

Nanometric layers of Cadmium sulphide by CBD and a Potential Application.

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Abstract: We presented an application of nanometric layers of Cadmium Sulphide developed using Chemical Bath Deposition (CBD) technique for the growth of the CdS thin films. The thin films of CdS were synthesized employing two different complexing agent, acetilacetone and glycine. In this paper we compare the results of the thin films characterization with both complexing agent and the CdS thin film growth with glycine was selected in order to develop a Thin Film Transistor (TFT). The energy bandgaps in these cases were $E_g = 2.53$ eV using glycine as complexig agent, and $E_g = 2.37$ eV using acetylacetone as complexing agent. An hexagonal polycrystalline structure concerning to the X-ray analysis results for both processes, from those studies were calculated their grain sizes by the model of Debye-Scherrer, resulting 3 nm for the acetylacetone process and 30 nm for the glycine process. Also we started with the computational analysis and preliminary results are presented.

Key-Words: - Cadmium Sulfide, Semiconductors, Thin Films, Acetylacetone, Glycine and Transistors.

1 Introduction

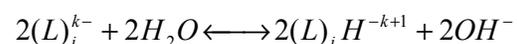
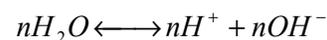
The recent advance in soft solution processing of inorganic materials offers an exciting opportunity to develop large-area manufacturing technologies for inorganic thin-film transistors (TFTs) [1, 2].

The chemical bath deposition (CBD) is used for deposition techniques that produce a solid film in a single immersion through control of the kinetics of formation of the solid. Film thickness and composition can be easily controlled by changing simple reaction parameters such as reactant concentration, reaction temperature and deposition time. The deposition medium for CBD consists of one or more salts of metal M^{n+} , a source for a chalcogenide (if the film to be deposited contains S, Se, etc), a source of oxygen for oxide films and a complexing agent (e.g. ammonia, NH_3 , EDTA, sodium citrate, etc.) all mixed in aqueous solution. For oxides, water normally provides the oxygen. The processes that occur in the CBD solution

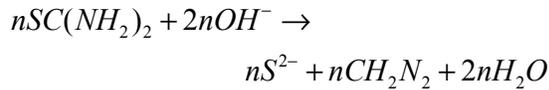
consist in general of the following steps: (1) Time zero to equilibrium between the complexing agent and water; (2) Hydrolysis of the chalcogenide source (if the film is not an oxide); (3) Formation and/or dissociation of ionic metal–ligand complexes $[M(L)_i]^{n-ik}$, where L^{k-} denotes one of more ligands, and (4), (5) Formation of the solids.

CBD for oxide or sulfide films from a metal cation M^{n+} complexed by i ligands L^{k-} , these steps can be depicted as follows [3]:

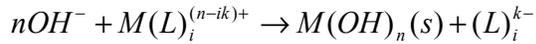
1) Dissociation of water and Complexant-water equilibrium:



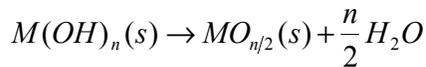
2) Thiourea hydrolyzes to form sulfides



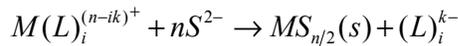
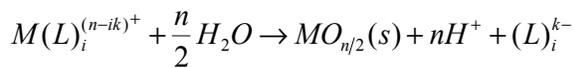
3) Displacement of ligands:



4) Deprotonation to form oxides



5) Total Reactions:



The complexant is optional and is usually chosen based on the affinity of its ligands toward the metal. This makes step 3 the rate-determining step, adding a degree of control for thin film thickness. The hydrolytic process of reactions 4 and 5 can be accelerated by heating the solution, which induces deprotonation of the hydrated metal species (reaction (4)). This eliminates the need to use a base in reactions 4 and 5. For the oxide systems to be evaluated in this proposal, hydrolysis can occur even in acidic solutions when the metal cation is easily hydrolysable, as in the case of some of the metals to be evaluated here (Al^{3+} , Zn^{2+} , In^{3+}). In contrast, CBD of non-oxides requires the addition of basic solutions. In all CBD processes, whether oxide or non-oxide, a solvated metal complex reacts with a chalcogenide source to form a desired solid product. The main difference is that for oxides, the ‘‘chalcogenide source’’ is water, so that tighter control must be exerted over just two parameters (pH, T) to achieve a similar degree of control over the rate of hydrolysis and, therefore, control over the film’s microstructure and properties. For non-oxide films, the supply of chalcogenide anions can be controlled, in addition to pH and temperature, with the concentration of the chalcogenide source.

