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Ionothermal syntheses and characterization of 2-D coordination polymers with 4-(1H-imidazol-1-yl) benzoic acid

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4-(1H-imidazol-1-yl) benzoic acid and its coordination polymers $[M(\text{IBA})\text{Cl}]_n$ ($M = \text{Zn}$ (**1**) and Co (**2**)) have been synthesized. Compounds **1** and **2** were obtained under ionothermal conditions and possess a 2-D laminar $[M(\text{IBA})]_n$ layer based on $M_2(\text{CO}_2)_2$ building blocks with coordinated chloride completing tetrahedral coordination of the metal. Compound **1** exhibits two photoluminescence emission peaks at 410 and 510 nm. The low-energy emission can be attributed to ligand-centered transition and the high-energy band can be assigned to ligand-to-metal charge transfer between the aromatic systems of the organic ligands and the 4s orbitals of Zn(II). The role of the ionic liquid in ionothermal synthesis and crystallization of **1** and **2** are briefly discussed.

Keywords: Ionothermal synthesis; Zinc coordination polymer; Cobalt coordination polymer; 4-(1H-imidazol-1-yl) benzoic acid; Photoluminescence

1. Introduction

Ionic liquids (ILs) have received attention in separations, electrochemistry, lubrications, life sciences, and catalytically active solvents for synthetic chemistry [1]. ILs can exhibit properties such as low vapor pressure, excellent thermal stability, and wide electrochemical window [2,3]. ILs have significant advantages over volatile and possibly toxic organic solvents, which make them suitable green solvents [4]. IL with completely dissociated molecular cation and anion and a strong ionic atmosphere can provide opportunities for chemical synthesis that differ significantly from conventional solvents such as water, ethanol, and DMF. Ionothermal synthesis has been employed to prepare crystalline solids, in which IL participates as both solvent and structure-directing agent, resulting in interesting materials [5–7]. For example, the Kwon group has demonstrated that the final products are controlled by halide of ILs in the Ni-1,3,5-benzenetricarboxylic acid system [8]. Morris's

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group utilized an enantiopure anion as one component of the IL to induce homochirality in a nickel(II) structure constructed of entirely achiral building blocks, despite the fact that the anion of the IL is not occluded by the material [9]. These results suggested that structural properties of the final products may be traced directly to the chemistry of the ILs. Herein, we report ionothermal syntheses and crystal structures of two coordination polymers of $[M(\text{IBA})\text{Cl}]_n$ ($M=\text{Zn}$ (**1**) and Co (**2**)) incorporating the 4-(1H-imidazol-1-yl) benzoic acid (HIBA) ligand. Photoluminescence properties of **1** are discussed.

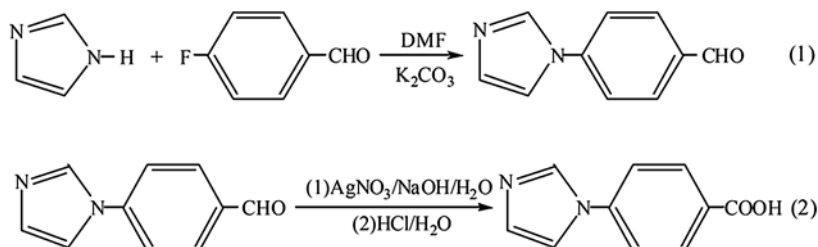
2. Experimental

2.1. Materials and instrumentation

All chemicals were obtained from commercial sources and used without purification. NMR analyzes were conducted on a Bruker Avance 400 spectrometer equipped with an automatic sample holder. Chemical shift data for each signal are reported in ppm units with DMSO as reference, where δ (DMSO) = 2.50 ppm. Elemental analyzes were carried out on an Elementar Vario EL III analyzer and IR spectra (KBr pellets) were recorded on a Perkin Elmer Spectrum One. Fluorescent spectra were measured at room temperature with a single-grating Edinburgh EI920 fluorescence spectrometer equipped with a 450 W Xe lamp, an nF900 lamp, and a R928P PMT detector. Thermogravimetric measurements were performed with a Netzsch STA449C apparatus under nitrogen with a heating rate of $10\text{ }^\circ\text{C min}^{-1}$ in Al_2O_3 containers.

