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The Influence of Different Complexing Agents on the Properties of SILAR-Deposited Cobalt Selenide Thin Films

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Abstract: The nanostructured thin films have good physical properties, and could be used in the various applications such as capacitor, photo detector, ultra violet opto electronics, light emitting diode, photonic integrated circuit and solar cell applications. The successive ionic layer adsorption and reaction (SILAR) method is one of the common chemical deposition techniques. This method has many advantages such as the simplest & the cheapest method, and required low temperature deposition process. This is the first time, preparation of cobalt selenide thin films onto soda lime glass slide at room temperature in the presence of various complexing agents (ammonia, triethanolamine and ethylenediaminetetraacetic acid disodium salt). Characterization of thin films was carried by using x-ray diffraction, atomic force microscopy and UV-visible spectrophotometer. AFM analysis showed that the cobalt selenide thin films prepared in the presence of ammonia exhibited uniform and completely covered the entire surface area of substrate. XRD data confirmed that obtained thin films (using ammonia and triethanolamine) were polycrystalline with well-developed phases. Optical properties indicated the band gap values of all films were in the range of 1.8 to 2 eV, suitable to be used in solar cell applications.

Keywords: Thin film, cobalt selenide, solar cell, band gap, absorption**.**

1 Introduction

The study of the properties of nanostructured thin films has received great attention all over the world. These semiconductor materials were used in different applications including biomedical [1], electro luminescence devices [2], anti-microbial activity, photonic devices, gas sensing [3], optical devices, field emission devices [4], laser devices [5], solar cell [6-9] and electronic devices. Characterization has been done in order to investigate the structural, morphological, optical and compositional of obtained films. These tools including as x-ray diffraction, scanning electron microscopy, energy dispersive x-ray analysis, spectroscopic ellipsometry, Raman Spectroscopy [10], electrochemical impedance spectroscopy, profilometer, Rutherford backscattering spectrometry, scanning tunneling microscopy [11], and atomic force microscopy [12]. Several deposition techniques have been used to produce thin films. These deposition techniques including chemical bath deposition [13-15], electro deposition [16], evaporation method [17], pulsed laser deposition [18], Spray pyrolysis [19], RF Sputtering [20], molecular beam epitaxy, chemical vapor deposition, sol gel method [21], and SILAR. Among the various deposition techniques, the successive ionic layer adsorption and reaction (SILAR) deposition method is the most commonly used due to simple [22], inexpensive [23], convenient for large area deposition in low temperature [24].

The main goal of this work is to investigate the influence of complexing agent (ammonia, triethanolamine and ethylenediaminetetraacetic acid disodium salt) on the cobalt selenide thin films deposited onto soda lime glass. Characterization of thin films by using X-ray diffraction, atomic force microscopy and UV-visible spectrophotometry was used for the first time.

2 Experimental Details

2.1 Preparation of Thin Films

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In this experiment, cobalt (II) chloride hexahydrate (acted as a source of Co^{2+} ion) and sodium selenite (supplied Se^{2-} ions) were used without further purification. Thin films were deposited onto soda lime glass slide via SILAR deposition method. The soda lime glass slide was cleaned by acetone, and de-ionized water before use. During the deposition process, the glass substrate immersed in the 0.2 M cationic solution (Co^{2+} ion) containing complexing agent for 35 seconds. After rinsing with de-ionized water for 6 seconds, it was immersed in 0.2 M anionic solution (Se^{2-} ions) containing complexing agent for 35 seconds. Then, rinsing with de-ionized water for 6 seconds again in order to remove the loose material on it. The sodium hydroxide (NaOH) and hydrochloric acid (HCl) were used to adjust pH value. Deposition was carried out at pH 3, at room temperature under different complexing agents such as (0.2 M triethanolamine) TEA, (0.2 M ethylenediaminetetraacetic acid disodium salt) Na_2EDTA and 0.2M ammonia. After the deposition process (after 18 cycles), the films were collected, rinsed by de-ionized water, and finally, put in the oven for 24 hours.