Since the early developments of TFT’s, metal chalcogenides such as Cu_2S , CdS and $CdSe$ have been applied as the semiconductor layer in MOS devices [4, 5]. In fact, the first MOS patent included Cu_2S as the semiconductor active layer [6]. Since CBD is allow temperature process it makes it appealing for application in flexible electronics

because the use of flexible plastic substrates adds a processing constraint: processing temperature. Maximum processing temperature must be less than $150^\circ C$, well below the temperature used for traditional inorganic materials deposition. While traditional solutions to the above constraint exist, most cannot meet all of the criteria, not to mention realistic processing cost. CBD is among the most inexpensive, simple and convenient deposition technique to obtain inorganic thin films on large area substrates. An additional advantage of CBD is that the highest processing temperature is $100^\circ C$, corresponding to the water boiling temperature, which is normally the main component of the reactive solution. Originally used for sulfide and selenide thin films, this technique has also been mostly used for materials for photovoltaic applications [7-13].

2 Problem Formulation

There are a lot of cadmium complexes as Cd-Sulfodichlorohydroxydimethylfuchsondicarboxylate, Cd-Glyoxal-bis-(2-hydroxyanil), $[CdCl_2(SC(N(CH_3)_2)SS(N(CH_3)_2)CS)_2]$, etc.

More and more complexes with cadmium can be synthesized for instance, Tetramethylthiuram monosulfide and tetramethylthiuram disulfide, are compounds that are extensively used in the biomedical field. The following thiuram derivatives; tetramethylthiuram disulphide (TMTD), tetraethylthiuram disulphide (TETD), tetramethylthiuram monosulphide (TMTM) and dipentamethylenethiuram disulphide (PTD) are sulphur containing compounds which speed up vulcanization, i.e. they are ‘‘accelerators’’. Thiram (tetramethylthiuram disulphide) is used as a fungicide to protect seeds, fruit, vegetables, ornamentals, turf crops, and herbs from a variety of fungal diseases. In a biological study it was observed that thiram reacts with copper and lead metals to enhance the uptake of these metals by the roots of the plants while titanium and vanadium tend to reduce the thiram as a ligand to dithiocarbamates. Complexes of thiodicarbonyl diamide silver and mercury derivatives ($C_{12}H_{24}Ag_2C_{12}N_4S_6$, $C_6H_{12}AgN_3O_3S_3$, $C_{42}H_{42}AgN_2P_2S_3NO_3$ and $C_6H_{12}Hg_{12}N_2S_3$) are prepared from the tetramethylthiuram monosulphide ligand, while the ligand tetramethylthiuram disulphide formed thioperoxydicarbonyl diamide complexes of zirconium and dysprosium. The use of these ligands as complexes with IIB metals, to form precursors for II-VI semiconductor nanoparticles has attracted some interest, due to the presence of sulphur atoms. The

thiuram disulphide unit consists of four sulfides and hence the binding to the metal centre upon formation of complexes would be through the sulfides. The stability of the complexes is enhanced by the chelating ability of the ligand to the metal ions. This resulted in an interest in tetramethylthiuram disulphide complexes of cadmium as potential single source precursors for the synthesis of CdS nanoparticles.

Metal xanthate complexes and their reaction products with a variety of Lewis bases have been extensively studied. The soluble alkali metal xanthates are widely used in extraction and separation of Hg, Ag, Cd, etc. Sodium and potassium ethyl xanthate have antidotal effects in acute mercurial poisoning. Transition metal xanthate complexes have been investigated for nonlinear optical applications, and Cd xanthate was demonstrated to have nonlinear optical properties and generated a very strong 2nd harmonic signal. The reaction products of Cd xanthate with Lewis bases have been much less extensively studied than other similar compounds. The synthesis and X-ray crystal structure of $[\text{Cd}(\text{C}_5\text{H}_5\text{N})_2(\text{S}_2\text{CO}-n-\text{C}_4\text{H}_9)_2]$. Like most bidentate dithiolate complexes, the reaction of $[\text{Cd}(\text{S}_2\text{CO}-n-\text{C}_4\text{H}_9)_2]_n$ with pyridine destroyed the original polymeric structure to yield the corresponding bis-(Lewis base) adduct.