2.2. Synthesis of HIBA

HIBA was prepared following a modified procedure (scheme 1) [10]. 0.2 ml tetrabutylammonium chloride was added dropwise to 20 mL of DMF containing a mixture of imidazole (4.54 g, 0.067 mol) and potassium carbonate (9.2 g, 0.067 mol) in a 50 mL three-neck flask under stirring. The reaction mixture was heated at $90\text{ }^\circ\text{C}$ for 5 min. Then, 4-fluorobenzaldehyde (8.3 g, 0.07 mol) was gradually added to a stirred solution of the reaction mixture. The resulting mixture was vigorously stirred at $90\text{ }^\circ\text{C}$ for 24 h to form a yellow precipitate. After cooling to room temperature, 100 mL ice water was added to the reaction mixture. The resulting yellow precipitate was collected by filtration and washed with Et_2O (10 mL). The product was dried at $60\text{ }^\circ\text{C}$ to afford 4-(imidazol-1-yl) benzaldehyde. 4-(Imidazol-1-yl) benzaldehyde (1.72 g, 0.01 mol) and AgNO_3 (3.4 g, 0.02 mol) were added to a



Scheme 1. Synthesis of HIBA.

60 mL 70% NaOH solution in a 150-mL flask. The reaction mixture was slowly heated to 60 °C and stirred for 24 h at this temperature. After cooling to room temperature, the insoluble material was removed by filtration and rinsed with H₂O (20 mL). The filtrate was acidified by addition of concentrated HCl to form a yellow precipitate; the yellow precipitate dissolved when further concentrated HCl was added. The final pH of the mixture is 2. Pale yellow crystals were obtained after one week. Yield: 1.45 g (56%). The crystal is formulated as its hydrochloride salt of HIBA·HCl·2H₂O [11]. ¹H NMR (400 MHz, DMSO-*d*₆, δ, ppm): 7.95 (d, 2H), 8.14 (d, 2H), 7.88 (s, 1H), 8.33 (s, 1H), and 9.73 (s, 1H). Anal. Calcd for C₁₀H₁₃ClN₂O₄ (Mr=260.67): C, 46.08; H, 5.03; N, 10.75%. Found: C, 45.95; H, 5.08; N, 10.62%. IR spectrum (cm⁻¹, KBr pellet): 3423 (m), 3122 (m), 1699 (s), 1639 (m), 1609 (vs), 1528 (s), 1493 (w), 1372 (w), 1331 (w), 1310 (s), 1254 (m), 1183 (w), 1116 (s), 1062 (s), 990 (w), 963 (m), 856 (w), 837 (w), 775 (m), 731 (m), 693 (w), 649 (w), 523 (w).

2.3. Synthesis of [Zn(IBA)Cl]_n (1)

Zn(NO₃)₂·6H₂O (0.0595 g, 0.2 mmol) and HIBA·HCl·2H₂O (0.0752 g, 0.29 mmol) were mixed with 0.6 g 1-ethyl-3-methylimidazolium tetrafluoroborate in a 20 mL Parr Teflon-lined stainless steel vessel. The vessel was sealed and heated to 160 °C. This temperature was kept for 5 days and then the mixture was cooled naturally to form colorless block crystals of **1**, which were washed with ethanol and dried in air (yield: 0.022 g, 38% on the basis of Zn). Anal. Calcd for C₁₀H₇ClN₂O₂Zn (Mr=288.00): C, 41.70; H, 2.45; N, 9.73%. Found: C, 41.64; H, 2.41; N, 9.71%. Main IR features (cm⁻¹, KBr pellet): 3448 (m), 3117(m), 3142(m), 1613 (vs), 1569 (m), 1526 (m), 1499 (w), 1420 (vs), 1384 (m), 1330 (w), 1305 (w), 1270 (w), 1250 (w), 1191 (m), 1126 (s), 1061 (s), 1012 (w), 966 (m), 947 (m), 862 (m), 847 (s), 821(w), 783 (m), 760 (m), 714(w), 693 (w), 648 (w), 625 (w), 539 (m).

