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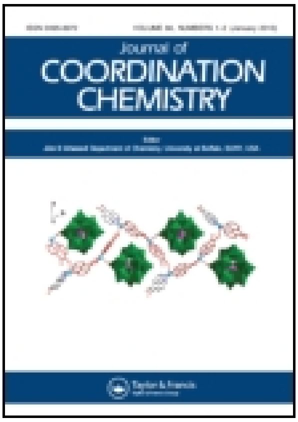

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Syntheses, structures and luminescence of three cadmium coordination polymers based on 1,2-bis(1,2,4-triazol-1-ylmethyl)benzene

Shan Zhao [a] , Yuan Yang [a] , Zhi Yang [a] , Bao-Long Li [a] & Yong Zhang [a]

[a] Key Laboratory of Organic Synthesis of Jiangsu Province , College of Chemistry, Chemical Engineering and Materials Science, Soochow University , Suzhou , P.R. China

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Syntheses, structures and luminescence of three cadmium coordination polymers based on 1,2-bis(1,2,4-triazol-1-ylmethyl)benzene

SHAN ZHAO, YUAN YANG, ZHI YANG, BAO-LONG LI* and YONG ZHANG

Key Laboratory of Organic Synthesis of Jiangsu Province, College of Chemistry, Chemical Engineering and Materials Science, Soochow University, Suzhou, P.R. China

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Three coordination polymers, $[Cd(obtz)(NCS)_2]_n$ (**1**), $[Cd(obtz)_2(dca)_2]_n$ (**2**), and $\{[Cd(obtz)_2(H_2O)_2]\cdot(SiF_6)\}_n$ (**3**), were synthesized using the flexible 1,2-bis(1,2,4-triazol-1-ylmethyl)benzene (obtz). In **1**, obtz connects the $[Cd(\mu\text{-NCS-}N{:}S)_2]_n$ 2-D networks into an unusual 3-D cubic network. **2** is comprised of neutral 1-D double chains. Each chain forms π–π stacking interactions with four adjacent chains (two above and two below) to extend a 3-D supramolecular network. **3** consists of 1-D cation chains $[Cd(obtz)_2(H_2O)_2]_n$ and SiF_6^{2-}. Three-dimensional supramolecular architecture is formed through hydrogen bond interactions between the coordination waters and uncoordinated 2-position nitrogens of obtz or SiF_6^{2-}. Luminescence and thermal stabilities of **1**, **2,** and **3** were measured.

Keywords: Coordination polymer; Cd(II) complex; Luminescence; Structure; Bis(1,2,4-triazol-1-ylmethyl)benzene

1. Introduction

Coordination polymers are of interest for potential applications as functional materials and intriguing topologies [1–5]. To achieve intriguing topologies and functional materials, the crucial step is to employ appropriate organic building blocks and metal ions. Contrary to rigid spacers, flexible ligands adopt different conformations according to geometric needs of different metal ions, affording coordination polymers with novel topologies and properties [6–10]. Anions serve more than merely balancing charges of a cationic complex, influencing the structure of a supramolecular system through coordination [11–15].

The N-containing ligands containing pyridine and imidazole have been widely used to construct coordination polymers [16–19]. Some coordination polymers with 1,2,4-triazole derivatives have been synthesized and characterized with magnetic properties and unusual topologies [20–24]. Our synthetic approach starts by focusing on construction of topological frameworks and potential functional materials using flexible poly(triazole) ligands [25–29]. The ligand 1,2-bis(1,2,4-triazol-1-ylmethyl)benzene (obtz) [30], a positional isomer of 1,4-bis(1,2,4-triazol-1-ylmethyl)benzene (bbtz) [31–33] that can adopt

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*Corresponding author. Email: libaolong@suda.edu.cn

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different conformations, is an attractive building block for constructing coordination polymers, yet is not well exploited (scheme 1).

Metal d^{10} coordination polymers exhibit interesting photoluminescent properties [15,26,29,34]. To investigate the effect of inorganic anions on topologies and syntheses of potential luminescent materials, we have synthesized three new coordination polymers, $[Cd(obtz)(NCS)_2]_n$ (**1**), $[Cd(obtz)_2(dca)_2]_n$ (**2**), and $\{[Cd(obtz)_2(H_2O)_2]\cdot(SiF_6)\}_n$ (**3**), using 1,2-bis(1,2,4-triazol-1-ylmethyl)benzene (obtz). Here, we report syntheses, crystal structures, luminescence properties, and thermal stabilities of the solid-state materials.