So we considered the idea to synthesize CdS thin films by CBD method using other compounds as complexing agents to the Cd, this in order to get a better control in its growth and high purity for applications to the nanoscaled optoelectronic devices. As a global result, we present two techniques to growing thin films using two different complexing agents: acetylacetone and glycine.

If we find a wide variety of complexing agents, then we increase the possibilities or ways to control the deposition rate of our films.

2.1 Experimental Method to produce CdS Thin Films using Acetylacetone

The acetylacetone was used as a ligand in the CBD in order to obtain CdS thin films, the nature of this agent is bidentate ligand and is convenient for the coupling with Cd ions [14]. The reaction conditions were determined experimentally.

The CdS thin films were deposited on glass slides substrates by means of the chemical bath technique. The reactive substances in the solution were: 2 ml of Cadmium Nitrate Tetrahydrated (0.1 M), 2 drops of 99% acetylacetone, 6 drops of buffer pH 11 ($\text{NH}_4\text{OH}/\text{NH}_4\text{Cl}$), 2 ml of Thiourea (1M) and two

amounts of 10 ml of waters, after the acetylacetone and before the thiourea.

The films were deposited in the substrate into the solution between 65 to 75°C, they were kept in the solution for 1 h. The obtained CdS films were flat homogeneous, green-yellowish, transparent and with very good adherence to the substrate. The gap energy of the films was 2.37 eV, for 10 minutes of reaction. Their thickness was approximately 100 nm, and they had a hexagonal crystalline structure.

The X-ray diffraction measurements were performed using a Rigaku Ultima III-XRD. Optical absorption spectra of the bilayers were recorded by an Ocean Optics USB4000-UV-VIS spectrometer. Atomic Force Microscopy AFM measurements were done in a DM09 Veeco Atomic Force Microscope.

2.2 Experimental Method to produce CdS Thin Films using Glycine.

Glycine is the smallest of the 20 amino acids commonly found in proteins. It has been used as complexing agent for the electrochemical deposition of Fe, Ni and Cr thin films on stainless steel and copper substrates [15].

The CdS films were deposited on glass slide substrates in a 100 ml beaker containing the reaction solution prepared by the subsequent addition of 31 ml of deionized water, 4 ml of 0.1M cadmium nitrate tetrahydrated ($\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$), 5 ml of 0.5 M glycine ($\text{NH}_2\text{CH}_2\text{COOH}$), 2 ml of pH 11 buffer, 5 ml of 1M thiourea ($(\text{NH}_2)_2\text{CS}$) and deionized water to complete 60 ml. The mixture was initially stirred to homogenize and then its temperature was set at 80°C in a thermal water bath. The substrates were immersed in the solution at this temperature and removed after 18 min when the reaction finished, according to its appearance. There was not stirring during the deposition process. The deposited CdS films were yellowish, homogeneous, specularly reflecting with very good adhesion to the substrate. The thickness of the films was about 60 nm as determined from depth profiles performed with Veeco Dektak 8 profilometer. The crystalline structure of the samples was analyzed by X-ray diffraction (XRD) measurements with a Rigaku Ultima III diffractometer. The absorption spectra of the films were measured in a Perkin Elmer Lambda 19 spectrophotometer in the 350–800 nm wavelength range. The morphology and roughness of the surface films were investigated by atomic force microscopy (AFM) using a JSPM-4210 scanning probe microscope (JEOL Ltd).

3 Problem Solution

Firstly were realized experimental essays in order to determine the reaction conditions to grow CdS thin films for both processes. As is usual in the Chemical Bath Deposition method, to work with in excess concentrations, then at begin it is chosen a handle and significant amount of the complexing agent or ligand compound and directly mixed with the cadmium source. After that were added the typical compounds to achieve the CdS thin films formation. Now we present here the Results and Discussion of these two processes in a comparative way [16]. We base our search for the reaction conditions in the general mechanism reaction presented in the introduction of this work.

In Fig. 1 it is shown the optical absorption spectra of these CdS thin films, which were grown using two different complexing agents, the gray line correspond the behavior for acetylacetonate and the black line for glycine, respectively. As can be observed a low level absorption was presented between 550 and 750 nm for acetylacetonate and for glycine its low absorbance range is from 505 to 750 nm, this is important for instance for the thin films solar cells purposes, where the CdS is normally used as a window material and it is required to have high transmission in the visible range, so that much more visible light penetrate into the active region.

