

## Research Article

# Synthesis and Characterization of Hexadecylamine Capped ZnS, CdS, and HgS Nanoparticles Using Heteroleptic Single Molecular Precursors

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Zn(II), Cd(II), and Hg(II) complexes of tetramethyl thiuram disulfides and 1-ethoxycarbonyl-1-ethylenecarbonyl-2-dithiolate were synthesized and characterized by elemental analysis, FTIR, and  $^1\text{H}$ - and  $^{13}\text{C}$ -NMR spectroscopy. The complexes were thermolysed in hexadecylamine as single molecule precursors to prepare HDA capped ZnS, CdS, and HgS nanoparticles. The optical and structural properties of the nanoparticles are reported. ZnS nanoparticles existed in the hexagonal phase with particle sizes of 8–15 nm; the CdS nanoparticles in the cubic phase have particle sizes in the range 4–7 nm and the HgS nanoparticles indexed to face-centered cubic phase have an average particle size of 7–12 nm.

## 1. Introduction

Dithiolate ligands are unsaturated bidentate ligands and react with transition metals to form dithiolene complexes [1, 2]. Metal complexes of dithiolene have excellent electronic functions as a result of their intermolecular charge transfer [3]. The ligand, 1-ethoxycarbonyl-1-cyanoethylene-2,2-dithiolate ( $\text{ecda}^{-2}$ ), is the analogue of 1,2-dithio analogue, maleonitriledithiolate ( $\text{mnt}^{-2}$ ), which is rare with average sulfur-sulfur bite angle of 3.074(2) Å for 1,1dithiolate [4]. More frequently reported structures consist of symmetrically 1,1dithiolate substituted ligand of the type  $\text{SCC}(\text{X})_2^{-2}$  but the high nuclearity metal cluster possessing asymmetrically substituted 1,1dithiolates,  $\text{S}_2\text{CC}(\text{X})(\text{Y})^{-2}$ , has not been satisfactorily characterized due to the difficulty of getting a good quality crystal suitable for X-ray diffractions [5]. This eventually militates against detailed characterization of the isolated complexes. They show exciting coordination properties by virtue of chelating and bridging behaviours, which have been demonstrated in its binary and heterobimetallic complexes [6]. It exhibits strong  $\pi$ -delocalization and its complexes

display fascinating structural and solid-state properties. The interest in dithiolate ligands is a result of their stabilization of transition metal ions in usual oxidation states, facile redox behaviour, and stabilization of square planar geometry around the transition metal ions, interesting spectral and magnetic properties in catalysis. The dithiolate chemistry of the group 12 elements is constrained to the +2 oxidation state. The ease and stability of the complexation reaction is due to the fact that group 12 metals act as strong Lewis acids and hence readily reacts with electron-rich sulfur containing ligands in the principle of hard-soft [Lewis] acid-base [7].

In this work, we have used the Zn(II), Cd(II), and Hg(II) complexes of mixed tetramethyl thiuram disulfides and 1-ethoxycarbonyl-1-ethylene-2-dithiolate as single molecule precursors for the synthesis of hexadecylamine capped metal sulfides nanoparticles [8–11]. HDA was employed as the capping agent because it can decrease growth rate and particle size and improve quantum efficiency of photoluminescence for the nanoparticles through passivation of the surface [12–14].

## 2. Experimental

**2.1. Materials and Physical Measurements.** Commercial reagents used were purchased from Sigma Aldrich and used without further purification. Potassium salt of 1-ethoxycarbonyl-1-cyanoethylene-2,2-dithiolate was prepared according to literature procedure [15]. The FTIR determination of the metal complexes was done using KBr discs on a Perkin Elmer Paragon 2000 FTIR spectrophotometer in the range of 4000–370  $\text{cm}^{-1}$ . The  $^1\text{H}$  and  $^{13}\text{C}$  NMR determination of the metal complexes was done using 400 MHz and 101 MHz Bruker NMR spectrophotometers, respectively. UV-Visible spectra of the metal complexes and the metal sulfide nanoparticles were recorded using Perkin Elmer Lambda 25 spectrophotometer in chloroform from 800 to 200  $\text{cm}^{-1}$ . Samples for scanning electron microscope (SEM) were prepared as follows: the respective nanoparticles were mounted on stub using carbon double-sided tape. They were coated with Au/Pd using the Eiko IB. 3 Ion Coater. They were observed using JOEL JSM-6390 LVSEM at a rating voltage of 15–20 kV at different magnifications as indicated on the SEM images. The photoluminescence measurement of the nanoparticles was done using a Perkin Elmer LS 45 Fluorimeter while powder X-ray diffraction patterns of the nanoparticles were recorded on a Bruker D8 Advanced, equipped with a proportional counter using Cu  $K\alpha$  radiation ( $\lambda = 1.5405 \text{ \AA}$ , nickel filter).