2.2 Characterization of Thin Films

The X-ray diffraction analysis was done to investigate the structure properties of the films. This technique was carried out by using a Malvern Panalytical diffractometer for the 2θ ranging from 10° to 80° with CuKα ($\lambda = 0.15418$ nm) radiation. The surface morphology, and roughness were analyzed by recording atomic force microscope (AFM) images with Bruker. The mode is Scanasyst peak force tapping. The cantilever is scanasyst-air (material: silicon tip on nitride lever with spring constants 0.4 N/m and resonance frequencies 70 kHz. The surface roughness was studied by on the R_q value. R_q is defined as the root mean square average of height deviation taken from the mean image data plane UV-visible spectrophotometer (Perkin Elmer UV/Vis Lambda 35) was used to investigate the optical properties of films. The thin films were placed across the sample radiation pathway while the cleaned microscope glass slide was put across the reference path. The band gap energy of the films was calculated based on the absorption data.

3 Results and Discussions

Thin film deposition techniques could be divided into two groups, namely physical technique and chemical method. One of the modern chemical deposition methods is called successive ionic layer adsorption and reaction (SILAR) method. SILAR method has many advantages [25] and can produce film thicknesses in the range of tenths of nanometers to several micrometers [26]. Researcher described that SILAR deposition technique comprised of two important processes [27], namely adsorption of ion onto the substrate and reaction of the adsorbed ion layer will be observed. During the deposition process, several processes could be observed [28, 29] such as dipping into cationic solution, rinsing in de-ionized water, dipping in an ionic solution and rinsing in de-ionized water.

The atomic force microscopy (AFM) measurement was done to study the surface topology of prepared sample. The figure 1 indicated the atomic force microscopy image of SILAR deposited cobalt selenide thin films in the presence of ammonia. This image was measured over 1 µm X 1 µm scanning range, indicating three-dimensional view of the sample. Based on the AFM image, cobalt selenide films composed of closely packed uniform crystal. It has dense morphology, compact structure and covers the entire surface area of substrate. The grains (average diameter is 0.05 µm) are nearly in spherical in shape and quite small. Film thickness and roughness were 1.1 µm and 0.026 µm, respectively. Sapna and co-workers [30] highlighted that the nature of grains has less porous and consisting of small spherical grains. Spherical or grain shaped semiconductor materials are considered as unique properties in thin films. These materials could be used in solar cell applications [31].

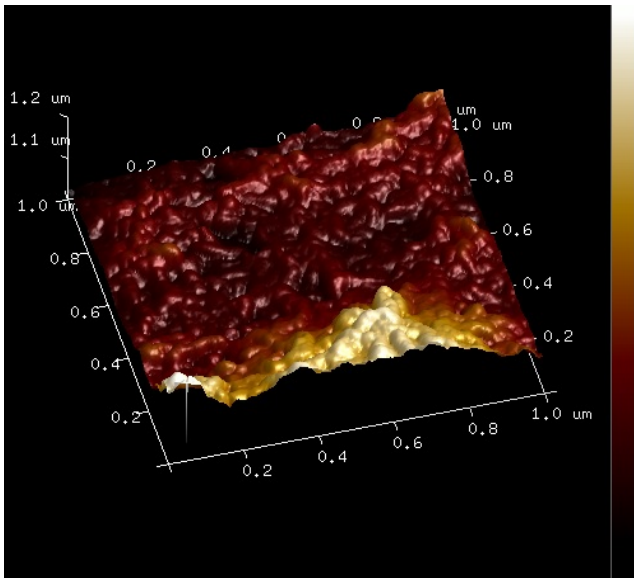

Fig. 1: Three-dimensional AFM view of SILAR deposited cobalt selenide thin films in the presence of ammonia.

Figure 2 indicated the X-ray diffraction (XRD) pattern for the cobalt selenide thin films prepared in the

presence of ammonia. Based on the figure, the XRD pattern showed several diffraction peaks at 2θ=24.5°, 28.7°, 33.9° and 37.8°, which can be indexed as reflection from the (022), (113), (004) and (133) plane of the cubic structure Co_9Se_8 compound. Other researchers have reported similar findings (cubic cobalt selenide structure) in the literature [32, 33]. The obtained X-ray diffraction pattern was well matched with the standard Joint Committee on Powder Diffraction Standards (JCPDS) (Reference code: 98-004-4857) as indicated in Table 1. According to the JCPDS data, lattice parameter values are a=b=c=10.431 Å. The crystal system, space group and space group number were cubic, Fm-3m and 225,respectively.

