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Synthesis and Characterization of Zinc Sulfide and Zinc-Iron Sulfide Nanoparticles from Zinc(II) Dithiocarbamate Complexes

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ABSTRACT

Bis(N-(pyrrol-2-ylmethyl)-N-(2-phenylethyl) dithiocarbamato-S, S') zinc(II) (1) and bis(N-methylferrocenyl-N-(2-phenylethyl) dithiocarbamato-S, S') zinc(II) (2) have been synthesized and characterized by elemental analysis and spectroscopy (IR and UV-vis). Complexes 1 and 2 have been used as precursors for the preparation of zinc sulfide and zinc-iron sulfide nanoparticles. Morphological characterization of nanoparticles was carried out using TEM.

Keywords : Zinc(II) Dithiocarbamate, Zinc Sulfide, Zinc-Iron Sulfide, Nanoparticles; Single Source Precursors

I. INTRODUCTION

A wide range of metal-dithiocarbamate complexes is known with examples finding use in applications as diverse as industry, agriculture, medicine and material science [1-3]. Metal sulfide nanoparticles have shown vital applications in many fields as an advanced material such as IR detectors [6], photocapacitors for energy conversion and storage [4], sensors [5], photonic materials [6] and advanced optoelectronic devices [7]. In recent years, transition metal dithiocarbamate complexes have received a great deal of attention because of their importance as single source precursors for the preparation of metal sulfide nanoparticles [8,9]. The N-bound organic moieties in dithiocarbamate ligands in metal complexes affect the morphology and size of the metal sulfide nanoparticles [10,11]. The photocatalytic activity of the metal sulfide nanoparticles depends on the morphology and size of the nanoparticles [12]. The single source precursor for the preparation of metal sulfide nanoparticles [13].

II. EXPERIMENTAL

2.1. Materials and techniques

All reagents and solvents were commercially available high-grade materials (Merck/ Sd fine/Sigma Aldrich) and used as received. IR spectra were recorded on a Thermo Nicolet Avatar 330 FT-IR spectrophotometer (range: 4000–400 cm^{-1}) as KBr pellets. EM images were recorded using TECNAI T2 G2 make-FEI, respectively. EDS were performed by SUPRA 55VP CARL. Photoluminescence spectra were recorded using Perkin Elmer 1555 fluorescence spectrophotometer at room temperature.

2.2. Preparation of complexes

Bis (N-(pyrrol-2-ylmethyl) – N - (2-phenylethyl) dithiocarbamato-S, S') zinc(II) (1) and bis (N-methylferrocenyl – N - (2-phenylethyl) dithiocarbamato - S, S') zinc(II) (2) were prepared by general methods reported earlier [9].

III. Preparation of zinc sulfide and zinc-iron sulfide

0.5 g of **2** was mixed in 15 ml triethylenetetra amine in a round bottom flask and then the content of the flask was refluxed for 15 minutes. The black precipitate obtained was filtered off and washed with methanol. Similar procedure was adopted for the preparation of zinc-iron sulfide from **2**.

IV. RESULTS AND DISCUSSION

4.1 Spectral studies of 1 and 2

4.1.1 IR spectral studies

IR spectra of complexes **1** and **2** show a distinct vibrational band in the region 1439–1499 cm^{-1}, which is attributed to the ν_{C-N} vibrations. The band is termediate between the stretching vibrations of the C–N single band at 1250-1350 cm^{-1} and C=N band at 1640-1690 cm^{-1}, suggesting partial double bond character and therefore, partial delocalization of π-electron density within NCS_2 moiety of the dithiocarbamate group. The ν_{C-S} bands appear around 1025 cm^{-1} without any splitting. This is an indication of the bidentate character of the dithiocarbamate ligands.