**2.2. Preparation of Complexes.** About 40 mL methanol solution containing 0.005 mol of the metal salts ( $\text{ZnCl}_2$ , 0.681 g;  $\text{CdCl}_2$ , 0.917 g;  $\text{HgCl}_2$ , 1.357 g) was added to tetramethyl thiuram disulfide (0.005 mol, 1.2012 g) in 30 mL methanol. The mixture was refluxed for 4 h followed by the addition of  $\text{K}_2\text{ecda}$  (0.005 mol, 1.201 g) in 30 mL methanol. The initial colourless solution turned slightly yellow and was refluxed further for 8 h, filtered, and redissolved in acetone. The product was washed with methanol and diethyl ether and dried at ambient temperature over  $\text{CaCl}_2$ .

**[Zn(tmtd)ecda].** Yield: 1.59 g (75.1%), m. p. 315–317°C.  $^1\text{H}$ -NMR (DMSO)  $\delta = 1.05$  (t, 3H,  $-\text{NH}-\text{CH}_3$ ), 4.02 (q, 2H,  $-\text{OCH}_2$ ).  $^{13}\text{C}$ -NMR (DMSO)  $\delta = 203.69$  (C=S), 168.68 (C=O), 122.94 (C $\equiv$ N), 59.29 ( $-\text{OCH}_2$ ). Selected IR,  $\nu$  ( $\text{cm}^{-1}$ ): 2245 (C $\equiv$ N), 1455 (C=S<sub>2</sub>), 382 (M-S), 1732 (C=O). Anal. calc. for  $\text{C}_{12}\text{H}_{17}\text{N}_3\text{O}_2\text{S}_6\text{Zn}$  (490.89): C, 29.44; H, 3.55; N, 9.01; S, 39.97. Found: C, 29.33; H, 3.49; N, 8.56; S, 39.08.

**[Cd(tmtd)ecda].** Yield: 1.58 g (72.8%), m. p. 337–339°C.  $^1\text{H}$ -NMR (DMSO)  $\delta = 1.27$  (t, 3H,  $-\text{OCH}_2\text{CH}_3$ ), 4.09 (q, 2H,  $-\text{OCH}_2$ ).  $^{13}\text{C}$ -NMR (DMSO)  $\delta = 208$  (C=S), 164 (C=O), 121.37 (C $\equiv$ N), 90.6 (C=C), 14.6 ( $-\text{CH}_3$ ), 58.5 ( $-\text{OCH}_2$ ). Selected IR,  $\nu$  ( $\text{cm}^{-1}$ ): 2233 (C $\equiv$ N), 1512 (C=S<sub>2</sub>), 384 (M-S), 1600 (C=O). Anal. calc. for  $\text{C}_{12}\text{H}_{17}\text{N}_3\text{O}_2\text{S}_6\text{Cd}$  (540.87): C, 26.35; H, 3.29; N, 8.13; S, 35.40. Found: C, 26.62; 3.17; N, 7.77; S, 35.47.