For the glycine process, after 18 minutes of deposition the films has reached enough thickness and the reaction finished, also from the Fig. 1 it is possible to conclude that this kind of films have an average transmittance of slightly major than 80 % within the optical region. While for the acetylacetonate process, after 10 minutes of deposition the films have reached enough thickness with an average transmittance of the order of 60 % within the optical region, in this case the reaction continue until around 40 minutes.

The Fig. 2 shows the $(\alpha h\nu)^2$ vs $h\nu$ plots which is typical for CdS thin films deposited by the CBD method or any other method. Since the absorption coefficient, α , for the allowed direct interband transition in a crystal is proportional to $(h\nu)^{-1}(h\nu - E_g)^{1/2}$, where $h\nu$ is the photon energy and E_g is the value of the energy bandgap, we can estimate E_g by plotting $(\alpha h\nu)^2$ as a function of $(h\nu - E_g)$. The energy bandgaps in these cases were $E_g = 2.53$ eV using glycine as complexing agent, and $E_g = 2.37$ eV using acetylacetonate as complexing agent. The CdS thin films can be considered good for use as a visible transmitting thin film, since the range of energies for a visible transmitting film is from 1.5eV to 3.0eV [17].

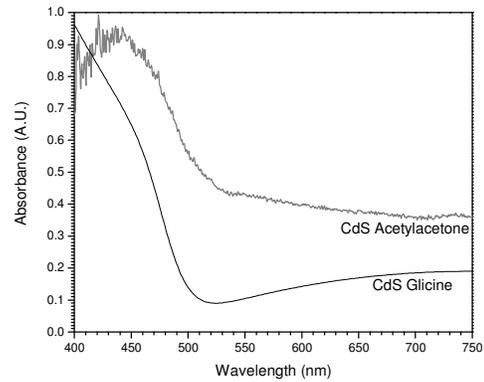


Fig.1. Optical absorption spectra of the CdS thin films using acetylacetonate and glycine as complexing agent respectively. The gray line corresponds to acetylacetonate process, and the black line for glycine.

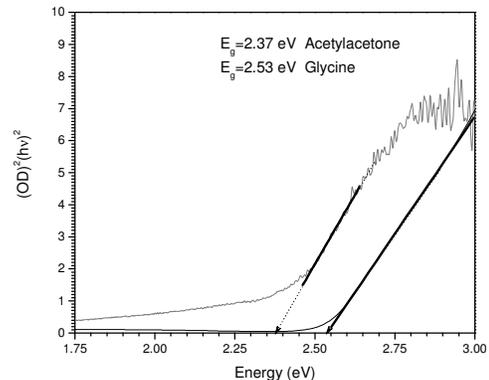


Fig. 2. $(OD)^2(h\nu)^2$ versus Energy, like utility to calculate Energy bandgap of the CdS thin films using acetylacetonate and glycine as complexing agents, respectively. The gray line shows the behavior for acetylacetonate and the dots for glycine.

The Fig. 3 shows the XRD patterns for the CdS thin films obtained by using acetylacetonate and glycine as complexing agent in the CBD process. A hexagonal polycrystalline structure concerning to the X-ray analysis results for both processes [18]. From those diffractograms were calculated their grain sizes by the model of Debye-Scherrer [19, 20], resulting 3 nm for the acetylacetonate process and 30 nm for the glycine process.

The pattern corresponding to the glycine process displays an intense peak at about 26.7° and weaker diffraction signals at about 28.6 , 48.3 and 52.3° ,

which can be assigned to the reflections by (002), (101), (103) and (112) planes of the CdS hexagonal crystalline phase. The relative large intensity of the (002) diffraction peak clearly evidences the preferred crystalline orientation of the CdS crystallites along the [002] direction. The lattice constant, c , calculated from this pattern is 6.677 Å, which is shorter than 6.713 Å, the lattice constant of bulk CdS. The percentage of variation of the value of c for the CdS films, related to that of the bulk, is -0.54 %. Because the (002) crystalline orientation of the CdS film, the [002] direction is perpendicular to the substrate. Since c is measured along the c -axis in the [002] direction, the shrinkage of this lattice constant can be related with tensile stress along the film-substrate interface, as has been observed in other chemically deposited CdS films [21].