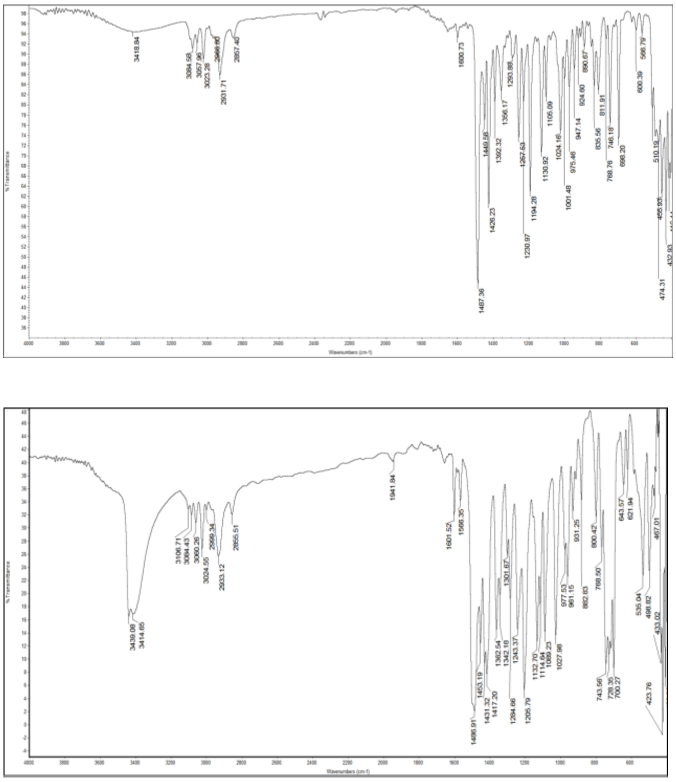


Fig. 1. IR spectrum of complex **1** and **2**

V. CHARACTERIZATION OF NANOPARTICLES

5.1. Powder X-ray diffraction analysis

The phase and structure of the samples were examined by powder X-ray diffraction. Nanoparticles obtained from **1** and **2** are represented as zinc sulfide, zinc-iron sulfide. The powder X-ray diffraction patterns for the nanoparticles are shown in Figs.2. Zinc sulfide displays peaks (Fig. 2.) which can be indexed to single phase rhombohedral with lattice constant comparable to the values of JCPDS file No. 89-2426. The peaks observed in the PXRD pattern of zinc-iron sulfide are due to cubic which are close to the data in JCPDS card No. 65-4384.

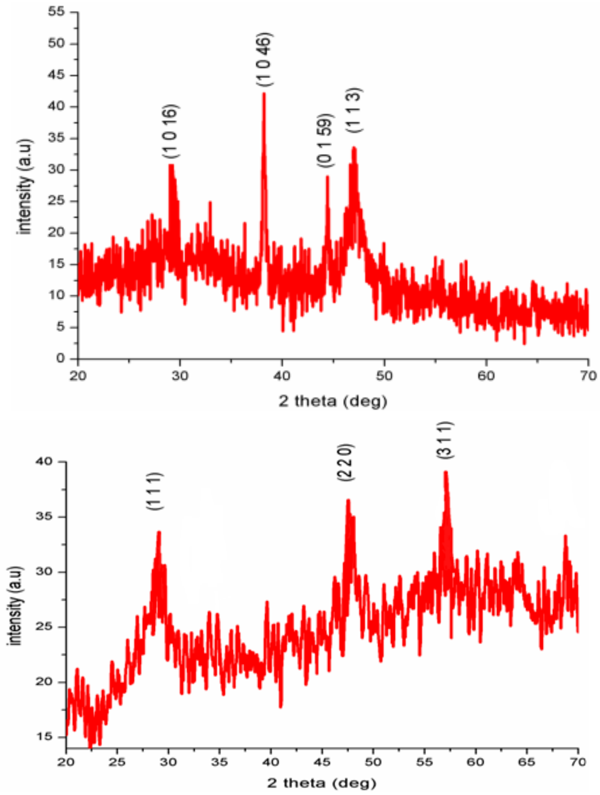
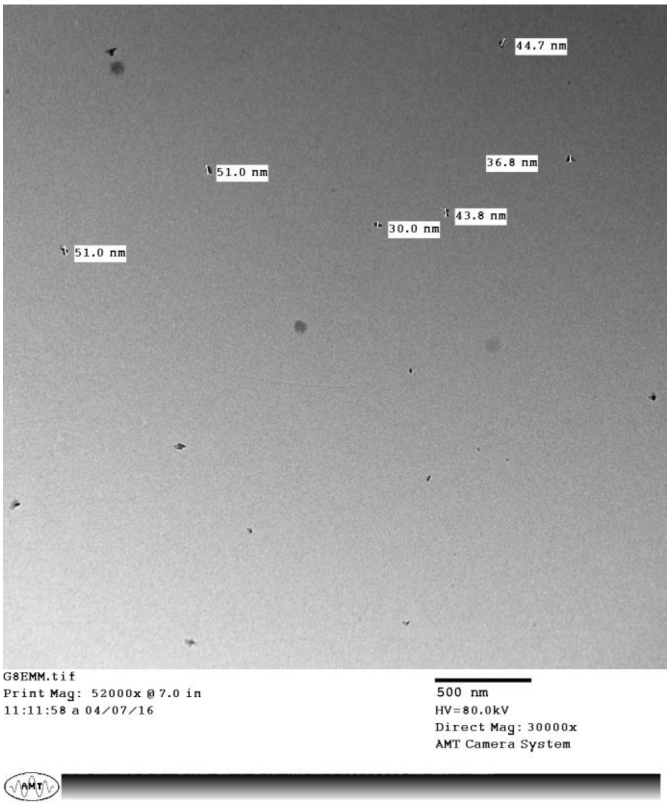


Fig.2. Powder X-ray diffraction pattern of zinc sulfide and zinc-iron sulfide

5.2. Morphological Characterization

The dimensions and morphologies of zinc sulfide and zinc-iron sulfide were studied by TEM measurements. The TEM images for the three samples are shown in Figs.3.