2.4. Synthesis of [Co(IBA)Cl]_n (2)

Co(NO₃)₂·6H₂O (0.1164 g, 0.4 mmol) and HIBA·HCl·2H₂O (0.0376 g, 0.145 mmol) were mixed with 0.6 g 1-ethyl-3-methylimidazolium tetrafluoroborate in a 20 mL Parr Teflon-lined stainless steel vessel. The vessel was sealed and heated to 120 °C. This temperature was kept for 5 days and then the mixture was cooled naturally to form purplish red rhombus crystals of **2**, which were washed with ethanol and dried in air (yield: 0.014 g, 36% on the basis of HIBA·HCl·2H₂O). Anal. calcd for C₁₀H₇ClN₂O₂Co (Mr=281.56): C, 42.66; H, 2.15; N, 9.95%. Found: C, 42.69; H, 2.11; N, 9.89%. Main IR features (cm⁻¹, KBr pellet): 3440 (s), 3131 (s), 2452 (w), 1690 (m), 1640 (m), 1609 (s), 1561 (m), 1526 (m), 1496 (w), 1385 (s), 1305 (m), 1259 (m), 1123 (s), 1070 (s), 997 (m), 956 (s), 863(s), 785 (w), 694 (w), 621 (m), 539 (s), 517 (s).

2.5. Single-crystal structure determination

Single-crystal X-ray diffraction data of **1** and **2** were collected on a Bruker Apex II CCD diffractometer equipped with graphite-monochromated Mo-K α radiation (λ =0.71073 Å). Data reduction was performed using SAINT and corrected for Lorentz and polarization

Table 1. Summary of crystal data and structural refinements for **1** and **2**.

Compound	1	2
Empirical formula	C ₁₀ H ₇ ClN ₂ O ₂ Zn	C ₁₀ H ₇ ClN ₂ O ₂ Co
Formula weight	288.00	281.56
Space group	<i>P</i> 2 ₁ / <i>c</i>	<i>P</i> 2 ₁ / <i>c</i>
<i>a</i> (Å)	8.3766(6)	8.4401(6)
<i>b</i> (Å)	15.4529(11)	15.4308(11)
<i>c</i> (Å)	8.6948(6)	8.6186(6)
<i>α</i> (°)	90	90
<i>β</i> (°)	108.5050(10)	108.3290(10)
<i>γ</i> (°)	90	90
<i>V</i> /Å ³	1067.29(13)	1065.52(13)
<i>Z</i>	4	4
<i>D</i> _{calcd} /g cm ⁻³	1.792	1.755
<i>μ</i> /mm ⁻¹	2.534	1.844
GOF on <i>F</i> ²	1.007	1.003
<i>R</i> ₁ , <i>wR</i> ₂ [<i>I</i> > 2σ(<i>I</i>)]	0.0323, 0.0763	0.0279, 0.0737
<i>R</i> ₁ , <i>wR</i> ₂ (all data)	0.0542, 0.0860	0.0336, 0.0776

Note: $R_1 = \sum ||F_o| - |F_c|| / \sum |F_o|$, $wR_2 = \{\sum w[(F_o)^2 - (F_c)^2]^2 / \sum w(F_o)^2\}^{1/2}$.

effects. Adsorption corrections were applied using SADABS [12]. The structures were solved by direct methods and successive Fourier difference syntheses and refined by full-matrix least-squares on *F*² (SHELXTL Version 5.1) [13]. All non-hydrogen atoms are refined with anisotropic thermal parameters. Hydrogens were assigned to calculated positions. The *R*₁ values are defined as $R_1 = \sum ||F_o| - |F_c|| / \sum |F_o|$ and $wR_2 = \{\sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2]\}^{1/2}$. The details of the crystal parameters, data collection, and refinement are summarized in table 1. More details about the crystallographic data have been deposited as Supplementary material.