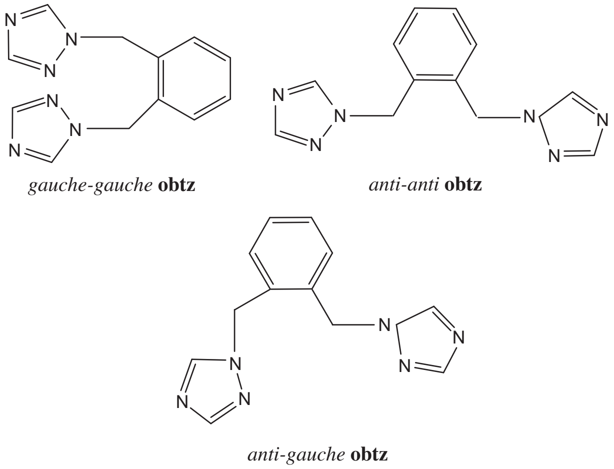
2. Experimental

2.1. *Materials and general methods*

All reagents were of analytical grade and used without purification. 1,2-Bis(1,2,4-triazol-1-ylmethyl)benzene (obtz) was synthesized according to literature method [35]. Elemental analyses for C, H, and N were performed on a Perkin-Elmer 240C analyzer. IR spectra were obtained for KBr pellets on a Nicolet 170SX FT-IR spectrophotometer from 4000 to 400 cm^{-1}. Luminescence measurements were carried out in the solid state at room temperature and spectra were collected with a Perkin Elmer LS50B spectrofluorimeter. Thermogravimetric Analysis (TGA) was carried out using a Thermal Analyst 2100TA Instrument and SDT2960 Simultaneous TGA–Differential Thermal Analysis Instrument in flowing dinitrogen at a heating rate of 10 °C/min.

2.2. *Synthesis of $[Cd(obtz)(NCS)_2]_n$ (1)*

A 15 mL CH_3OH/H_2O solution of $Cd(NO_3)_2\cdot 4H_2O$ (0.2 mmol) and KSCN (0.4 mmol) was added to one tube of the "*H*-shaped" tube. The obtz (0.2 mmol) was added to the other tube. Colorless block-shaped crystals of **1** were obtained after three weeks at room temperature in 68% yield (0.064 g). Anal. Calcd for $C_{14}H_{12}CdN_8S_2$ (**1**): C, 35.87; H, 2.58; N, 23.91%. Found: C, 35.84; H, 2.60; N, 23.87. IR data (cm^{-1}): 2099vs, 1581w, 1520m, 1281m, 1204w, 1134m, 980w, 872w, 718m, 671w, 648w, and 455w.



Scheme 1. Three conformations of obtz.

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2.3. *Synthesis of [Cd(obtz)$_2$(dca)$_2$]$_n$ (2)*

A 10 mL aqueous solution of obtz (0.2 mmol) and Na[N(CN)$_2$] (0.2 mmol) was added to a tube. Then 10 mL 1 : 1 (v/v) H_2O/CH_3OH was slowly added to the tube. Finally 10 mL CH_3OH solution of $Cd(NO_3)_2 \cdot 4H_2O$ (0.1 mmol) was slowly added to the tube. Colorless block-shaped crystals of **2** were obtained after two weeks at room temperature in 56% yield (0.041 g). Anal. Calcd for $C_{28}H_{24}CdN_{18}$ (**2**): C, 46.38; H, 3.34; N, 34.78%. Found: C, 46.34; H, 3.37; N, 34.81. IR data (cm^{-1}): 2345s, 2291s, 2163vs, 1528vs, 1350m, 1292m, 1217m, 1130s, 1018m, 989m, 906m, 723s, 674s, 520m, and 412w.