Fig.3. Indicates that the zinc sulfide particles are spherical. The average particle diameter as determined from the TEM images is around 40 nm. Nearly spherical zinc-iron sulfide particles can be seen in the TEM image (Fig.3) of the sample prepared from complex **2**.



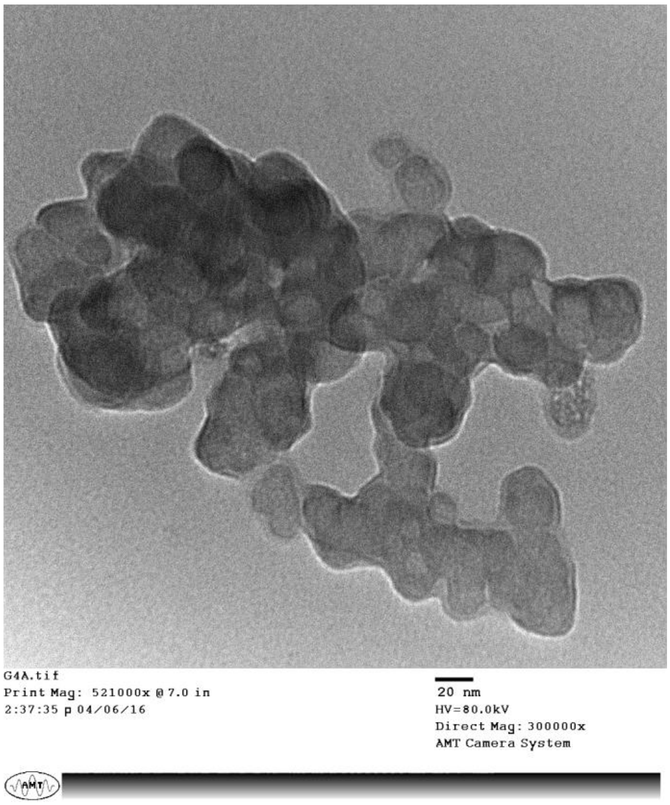
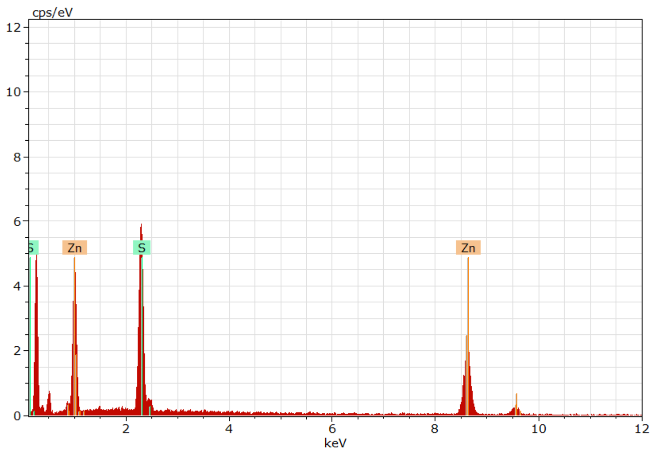


Fig.3. TEM image of zinc sulfide and zinc-iron sulfide

5.3. Energy dispersive X-ray spectral (EDS) analysis

The energy dispersive X-ray spectra of zinc sulfide and zinc-iron sulfides are shown in Figs.4. Energy dispersive X- ray spectroscopy analysis were performed to confirm the formation of zinc sulfide and zinc-iron sulfides. The atomic ratio of Zn/S of the products obtained from 1 calculated from the EDS is 1: 1.09. The excess sulfur comes from the S impurities. Zn, Fe and S elements are found to present in the EDAX of zinc-iron sulfide the atomic ratio of Zn, Fe and S is 3.0: 2.05: 4.95 respectively. This provides the evidence for the formation bimetallic sulfides.