3. Results and discussion

3.1. Syntheses

IL comprising dissociated molecular cation and anion with a strong ionic atmosphere participates as both solvent and structure-directing agent in ionothermal reactions. Compounds **1** and **2** were obtained from 1-ethyl-3-methylimidazolium tetrafluoroborate IL solvent and are structurally distinct from [Zn(IBA)₂]_{*n*} and [Co(IBA)₂]_{*n*} synthesized under hydrothermal conditions [14,15]. Clearly, use of IL causes the observed differences. IL can play multiple roles in ionothermal syntheses and crystallizations [16]. In the present case, neither cation nor anion of IL is incorporated in **1** and **2**, indicating 1-ethyl-3-methylimidazolium tetrafluoroborate IL serves only as a solvent. Many compounds with incorporation of cations or anions of the ILs have been documented [17–19], commonly resulting in ionic compounds. Both components of ILs can be incorporated in the structures [20].

3.2. Structure description for [M(IBA)Cl]_{*n*} (M=Zn(**1**) and Co(**2**))

Compounds **1** and **2** crystallize in the monoclinic *P*2₁/*c* space group and are a 2-D laminar grid network based on dinuclear M₂(CO₂)₂ (M=Zn(**1**) and Co(**2**)) units. Compounds **1** and

2 are isomorphic, so only the structure of **1** is discussed here representatively. The asymmetric unit of **1** contains one Zn(II), one IBA⁻, and one Cl⁻. As depicted in figure 1, Zn is four-coordinate by two carboxylate oxygens (O1 and O2A, symmetry code: A $-x + 3, -y + 1, -z + 1$) from two IBA⁻, one nitrogen of a IBA⁻, and one chloride in a distorted tetrahedral geometry with a long Zn–Cl bond, 2.2120(9) Å. The Zn–O bond lengths are 1.9457(19) and 1.9546(18) Å and the Zn–N bond length is 2.005(2) Å, comparable with values in related zinc compounds [21–24]. Coordinated chloride is derived from HIBA·HCl·2H₂O. As shown in figure 2, a pair of Zn ions related by an inversion center are bridged by two carboxylates to generate a dinuclear Zn₂(CO₂)₂ unit with a Zn···Zn separation of 3.596 Å. Such a Zn···Zn separation does not indicate any significant direct interactions between the metals and is constrained by the bridging geometry. The HIBA⁻ is tridentate, bridging three zinc ions through the bidentate carboxylate and the imidazole