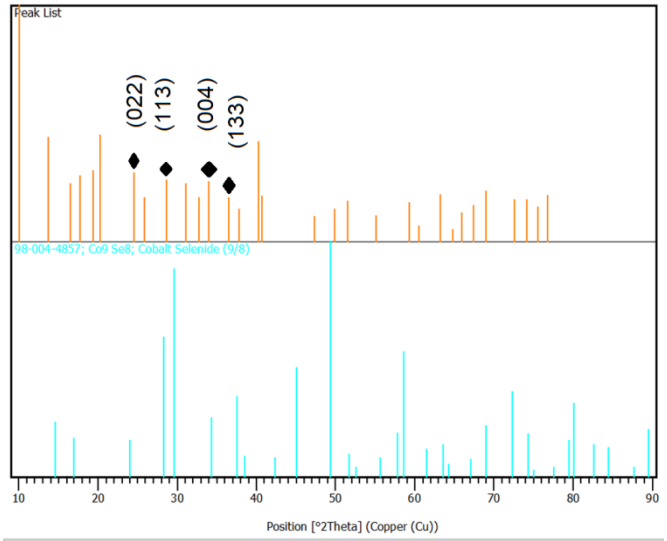

Fig. 2: X-ray diffraction pattern for the cobalt selenide thin films prepared in the presence of ammonia.

Table 1: Comparison of observed d-spacing values with standard d-spacing values of cobalt selenide thin films by using ammonia.

2θ (°)	*hkl*	Observed d-spacing values (Å)	Standard d-spacing values (Å)
24.5	022	3.6	3.6
28.7	113	3.1	3.1
33.9	004	2.6	2.6
37.8	133	2.4	2.4

Figure 3 indicates absorbance spectrum (in the wavelength of 300 to 1000 nm) of cobalt selenide thin films prepared in the presence of ammonia. Generally, the obtained sample showed high absorption in the visible range, which make these materials possible to use in the photo electrochemical cell and solar cells. Following that, absorbance reduces with the increasing of wavelength. A similar tendency was also seen by other researchers [34, 35]. The band gap was calculated based on the Stern equation which recommended by many researchers [36-42].

$$A = \frac{[k(hv-E_g)^{n/2}}{hv} \quad [equation\ 1]$$

where *v* is the frequency, *h* is the Planck's constant, *k* equals a constant while *n* carries the value of either 1 or 4. The *n* value is 1 for a direct gap material and *4* for indirect gap material. The plot of $(Ahv)^2$ against *hv* is indicated in figure 4. Extrapolation of the linear portion of the curve to $(Ahv)^2=0$ produce the band gap energy. The band gap was calculated to be 1.8 eV. These semiconductor materials could be used in solar cell applications because of direct band gap between 1 to 2 eV [43, 44]. Other scientist groups have reported similar band gap values (Table 2) for the cobalt selenide thin films prepared under various deposition techniques including chemical bath deposition, electro deposition, magnetron sputtering method and mechano chemical method.

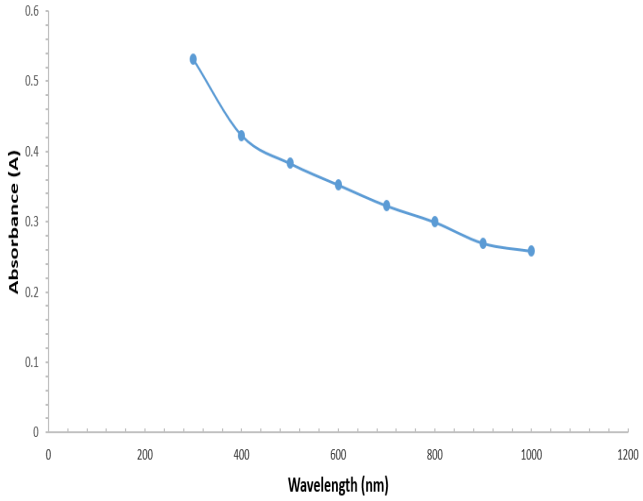

Fig. 3: Optical absorbance spectrum of cobalt selenide thin films prepared in the presence of ammonia.