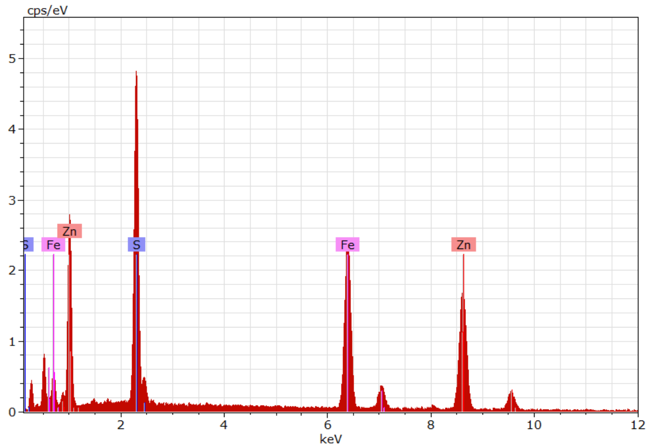


Fig.4. EDS spectrum of zinc sulfide and zinc-iron sulfide

VI. CONCLUSION

In this contribution two new zinc(II) dithiocarbamate complexes have been synthesized and characterized by spectroscopic techniques. These complexes have been exploited as single source precursors for the preparation of zinc sulfide and zinc-iron sulfide nanoparticles. Zinc sulfide and zinc-iron sulfide nanoparticles were characterized using pXRD, TEM and EDS spectroscopy. TEM image of zinc-iron sulfide nanoparticles demonstrated that the particles are nearly spherical shapes. We expect this simple approach can be used for the synthesis of monometallic and bimetallic sulfide semiconductor nanoparticles with different morphologies, compositions and properties from single source precursors.

VII. CONFLICT OF INTEREST

The authors declare no conflict of interest.

VIII. ACKNOWLEDGMENTS

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IX. REFERENCES

[1]. Z. Leka, D. Voja, M. Kosovi, N. Latinovic, M. Dakovic, A. Visnjevac, Synthesis, structure and antifungal activities of noval Co, Mo and Pt complexes with triammonium N, N-diacetatedithiocarbamate, Polyhedron. 80 (2014) 233- 242:

[2]. G.N. Kaludjerovic, V.M. Ojinovic, S.R. Trifunovic, I.M. Hodzic and T.J. Sabo, Synthesis and characterization of trisbutyl-(1-methyl-3-phenyl-propyl)-dithiocarbamato]-cobalt(III)seskvitoluene, J. Serb. Chem. Soc. 67 (2002) 123-126:

[3]. R. Abu-El-Halawa, S.A. Zabin, Removal efficiency of Pb, Cd, Cu and Zn from polluted water using dithiocarbamate ligands, J. Taiba Univ. Sci. (2015) 1- 9:

[4]. M. Gong, A. Kiokeminde, N.Kumar, H.Zhao and S.Ren, Ionic-passivated FeS2 photocapactors for energy conversion and storage, Chem. Commun. 49 (2013) 9260-9262:

[5]. N. Tokyo, K. Azkio, J Pn. Kokai Pat. 7855478 C1.C23 C15/00, (1978):

[6]. N. Tokya, JPn. Kokai pat. 75130378 C1 H0 1L CO 1B, (1975):

[7]. D. J. Asunskis, I.L. Boltin, A. T. W Rolde, A.M. Zachary and L.Hanley, Lead sulfide nanocrystals-polymer composition for optoelectronic applications, Macromol.

[8]. Symp. 268 (2008) 33-37:

[9]. S. Khalid, E. Ahmed, M.A. Malik, S. Abu bakar, Y. Khan and P.O. Brien, Synthesis of pyrite thin films and transition metal doped pyrite thin films by aerosol-assisted chemical vapour deposition, New. J. Chem. 39 (2015) 1013-1021:

[10]. E. Sathiyaraj, G. Gurumoorthy and S. Thirumaran, Nickel(II) dithiocarbamate complexes containing the pyrrole moiety for sensing anions and synthesis of nickel sulfide and nickel oxide nanoparticles, New. J. Chem. 39 (2015) 5336-5349:

[11]. N.L. Pickett and P.O. Brien, Synthesis of semiconductor nanoparticles using single-molecular precursors, Chem. Rec. 1 (2001) 467-479:
[12]. N. Srinivasan, S. Thirumaran, Effect of pyridine as a ligand in precursor on morphology of CdS nanoparticles, Superlattices Microstruct. 51 (2012) 912-920:
[13]. S. Rengaraj, S. Venkatraj, S.H. Jec, Y.H. Kim, C. Tai, E. Repo, A. Koistienen, A. Ferancova, M. Sillpaa, Self-Assembled Mesoporous Hierarchial-like In2S3 Hallow Microspheres Composed of Nanofibers and Nanosheets and their Photocatalytic Activity, Langumuir 27 (2011) 352- 358:
[14]. R. Chauhan, A. Kumar, R.P. Chaudhary, Photocatalytic degradation of methylene blue with Cu doped ZnS nanoparticles, J. Lumin. 145 (2014) 6-12:
[15]. S. Lee, D. Song, D. Kim, J. Lee, S. Kim, I.Y. Park, Y.D. Choi, Effects of synthesis temperature on particle size/shape and photoluminescence characteristics of ZnS:Cu nanocrystals, Mater. Lett. 58 (2004) 342.

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