Syntheses, crystal structures, and fluorescent properties of three coordination polymers with 5-sulfoisophthalic acid anion and 4-phenylpyridine

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Abstract

Three coordination polymers—\([\text{Ag}_2(\text{SIP})(\text{4-pp})]\cdot\text{H}_2\text{O}]_n, [\text{Pb}_4(\text{SIP})_2(\mu_3-\text{OH})_2(\text{H}_2\text{O})]_n,\) and \([\text{Zn}_2(\text{SIP})_2(\text{4-pp})_4]([\text{Zn}(\text{4-pp})_2(\text{H}_2\text{O})_4]\cdot4\text{H}_2\text{O}]_n\) (SIP = 5-sulfoisophthalic acid anion, 4-pp = 4-phenylpyridine)—are constructed under hydrothermal or solvothermal conditions using sodium m-phthalate 5-sulfonate (NaH_2SIP) and 4-phenylpyridine as ligands. The structures of the three complexes are determined by single-crystal X-ray diffraction, powder X-ray diffraction, thermogravimetric analyses, Fourier-transform infrared analysis, and photoluminescence. The fluorescent properties of the complexes in the solid state are investigated at room temperature, and the results indicate that they all show photoluminescent properties.

Keywords

4-phenylpyridine, 5-sulfoisophthalic acid, coordination polymers, fluorescent properties, single-crystal X-ray diffraction

Introduction

The design and construction of coordination polymers (CPs) have been a very active area of materials research in recent years. The intense interest in these materials is driven by their potential applications as functional materials for luminescence sensing, gas storage and separation, drug delivery, catalysis, magnetism, and due to their intriguing structural diversity.1–6 It is well-known that many factors such as synthetic methods, central metals, ligands, and pH can influence the final structures of CPs.7–10 There is no doubt that organic ligands and the metal center play a significant role in the design and synthesis of CPs, while selecting the coordination geometry of the metal centers and organic ligands containing appropriate functional groups (such as polycarboxylic acid or multipyridine ligands) are also key for constructing CPs. Due to the high coordination numbers and flexible coordination geometries of transition-metal ions, they are able to produce a variety of structures. Moreover, the properties of CPs are highly dependent on the structures of the free ligands and the nature of the metal ions. The assembly of these metal–organic CPs allows for a wide choice of different parameters, including diverse electronic properties. A wide variety of CPs with interesting structures and desirable properties have been prepared through judicious choice of organic linkers and metal ions.

Recently, many CPs have been synthesized by employing polycarboxylate aromatic ligands because of their versatile coordination modes and thermal stability.11–15 Sodium m-phthalate 5-sulfonate (NaH_2SIP) has inspired significant research efforts toward assembling coordination architectures. The approach to the supramolecular frameworks employed in this work involves the use the trifunctional linker NaH_2SIP for the following reasons: it contains two carboxylate groups and one sulfonate group and is one of the most useful multifunctional ligands due to (1) structural rigidity, (2) strong bonding interactions, and (3) a rich diversity of coordination modes that can be utilized for extension of the metal ions into high-dimensional structures.16 The sulfonate group is generally perceived as a weaker group with respect to its coordinating ability, and it has one more potentially coordinating oxygen atom. The weak coordination nature of sulfonate makes its coordination mode very...
flexible. In addition, the sulfonate group is a suitable hydrogen-bond acceptor. It can also be used as short bridge and long bridge between metal centers to construct a variety structures. It can be partially or completely deprotonated, and normally serves as a trigonal-shaped connector to construct diverse metal-supramolecular systems via different coordination and hydrogen-bonding modes.\textsuperscript{17–22} 