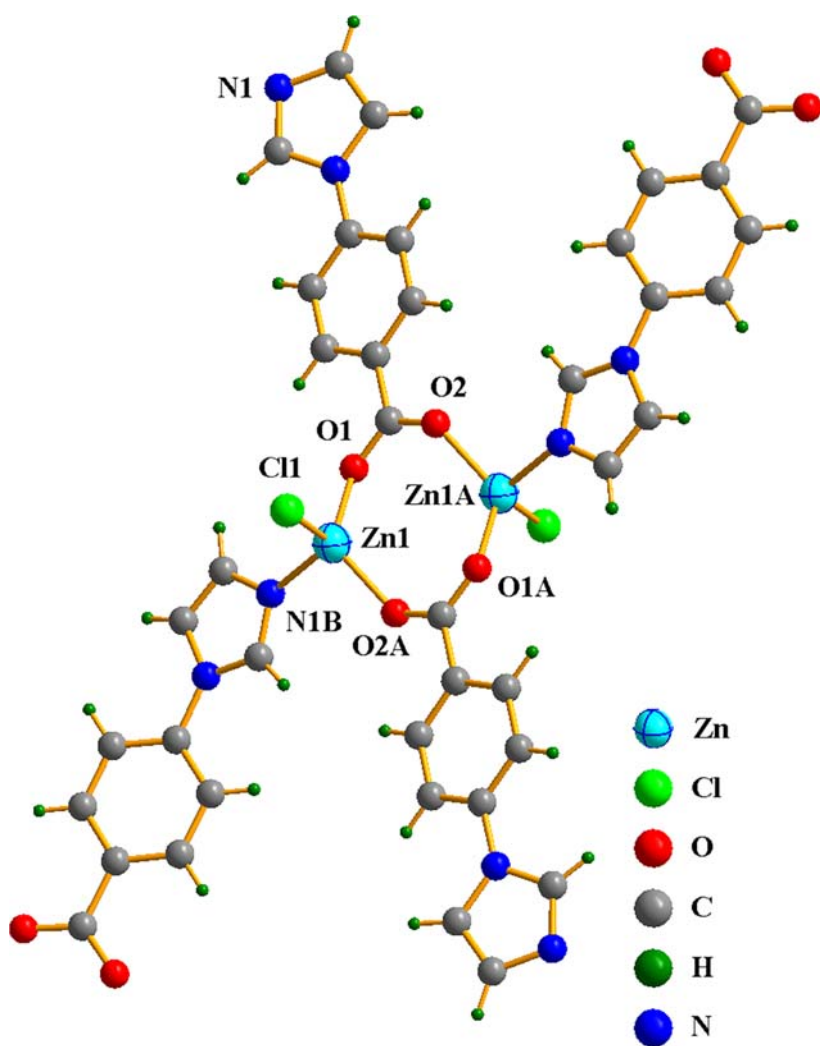


Figure 1. Molecular structure of **1**. Symmetry codes for the generated atoms: A. $-x + 3, -y + 1, -z + 1$; B. $-x + 2, y + 1/2, -z + 3/2$.