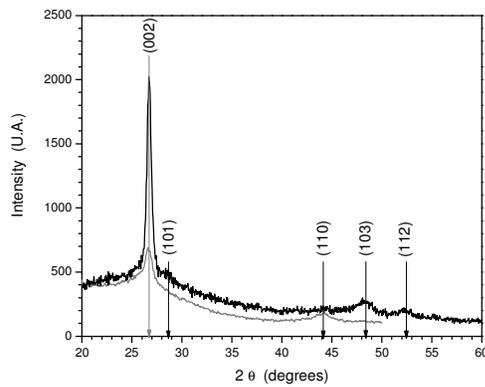


Fig. 3. X-Ray diffraction patterns of the CdS thin films using acetylacetone and glycine as complexing agent respectively. The gray line corresponds to acetylacetone and the black line for glycine.

By another hand, the surface morphology that conforms the thin film growth using acetylacetone as complexing agent was analyzed by the Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM), as it is illustrated in Fig. 4 and Fig. 5, respectively. The image obtained by SEM can be interpreted as changes in the flatness of the surface film, showing a global growing and clusters formation of nanocrystals, while the image obtained by AFM shows some regular surface morphology which is representative across the overall sample extension.

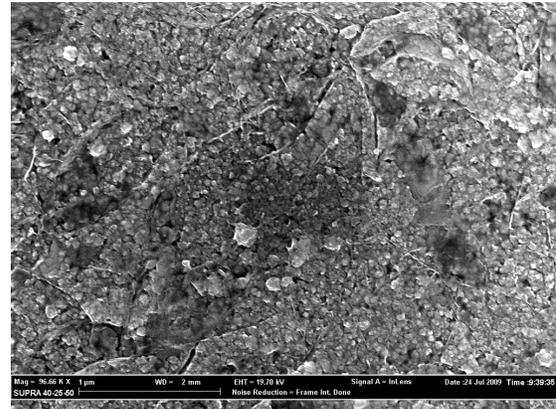


Fig. 4. SEM image of CdS thin film growth with acetylacetone as complexing agent.

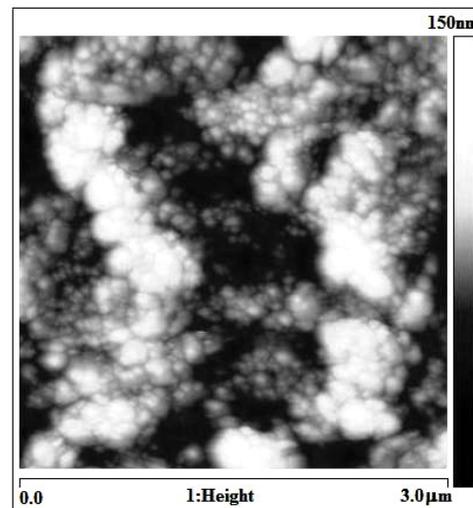


Fig. 5. AFM image of CdS thin film growth with acetylacetone as complexing agent.

In Fig. 6 are shown AFM images of the CdS film surface grown using glycine as complexing agent, (a) A two-dimensional top view and (b) A three-dimensional in perspective view. The smooth surface of the films exhibits a well-defined granular structure with nanometric-sized grains. The rms average roughness of the film measured in the displayed area is 2.3 nm [22]. Due the low roughness of this thin films the optical transmission increase in the most part of the visible region or its absorbance decrease