\[\pi-\pi\] stacking interactions between the aromatic rings may facilitate ordered, non-interpenetrated open frameworks. In this work, three complexes based on 5-sulfoisophthalic acid and 4-phenylpyridine—namely, \([\text{Ag}_2(\text{SIP})(4-\text{pp})]\cdot[\text{Ag}(4-\text{pp})_2]\cdot\text{H}_2\text{O}]_n\) (1), \([\text{Pb}_4(\text{SIP})_2(\mu_3-\text{OH})(\text{H}_2\text{O})_2]_n\) (2), and \([\text{Zn}_2(\text{SIP})_2(4-\text{pp})_4][\text{Zn}(4-\text{pp})_2(\text{H}_2\text{O})_4]\cdot4\text{H}_2\text{O}]_n\) (3)—have been synthesized. The thermogravimetric analyses (TGA) and luminescent properties of CPs 1–3 have been investigated in detail.

Results and discussion

Description of structure 1

Single-crystal X-ray structural analysis reveals that the asymmetric unit of complex 1 contains one positive and one negative ion units and one lattice water molecule. The unit consists of three Ag cations, one SIP\textsuperscript{3−}, three 4-pp ligands, and one free water molecule (Figure 1(a)). Ag(1) and Ag(2) lie in a three-coordinated environment, while the Ag(3) ion lies in a two-coordinated environment. The Ag(1) ion is coordinated by three oxygen atoms from three 5-sulfoisophthalic anions. The Ag–O bond lengths range from 2.146(1) Å to 2.432(1) Å (Table 1). The Ag(1) and Ag (1A) ions form an eight-membered ring via two SIP\textsuperscript{3−} carboxyl groups. The Ag(2) ion is coordinated by one nitrogen atom from the 4-pp ligand and two oxygen atoms from two SIP\textsuperscript{3−} units. In addition, the Ag(3) ion is coordinated by two nitrogen atoms from 4-pp ligands. As shown in Figure 1(a), in the eight-membered rings, the Ag ions interact with each other and exhibit a short Ag–Ag interaction of 2.8528(10) Å and 2.9853(4) Å. The Ag ions are linked to one-dimensional (1D) chain structure through the coordination of 5-sulfoisophthalic bridges (Figure 1(b)). The 1D chain structures are linked via the sulfonate ions of SIP\textsuperscript{3−} to give a two-dimensional (2D) structure (Figure 1(c)).

Description of structure 2

Single-crystal X-ray diffraction analysis reveals that complex 2 crystallizes in the triclinic space group \(P\overline{1}\). The asymmetric unit of complex 2 contains two Pb(II) ions, one 5-sulfoisophthalic acid anion, one water molecule, and one hydroxy ion (Figure 2(a)). The six-coordinated Pb(1) ion adopts a distorted octahedral geometry surrounded by three oxygen atoms from two 5-sulfoisophthalic acid anions (Figure 2(b)), one oxygen from one coordinated water molecule, and two oxygens from two hydroxy ions. The Pb–O distances vary from 2.6465(2) Å to 2.7026(2) Å (Table 2). The five-coordinated Pb(2) ion is coordinated by three oxygen atoms from two SIP\textsuperscript{3−} anions, one oxygen from one coordinated water molecule, and one oxygen from one hydroxy group. The Pb–O bond distances range from 2.3995(2) Å to 2.7588(2) Å. The coordination geometry around the Pb(2) ion may be regarded as midway between square pyramidal and trigonal bipyramidal as described by the \(\tau\) parameter of 0.59. In complex 2, the Pb\textsuperscript{2+} ions are bridged by SIP\textsuperscript{3−} to give a 2D layer structure (Figure 2(c)).