**[Hg(tmtd)ecda].** Yield: 1.9 g (91.6%), m. p. 309–311°C.  $^1\text{H}$ -NMR (DMSO)  $\delta = 1.23$  (t, 3H- $\text{CH}_3$ ), 4.35 (q, 2H,  $-\text{OCH}_2$ ).  $^{13}\text{C}$ -NMR (DMSO)  $\delta = \text{NA}$ . Selected IR,  $\nu$  ( $\text{cm}^{-1}$ ):

2244 (C $\equiv$ N), 1492 (C=S<sub>2</sub>), 449 (M-S), 1738 (C=O). Anal. calc. for  $\text{C}_{12}\text{H}_{17}\text{N}_3\text{O}_2\text{S}_6\text{Hg}$  (628.94): C, 22.95; H, 2.76; N, 6.64; S, 30.44. Found: C, 22.90; H, 2.72; N, 6.68; S, 30.50.

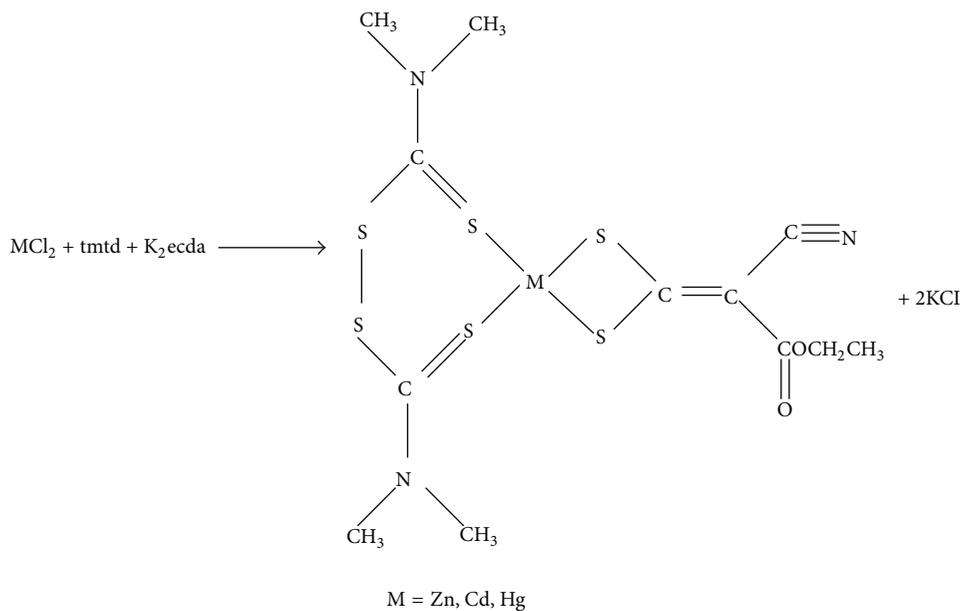
**2.3. Synthesis of HDA Capped Metal Sulfides Nanoparticles.** In a typical experiment, about 0.4 g of the precursor complex was dissolved in 10 mL of trioctylphosphine oxide (TOP). The obtained solution was then injected into 4 g of hot HDA at a temperature of 120°C. A subsequent decrease in temperature of 20–30°C was observed and the solution was allowed to stabilize at 120°C. Further heating was done for 60 min and the reaction was terminated. The solution was then allowed to cool to about 70°C, and about 100 mL methanol was added to remove the excess HDA. The flocculent precipitate formed was centrifuged and the supernatant was decanted, after which the isolated solid was dispersed in toluene. The above centrifugation and isolation procedure was repeated three times for the purification of the prepared metal sulfide nanoparticles.

## 3. Results and Discussion

Reaction of one molar equivalent of tmtd and ecda with one mole of  $\text{ZnCl}_2$ ,  $\text{CdCl}_2$ , and  $\text{HgCl}_2$  at room temperature gave the respective metal complexes. All compounds are air-stable and insoluble in most solvents but some are soluble in polar coordinating solvents such as DMSO and DMF. The formation of the compounds is shown in Scheme 1. The analytical and spectroscopic data are consistent with the proposed formulation for the complexes.