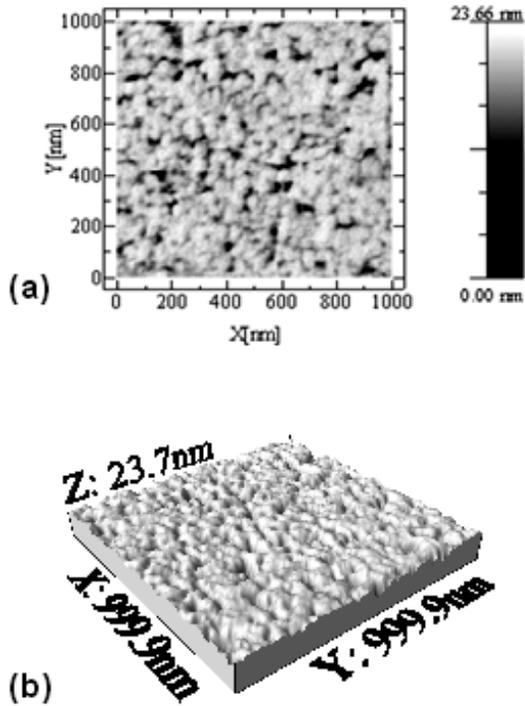
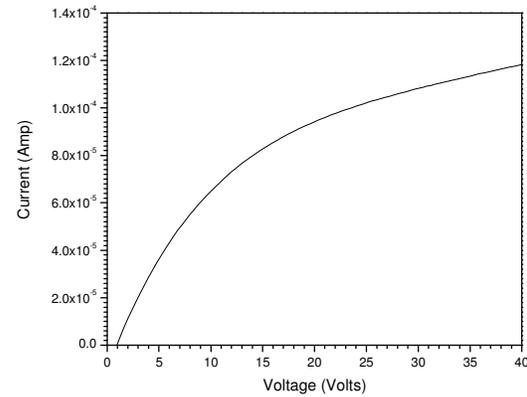
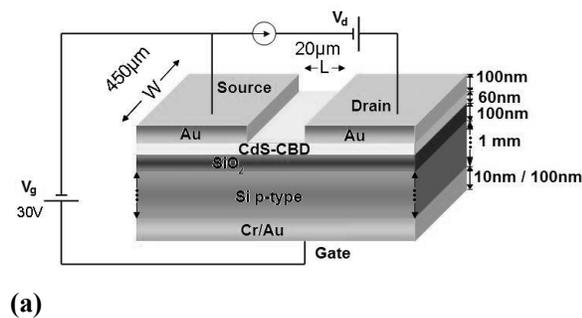


Fig. 6. AFM images of CdS thin films growth with glycine as complexing agent. (a) Top view AFM image of a representative area of the CdS thin film, showing its nanometric grain sizes, and (b) Perspective view of AFM image of the same representative area of the CdS thin film, showing its nanometric grains sizes.

Finally an appliance was suggested where it was used the process of CdS thin film with glycine, but growth on SiO₂/ Si p-type. This was the base to create a Thin Film Transistor as the shown one in schematic form in Fig. 7 (a). The plot in the Fig. 7 (b) shows the typical transistor behavior, here we can see the correlation between the current source-drain ($I_{sd}=I_d$) and the voltage source-drain ($V_{sd}=V_d$).



(b)

Fig. 7 Application of CdS thin film growth with glycine. (a) Schematic Thin Film Transistor, and (b) Current vs. voltage of CdS thin film growth with glycine as complexing agent.