2.4. *Synthesis of {[Cd(obtz)$_2$(H$_2$O)$_2$]·(SiF$_6$)}$_n$ (3)*

A 15 mL CH_3OH solution of obtz (0.2 mmol) was added to 15 mL aqueous solution of Cd $(NO_3)_2 \cdot 4H_2O$ (0.1 mmol) and K_2SiF_6 (0.1 mmol). The resulting solution was stirred for 20 min and filtered. Colorless block-shaped crystals of **3** were obtained when the filtrate stood on the desk at room temperature for three weeks in 73% yield (0.056 g). Anal. Calcd for $C_{24}H_{28}CdF_6N_{12}O_2Si$ (**3**): C, 37.38; H, 3.66; N, 21.80%. Found: C, 37.36; H, 3.69; N, 21.78. IR data (cm^{-1}): 3423m, 1528m, 1462w, 1341m, 1288m, 1134m, 1026w, 986m, 914w, 744vs, 682m, and 466w.

2.5. *X-ray structure determination*

Single crystals of **1**, **2**, and **3**, suitable for X-ray crystallography, were carefully selected under an optical microscope and glued to thin glass fibers. The diffraction data were collected on a Rigaku Mercury CCD diffractometer with graphite monochromated MoKα radiation. Intensities were collected by the ω scan technique. The structures were solved by direct methods and refined with full-matrix least-squares (SHELXTL-97) [36]. The positions of hydrogens of obtz were determined with theoretical calculation; hydrogens of water were obtained from a difference map. The parameters of the crystal data collection and refinement of **1**, **2**, and **3** are given in table 1. Selected bond lengths and angles are listed in table 2.

3. Results and discussion

3.1. *The crystal structures*

The structure of **1** is a 3-D cubic network. **1** crystallizes in the monoclinic space group *C2/c*. The asymmetric unit contains half Cd(II) located on the inversion center, half obtz, and one NCS$^-$. The coordination geometry of Cd(II) of **1** is a 4 + 2 tetragonally distorted octahedron, coordinated by four nitrogens from two isothiocyanate and obtz in the equatorial plane and two sulfurs in axial positions (figure 1(a)). Each NCS$^-$ coordinates to two Cd(II) ions in a μ–NCS-*N:S* mode, and a single thiocyanate bridges Cd(II) center into a 2-D network [Cd(NCS)$_2$]$_n$, resulting in an "hourglass-shaped" 16-membered [Cd$_4$(μ-NCS-*N:S*)$_4$] metallocycle (figure 1(b)). The Cd· · ·Cd distances are 6.1357(9) and 12.9257(25) Å through NCS$^-$ and obtz bridges, respectively.

Each obtz exhibits *gauche-gauche* conformation. The C1D–C1–C4–N1 torsion angle is 89.3(4)°. The dihedral angle between two triazole ring planes is 67.5(2)°. The dihedral

Table 1. Crystallographic data for **1**, **2**, and **3**.

	1	**2**	**3**
Formula	$C_{14}H_{12}CdN_8S_2$	$C_{28}H_{24}CdN_{18}$	$C_{24}H_{28}CdF_6N_{12}O_2Si$
Fw	468.84	725.05	771.07
Crystal system	Monoclinic	Triclinic	Triclinic
Space group	$C2/c$	$P\bar{1}$	$P\bar{1}$
Temp. (K)	293(2)	293(2)	293(2)
a (Å)	23.410(5)	8.559(3)	8.082(2)
b (Å)	9.1836(18)	9.301(3)	8.388(2)
c (Å)	8.1392(17)	9.768(3)	11.349(3)
α (°)	90	94.515(8)	87.853(9)
β (°)	98.147(4)	95.466(9)	87.533(10)
γ (°)	90	97.572(10)	87.018(9)
V (Å^3)	1732.2(6)	764.0(4)	767.2(3)
Z	4	1	1
ρ_{calc} (g/cm^3)	1.798	1.576	1.669
μ (mm^{-1})	1.517	0.768	0.833
F(000)	928	366	388
Reflections collected	8144	7427	7465
Unique reflections	1579 [R(int) = 0.0298]	2779 [R(int) = 0.0238]	2800 [R(int) = 0.0201]
Parameters	115	214	219
Goodness of fit	1.088	1.050	1.023
R_1 [$I > 2\sigma(I)$]	0.0296	0.0286	0.0233
wR_2 (all data)	0.0736	0.0686	0.0616

Table 2. Selected bond lengths (Å) and angles (°) for **1**, **2**, and **3**.