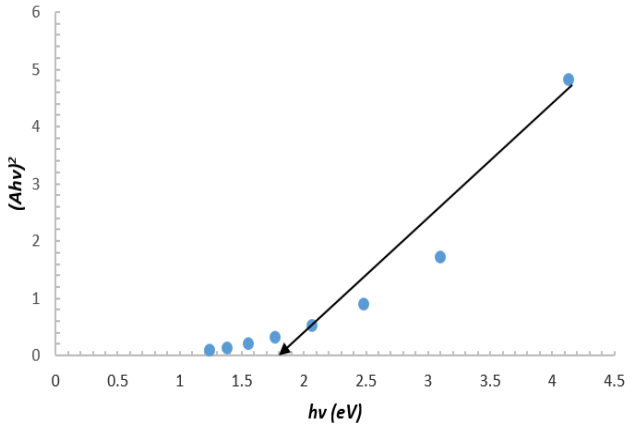

Fig. 4: Plot of $(Ahv)^2$ against *(hv)* of cobalt selenide thin films prepared in the presence of ammonia.

Table 2: Band gap energy of cobalt selenide thin films prepared under different deposition techniques.

Remarks	Band gap value (eV)
Thin films were produced onto glass substrate by using chemical bath deposition method in the presence of ammonia, cobalt (Ii) acetate and sodium selenosulphite as reported by Ghobadi and co-workers [45].	1.8 to 3.6
Thin films were synthesized onto tin oxide glass substrate via electro deposition technique in the presence of H_2SeO_3 and $Co(CH_3COO)_2$ solutions as described by Liu and co-workers [46].	1.53
Thin films were prepared onto non-conducting micro glass slide through chemical bath deposition method, in the presence of cobalt nitrate, ammonia and sodium selenosulphate as highlighted by Muddsar and co-workers [47].	1.7
Thin films were produced using magnetron sputtering method as pointed out by Zhu and co-workers [48].	1.53
Thin films were synthesized onto tin oxide coated glass by using electrodeposition method as concluded by Wang and co-workers [49].	1.53
Thin films were grown using the mechanochemical method as reported by Nina and co-workers [50].	1.7

The figure 5 exhibited the 3-dimensional atomic force microscopy image (1 µm X 1 µm scanning range) of SILAR deposited cobalt selenide thin films with the addition of triethanolamine (TEA). The AFM image showed the prepared films have non-uniform (0.03 to 0.1 µm) surface and cover the entire surface area of substrate. The formation of films modeled as mixture of small and big nano-grains. Film thickness and roughness were about 1.2 µm and 0.0245 µm, respectively. The influence of triethanolamine on the surface roughness and morphological properties of films have been studied by many researchers. Manikandan and co-workers [51] have reported that FeS films prepared by using 0.15 M TEA showed excellent structure. Preetha and co-workers [52] have highlighted that densely packed with bigger grains for the films prepared using TEA. Kishorkumar and co-workers [53] have pointed out that several obvious differences in morphology (nanoflake, nano sphere and nanocoral-like morphology) can be observed after addition of TEA.

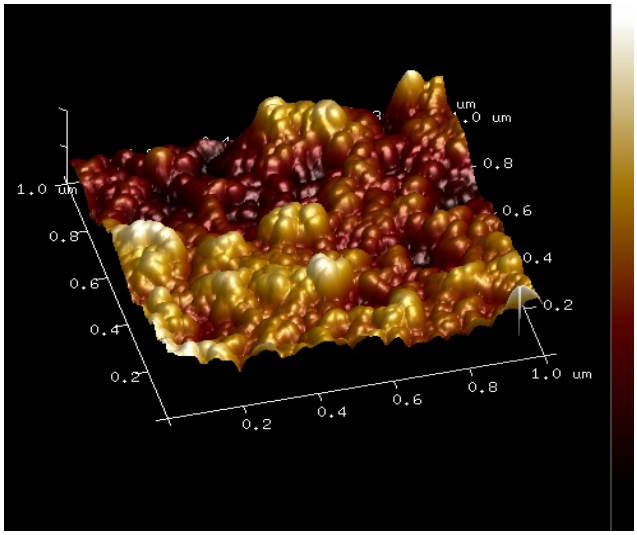

Fig.5: Three-dimensional AFM view of SILAR deposited cobalt selenide thin films in the presence of TEA.

