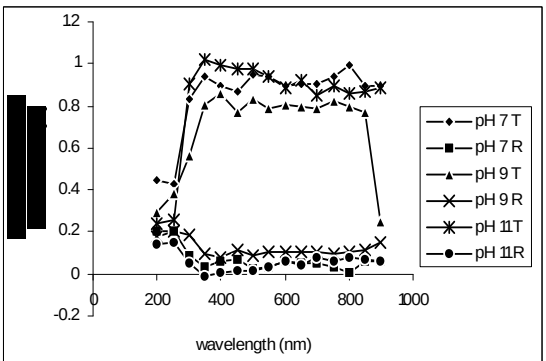
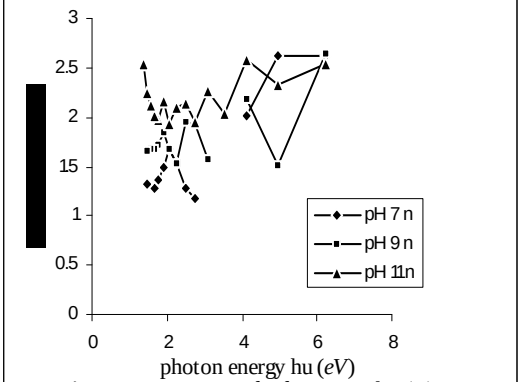


Figure 4: Transmittance (T) Reflectance (R) spectra for CuO thin films produced at 320K and pH of 7, 9 and 11.



Fi gure 5: Variation of refractive index (*n*) against photon energy for CoO thin films produced at 320K and pH of 7, 9 and 11

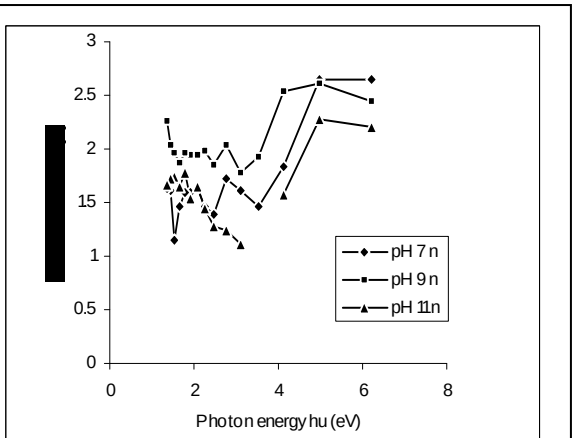


Figure 6: Variation of refractive index (n) against photon energy for CuO films produced at 320k and pH of 7, 9 and 11.

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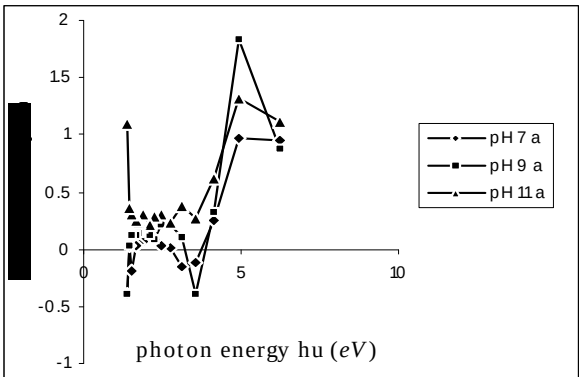

Figure 7: Variation of coefficient of absorption against photon energy for CoO thin films produced at 320k and pH of 7, 9 and 11

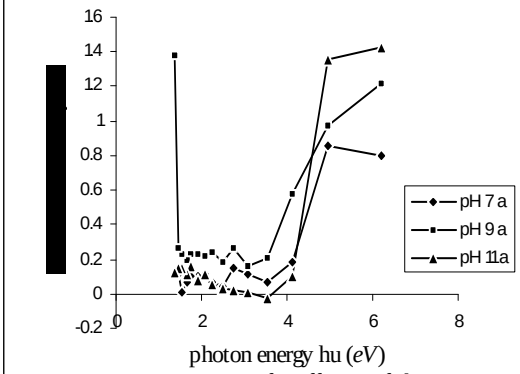

Figure 8: Variation of coefficient of absorption against photon energy for CuO thin films produced at 320k and pH of 7, 9 and 11.