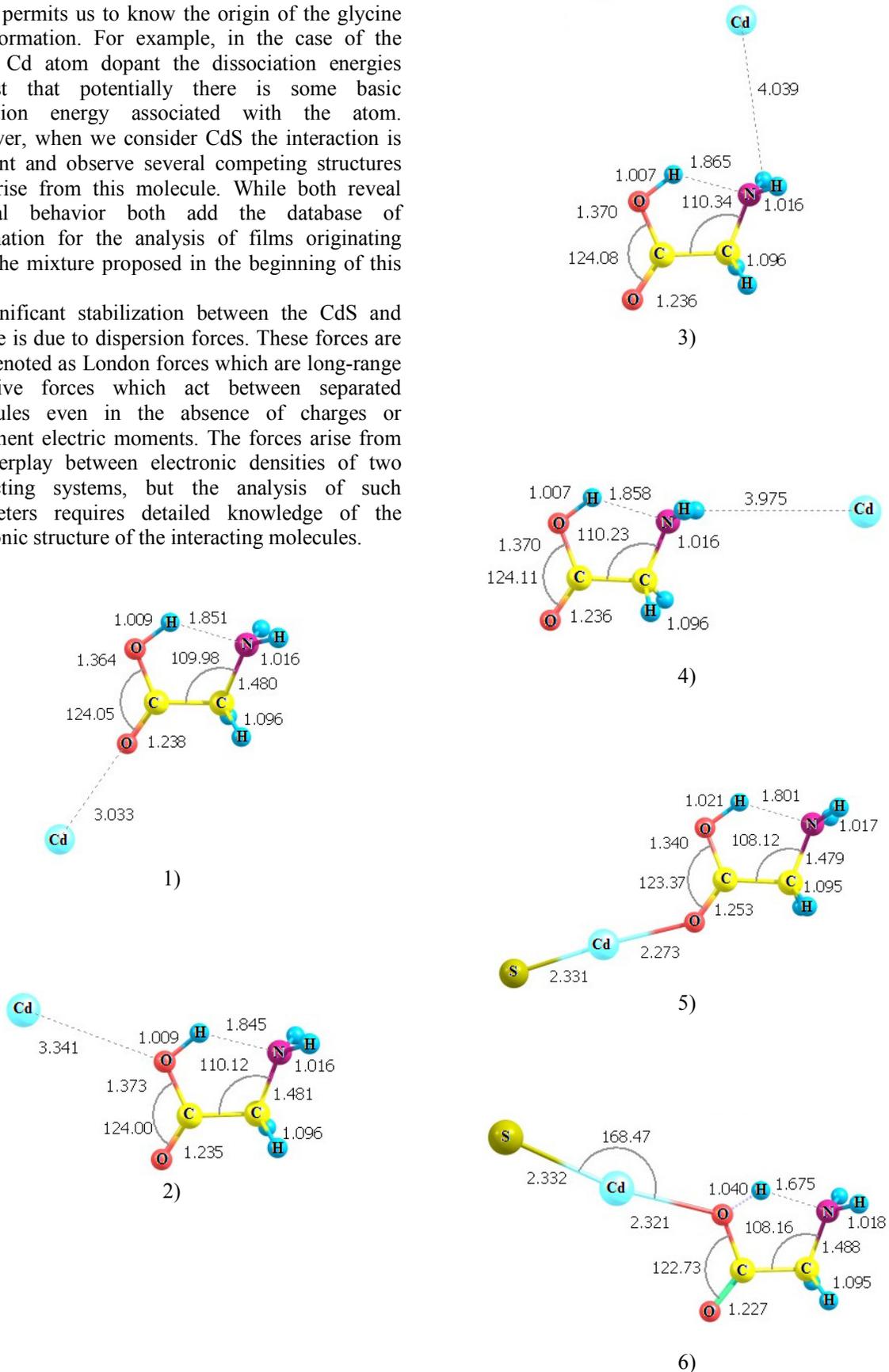
Quantum chemical computations were also performed using the GAUSSIAN03 [24] suite of programs. These computations serve as complements to the experimental work to understand the principles of the work described. Geometry optimizations will be carried out with the B3LYP general-gradient potential approximation in conjunction with the LanL2DZ pseudopotential basis set. Furthermore MP2 single point energies will be used to assess a higher accurate energy comparison for the principles observed. Hessian matrix elements must be evaluated to determine whether the structures computed are in fact minimum energy structures along the potential energy surface.

This will be used for the initial structures presented in this work. What we will call training set is a set of configurations that correspond to local minimum structures along the potential energy surface. These will be used to verify the exact structures to consider in further modeling. Several minimum energy structures will be considered and investigated in this project.

In this part of the discussion we have attempted to form an understanding for the underlying principles being described (see Figure 8 and Table 1). We will consider two examples in this work to analyze the interactions between Cd, CdS and glycine. It is imperative to know the basic interactions between multiple systems in order for us to fully appreciate the chemistry behind the observed phenomena. To distinguish these basic

forces permits us to know the origin of the glycine film formation. For example, in the case of the single Cd atom dopant the dissociation energies suggest that potentially there is some basic formation energy associated with the atom. However, when we consider CdS the interaction is covalent and observe several competing structures that arise from this molecule. While both reveal internal behavior both add the database of information for the analysis of films originating from the mixture proposed in the beginning of this work.

Significant stabilization between the CdS and glycine is due to dispersion forces. These forces are also denoted as London forces which are long-range attractive forces which act between separated molecules even in the absence of charges or permanent electric moments. The forces arise from an interplay between electronic densities of two interacting systems, but the analysis of such parameters requires detailed knowledge of the electronic structure of the interacting molecules.