Structural studies confirm that the films prepared using triethanolamine were polycrystalline in nature (Figure 6). Several diffraction peaks such as (002), (022), (113), (222), (135), (006), (246), (355), (337) and (066) could be observed. The obtained d-spacing values matched well with JCPDS data (Reference: 98-004-4857) as shown in Table 3. The number of diffraction peaks increased to ten peaks if compared to films prepared using ammonia (4 peaks), indicating more favourable condition for the formation of cobalt selenide (Co_9Se_8) thin films. The TEA complex resulted in better crystallinity as reported by other researchers [54-56].

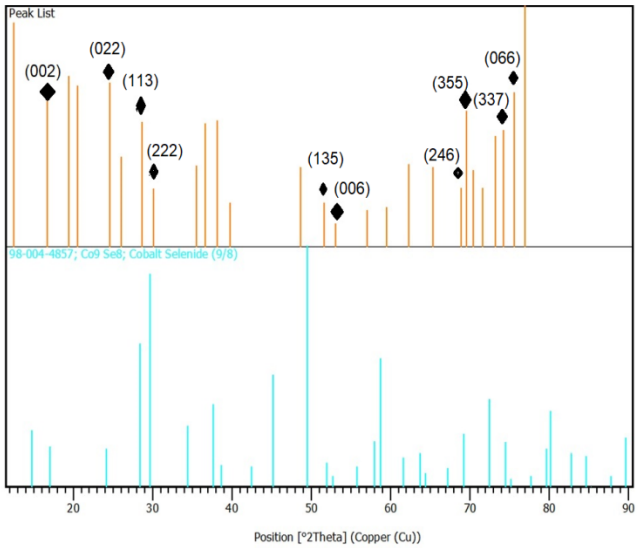

Fig.6: X-ray diffraction pattern for the cobalt selenide thin films prepared in the presence of TEA.

Table 3: Comparison of observed d-spacing values with standard d-spacing values of cobalt selenide thin films by using TEA.

2θ (°)	*hkl*	Observed d-spacing values (Å)	Standard d-spacing values (Å)
16.7	002	5.3	5.2
24.5	022	3.6	3.7
28.6	113	3.1	3.1
30.1	222	3.0	3.0
51.7	135	1.8	1.8
53.1	006	1.7	1.7
68.9	246	1.4	1.4
69.6	355	1.3	1.3
74.2	337	1.28	1.27
75.6	066	1.25	1.23

The optical properties of cobalt selenide thin films were studied and the absorption spectrum was displayed in Figure 7 (in the wavelength of 300 to 1000 nm). The results confirmed that high absorption in the visible range, indicating these films could be used in solar cell applications. The plot of $(Ahv)^2$ against hv is indicated in figure 8. Band gap could be measured when extrapolation of the linear portion of the curve to $(Ahv)^2$=0. The band gap was about 1.9 eV. The band gap has vital importance in photovoltaic related applications. The thin films have band gap of 1 to 2 eV are most suitable for solar cell applications [57-59].

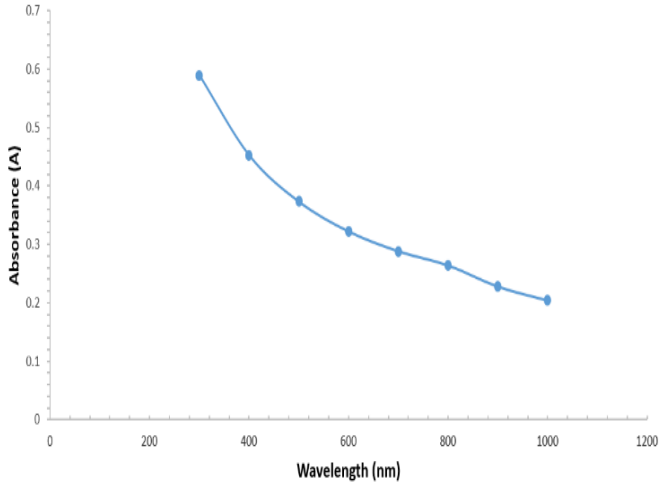

Fig. 7: Optical absorbance spectrum of cobalt selenide thin films prepared in the presence of TEA.