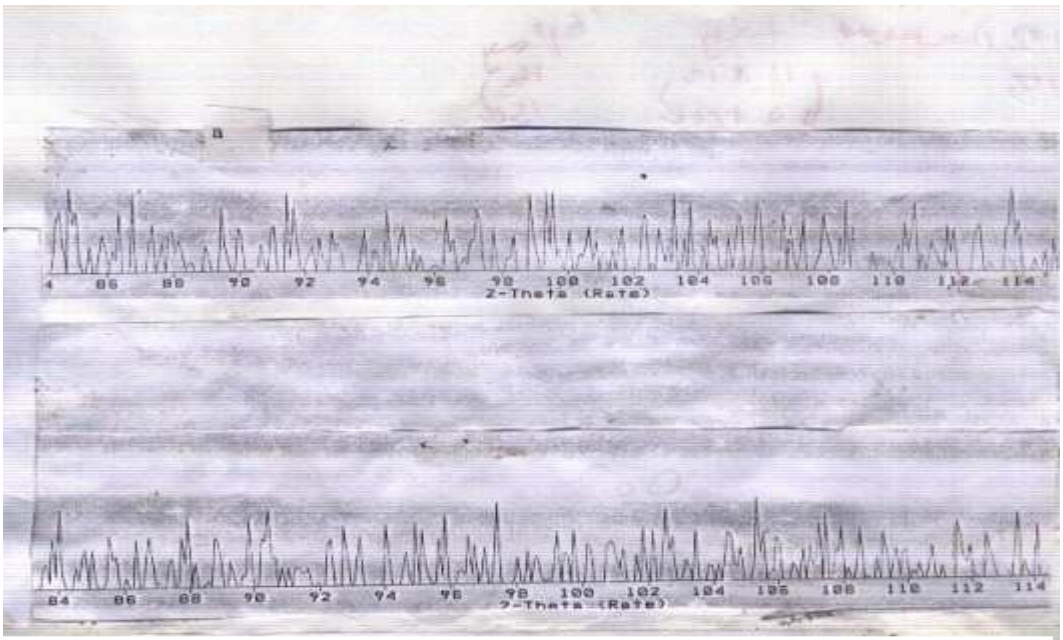

Figure 9: X-ray diffraction results for (a) uncoated glass slide and (b) CoO thin films produced at 320K

