



# Syntheses and structural characterization of two new nanostructured Bi(III) supramolecular polymers via sonochemical method



Xiao-Wei Yan<sup>a</sup>, Ensieh Haji-Hasani<sup>b</sup>, Ali Morsali<sup>c,\*</sup>

<sup>a</sup> College of Chemical and Biological Engineering, Hezhou University, Hezhou 542800, China

<sup>b</sup> Department of Chemistry, Payame Noor University, Abhar, Zanjan, Islamic Republic of Iran

<sup>c</sup> Department of Chemistry, Faculty of Sciences, Tarbiat Modares University, P.O. Box 14155-4838, Tehran, Islamic Republic of Iran

## ARTICLE INFO

### Article history:

Received 19 October 2015

Received in revised form 11 November 2015

Accepted 12 November 2015

Available online 26 November 2015

### Keywords:

Coordination polymer

Bismuth(III)

Supramolecular compound

Bipyridyl ligand

Nano-structure

## ABSTRACT

Two new bismuth(III) coordination supramolecular polymers,  $\{[Bi_2(Hbpp)(bpp)(\mu-1)_2I_6](Hbpp)\cdot MeOH\}_n$  (**1**) and  $[Bi(Hbpp)(Br_4)]$  (**2**), ( $bpp = 1,3\text{-di(pyridin-4-yl)propane}$ ) were prepared and were structurally characterized by single crystal X-ray diffraction. Single crystalline one-dimensional materials were prepared using a heat gradient applied a solution of the reagents using the branched tube method. The structural determination by single crystal X-ray crystallography shows that compounds **1** and **2** form monoclinic polymers with symmetry space group  $P2_1$  in the solid state. These new nanostructured Bi(III) supramolecular compounds,  $\{[Bi_2(Hbpp)(bpp)(\mu-1)_2I_6](Hbpp)\cdot MeOH\}$  (**1**) and  $[Bi(Hbpp)(Br_4)]$  (**2**), were also synthesized by sonochemical method. The nanostructures were characterized by Field Emission-scanning electron microscopy (FE-SEM), powder X-ray diffraction (PXRD) and IR spectroscopy.

© 2015 Elsevier B.V. All rights reserved.

## 1. Introduction

In recent years, the intelligent design and synthesis of coordination polymers have received extensive attention not only owing to intriguing structural motifs of the coordination polymers but also because of their potential applications in many fields, such as catalysis, optical devices and magnets [1–11]. The arrangement of coordination polymers depends on some factors for example temperature, the metal atoms and so on [12–13]. Between these factors, organic ligands perform important function in the construction of coordination polymers with distinctive structures. The 1,3-di(pyridin-4-yl)propane ( $bpp$ ) ligand is greatly used for the synthesis of coordination polymers [14–15].

Bismuth(III) because of the presence of  $6s^2$  lone pair on its valence shell is of interest for preparation of coordination supramolecular compounds. The coordination chemistry of bismuth(III) is widely spaced when compared with other metals [16–18], and even it is perfectly true for supramolecular based on Bi(III) compounds. The bismuth(III) complexes with the bipyridyl ligands rarely reported until recently [19–21]. It may be a consequence of low solubility of these complexes [22]. However, the larger bismuth atoms can exhibit higher coordination and much variable coordination numbers from 3 to 10, which provides

singular opportunities for the discovery of new network topologies and singular physical properties [23].

Also usage of ultrasound for organic synthesis and the preparation of nanomaterials has been grown at an enormous rate [24]. Sonochemistry is the research area in which molecules undergo a reaction due to the utilization of powerful ultrasound radiation (20–10 MHz). Ultrasound irradiation is facile, environmentally friendly and cost effective synthesis of nanoscale coordination polymers [25].

In this work, we have synthesized and characterized of two new Bi(III) supramolecular structures,  $[Bi_2(Hbpp)(bpp)(\mu-1)_2I_6](Hbpp)\cdot MeOH$  (**1**) and  $[Bi(Hbpp)(Br_4)]$  (**2**) ( $bpp = 1,3\text{-di(pyridin-4-yl)propane}$ ) by brunched tube method and also their nanostructures were prepared by a simple synthetic sonochemical method.