**3.1. Spectroscopic Studies of the Metal Complexes.** The IR of the metal complexes synthesized from ecda and tetramethyl thiuram disulfide shows characteristic changes in the functional group when compared with the spectra of the ligands (Figure 1). The ecda has four important absorption frequencies which are 2161  $\text{cm}^{-1}$  due to  $\nu(\text{C}\equiv\text{N})$ ; 1681  $\text{cm}^{-1}$  due to  $\nu(\text{C}=\text{O})$ ; 1360  $\text{cm}^{-1}$  due  $\nu(\text{C}=\text{CS}_2)$ ; and 1160  $\text{cm}^{-1}$  due to  $\nu(\text{C}-\text{S})$  (Table 1) [16]. These stretching vibrations were observed in the metal complexes at 2245, 1732, 1438, and 1142  $\text{cm}^{-1}$  for  $[\text{Zn}(\text{tmtd})\text{ecda}]$ ; 2233, 1600, 1512, and 1147  $\text{cm}^{-1}$  for  $[\text{Cd}(\text{tmtd})\text{ecda}]$ ; and 2244, 1738, 1492, and 1191  $\text{cm}^{-1}$  for  $[\text{Hg}(\text{tmtd})\text{ecda}]$ , respectively. There are four vital bands in thiuram disulfide metal complexes and they appear in the region of 800–1510  $\text{cm}^{-1}$ : a strong thioureide C-N band, the band at 1200  $\text{cm}^{-1}$  which is due to C-N vibration of the alkyl group, the band responsible for the C=S stretching mode around 973  $\text{cm}^{-1}$ , and the band around 822  $\text{cm}^{-1}$  that can be ascribe to the stretching C-S mode.

All these band are observable in  $[\text{Zn}(\text{tmtd})(\text{ecda})]$ ,  $[\text{Cd}(\text{tmtd})(\text{ecda})]$ , and  $[\text{Hg}(\text{tmtd})(\text{ecda})]$  at vibration frequencies of 1520, 1512, and 1530  $\text{cm}^{-1}$ ; 1200, 1209, and 1201  $\text{cm}^{-1}$ ; 973, 95, and 965  $\text{cm}^{-1}$ ; 822, 820, and 830  $\text{cm}^{-1}$ , respectively. The absorption bands assigned to M-S in these complexes are observed at 384, 449, and 416  $\text{cm}^{-1}$  for  $[\text{Zn}(\text{tmtd})(\text{ecda})]$ ,  $[\text{Cd}(\text{tmtd})(\text{ecda})]$ , and  $[\text{Hg}(\text{tmtd})(\text{ecda})]$ , respectively.



SCHEME 1: Synthesis of the metal complexes.