Figure 10: Electaron micrograph of CoO thin films produced at 320K

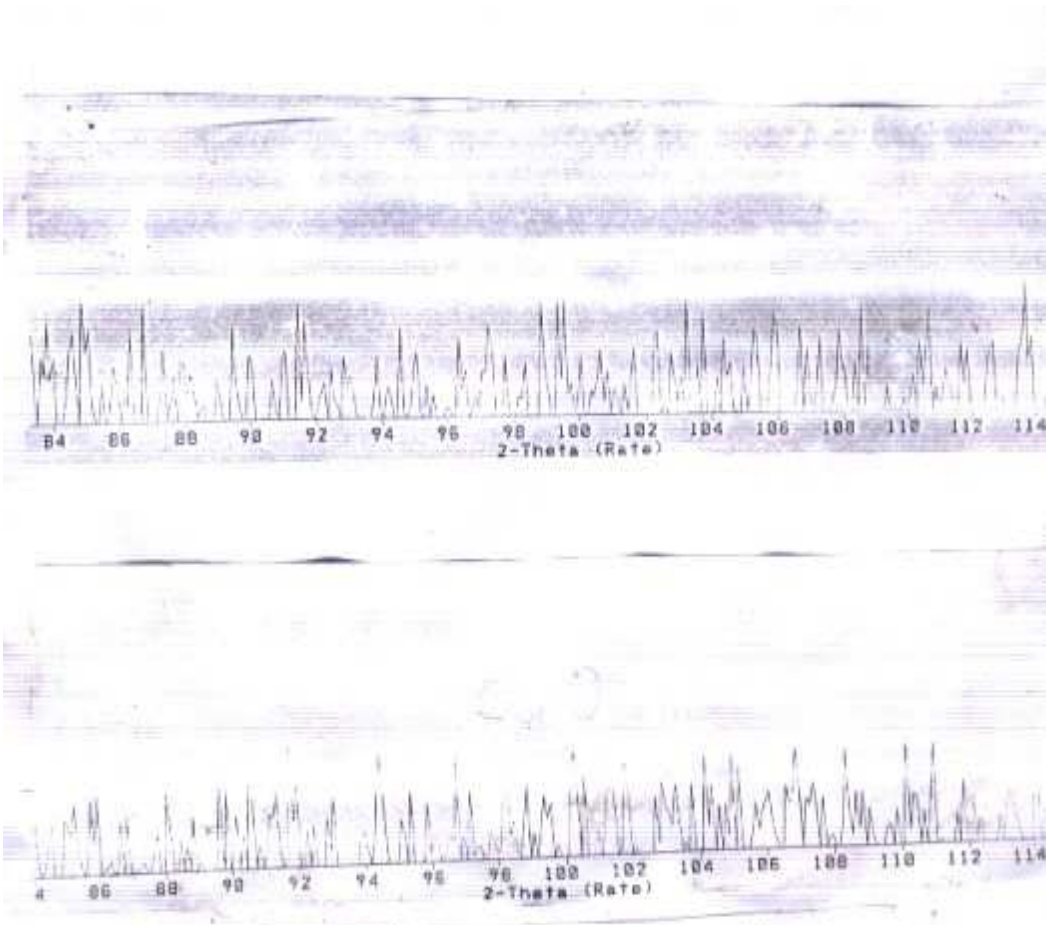

Figure 11: X-ray diffraction results for (a) uncoated glass slide and (b) CuO thin films produced at 320K

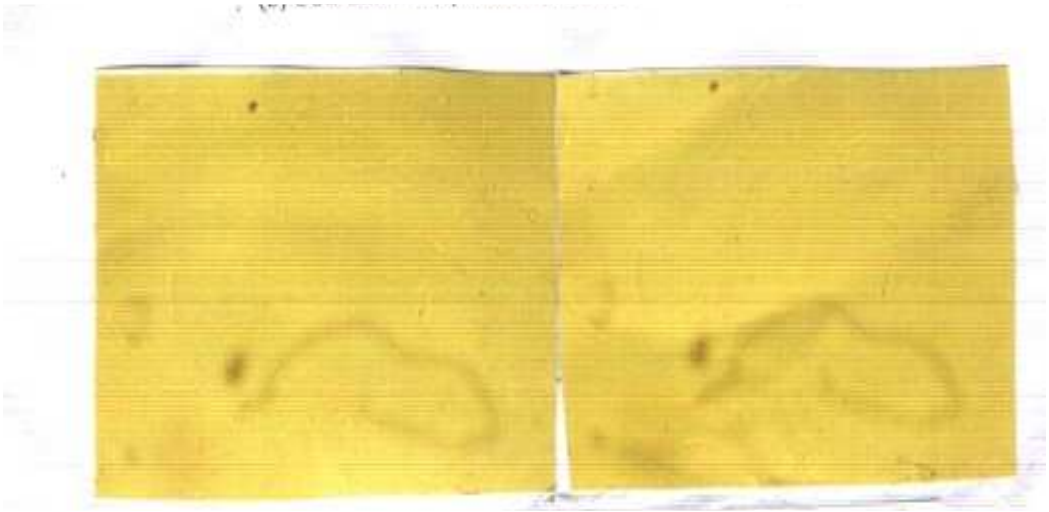

Figure 12: Electaron micrograph of CuO thin films produced at 320K

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