1			
Cd(1)–N(3)	2.317(3)	Cd(1)–N(4A)	2.297(3)
Cd(1)–S(1)	2.7338(10)		
N(4A)–Cd(1)–N(3)	88.84(12)	N(3)–Cd(1)–S(1)	89.22(7)
N(4A)–Cd(1)–S(1)	87.51(8)		
2			
Cd(1)–N(3)	2.336(2)	Cd(1)–N(6B)	2.349(2)
Cd(1)–N(8)	2.329(2)		
N(6B)–Cd(1)–N(3)	91.74(7)	N(8)–Cd(1)–N(3)	92.36(8)
N(8)–Cd(1)–N(6B)	91.90(8)		
3			
Cd(1)–N(3)	2.2888(18)	Cd(1)–N(6B)	2.3586(17)
Cd(1)–O(1)	2.3233(19)		
N(3)–Cd(1)–N(6B)	87.50(6)	N(3)–Cd(1)–O(1)	87.71(8)
N(6B)–Cd(1)–O(1)	91.50(7)		

Symmetry transformations used to generate equivalent atoms: A $x+3/2$, $-1/2$, $z+3/2$; B x, $y+1$, $-1/2$; C $-x+3/2$, $y+1/2$, $z+1$ for **1**; A $-x+2$, $-y$, $-z+2$; B x, y, $z+1$; C $-x+2$, $-y$, $-z+1$ for **2**; A $-x+2$, $-y$, $-z+2$; B $-x+2$, $-y+1$, $-z+2$; C x, $y-1$, z for **3**.

angles between the triazole ring planes and benzene ring plane are both 71.5(2)°. Each obtz coordinates two Cd(II) ions through its two 4-position triazole nitrogen to further extend the [Cd(μ-NCS-*N:S*)$_2$] 2-D networks into an unusual 3-D cubic network (figure 1(c)).

2 consists of neutral 1-D double chains and crystallizes in the triclinic space group $P\bar{1}$. The asymmetric unit contains a half Cd(II), one obtz, and one dca. The coordination geometry of Cd(II) is a distorted octahedron, coordinated by four triazole nitrogens from four obtz and two nitrogens from two dca$^-$ (figure 2(a)). Two obtz are wrapped around each other and held together by Cd(II) ions to form a 1-D double chain (figure 2(b)).

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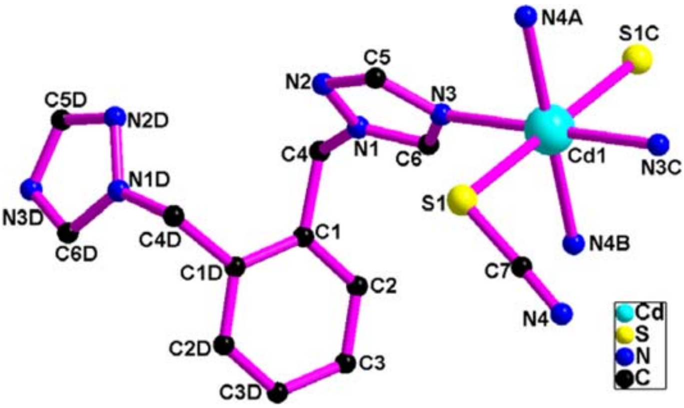

Figure 1(a). The coordination environment of Cd(II) in **1**.

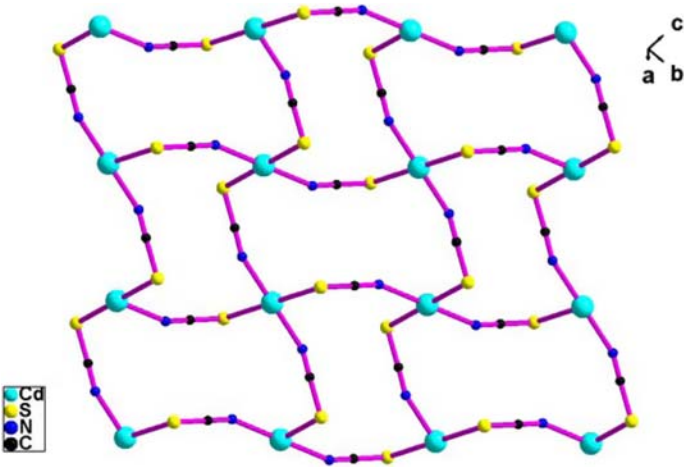

Figure 1(b). $[Cd(NCS)_2]_n$ 2D network in **1**.