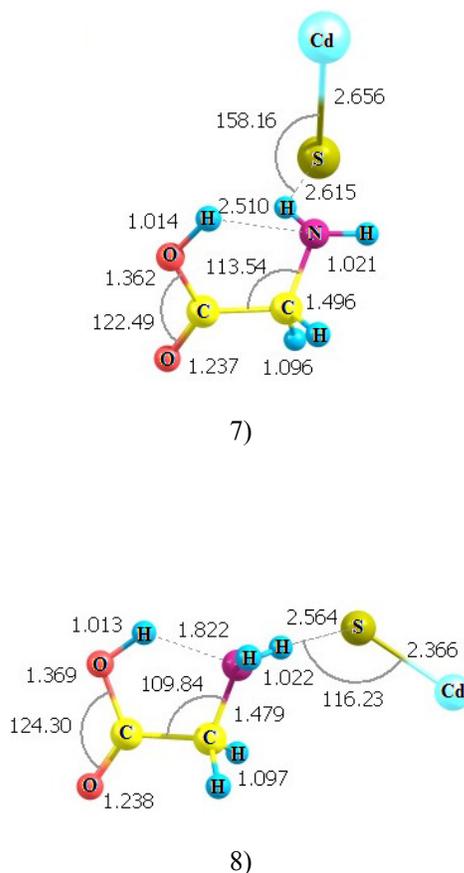


Fig. 8. Selected geometrical parameters for eight species described whereby bond lengths are in angstroms and bond angles in degrees.

System		$\Delta E(^{\circ}K)$	GAP
Cd-Gly	1	-1.937	8.537
	2	-1.635	8.685
	3	-0.579	8.943
	4	-0.478	8.959
CdS-Gly	5	0.000	8.419
	6	0.509	8.241
	7	18.799	7.855
	8	23.223	7.057

Table 1. MP2 Dissociation energies with B3LYP zero-point corrections are denoted by ΔE ($^{\circ}K$) in kcal/mol, HOMO/LUMO band gaps in eV are listed under GAP in the table for the Cd-Gly and CdS-Gly systems.

From the first structure in the figure we can see from structure 1 that the Cd atom forms an interaction with the oxygen atom of the carboxyl group of the glycine molecule with an interaction of

about -1.94 kcal/mol with a band gap of 8.54 kcal/mol. The intermolecular separation is about 3.0 angstroms with internal hydrogen bonding strong in the species. As we move to species 2 we can see that the dissociation energies decrease to around -1.64 kcal/mol as a result of attachment to the OH group of the COOH terminus of the glycine molecule. The band gap is slightly increased as a result of this interaction. There is a general trend that as dissociation energies decrease the molecules become slightly more organic in chemical behavior from the interaction which is visible from the band gap trends. For the next structure the dissociation energy decreases to about -0.58 kcal/mol with a decrease in the bonding pattern. As we can see the distances are slightly larger at around 4 angstroms with an interaction to the NH_2 group.

The final structure with the Cd interaction is 4 with a an interaction energy of -0.48 and 8.96 kcal/mol for the bandgap. The distance is similar to 3 but the OH group in this former structure had a favorable effect in the stabilization of the atom. In structure 4 the dipoles align which leads to partial stabilization but it is not as large as in 3 due to the OH and NH_2 angles of interaction. In all structures the general energy of interaction is indeed favorable. For the next series of structures since we observe that the interaction is indeed covalent we have compared four competing structures in the energy order of 5, 6, 7 and 8 with descending stabilities. As we can see the CdS molecule aligns with oxygen atom of the COOH group to yield a low energy species. The band gap for this species is the lowest of all the structures which means it shall be less reactive with respect to outside perturbation. The next structure 6 is around 0.51 kcal/mol higher in energy as a result of its binding to the OH group, which due to torsional strain has a slightly higher energy, for the interactions with 7 and 8 we get interaction energy of about 18.8 and 23.22 kcal/mol, respectively. The primary destabilization of structure 8 is due to the torsional strain from the CdS interaction to the nitrogen atom which is minimized in 7 from the offset of such effects by dipole modifications. The band gaps for these species are significantly lower than the former molecules revealing a more reactive state.

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4 Conclusion

From this research we developed two new processes to synthesize CdS thin films of very good quality, comparable to the obtained with other

techniques. So, we can conclude that the Chemical Bath Deposition technique has a high potential to contribute into built appliances in the actual nanometric scale order, such as the TFT. Here we purpose the use of Acetylacetone and Glycine by separate, both as complexing agents for the cadmium ion. We can recall that the respective grain sizes were 3 and 30 nm, their respective energy bandgaps were 2.37 and 2.53 eV, and their almost constant transmittance levels were 80% and 60%. For both processes the polycrystalline structure corresponds to a hexagonal phase. Also we are starting to consider appropriate to use a theoretical computing method, as tools to approach the explanation of the complexation chemical behave, for this reason it is presented some preliminary structures and Energy calculations.

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