



Sonochemical synthesis, characterization, and effects of temperature, power ultrasound and reaction time on the morphological properties of two new nanostructured mercury(II) coordination supramolecule compounds



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ABSTRACT

Two new mercury(II) coordination supramolecular compounds (CSCs) (1D and 0D), $[\text{Hg}(\text{L})(\text{I})_2]_n$ (**1**) and $[\text{Hg}_2(\text{L}')_2(\text{SCN})_2] \cdot 2\text{H}_2\text{O}$ (**2**) ($\text{L} = 2\text{-amino-4-methylpyridine}$ and $\text{L}' = 2,6\text{-pyridinedicarboxylic acid}$), have been synthesized under different experimental conditions. Micrometric crystals (bulk) or nano-sized materials have been obtained depending on using the branch tube method or sonochemical irradiation. All materials have been characterized by field emission scanning electron microscope (FESEM), scanning electron microscopy (SEM), powder X-ray diffraction (PXRD) and FT-IR spectroscopy. Single crystal X-ray analyses on compounds **1** and **2** show that Hg^{2+} ions are 4-coordinated and 5-coordinated, respectively. Topological analysis shows that the compound **1** and **2** have 2C1, **sql** net. The thermal stability of compounds **1** and **2** in bulk and nano-size has been studied by thermal gravimetric (TG), differential thermal analyses (DTA) for **1** and differential scanning calorimetry (DSC) for **2**, respectively. Also, by changing counter ions were obtained various structures **1** and **2** (1D and 0D, respectively). The role of different parameters like power of ultrasound irradiation, reaction time and temperature on the growth and morphology of the nanostructures are studied. Results suggest that increasing power ultrasound irradiation and temperature together with reducing reaction time and concentration of initial reagents leads to a decrease in particle size.

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1. Introduction

In recent years, the design and synthesis of CSCs have drew more and more attention not only due to their tremendous potential applications in molecular recognition [1], nonlinear optics [2], magnetism [3], metal ion selection [4], heterogeneous catalysis [5], and gas storage [6], but also for their intriguing structural diversity and interesting hydrogen bonding network

[7,8]. Generally, the kind of metal ions, the selection of auxiliary ligand, geometry and number of coordination sites provided by organic ligands are all important parameters in the self-assembly processes of CSCs. Among these parameters, the selection of organic ligand with suitable binding groups is especially crucial [9–16].

Mercury(II) is a well-known metal with a wide range of applications in different areas. However, mercury has not been used widely for the fabrication of coordination polymers in the literature [15–22]. The d^{10} configuration of the Hg(II) ion is associated with a flexible coordination environment so that different geometries can be generated to tailor-make materials [18]. The CSCs of

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this metal ion can be synthesized through different methods, such as the diffusion-based method, layering technique, evaporation route, hydrothermal synthesis, crystallization technique and ultrasonic irradiation method [23–29].

Sonochemical synthesis of various types of nanoparticles and nanostructured materials composed of noble metals [30–32], transition metals [33–35], semiconductors [36], carbon materials [37], and polymeric materials [38], have received much attention in recent years. This is due to the unique reaction routes induced by acoustic cavitation in solution, which provides extreme conditions of transient high temperature and high pressure within the collapsing bubbles, shock wave generation, and radical formation [38,64–66].

Nanostructured materials have been intensively studied in recent years because the physical properties of these materials are often quite different from those of the bulk [39–42]. Nano size CSCs are attractive to explore, since controlling the growth of materials at the sub-micrometer scale is of the central importance in the emerging field of nanotechnology [43]. CSCs with various morphologies, e.g., nanospheres, nanocubes, nanosheets, and nanorods have been prepared by various synthetic techniques, such as precipitation, microemulsion, and solvothermal techniques, as well as microwave-assisted methods [44,45]. The development of mild, green, low-cost, large-scale, environmentally responsible, and more flexible methods for creating controllable morphologies of nano/microstructures CSCs are strongly desired [46–48].

The effects of ultrasound radiation on chemical reactions were reported in the recent works [49–51]. In this manuscript, we have developed a simple sonochemical to prepare nanostructures of $[\text{Hg}(\text{L})(\text{I})_2]_n$ (**1**) and $[\text{Hg}_2(\text{L}')_2(\text{SCN})_2] \cdot 2\text{H}_2\text{O}$ (**2**). The power of ultrasound irradiation, sonication time, temperature of reaction, and concentration of initial reactants were the parameters evaluated for reaching the optimized condition. Scanning electron microscopy (SEM) and powder X-ray diffraction (PXRD) were used for the characterization of the products. In addition, we have investigated the influence of different properties (sonication power, reaction time and temperature) on the particle morphology and size. This was achieved by employing a sonochemical method.

2. Experimental

2.1. Materials and physical techniques

Starting reagents for the synthesis were purchased and used without any purification from industrial suppliers (Sigma–Aldrich, Merck and others). Elemental analyses (carbon, hydrogen, and nitrogen) were performed employing a Heraeus Analytical Jena, Multi EA 3100 CHNO rapid analyzer. Fourier transform infrared spectra were recorded on a FT-IR JASCO 680-PLUS spectrometer as KBr pellets in the 4000–400 cm^{-1} spectral range, also infrared spectra (IR) were obtained using the instrument Bruker Tensor 27 FT-IR with a single window reflection of diamond ATR (Attenuated total reflectance) model MKII Golden Gate, Specac and the OPUS data collection program software. The instrument is equipped with a room temperature detector, and a mid-IR source (4000 to 400 cm^{-1}). Since it is a single beam instrument, it was needed to run a background spectrum in air before the measurement.