## 2. Experimental

### 2.1. Physical measurements

IR spectra were recorded using Thermo scientific Nicolet\_is10 spectrophotometers. Elemental analyses were carried out using a FAMX analyzer. Melting points were measured on an Electrothermal 9100 apparatus. X-ray powder diffraction (XRD) measurements were performed using an X'pert diffractometer of Philips Company with monochromatized  $Cu\alpha$  radiation. Crystallographic measurements were made at 100(2) and 273(2) K using a Bruker APEX area-detector diffractometer for compounds **1** and

\* Corresponding author.

E-mail address: [morsali\\_a@modares.ac.ir](mailto:morsali_a@modares.ac.ir) (A. Morsali).

**Table 1**  
Crystal data and structure refinements for compounds **1–2**.

Identification code	<b>1</b>	<b>2</b>
Empirical formula	C <sub>40</sub> H <sub>48</sub> Bi <sub>2</sub> I <sub>8</sub> N <sub>6</sub> O	C <sub>26</sub> H <sub>30</sub> Bi Br <sub>4</sub> N <sub>4</sub>
Formula weight	2062.00	927.16
Temperature (K)	100(2)	273(2)
Wavelength	0.71073	0.71073
Crystal system	Monoclinic	Monoclinic
Space group	P2 <sub>1</sub>	P2 <sub>1</sub>
Unit cell dimensions	<i>a</i> = 12.870(6) Å <i>b</i> = 17.000(9) Å <i>c</i> = 13.348(7) Å $\beta$ = 100.862(9)°	<i>a</i> = 8.8758(8) Å <i>b</i> = 18.0085(15) Å <i>c</i> = 10.1470(8) Å $\beta$ = 111.288(2)°
Volume	2868(3) Å <sup>3</sup>	1511.2(2) Å <sup>3</sup>
Z	2	2
Density (calculated)	2.388 g/m <sup>3</sup>	2.038 g/m <sup>3</sup>
<i>F</i> (000)	1856	874
Theta range for data collection	2.42–28.37°	2.26–25.10°
Index ranges	–15 ≤ <i>h</i> ≤ 15 –20 ≤ <i>k</i> ≤ 20 –16 ≤ <i>l</i> ≤ 16	–9 ≤ <i>h</i> ≤ 10 –21 ≤ <i>k</i> ≤ 21 –12 ≤ <i>l</i> ≤ 10
Reflections collected	10,529	2692
Independent reflections	9766	2184
Absorption correction	Integration	Integration
Refinement method	<i>F</i> <sup>2</sup> > 2σ( <i>F</i> <sup>2</sup> )	<i>F</i> <sup>2</sup> > 2σ( <i>F</i> <sup>2</sup> )
Data/restraints/parameters	10,529/1/516	2692/1/163
Goodness-of-fit on <i>F</i> <sup>2</sup>	1.048	1.058
Final <i>R</i> indices [ <i>I</i> > 2σ( <i>I</i> )]	<i>R</i> <sub>1</sub> = 0.0298 and <i>wR</i> <sub>2</sub> = 0.0788	<i>R</i> <sub>1</sub> = 0.0417 and <i>wR</i> <sub>2</sub> = 0.1179
<i>R</i> indices (all data)	<i>R</i> <sub>1</sub> = 0.0339 and <i>wR</i> <sub>2</sub> = 0.0843	<i>R</i> <sub>1</sub> = 0.0521 and <i>wR</i> <sub>2</sub> = 0.1225

**2**, respectively. TGA and DTA curves were recorded using a PL-STA 1500 device manufactured by Thermal Sciences. The samples were characterized with a Field Emission-scanning electron microscopy (FE-SEM) Mira3 TESCAN-XMU with gold coating.

## 2.2. Preparation of [Bi<sub>2</sub>(Hbpp)(bpp)(μ-I)<sub>2</sub>I<sub>6</sub>](Hbpp)·MeOH (**1**) and [Bi(Hbpp)(Br<sub>4</sub>)] (**2**)

Compound **1** was prepared by the following method (the branched tube method): Potassium iodide (0.249 g, 1.5 mmol)

was placed in one arm of the branched tube and mixture of bpp (0.198 g, 1 mmol) and bismuth(III) chloride (0.1577 g, 0.5 mmol) in the other. Methanol was carefully added to fill both arms. Be careful, do not enter primal materials in the small arm. The tube closed tightly and ligand-containing arm submerged in a bath at 60 °C while the other was at room temperature. After 21 days, orange crystals (m.p. = 152 °C), had deposited in the cooler arm which were filtered off, and air dried. Synthesis of compound **2** is similar to it in compound **1**, with difference that here KBr be used instead of KI. After 21 days, white crystals (m.p. = 187 °C), had deposited in the cooler arm which were filtered off, and air dried.