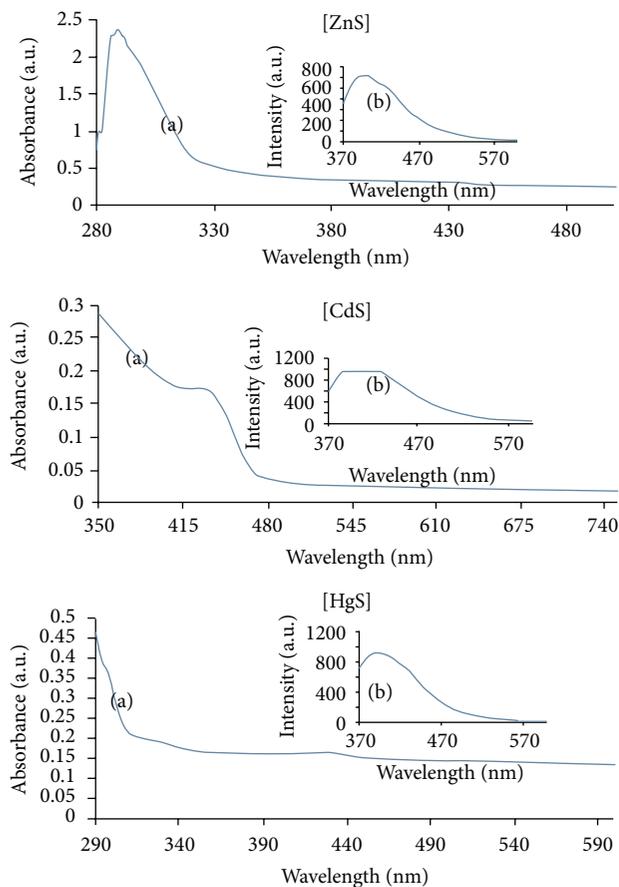


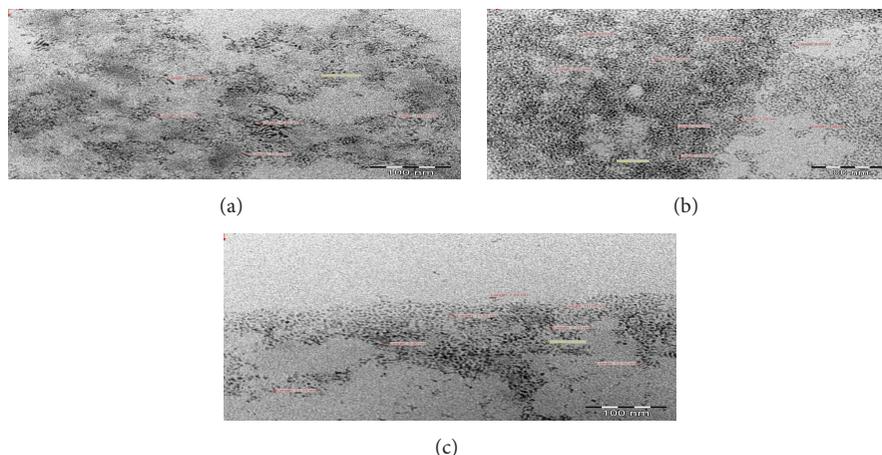
FIGURE 1: Optical absorption (a) and corresponding photoluminescence spectra (b) of HDA capped ZnS, CdS, and HgS nanoparticles synthesized from the metal complexes.

The  $^1\text{H-NMR}$  of the Zn complex shows a triplet at  $\delta$  1.05 and a quartet at  $\delta$  4.02 ppm that can be assigned to  $\text{CH}_3$  and  $\text{OCH}_2$  of the  $\text{ecda}^{2-}$ . The  $^{13}\text{C-NMR}$  spectrum of  $[\text{Zn}(\text{tmtd})\text{ecda}]$  shows peaks around  $\delta$  203.69, 168.68, 122.94, 59.29, and 18.51 ppm corresponding to C-S, C=O, C $\equiv$ N, -OCH $_2$ , and CH $_3$ , respectively, for carbon of the  $\text{ecda}^{2-}$ . The  $^1\text{H-NMR}$  spectra of the Cd complex contained a triplet at  $\delta$  1.27 for the -CH $_3$  protons of the tetraethyl thiuram disulfide (tmtd) and the ester group of the ecda ligand. The protons of the -OCH $_2$  group are observed as a quartet at 1.27–4.09 ppm. The  $^1\text{NMR}$  spectra of this complex,  $[\text{Hg}(\text{tmtd})\text{ecda}]$ , show triplet peaks 1.05 and 4.35 for -CH $_3$  protons of the thiuram disulfide and  $\text{ecda}^{2-}$ . The other peaks at 2.5 and 3.4 are due to solvent peaks of DMSO and water. The complex is partially soluble in DMSO and hence the spectrum was very weak. The complex was run for solid state on 600 MHz NMR but the complex could not be used for calibration and all effort to do it proved abortive. As a result of this, the  $^{13}\text{C}$  NMR was not reported.

**3.2. Optical Properties of Metal Sulfide Nanoparticles.** UV-Visible spectra analysis has served as a useful technique for the characterization of semiconductor nanoparticles, which exhibit quantum size effect. The photogenerated electron-hole pairs causes the quantum size effect. The UV-Visible absorption spectrum of semiconductor nanoparticles depends on their size and the absorption maximum decreases with the nanoparticle size [16]. The UV-Vis absorption spectra (Figure 1) showed that the absorption peaks of analyzed ZnS, CdS, and HgS particles are at 288, 439, and 296 nm, respectively, a relative blue-shift to the band gap of the bulk, which are at 340, 515, and 563 nm, respectively [17, 18].

TABLE I: Relevant IR spectra of  $[M(\text{tmtd})\text{ecda}]$ .