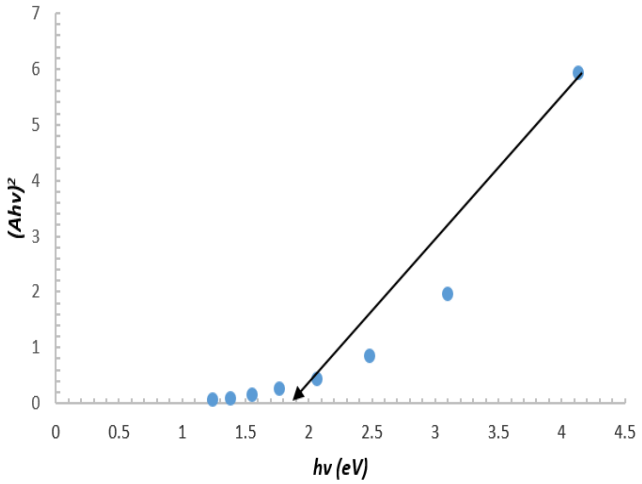

Fig. 8: Plot of $(Ahv)^2$ against (hv) of cobalt selenide thin films prepared in the presence of TEA.

The AFM measurements were scanned in 1 µm X 1 µm area. In the AFM image, several large grains (average diameter is 0.05 µm) were clearly apparent in the surface of substrate (figure 9). Non-uniform grain size could be observed for the films prepared using Na_2EDTA (Ethylenediaminetetraacetic acid disodium salt), indicating this complexing agent is not favorable. The film thickness and surface roughness were about 1.5 µm and 0.0046 µm, respectively.

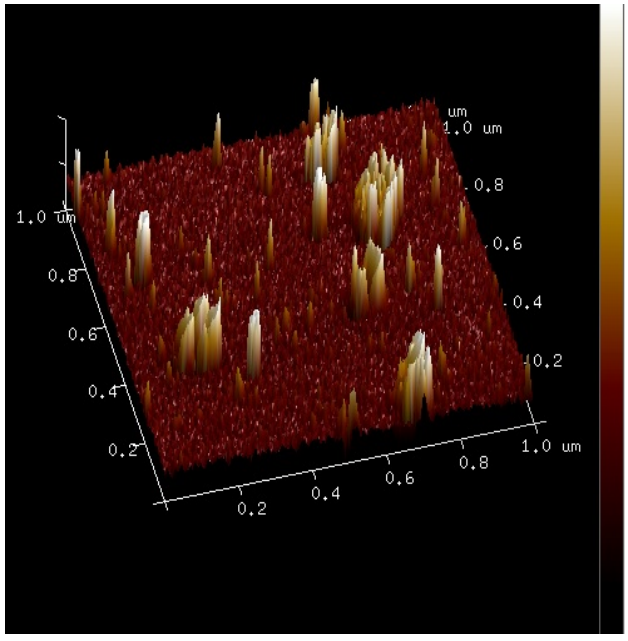

Fig.9: Three-dimensional AFM view of SILAR deposited cobalt selenide thin films in the presence of Na_2EDTA.

The XRD results reveal that the cobalt selenide (Co_9Se_8) films prepared by addition Na_2EDTA exhibited poor crystallinity. In this XRD analysis, prepared sample

indicated single diffraction peak (figure 10), attributed to (111) plane. The observed d-spacing value was matched [Table 4] with JCPDS data [Reference: 98-004-4857].