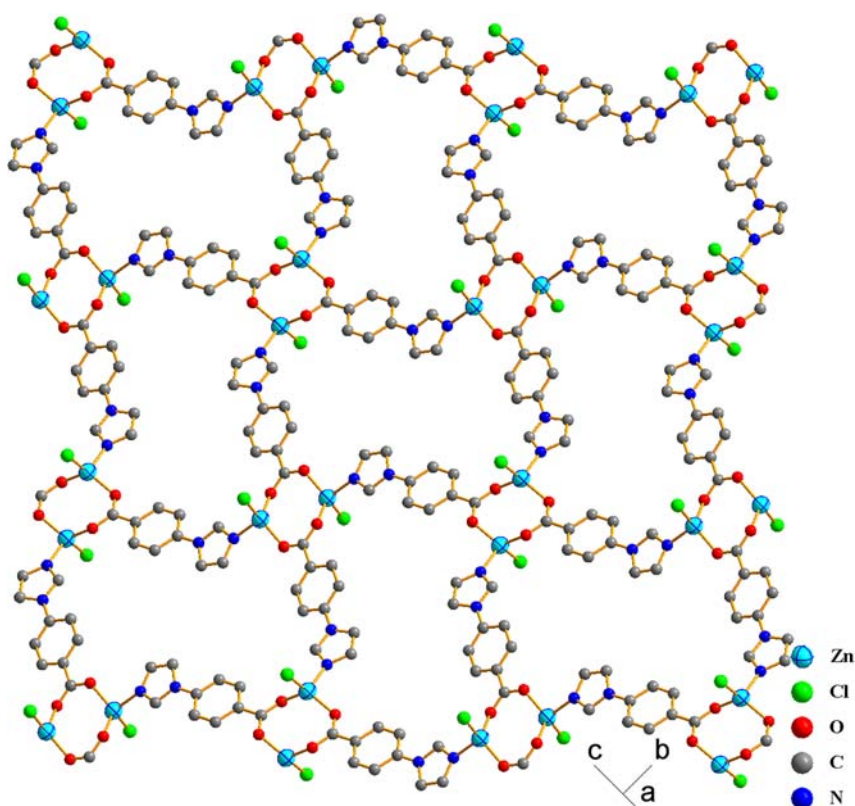


Figure 2. View of the 2-D layer of **1**.

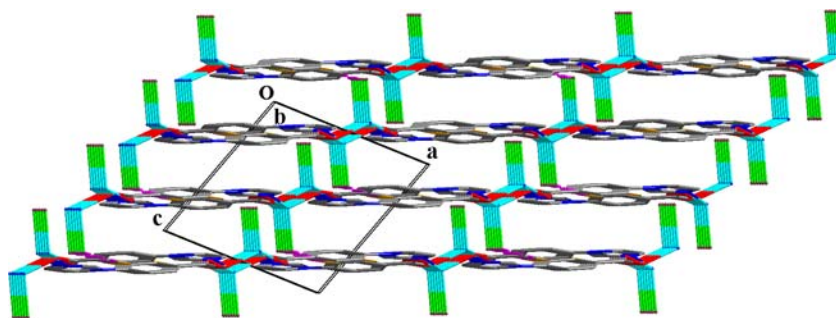


Figure 3. The 2-D layers linked by C3-H3A \cdots Cl11 hydrogen bonds to form a 3-D structure in **1**. Hydrogen bonds are represented by dashed lines.

nitrogen. Each $\text{Zn}_2(\text{CO}_2)_2$ is linked to four adjacent neighbors in two orthogonal directions through IBA^- generating a 2-D network, as shown in figure 2. The 2-D network features metal-organic rectangular grids with the rectangular cavity size of $12.1 \times 11.5 \text{ \AA}$. The layers in **1** are assembled into a 3-D structure via weak interlayer C3-H3A \cdots Cl11 hydrogen bonds with a C \cdots Cl distance of $3.617(2) \text{ \AA}$ (figure 3).

The unique physicochemical properties of the IL lead to framework structures that are different from those obtained by more extensively utilized hydro(solvo)thermal methods. Compound **1** obtained from ionothermal reaction is much different from $[\text{Zn}(\text{IBA})_2]_n$ synthesized under hydrothermal conditions [14]. $[\text{Zn}(\text{IBA})_2]_n$ crystallizes in a chiral space group $P4_122$ and has a twofold interpenetrated 2-D network, in which each zinc is tetrahedrally coordinated by four bidentate IBA^- to form a 2-D bilayer network based on single metal centers, much different from the flat laminar 2-D layer of **1** based on dinuclear zinc units. The zinc ion in **1** is also four-coordinate but surrounded by three IBA^- and one chloride, which is nearly perpendicular to the flat 2-D layer of $[\text{Zn}(\text{IBA})_2]_n$. Chloride plays a significant role in formation of the final 2-D layered structure of **1**. The same situation exists for **2**, which is different from the structure of $[\text{Co}(\text{IBA})_2]_n$ synthesized through hydrothermal method [15]. Reaction of an analog of HIBA, 2-(4,5-diphenyl-1H-imidazol-2-yl)benzoic acid, bearing large substituent groups with zinc ion afforded a mononuclear zinc structure [25].