Description of structure 3

X-ray diffraction analyses revealed that 3 crystallizes in the triclinic space group \(P\overline{1}\). The basic structural unit of 3 contains three Zn(II) ions, two SIP\textsuperscript{3−} anions, six 4-pp ligands, four coordinated water molecules, and four free water molecules. The oxygen atoms from SIP\textsuperscript{3−} adopt bidentate bridging and monodentate modes, and the nitrogen atoms from the 4-pp adopt a monodentate pattern. As shown in...
Figure 3(a), the central Zn(1) ion is coordinated by three oxygen atoms (O1, O2A, and O3A) from three SIP3− anions and two nitrogen atoms (N1 and N2) from the 4-pp, respectively. The coordination geometry around the Zn(1) ion can be regarded as an intermediate between square pyramidal and trigonal bipyramidal \( (\tau = 0.55) \). The central Zn(2) ion is coordinated by two nitrogen atoms (N3 and N3A) from two 4-pp units and four water molecules (O8, O9, O8A, and O9A). The Zn(2) is six-coordinated in a distorted octahedral configuration. Figure 3(b) shows the coordination pattern of the Zn ions. The central Zn ions form a 1D chain structure through the tridentate bridge of SIP3− (Figure 3(c)). The 1D chain structures extend into a three-dimensional (3D) framework through π–π stacking, intermolecular hydrogen bonding, intermolecular van der Waals interactions, and other weak interactions. Selected bond lengths and angles are listed in Table 3. Selected hydrogen bonds and angles are listed in Table 4.

**IR spectra**

IR spectroscopy is an excellent approach to characterize and study crystallization, and hence, the IR spectra of complexes 1–3 in the 4000–500 cm\(^{-1} \) region have been studied (Figure 4). The peaks at 3477, 3491, and 3406 cm\(^{-1} \) in the three compounds can be assigned to the \( \nu_{\text{O–H}} \) stretching vibrations, suggesting the presence of water molecules. Strong characteristic bands are observed in the ranges of 1602, 1596, and 1608 cm\(^{-1} \), while the absence of the expected absorption bands at around 1700 cm\(^{-1} \) for the protonated carboxyl group indicates that all the carboxyl groups of SIP3− have been deprotonated. Skeletal vibrations for the phenyl and N-heterocyclic rings appear at 617, 621, and 765 cm\(^{-1} \).

**Powder X-ray diffraction and thermal analyses**

The Powder X-ray diffraction (PXRD) analyses of the synthesized crystalline samples of 1–3 are in good agreement with the simulated patterns, demonstrating the phase purity of the synthesized products (Figure 5).

TGA were carried out for complexes 1–3 in order to characterize the polymers more fully in terms of thermal stability. For complex 1, a weight loss of 1.78% corresponding to the release of a water molecule is observed from room temperature to 172 °C (calcd 1.71%) (Figure 6). On further heating, the framework of complex 1 starts to collapse. For complex 2, a weight loss of 3.1% corresponding to the release of water molecules and hydroxy ions is observed from room temperature to 113 °C (calcd 2.9%).

**Table 1.** Selected bond lengths (Å) and angles (°) for complex 1.

| Bond                  | Length (Å) | Angle (°)     | Symmetry Codes |
|-----------------------|------------|---------------|----------------|
| Ag1-O6                | 2.432(1)   |               | A: 4−X, 1−Y, −Z; B: 1+X, +Y, +Z; C: 3−X, 1−Y, −Z. |
| Ag2-O2A               | 2.231(2)   |               |                |
| O4B-Ag1-O6            | 95.735(27) |               |                |
| Ag1-O3C               | 2.146(1)   |               |                |
| Ag3-N1                | 2.150(1)   |               |                |
| O2-Ag2-O1A            | 162.413(38)|               |                |
| Ag1-O4B               | 2.162(1)   |               |                |
| Ag3-N2                | 2.155(2)   |               |                |
| O2-Ag2-N3             | 106.121(16)|               |                |
| Ag2-O2               | 2.211(2)   |               |                |
| O3C-Ag1-O4B           | 160.61(2)  |               |                |
| O1A-Ag2-N3            | 91.113(21) |               |                |
| Ag2-N3                | 2.351(1)   |               |                |
| O3C-Ag1-O6            | 103.597(13)|               |                |

Symmetry codes: A: 4−X, 1−Y, −Z; B: 1+X, +Y, +Z; C: 3−X, 1−Y, −Z.
On further heating, the framework of complex 2 starts to collapse. The TGA curve of 3 shows two main weight loss steps. The first weight loss of 3.94% from room temperature to 70 °C corresponds to the release of lattice water molecules (calcd 4.13%). The second weight loss of 3.94% from 70 to 180 °C is attributed to the removal of coordinated water molecules (calcd 4.0%). On further heating, the framework gradually decomposed.

