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Nano-sized Mercury(II) Schiff Base Complexes: Synthesis, Characterization and Thermal Studies

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ABSTRACT

In this paper, nano-sized of two mercury(II) complexes with the general formula of $HgLX_2$ (X=Br (**1**) and I (**2**)), **L**=*N,N'*-bis{(paramethylamino)benzylidene}ethylenediamine have been prepared by ultrasonic bath assisted from the reaction of HgX_2 and **L** in molar ratio 1:1 in methanol solution. The complexes were characterized by elemental analyses (CHN) and FT-IR spectroscopy, XRD and SEM. In addition, thermal stability of complexes was studied.

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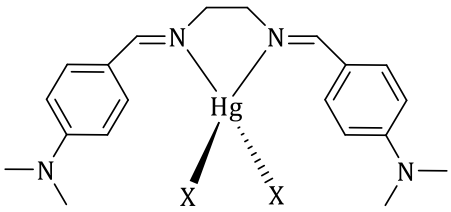
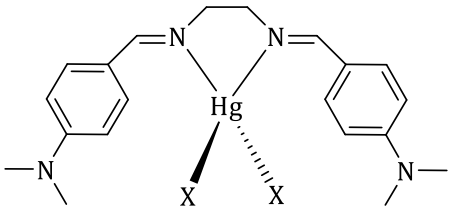
Graphical Abstract



Introduction

Metal complexes containing d^{10} configuration electron such as Zn(II) and Hg(II) have extensively been investigated due to their various applications and structures [1-5], such as new biological active agents [3, 6, 7] and dye-sensitized solar cells [8, 9]. In this sense, Basu Baul *et al.* [2] reported the effect of ligand *R*-group on structure and optical properties of mercury(II) halides complexes. Also, Ghoreishi Amiri *et al.* [9], reported the effect of rigid and flexible organic ligand to the coordination polymers of zinc(II). Recently, preparation of nano-size metal complexes is attractive because of their unique properties [3, 10] and generally due to the fact that they are used as precursors for the synthesis of metal oxide nanoparticles [11, 12]. A literature review confirmed that transition metal complexes with bidentate Schiff base ligand have shown notable biological properties [6, 7]. Recently, Sheikhshoaie *et al.* [13, 14] reported the preparation of nano-sized Zn(II) Schiff base complexes *via* sonochemical method and used them as new precursor for synthesized of ZnO nanoparticles.

Herein, the synthesis and characterization of nano-size of mercury(II) Schiff base complexes (Scheme 1) by a simple ultrasonic bath assisted are described.



Scheme 1. Chemical structures of the title complexes, X = Br (**1**) and I (**2**)

Experimental

Materials and methods

All chemical reagents and solvents were purchased from Merck Company. The Schiff base ligand was freshly synthesized based on what has been described before [15]. Elemental analyses were performed

on a Heraeus CHNeO-Rapid analyzer and the results agreed with the calculated values. X-ray powder diffraction (XRD) pattern of the complex was recorded on a Bruker AXS diffractometer D8 ADVANCE with Cu-Kα radiation with nickel beta filter in the range 2θ=10°–80°. Fourier transform infrared (FT-IR) spectra were recorded as a KBr disk on a FT-IR Perkin–Elmer spectrophotometer. The scanning electron microscopy (SEM) images were obtained from a Philips XL-30ESEM. The TG/DTA were performed on a Perkin-Elmer TG/DTA lab system 1 (Technology by SII) in argon atmosphere (flow rate 16.66 cm^3/min^{-1}) with a heating rate of 20 °C/min in the temperature span of 25–800 °C. The ultrasonic bath with a power output of 40 KHz has been used for preparation of complexes.

A methanolic solution of the Schiff base ligand (1 mmol in 5 mL) was drop by drop (during 20 min) added to the stoichiometric amount of methanolic solution of $HgBr_2$ (**1**) and HgI_2 (**2**) (1 mmol in 10 mL) under ultrasonic bath irradiation. After completed addition, the reaction mixture was kept in the ultrasonic bath for a period of 60 min. The obtained color less precipitate was filtered and dried at room temperature and, then, characterized.

$Hg((Me_2N\text{-}ba)_2en)Br_2{\cdot}2H_2O$ (**1**): Yield, 73%. Anal. Calc. for $C_{20}H_{30}N_4HgO_2Br_2$: C, 33.41; H, 4.17; N, 7.79%. Found: C, 33.32; H, 4.23; N, 7.85%. FT-IR (cm^{-1}): 2825-3015 (H-C aliphatic and aromatic), 1588 (C=N).