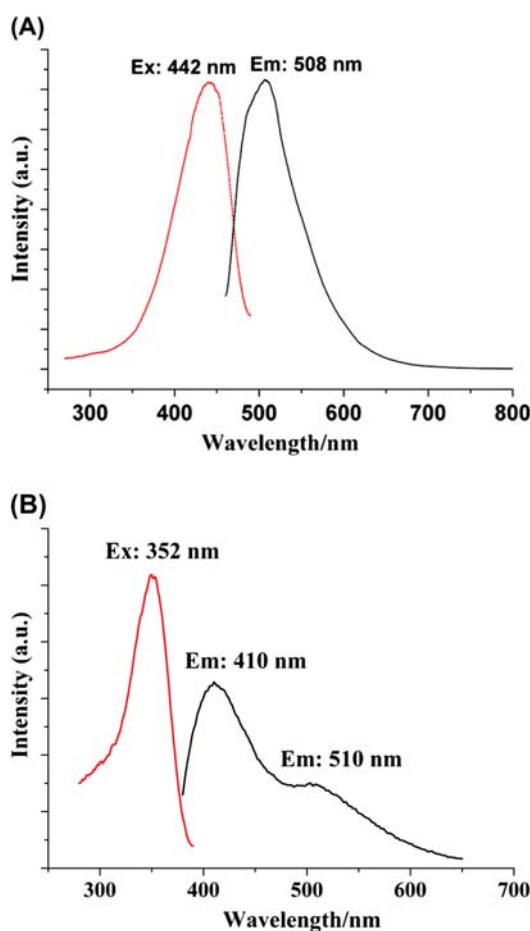


Figure 4. Room-temperature excitation and emission spectra for HIBA (A) and **1** (B) in the solid state.

3.3. Photoluminescence properties of **1**

Coordination polymers are promising photoluminescent materials because of their high thermal stability, and structure- and metal-dependent emission. The d^{10} metal compounds have interesting photoluminescent properties [26,27]. Photoluminescent properties of **1** and the free ligand have been investigated in the solid state at room temperature. As shown in figure 4(A), HIBA displays a photoluminescent emission at 508 nm upon excitation at 442 nm, which is green. The main chromophore of HIBA is the five-membered imidazole ring and six-membered benzene ring. This emission observed for free ligand can be assigned to typical ligand-centered $\pi^* \rightarrow \pi$ transition. Under UV radiation ($\lambda_{\text{ex}} = 352$ nm) at room temperature, solid **1** displays a broad emission band with emission maxima at 410 nm and with a shoulder at 510 nm (figure 4(B)), resulting in an overall emission color of bluish green. The low-energy emission centered at 510 nm corresponds to ligand-centered emission. The high-energy emission centered at 410 nm can be attributed to ligand-to-metal charge transfer (LMCT) between aromatic π systems and the 4s orbitals of zinc (II) centers. This is consistent with Zn(II) coordination complexes with aromatic ligands possessing an LMCT photoluminescence [28,29].

3.4. IR and TGA Studies

IR spectra of $[\text{M}(\text{IBA})\text{Cl}]_n$ show the typical asymmetric (1613 and 1569 cm^{-1} for **1** and 1609 and 1561 cm^{-1} for **2**, respectively) and symmetric (1420 cm^{-1} and 1385 cm^{-1} for **1** and **2**, respectively) stretches of carboxylate. To examine the thermal stability of **1** and its structural variation as a function of temperature, thermogravimetric analysis (TGA) was performed on crystalline samples of **1**. TGA curve shows that **1** is stable to 400 $^\circ\text{C}$, and then, the framework starts to collapse (Supplementary material).

4. Conclusions

We present two coordination polymers synthesized under ionothermal reaction. $[\text{M}(\text{IBA})\text{Cl}]_n$ is a 2-D laminar layer based on $\text{M}_2(\text{CO}_2)_2$ building blocks. This article demonstrates that the ionothermal reaction and chloride facilitate formation of the present laminar layer of $[\text{M}(\text{IBA})\text{Cl}]_n$. More experimental data are needed to fully understand how IL affects the reaction outcome in ionothermal synthesis. In addition, the thermal stability and the photoluminescent properties of **1** have been investigated.

Acknowledgement

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