Thermal gravimetric analysis (TGA), differential thermal analysis (DTA) and differential scanning calorimetry (DSC) of the title compound were performed on a computer-controlled STA - PT 1500 apparatus, also thermal measurements were performed with PerkinElmer Pyris instrument by using platinum

pans. The curves were recorded in temperature range from 30 °C to 650 °C at 10 °C/min under nitrogen atmosphere with flow rate 60 mL/min. The data obtained was processed with Pyris software and calorimetry measurements were done by PerkinElmer 8000 equipment by using aluminum and platinum pans under nitrogen atmosphere in the temperature range between 10–400 °C. Single phase powder sample of 1–1, 2 – 1, 3 – 1 and 4 – 1 crystal structure compound **1** and **2** were loaded into alumina pans and heated with a ramp rate of 10 °C/min from room temperature to 650 °C under argon atmosphere. Single crystal X-ray diffraction experiments were carried out for compounds **1** and **2** with MoK α radiation ($\lambda = 0.71073 \text{ \AA}$) at ambient temperature. A microfocused Rigaku mm003 source with integrated confocal caxFlux double bounce optic and HPAD

Table 1

Crystal data and structures refinement for $[\text{Hg}(\text{L})(\text{I})_2]_n$ (**1**) and $[\text{Hg}_2(\text{L}')_2(\text{SCN})_2] \cdot 2\text{H}_2\text{O}$ (**2**).

Empirical formula	$\text{C}_6 \text{H}_8 \text{Hg} \text{I}_2 \text{N}_2$	$0.4(\text{C}_{16}\text{H}_8\text{Hg}_2\text{N}_4\text{O}_8\text{S}_2) \cdot 0.8 \text{H}_2\text{O}$
Formula weight	562.53	354.24 g/mol
Temperature	203(2) K	293(2) K
Wavelength	0.71075 Å	0.71073 Å
Crystal system	Monoclinic	Monoclinic
Space group	P 21/c	P2 ₁ /c
Unit cell dimensions	a = 4.3480(2) Å, $\alpha = 90.000^\circ$ b = 12.0700(9) Å, $\beta = 91.500(3)^\circ$ c = 20.3618(14) Å, $\gamma = 90.000^\circ$	a = 7.0679(2) Å, $\alpha = 90^\circ$ b = 17.9631(4) Å, $\beta = 116.336(4)^\circ$ c = 20.1454(7) Å, $\gamma = 90^\circ$
Volume	1068.23 (12) Å ³	2292.23 (14) Å ³
Z	4	10
Density(calculated)	3.50853 g/cm ³	2.566 g/cm ³
Absorption coefficient	22.135 Mg/m ³	13.58 Mg/m ³
F(0 0 0)	976.0	1632
Theta range for data collection	2.00 to 29.08°	4.1 to 31.3°
Reflections collected	3864	26544
μ	20.14 mm ⁻¹	13.621 mm ⁻¹
Index ranges	$-4 \leq h \leq 6$ $-16 \leq k \leq 16$ $-27 \leq l \leq 27$	$-9 \leq h \leq 10$ $-24 \leq k \leq 26$ $-29 \leq l \leq 28$
h, k, l (max)	6, 16, 27	9, 27, 16
(sin θ)/ λ max	0.698 Å ⁻¹	0.734 Å ⁻¹
Theta(max)	26.8°	31.3°
Radiation type	Mo K α	Mo K α
Refinement method	Full-matrix least-squares on F ²	Full-matrix least-squares on F ²
Goodness-of-fit-on F ²	1.080	1.054
Refinement	R[F ² > 2 σ (F ²)] = 0.56 wR(F ²) = 0.136 S = 1.08	R[F ² > 2 σ (F ²)] = 0.031 wR(F ²) = 0.078 S = 1.05
Extinction coefficient	n/a	0.00081(9)
Largest diff. peak and hole	3.40, -3.00 e Å ⁻³	2.44 and -1.61 e Å ⁻³
CCDC no.	1510179	1501127

Table 2

Selected bond lengths/Å for compound $[\text{Hg}(\text{L})(\text{I})_2]_n$.

Hg(1)–N(1)	2.318(12)	N(1)–C(1)	1.334(18)
Hg(1)–I(2)	2.6569(11)	N(1)–C(5)	1.389(17)
Hg(1)–I(1) ⁱ	2.7370(11)	N(2)–C(5)	1.385(18)
Hg(1)–I(1)	2.9986(11)	N(2)–H(2)A	0.89(2)
I(1)–Hg(1) ⁱⁱ	2.7371(11)	N(2)–H(2)B	0.88(2)
C(2)–C(1)	1.37(2)	C(1)–H(1)	0.9400
C(2)–C(3)	1.43(2)	C(6)–C(3)	1.48(2)
C(2)–H(2)	0.9400	C(6)–H(6)A	0.9700

Symmetry transformations used to generate equivalent atoms: (i) –1+x, y, z; (ii) 1+x, y, z.

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