$Hg((Me_2N\text{-}ba)_2en)I_2$ (**2**): Yield, 75%. Anal. Calc. for $C_{20}H_{26}N_4HgBr_2$: C, 30.98; H, 3.36; N, 7.23%. Found: C, 31.03; H, 3.41; N, 7.19%. FT-IR (cm^{-1}): 2820-3011 (H-C aliphatic and aromatic), 1589 (C=N).

Results and discussion

In a convenient one-pot reaction by an assisted ultrasonic bath, three nano-sized complexes with the general formula of $Hg((Me_2N\text{-}ba)_2en)X_2$ [X=Br (**1**) and I (**2**)] have been prepared and characterized. All complexes are insoluble in common organic solvent such as methanol, ethanol and chloroform. They are stable at room temperature in solid state for several months. The compounds contained Br adsorbed water molecules as the elemental analysis and the TG of these compounds confirmed this matter.

In the FT-IR spectra of complexes, some weak vibrations at about 3000 cm^{-1} are found and are attributed to the stretching vibrations of C-H bonds. A sharp peak at about 1590 cm^{-1} assigned to C=N (azomethine) of ligand (1612 cm^{-1}), and shifted to lower wave numbers (≈ 22 cm^{-1}). This change confirmed the coordinated of azomethine nitrogens to metal ions [3, 7-9]. Also, the FT-IR spectrum of the complexes did not show the stretching vibration of carbonyl and amine functional groups, confirmed the presence of Schiff base ligand in the complexes.

Thermal study (TG-DTA) of the title compounds was investigated from room temperature to 800 °C under argon atmosphere with the heating rate of 20 °C/min. The TG-DTA curves of complexes are

displayed in Figures 1 and 2, and show various steps of mass losing against temperature. In the TG curves of complex **1**, there is weight loss up to 250 °C, which confirmed the adsorb of water molecules in the surface of complexes, while for complex **2** there is no weight loss up to 200 °C confirming the absence of any coordinated or crystalline water molecules in the complex [7]. After that, the complexes were decomposed in two steps at temperature ranges 205-450 °C and 450 – 780 °C.

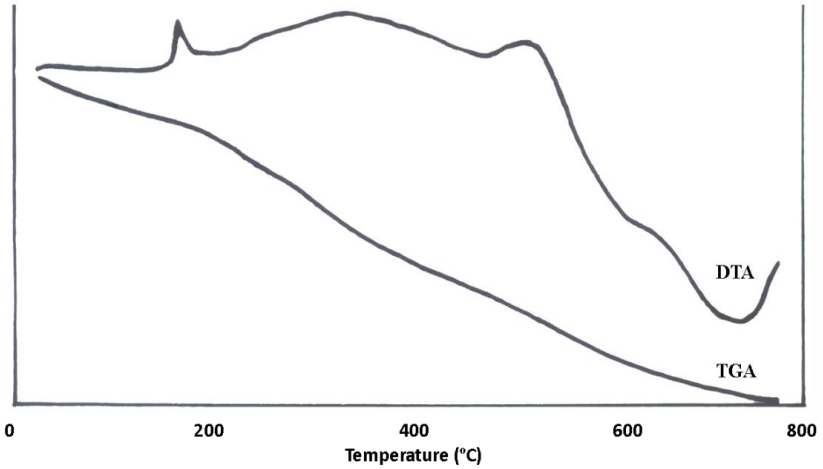


Figure 1. The TG-DTA curves of the complex **1**

For the study of the particle size of the complexes the XRD patterns have been recorded and presented in Figures 3 and 4. The XRD patterns have shown that the sharp crystalline peaks indicate the crystalline phase. The average size of the nano-sized complexes were calculated by Scherrer's formula, and found to be <50 nm, confirmed by SEM images (Figures 3 and 4).