	$\nu(\text{C}\equiv\text{N})$	$\nu(\text{C}=\text{O})$	$\nu(\text{C}=\text{S}_2)$	$\nu(\text{C}-\text{S})$	$\nu(\text{C}=\text{S})$	$\nu(\text{M}-\text{S})$
$[\text{Zn}(\text{dmtu})_2\text{ecda}]$	2245	1732	1138	1142	970	382
$[\text{Cd}(\text{dmtu})_2\text{ecda}]$	2233	1600	1512	1147	957	384
$[\text{Hg}(\text{dmtu})_2\text{ecda}]$	2244	1738	1492	1170	X	449

FIGURE 2: TEM images of (a) ZnS; (b) CdS; and (c) HgS nanoparticles, prepared from their respective precursor complexes at  $120^\circ\text{C}$ .

The peaks positions can be related to the mean diameter of the particles, smaller diameter meaning lower wavelength. The results showed the effect of quantum confinement [19]. The absorption shift represents a change in band gap along with exciton features, which can be used as a measure of particle size and size distribution. The calculated gap for the ZnS, CdS, and HgS particles are  $E_g = 3.78, 2.82,$  and  $3.89$  eV, respectively.

In semiconductor nanoparticles, the position of absorption band edge can be taken as an indicative of their sizes. The approach has proven very useful since the result obtained from this method has not been affected by the presence of aggregates. The electronic coupling between particles in an aggregate is sufficiently weak so that the resultant spectrum is determined by the primary particles only [20]. Brus equation [21] was used to calculate the particle size from the absorption edge. From the characteristic absorption spectra, the size of CdS and ZnS nanoparticles was estimated to be about 5.68 and 7.88 nm in diameter. The photoluminescence spectra of the as-prepared ZnS, CdS, and HgS from the complexes of  $[\text{Zn}(\text{tmtd})\text{ecda}]$ ,  $[\text{Cd}(\text{tmtd})\text{ecda}]$ , and  $[\text{Hg}(\text{tmtd})\text{ecda}]$  were recorded at room temperature at an excitation wavelength of 370 nm and they exhibit emission peak maxima at 398, 394, and 393 nm, respectively. The CdS and HgS are blue shifted compared with their bulk materials.

**3.3. Structural Properties of Metal Sulfide Nanoparticles.** TEM images of the ZnS, CdS, and HgS nanoparticles synthesized are presented in Figure 2. It is observed that the particles of the ZnS are dot shaped and the size is rather evenly distributed. The particle sizes of the ZnS nanoparticles are in the range 8–15 nm. The CdS particles display a spherical shaped morphology with particles sizes in the range 4–7 nm while,

in the HgS sample, agglomerated oval shaped particles were observed. TEM showed that the product was composed of 40% of isolated particles and 60% of agglomerated particles. The average size of HgS nanoparticles is in the range 7–12 nm. It is hard to determine the exact dimension of HgS particles by only observing the TEM image because the extremely small particles aggregate to secondary particles [22].

The microstructure of the nanocrystals was examined by scanning electron microscopy (SEM) and the elemental compositions of the nanoparticles formed were confirmed by energy dispersive X-ray (EDX) analysis as shown in Figure 3. It can be seen that the surface of the particles appears smooth and spherical. The powder XRD patterns of as-prepared HDA capped ZnS, CdS, and HgS nanoparticles samples are shown in Figure 4. These XRD patterns of the ZnS, CdS, and HgS exhibited different structures. For ZnS, the three peaks with  $2\theta$  values of 26.78, 44.43, and 52.36 correspond to the presence of hexagonal ZnS. The indicator is the peak value 26.78 planes which correspond to (1 0 1) and is in good agreement with joint committee of powder diffraction standard (JCPDS) data belonging to hexagonal ZnS [23] and the presence of 52.36 that suggests the hexagonal phase of ZnS [24]. The broad peaks are due to finite size effects that also indicate that the particles sizes are in nanometer range.