**Luminescent properties**

It is well-known that CPs constructed with a d10 metal center and conjugated organic linkers are promising candidates for photoactive materials,23–27 with potential applications such as chemical sensors and electro-luminescent materials. Therefore, the photoluminescence (PL) properties of the three complexes, together with the free ligands, are investigated at room temperature for comparison (Figure 7). The free NaH2SIP ligand exhibits a broad weak luminescent emission band with one emission peak at 310 nm ($\lambda_{ex} = 262.4$ nm). The 4-pp ligand exhibits a broad weak luminescent emission band with one emission peak at 320 nm ($\lambda_{ex} = 264.4$ nm) that can be ascribed to the $\pi^*\rightarrow n$ or $\pi^*\rightarrow \pi$ transitions, as previously reported.28,29 Complexes 1 and 2 have weak emission bands at 399 nm ($\lambda_{ex} = 359$ nm) for 1 and 384.4 nm ($\lambda_{ex} = 352$ nm) for 2, respectively. Noticeably, complex 3 has a stronger emission at 380 nm ($\lambda_{ex} = 300$ nm), and has a significant luminescence enhancement compared with the other tested complexes. This can be attributed to the coordination interactions between the ligands and the metal Zn center, which may increase the conjugational rigidity and decrease the non-radiative energy loss of the entire system. On comparison with SIP3− and 4-pp ligands, there appears to be differences in the degree of red-shifts for the emission bands of three complexes. Since the d10 metal ions are difficult to oxidize or reduce, the emission bands of the complexes may be attributed to ligand-to-ligand charge transfer (LLCT),30,31 which is neither metal-to-ligand charge transfer (MLCT) nor

**Table 2.** Selected bond lengths (Å) and angles (°) for complex 2.

| Bond Lengths | Angle Degrees |
|--------------|---------------|
| Pb1-O9       | 2.3452(2)     | O9-Pb1-O7 77.849(5) | O6-Pb1-O5 66.445(4) |
| Pb1-O6       | 2.6465(2)     | O9-Pb1-O6 72.605(4) | O6-Pb2-O1B 81.804(4) |
| Pb1-O7       | 2.7026(2)     | O9-Pb1-O5A 73.130(5) | O6-Pb2-O1C 77.260(4) |
| Pb1-O5A      | 2.6936(2)     | O9A-Pb1-O8 84.315(4) | O6-Pb2-O2C 90.081(5) |
| Pb1-O8       | 2.6700(2)     | O9A-Pb1-O7 141.673(6) | O6-Pb2-O9 75.388(5) |
| Pb2-O9       | 2.3427(2)     | O9A-Pb1-O6 130.230(5) | O1B-Pb2-O1C 71.109(4) |
| Pb2-O1C      | 2.7068(2)     | O9A-Pb1-O5A 73.510(5) | O1B-Pb2-O2C 119.085(5) |
| Pb2-O2C      | 2.3993(2)     | O8-Pb1-O7 79.094(4) | O1B-Pb2-O9 154.441(5) |
| Pb2-O1B      | 2.7588(2)     | O8-Pb1-O6 126.930(5) | O1C-Pb2-O2C 48.325(4) |
| Pb2-O6       | 2.5025(2)     | O8-Pb1-O5A 156.516(6) | O2C-Pb2-O9 72.873(5) |
| O9-Pb1-O9A   | 68.340(4)     | O7-Pb1-O6 48.395(4) | O8-Pb1-O5A 113.863(6) |
| O9-Pb1-O8    | 91.780(5)     |                       |                     |

Symmetry codes: A: 1 − X, − Y, − Z; B: 1 − X, 1 − Y, 1 − Z; C: 1 + X, −1 + Y, + Z.