The chains extend along the *c*-axis. Cd···Cd separation by obtz is equal to the *c*-axis translation (9.768(3) Å). Each obtz exhibits *anti*-*gauche* conformation. The C2–C1–C7–N1 and C1–C2–C8–N4 torsion angles are 132.8(2)° and −74.2(3)°, respectively. The dihedral angles between two triazole ring planes, N1–N3/C9/C10, N4–N6/C11/C12 triazole ring plane, and benzene ring planes are 79.0(2)°, 79.7(2)°, and 79.6(2)°, respectively.

Chains of **2** are parallel stacked along the *a* direction with the distances corresponding to the *a*-axis translation (8.559(3) Å), not exhibiting $\pi\cdots\pi$ stacking interactions between the obtz ligands. However, benzene rings of adjacent chains along the *b* direction overlap to generate (3.80, 3.83 Å) $\pi\cdots\pi$ stacking interactions. Each chain forms $\pi\cdots\pi$ stacking interactions with four adjacent chains (two above and two below chains along the *b* direction). A 3-D supramolecular network is constructed through these $\pi\cdots\pi$ stacking interactions (figure 2(c)).

3 consists of 1-D cation chains $[Cd(obtz)_2(H_2O)_2]_n$ and SiF_6^{2-}. The coordination geometry of Cd(II) is distorted octahedral; in the equatorial plane are located four nitrogens from the four obtz and axial positions are occupied by two oxygens from two waters (figure 3(a)).

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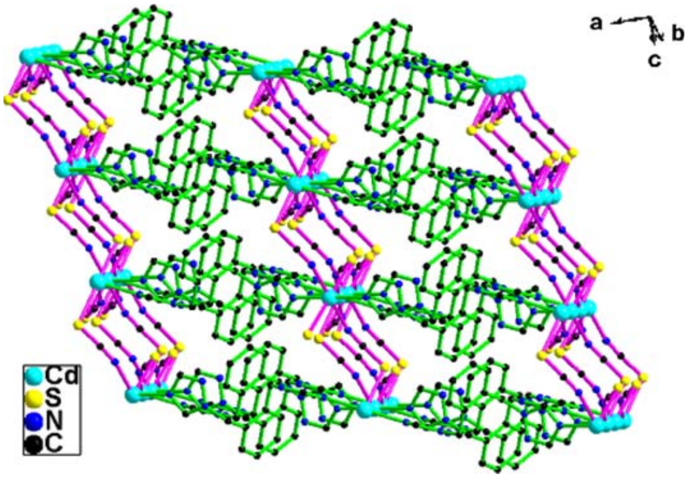

Figure 1(c). The 3-D network of **1**.

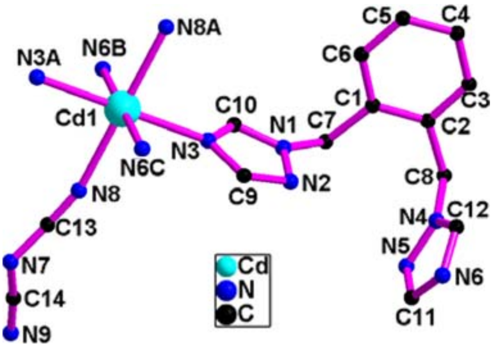

Figure 2(a). The coordination environment of Cd(II) in **2**.

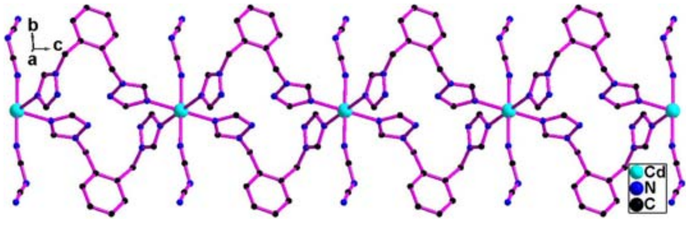

Figure 2(b). The 1-D double chain in **2**.

Two obtz ligands are wrapped around each other and held together by Cd(II) to form the 1-D cation chain $[Cd(obtz)_2(H_2O)_2]_n$ (figure 3(b)). The chains extend along the *b*-axis and the Cd···Cd separation by obtz is equal to the *b*-axis translation 8.388(2). The obtz shows *anti*-*gauche* conformation. The C2–C1–C7–N1 and C1–C2–C8–N4 torsion angles are 111.3(2)° and −73.0(3)°, respectively. The dihedral angles between two triazole ring planes, N1–N3/C9/C10, N4–N6/C11/C12 triazole ring plane, and benzene ring planes are 79.4(2)°, 98.4(2)°, and 98.4(2)°, respectively.