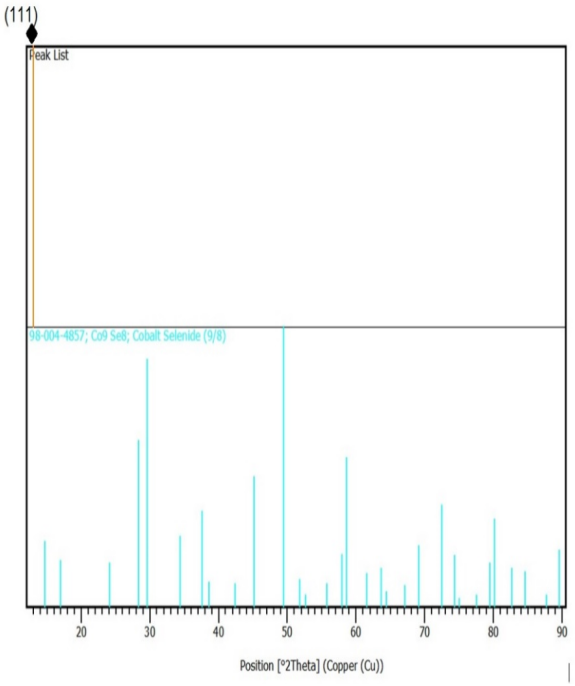

Fig.10: X-ray diffraction pattern for the cobalt selenide thin films prepared in the presence of Na_2EDTA.

Table 4: Comparison of observed d-spacing value with standard d-spacing value of cobalt selenide thin films by using Na_2EDTA.

2θ (°)	*hkl*	Observed d-spacing value (Å)	Standard d-spacing value (Å)
13.03	111	6.7	6.0

The optical absorption spectrum was recorded in the wavelength range between 300 to 1000 nm using UV-visible spectrophotometer. From the figure 11, noted that the highest absorbance value could be observed in the short wavelength. Similar results have been described in binary, ternary and quaternary thin films [60,61]. However, absorbance reduces with the increase in the wavelength. In the near infra-red region, the curves reveal a very low absorption of energy [62]. The band gap is 2 eV as shown in figure 12.

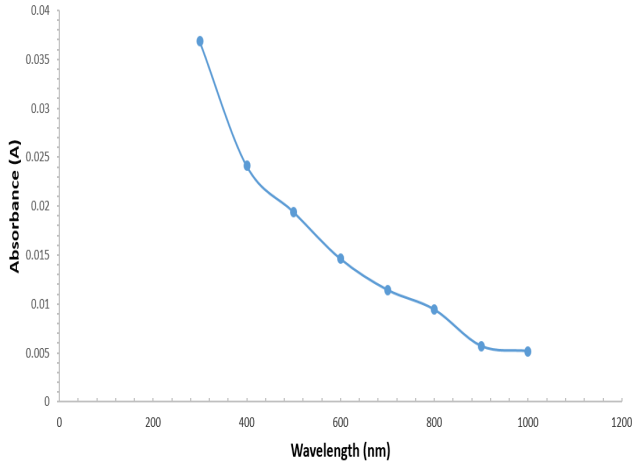

Fig.11: Optical absorbance spectrum of cobalt selenide thin films prepared in the presence of Na_2EDTA.

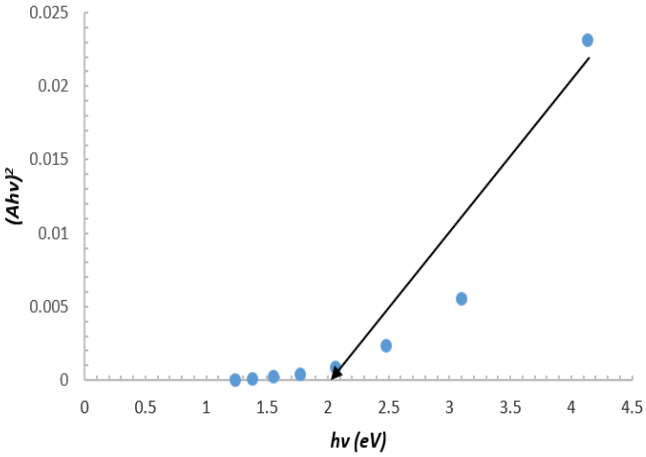

Fig.12: Plot of $(Ahv)^2$ against (hv) of cobalt selenide thin films prepared in the presence of Na_2EDTA.