The XRD pattern of CdS of this complex,  $[\text{Cd}(\text{tmtd})\text{ecda}]$ , is shown in Figure 4 and shows a single-phase sphalerite crystal structure. The  $2\theta$  values are 26.9, 36.16, 43.6, and 51.83 and they can be indexed to the following planes: (1 1 1), (2 0 0), (2 2 0), and (3 1 1) of the cubic phase  $\beta$ -CdS (JCPDS 80-0019). The peak broadening of XRD pattern indicates that small nanocrystals are present. The listed peaks as observed in the XRD pattern of HgS of this complex at  $2\theta$  are 26.49,

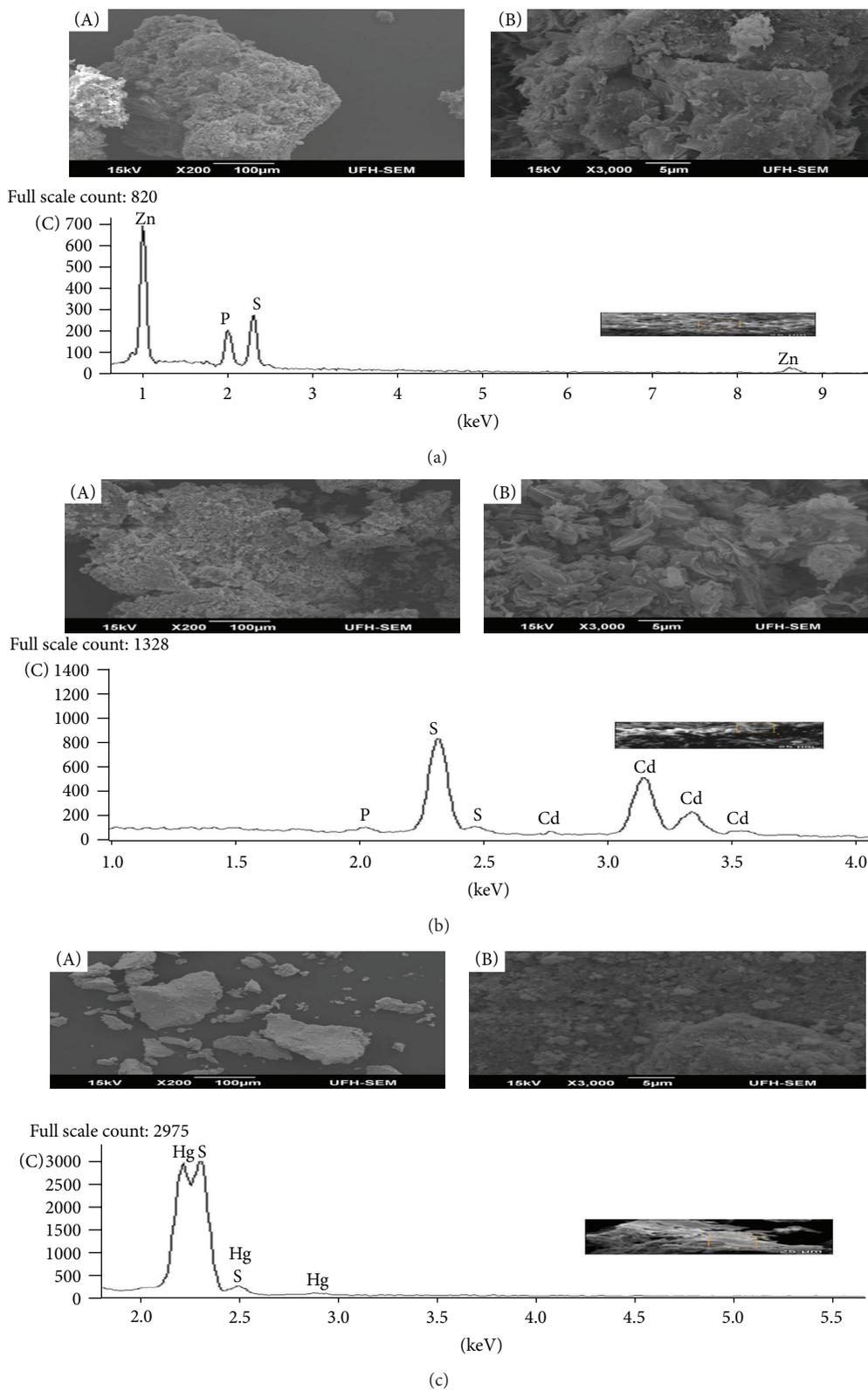


FIGURE 3: (a) SEM micrograph of ZnS nanoparticles from  $[Zn(tmd)ecda]$  complex, at  $120^{\circ}C$  at (A) low magnification, (B) high magnification, and (C) EDX spectrum of the ZnS sample. (b) SEM micrograph of CdS nanoparticles from  $[Cd(tmd)ecda]$  complex, at  $120^{\circ}C$  at (A) low magnification, (B) high magnification, and (C) EDX spectrum of the CdS sample. (c) SEM micrograph of HgS nanoparticles from  $[Hg(tmd)ecda]$  complex, at  $120^{\circ}C$  at (A) low magnification, (B) high magnification, and (C) EDX spectrum of the HgS sample.

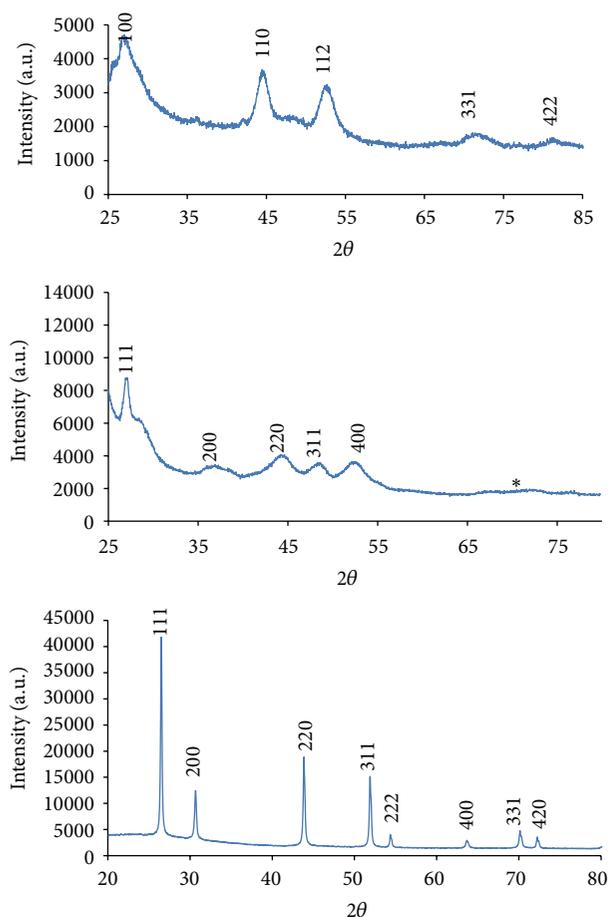


FIGURE 4: XRD patterns of ZnS, CdS, and HgS nanoparticles synthesized from the complex.

30.64, 43.85, 51.89, and 54.33. They match the peaks of known metacinnabar [25]. The peaks are intense and all orient along the planes (1 1 1), (2 0 0), (2 2 0), (3 1 1), and (4 0 0). All these peaks are to be indexed to face-centered cubic HgS and they are relatively broad which show that the particles are in nanosize dimension.

#### 4. Conclusion

Zn (II), Cd (II), and Hg (II) complexes of mixed tetra-methyl thiuram disulfides and 1-ethoxycarbonyl-1-ethylenecarbonyl-2-dithiolate were synthesized and characterized by elemental analyses, FTIR, and NMR spectroscopy. The complexes are formulated as four coordinate species and were used as single source precursors to synthesize metal sulfides nanoparticles. The sizes of the metal sulfides nanoparticles obtained from the TEM are in the range 4–15 nm in diameter. The XRD patterns confirm the hexagonal phase for ZnS; the CdS is in cubic phase while the HgS nanoparticles are indexed to face-centered cubic phase. The relatively broad peaks of their XRD patterns are due to finite size effects that are indicative of particle sizes in nanometer range.

#### Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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