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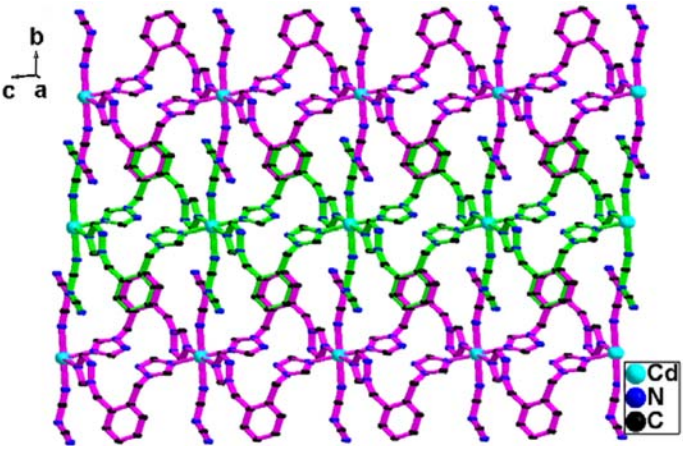

Figure 2(c). The 3-D network in **2**.

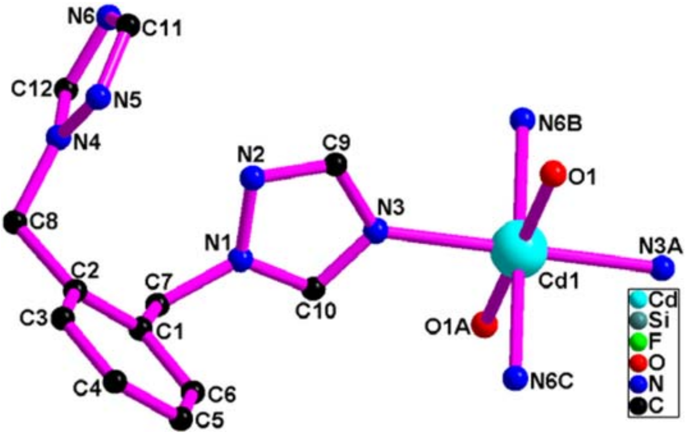

Figure 3(a). The coordination environment of Cd(II) in **3**.

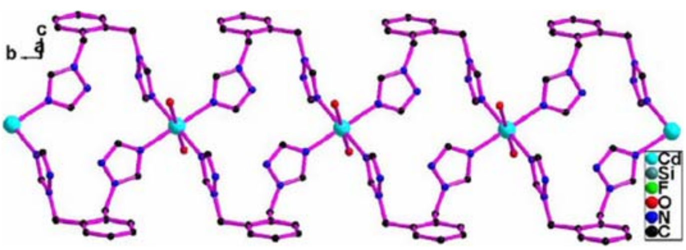

Figure 3(b). The 1-D double chain in **3**.

The cation chains $[Cd(obtz)_2(H_2O)_2]_n$ are parallel stacked along the *a* direction with distances corresponding to the *a*-axis translation (8.082(2) Å), showing no $\pi\cdots\pi$

Table 3. Hydrogen bond parameters (Å, °) for **3**.

D–H···A	d(D–H)	d(H···A)	D(D···A)	∠(DHA)
O(1)–H(1W)···F(2)[a]	0.71(3)	1.93(3)	2.641(2)	173(3)
O(1)–H(2W)···N(5)[b]	0.76(3)	2.18(3)	2.907(3)	163(3)

Symmetry transformations used to generate equivalent atoms: [a]x, y, $z+1$; [b]i $-x+1$, $-y+1$, $-z+2$.

stacking interactions between obtz. Hydrogen bonding interactions play an important role in formation of the supramolecular structure. There are hydrogen bonds between coordination waters and uncoordinated 2-position nitrogens of obtz between adjacent chains (O(1)–H(2 W)···N(5) ($-x+1$, $-y+1$, $-z+2$) 2.907(3) Å) (table 3). A 2-D hydrogen bond network is, thus, formed (figure 3(c)). There are hydrogen bond interactions between coordination waters and SiF_6^{2-} (O(1)–H(1 W)···F(2) (x, y, $z+1$) 2.641(2) Å). Three-dimensional supramolecular architecture is formed through the interactions (figure 3(d)).