The role of complexing agent in synthesis of thin films has been reported by many researchers. Several types of complexing agents such as hydrazine hydrate, triethylamin, nitrilotriacetic acid, glycine, tri-sodium citrate and tartaric acid were used during the deposition of thin films. Thin films prepared in the presence of complexing agent reduced the deposition rate due to higher complexation, improved the quality of thin films. Table 5 indicated that the obtained experimental results strongly depended on the amount of complexing agent and the nature of complexing agent.

Table 5: Thin films have been prepared by using various different complexing agents.

Thin films	Highlighted results	Complexing agent
zinc selenide	• Deposition was carried out in alkaline conditions, low temperature via chemical bath deposition method as reported by Hile and co-workers [63]. • Morphological studies revealed that spherical grain changed to nano-flakes when the complexing agent volume was increased (5 to 35 mL).	hydrazine hydrate
Lead sulphide	• Chemical bath deposition method was used to synthesis thin films onto glass slide as highlighted by Fekadu and Dejene [64]. • The major diffraction peak could be observed in the (111) plane, higher intensity of the diffraction peaks could be seen with increasing the concentration of complexing agent.	triethylamin
Ho doped (Cd-Ag)S films	• The growth thin films at room temperature onto glass slide through chemical bath deposition method as described by Yadu and co-workers [65]. • Ho-doped (Cd-Ag)S films have smaller particle size if compared to as-deposited films. Also, these films showed globular ball type and flakes type structure.	Ethylenediamine tetraacetic, triethanolamine
ZnS	• The complexing agents that produce stable complexes with the Zn^{2+} ions in the reaction bath. S^{2-} has been released from the decomposition of thiourea in the presence of hydroxide as pointed out by Kazi and co-workers [66]. • A high concentration of the complexing agent, causes to a heterojunction reaction on the substrate surface.	Tri-sodium citrate and tartaric acid
PbS	• Thin films were prepared using chemical bath deposition method as explained by Chalapathi and co-workers [67]. • The films prepared in the presence of complexing agent showed large grained with improved hole mobilities.	ethylenediamine tetraacetic acid
SnS	• Chemical bath deposition method was used to produce thin films as reported by Gaitan and co-workers [68]. • The films prepared using complexing agent formed thicker films, better grain connectivity, and the formation of cubic SnS.	nitrilotriacetic acid

Pr doped $CuInS_2$ film	• Thin films were synthesized using chemical bath deposition at 80 °C as highlighted by Sengupta and Pateria [69]. • The obtained films indicated high transmittance, low reflectance found in the visible region. Also, results exhibited high photocurrent is found, and good photosensitivity is observed in films.	triethanolamine
Cu_2ZnSnS_4 films	• Electro deposition was used to prepare thin films as pointed out by Demir [70]. • Deposition was carried out onto indium tin oxide glass, pH 5, deposition time (2700 seconds), and deposition potential of -1.05 V. • Formation of stoichiometric kesterite structure as indicated in XRD analysis.	Trisodium citrate
PbS	• Thin films have been synthesized onto glass slide in alkaline conditions, using chemical bath deposition method as concluded by Yesica and Luis [71]. • The films showed unique properties such as highly compact and adherent films, pyramid-like particles, cubic structure.	glycine
Cu_2ZnSnS_4 films	• Solvothermal method was used to produce thin films as described by Hadeethi and co-workers [72]. • The films prepared showed single phase nanocrystals, high purity and excellent crystallinity.	glycine
ZnS	• Chemical spray pyrolysis method was used to produce thin films as pointed out by Offor and co-workers [73]. • Morphology studies revealed that nano dot-like and nano rod-like crystals were observed with increasing molar concentration (complexing agent)	trisodium citrate

5 Conclusions

The cobalt selenide thin films were deposited onto soda lime glass by using SILAR method under various complexing agents. AFM studies demonstrated that surface morphology was markedly affected by the nature of the complexing agents. XRD data confirmed that obtained thin films (using ammonia and triethanolamine) were polycrystalline with well-developed phases. Experimental results confirmed that complexing agent played an important role in the formation of the thin films.

Acknowledgements

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