![Figure 3.](image-url)
results show that the central metal atoms make significant contributions to the structural diversities of resulting complexes. Moreover, the variations in the coordination numbers and the atomic radii of the central metal atoms also have an impact on the final products. In addition, we have studied the luminescent properties of the complexes, with the results indicating that the three complexes show PL properties, indicating that ligand-to-metal charge transfer (LMCT) in nature. The differences in emissions of the three complexes are probably due to the differences in the coordination environments of the central metal ions.

**Conclusion**

In summary, three new metal complexes—[[Ag$_2$(SIP)(4-pp)]][Ag(4-pp)$_2$]·H$_2$O]$_n$, [Pb$_2$(SIP)$_2$(μ$_3$-OH)$_3$]$_2$H$_2$O]$_n$, and [[Zn$_2$(SIP)$_2$(4-pp)$_4$]·[Zn(4-pp)$_2$(H$_2$O)$_4$]·4H$_2$O]$_n$—have been synthesized and characterized. These complexes display intriguing and versatile coordination features with 3D frameworks. The structural differences show that the central metal atoms make significant contributions to the structural diversities of resulting complexes. Moreover, the variations in the coordination numbers and the atomic radii of the central metal atoms also have an impact on the final products. In addition, we have studied the luminescent properties of the complexes, with the results indicating that the three complexes show PL properties, indicating that they are potential candidates for luminescent materials. The obtained results show that NaH$_2$SIP is a useful ligand that can direct the assembly of metal–organic materials.

**Experimental**

**Reagents and physical measurements**

All chemicals for the syntheses were purchased from commercial sources (Aldrich, Aladdin, or Alfa Aesar) and were used as received without further purification. The structures of the complexes were confirmed by G8910A CCD single-crystal X-ray diffraction analyses. Infrared spectra were obtained using a PerkinElmer spectrometer. PL spectra were acquired on an RF-5301 fluorescence spectrophotometer (4000–400 cm$^{-1}$). TGA were performed on a TGA Q600 Thermogravimetric Analyzer and heated from room temperature to 800°C at a rate of 10°C min$^{-1}$. The PL spectra for the solid samples were measured at room temperature.

**Synthesis of complexes 1–3**

1. A mixture of NaH$_2$SIP (0.0268 g, 0.1 mmol), NaOH (0.008 g, 0.2 mmol), Ag(NO)$_3$ (0.0170 g, 0.1 mmol), and 4-phenylpyridine (0.0156 g, 0.1 mmol) in distilled water (10 mL) was stirred for 10 min at room temperature. The resulting solution was sealed in a 25-mL Teflon-lined stainless steel container, which was then heated at 120°C for 3 days. After the sample had cooled to room temperature at 10°C h$^{-1}$, colorless rod-shaped crystals were isolated. Yield: 55% (based on Ag). FTIR: (KBr, v, cm$^{-1}$): 3477(w), 1597(s), 1536(s), 1435(s), 1359(s), 1198(m), 1039(s), 764(s), and 617(s). Anal. calcd for C$_{41}$H$_{32}$Ag$_3$N$_3$O$_8$S (%): C, 46.84; H, 0.86, S, 4.62; found: C, 46.84; H, 0.89; S, 4.67.

2. Colorless lump-shaped crystals were obtained following the same method used for the preparation of complex 1 and using Pb(NO)$_3$ instead of Ag(NO)$_3$. Yield: 35% (based on Pb). FTIR: (KBr, v, cm$^{-1}$): 3491(w), 1597(s), 1536(s), 1435(s), 1359(s), 1198(m), 1112(s), 1047(s), 725(s), and 617(s). Anal. calcd for C$_{41}$H$_{26}$Ag$_3$N$_3$O$_8$S (%): C, 46.84; H, 0.86; S, 4.62; found: C, 46.88; H, 0.89; S, 4.67.