1 forms an unusual 3-D cubic network. **2** is comprised of neutral 1-D double chains and a 3-D supramolecular network through the π–π stacking interactions. **3** consists of 1-D cation chains $[Cd(obtz)_2(H_2O)_2]_n$ and SiF_6^{2-} and forms a 3-D hydrogen bond architecture. The structural variety of **1**, **2**, and **3** depends on the counter anions and obtz ligands. Cadmium(II) coordination compounds containing triazole ligands exhibit a variety of architectures and topologies. For example, $\{[Cd(ttmb)_2](SO_4)(H_2O)_{16}\}_n$, $\{[Cd(ttmb)_2(H_2O)_2](NO_3)_2(H_2O)_6\}_n$ and $\{[Cd(ttmb)_2(H_2O)_2](ClO_4)_2(H_2O)_7\}_n$ (ttmb = 1,3,5-tri(1,2,4-triazol-1-ylmethyl)-2,4,6-trimethylbenzene) exhibit the (3,6)-connected $CdCl_2$-type 2-D network, 2-D (4,4) network, and 1-D double chain, respectively [34]. $\{[Cd_3Cl_2(Trz)_4]H_2O\}_n$ (Trz = 1,2,4-triazole) shows an unusual (3,9)-connected network with Schläfli symbol of $(4^{23}\cdot6^{13})(4^3)$ [37]. $\{[Cd(Htmb)(Cl)(CH_3CO_2)]CH_3OH\}_n$ (Htmb = 2-(1H-triazol-1-ylmethyl)-1H-benzoimidazole) consists of 1-D infinite double chains [38]. $[Cd(Htmb)_2(Cl)_2(H_2O)_2]4H_2O$ displays a mononuclear structure, wherein an interesting water chain is formed. $[Cd(Htmb)_2(I)_2]$ is a mononuclear structure and forms a 3-D hydrogen bond network [38]. $\{[Cd_2(btrm)(ip)_2(H_2O)_2]2H_2O\}_n$ and $\{[Cd_2(btrm)(hip)_2(H_2O)_4]3H_2O\}_n$ (btrm = bis(1,2,4triazol-1-yl)methane, H_2ip = isophthalic acid, H_2hip = 5-hydroxy isophthalic acid) exhibit a 3-D network with $CdSO_4$ topology and 1-D ladder structure, respectively [39].

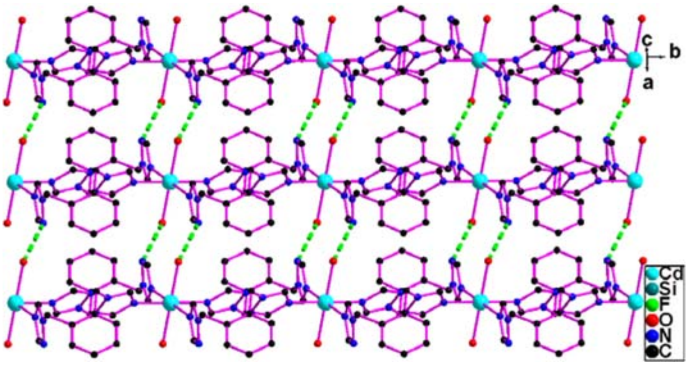

Figure 3(c). The 2-D supramolecular network in **3**.

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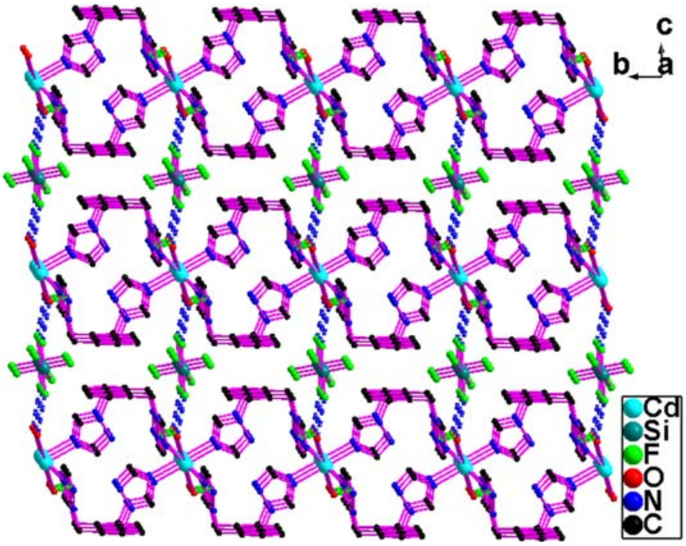

Figure 3(d). The 3-D supramolecular network in **3**.