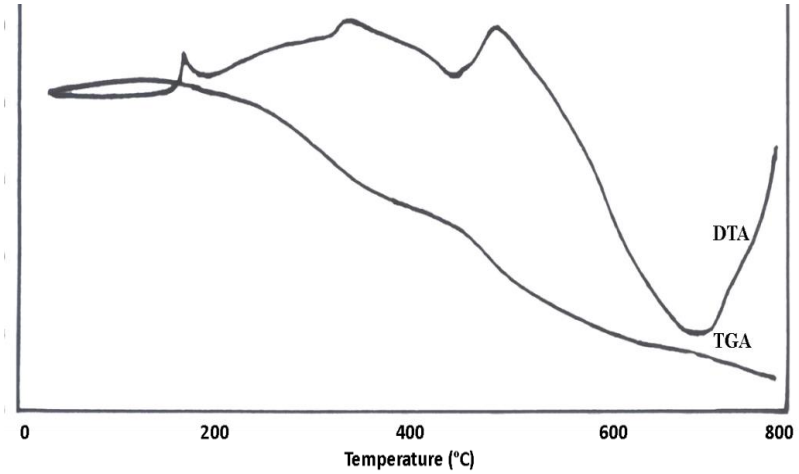


Figure 2. The TG-DTA curves of the complex **2**

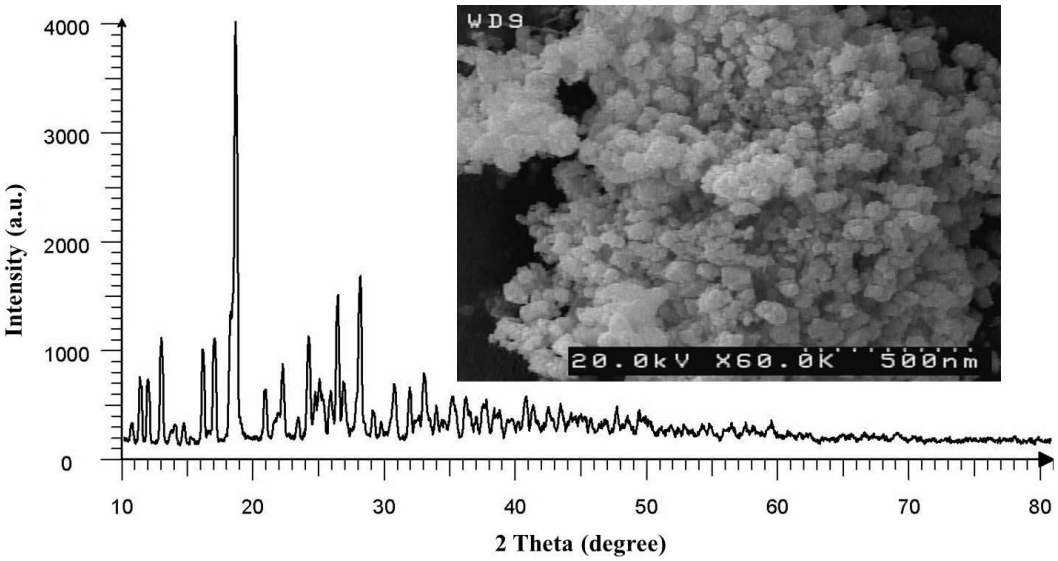


Figure 3. XRD pattern and SEM image of complex **1**

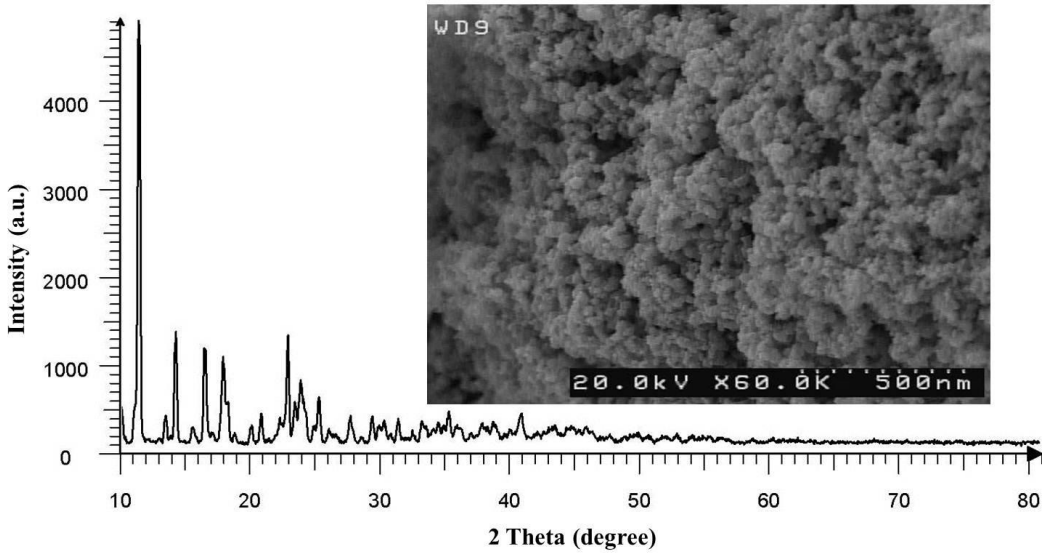


Figure 4. XRD pattern and SEM image of complex **2**

Conclusion

Nano-sizes of mercury(II) Schiff base complexes have been synthesized using ultrasonic bath assisted. Morphology and size of the products depend on the initial salt reagents ($HgBr_2$ and HgI_2). FT-IR, TGA, XRD and SEM techniques were used to characterize the synthesize samples. SEM images showed that the morphology of the particles is different, and the size of the particles is within the nano scale.

Acknowledgment

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Conflict of Interest

We have no conflicts of interest to disclose.

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