### 2.2.1. Compound **1**

IR (selected bands; in cm<sup>-1</sup>): 457 m, 521 m, 543 s, 1007 s, 1063 w, 1221 m, 1425 w, 1500 w, 1608 s, 2922 w, and 3447 w.

<sup>1</sup>HNMR (DMSO): δ = 8.6–8.65 (d, 1H), 7.4–7.48 (d, 1H), 5.4–5.7 (s, 1H), 2.7–2.9 (t, 2H), 1.8–2.1 (q, 2H) ppm.

<sup>13</sup>CNMR (DMSO): δ = 155, 147, 123, 35, 30 ppm.

### 2.2.2. Compound **2**

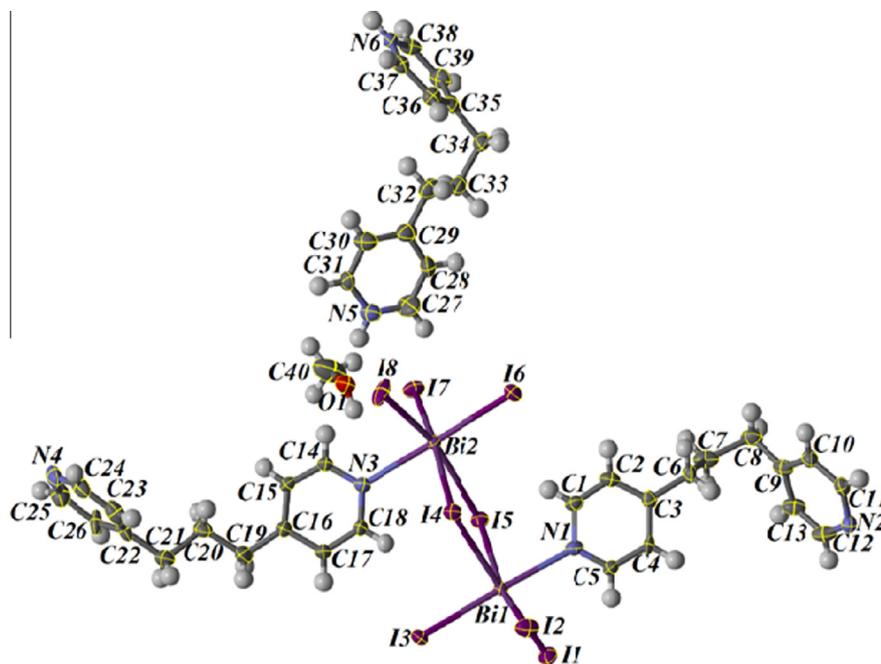
IR (selected bands; in cm<sup>-1</sup>): 510 m, 812 m, 1008 m, 1635 s, 2924 s, and 3423 w.

<sup>1</sup>HNMR (DMSO): δ = 8.6–8.65 (d, 1H), 7.4–7.48 (d, 1H), 5.4–5.44 (s, 1H), 2.62–2.68 (t, 2H), 1.9–1.98 (q, 2H) ppm.

<sup>13</sup>CNMR (DMSO): δ = 155, 147, 123, 35, 30 ppm.

## 2.3. Synthesis of compounds **1–2** nanostructures

Ultrasonic syntheses of compounds **1–2** were carried out in an ultrasonic bath at ambient temperature and atmospheric pressure. To prepare the nanostructure of compounds **1–2**, a solution of BiCl<sub>3</sub> (0.123 mmol) and KI or KBr (0.37 mmol) were dissolved in 10 mL CH<sub>3</sub>OH in a 25 mL flask and it was positioned in the ultrasonic bath. Into this solution 10 mL of 0.25 mmol ligand 1,3-di(pyridin-4-yl)propane was added dropwise. The obtained precipitates were filtered off and then dried in air.



**Fig. 1.** The molecular structure of the asymmetric  $\{[Bi_2(Hbpp)(bpp)(\mu-I)_2I_6](Hbpp) \cdot MeOH\}_n$  (**1**).

Download English Version:

<https://daneshyari.com/en/article/1265654>

Download Persian Version:

<https://daneshyari.com/article/1265654>

[Daneshyari.com](https://daneshyari.com)