3. A mixture of NaH$_2$SIP (0.0536 g, 0.2 mmol), 4-pp (0.0310 g, 0.2 mmol), and Zn(NO)$_3$·6H$_2$O (0.0595 g, 0.2 mmol) was added to a mixed solvent system of ethanol (3 mL) and H$_2$O (10 mL). The solution was kept at 120°C for 3 days. Colorless needle-shaped crystals were obtained. Yield: 43% (based on Zn). FTIR: (KBr, v, cm$^{-1}$): 3403(s), 1614(s), 1556(s), 1422(m), 1366(w), 1224(s), 1196(m), 1044(m), 766(m), 696(m), and 625(m). Anal. calcd for C$_{16}$H$_{12}$O$_{18}$Pb$_4$S$_2$ (%): C, 13.86; H 0.86, S, 4.62; found: C, 13.82; H, 0.89; S, 4.67.
S₂Zn₃ (%): C, 53.78; H, 4.59; N, 4.59; S, 3.50; found: C, 53.79; H, 4.53; N, 4.55; S, 3.52.

Crystal structure determinations

The crystal and molecular structures of the complexes were determined by single-crystal X-ray diffraction. Diffraction intensities for the complexes were collected at 298(2) K using an Agilent G8910A CCD area-detector diffractometer with MoKα (λ = 0.71073 Å) radiation. Using Olex2, the structure was solved with the SHELXL refinement package using full-matrix least-squares refinement techniques. All hydrogens were added theoretically. Crystal data as well as details of the data correction and refinement for the three complexes are summarized in Table 5.

Declaration of conflicting interests

The author(s) declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.
**Table 5. Crystallographic and experimental data for three complexes 1–3.**

| Complex | 1 | 2 | 3  |
|---------|---|---|----|
| Empirical formula | \( \text{C}_4\text{H}_3\text{Ag}_3\text{N}_3\text{O}_8\text{S} \) | \( \text{C}_1\text{H}_2\text{O}_8\text{Pb}_4\text{S}_2 \) | \( \text{C}_3\text{H}_4\text{N}_6\text{O}_2\text{Zn}_3 \) |
| Formula weight | 1050.37 | 1385.14 | 1829.78 |
| Crystal system | Triclinic | Triclinic | Triclinic |
| Space group | \( \text{P} \) | \( \text{P} \) | \( \text{P} \) |
| \( a \) (Å) | 9.8425(10) | 6.9328(6) | 10.1420(5) |
| \( b \) (Å) | 11.752(2) | 8.3234(7) | 12.6217(7) |
| \( c \) (Å) | 18.115(10) | 10.8413(8) | 18.7215(12) |
| \( \alpha \) (°) | 101.85(3) | 90.693(5) | 70.462(5) |
| \( \beta \) (°) | 93.52(3) | 107.775(7) | 87.406(5) |
| \( \gamma \) (°) | 107.52(13) | 91.040(7) | 73.131(5) |
| \( V \) (Å³) | 1938.5(12) | 595.53(8) | 2157.8(2) |
| \( Z \) | 2 | 2 | 1 |
| \( F \) | 1040 | 612 | 960 |
| \( D_\text{c} \) (g cm\(^{-3}\)) | 1.800 | 3.862 | 1.428 |
| \( \mu \) (mm\(^{-1}\)) | 1.612 | 28.452 | 0.957 |
| 2\( \theta \) range (°) | 6.44–52.74 | 7.792–58.25 | 6.736–49.99 |
| Obs. Ref. (\( I > 2\sigma(I) \)) | 13,213 | 3838 | 12,832 |
| \( R_m \) | 0.0307 | 0.0985 | 0.0414 |
| \( R_b \) (\( I > 2\sigma(I) \)) | 0.0448 | 0.1196 | 0.0759 |
| \( wR_2 \) (all data) | 0.1018 | 0.3230 | 0.2448 |
| Gof | 1.055 | 1.289 | 0.981 |

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