3.2. *Luminescent properties*

The solid-state luminescent properties of **1**, **2**, **3**, and free obtz were investigated at room temperature (figure 4). Free obtz shows a luminescence peak at 487 nm with excitation at 450 nm. **1**, **2**, and **3** display luminescence maxima around 405, 415, and 306 nm, upon excitation at 344, 353, and 250 nm, respectively. The emissions of **1**, **2**, and **3** have 82, 72, and 181 nm blue-shift, respectively. The emissions can be attributed to ligand-to-metal charge transfer [15, 26, 29, 34]. **1** and **2** have similar emission but **2** has relatively stronger emission than that of **1**, which may be assigned to different conformation of obtz ligands (the *gauche-gauche* conformation in **1** and the *anti-gauche* conformation in **2**). The

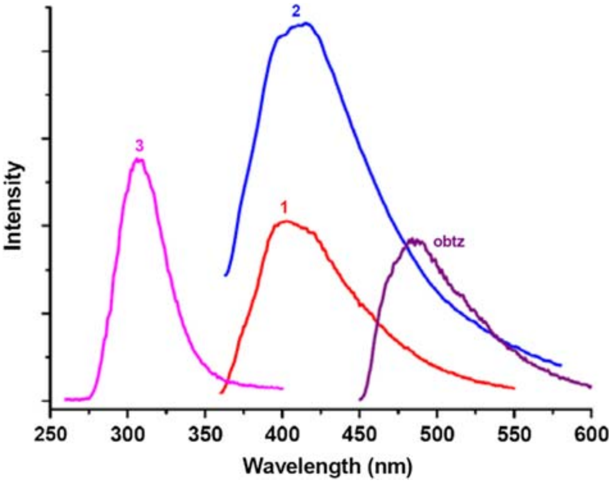

Figure 4. Solid-state photoluminescence spectra of **1**, **2**, **3** and free obtz.

emission of **3** has the bigger blue-shift than **1** and **2** which can tentatively be attributed to hydrogen bond interactions.

3.3. *Thermal stability*

The compounds are stable and retain crystallinity at room temperature for at least several weeks. TG experiments were carried out to explore thermal stabilities (Supplementary material). In the TG curves, **1** and **2** were stable to 231 and 256 °C, respectively. Then weight loss occurred and did not stop at 500 °C. In **3**, the coordination waters were lost from 115 to 172 °C (calculated: 4.67%, found: 4.82%). The remaining substance was thermally stable upon heating to 232 °C. Then **3** exhibited rapid decomposition at 260 to 340 °C and the weight loss occurred continuously and did not stop at 500 °C.

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4. Conclusion

In order to investigate effects of inorganic anions on topologies and syntheses of potential luminescent materials, the coordination polymers $[Cd(obtz)(NCS)_2]_n$ (**1**), $[Cd(obtz)_2(dca)_2]_n$ (**2**), and $\{[Cd(obtz)_2(H_2O)_2]\cdot SiF_6\}_n$ (**3**) were synthesized using 1,2-bis(1,2,4-triazol-1-ylmethyl)benzene (obtz). The ligands obtz exhibit *gauche-gauche*, *anti-gauche,* and *anti-gauche* conformations in **1**, **2**, and **3**, respectively. The topologies of **1**, **2**, and **3** show conformations of flexible ligands and inorganic anions (bridging NCS^-, monodentate dca^-, and uncoordinated SiF_6^{2-} anions which forms hydrogen bonds) play a key role in formation of different coordination polymers. **1**, **2**, and **3** may be candidates as luminescent materials. Further syntheses and structural studies of coordination polymer with flexible triazole ligands are underway in our laboratory.

Supplementary material

Crystallographicdata for the structural analyses have been deposited with the Cambridge Crystallographic Data Center with CCDC reference numbers CCDC-891882, 891883, and 891884.

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