

SOLVOTHERMAL SYNTHESIS, CRYSTAL STRUCTURE AND PHOTOLUMINESCENCE OF Cd(II) COORDINATION POLYMER DERIVED FROM A 1,2,3-TRIAZOLE-BASED TRICARBOXYLATE LIGAND

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Abstract. A new polymeric complex of cadmium(II) was synthesized under solvothermal conditions by the reaction of 5-(4-carboxy-5-methyl-1H-1,2,3-triazol-1-yl) isophthalic acid (H₃L) with cadmium nitrate tetrahydrate in a mixture of *N,N*-dimethylacetamide and water. The obtained compound was investigated using single crystal X-ray diffraction, thermogravimetry, infrared and photoluminescence spectroscopies. The polymeric complex obtained is an infinite 2D coordination polymer with the general formula $[\text{Cd}_3\text{L}_2(\text{H}_2\text{O})_6 \cdot 2\text{H}_2\text{O}]_n$ (**1**). The intense photoluminescence emission of cadmium (II) complex was observed in the blue-violet region of the spectrum.

Keywords: coordination polymer, 1,2,3-triazole, thermogravimetry, photoluminescence.

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Introduction

Coordination polymers are chemical entities assembled from metal ions and organic ligands through coordinative bonds or supramolecular interactions, which form well-ordered structures such as 1D, 2D or 3D polymeric networks. The structure of coordination polymers can be influenced by various factors, such as the nature of the metal ion [1], organic ligands [2], solvent [3], pH [4] and reaction temperature [5]. In last decades, a new type of coordination polymer compounds known as metal-organic frameworks (MOFs) has garnered significant attention, while the research direction pertaining to them has constantly shown a groundbreaking, rapid, quantitative and qualitative development [6]. As infinite crystalline networks containing metal ions connected with organic ligands and containing voids, these structurally interesting coordination polymers are in most cases prepared through rather simple synthetic approaches [7,8]. In addition to their fascinating structures, MOFs also exhibit unique properties that enable various potential applications [9,10]. Numerous studies have demonstrated that multifunctional ligands containing both nitrogen and oxygen as donor atoms are particularly interesting for the generation

of MOFs, as these heteroatoms have the ability to coordinate in different ways, thus allowing the preparation of distinct polymeric architectures with diverse geometries and properties starting from one given ligand [11].

Polycarboxylate ligands containing various central units and cores are widely used in the synthesis of MOFs with different porosities [12]. The structure-activity correlation of metal-organic frameworks plays an important role in their applicability in different fields [13] including gas storage and separation [14,15], catalysis [16], sorption [17], drug delivery [18], optoelectronics [19], proton conduction [20], sensory materials [21], magnetism [22] or chemical and physical sensing [23], to name only a few. Also, the chemistry of 1,2,3-triazoles has become increasingly attractive due to their important role by acting as a structural linker that connects metal nodes to form 1D, 2D, and 3D framework coordination polymers [24], their diverse biological activities [25–27], while their intrinsic fluorescence makes 1,2,3-triazoles useful scaffolds in the structure of compounds for optical applications [28–30]. In an effort to combine both features in a single ligand, triazole-containing polycarboxylates have been successfully employed

for the generation of coordination polymers. Thus, the synthesis through a solvothermal approach and magnetic or luminescent properties of a series of coordination polymers derived from 5-(4-carboxy-1*H*-1,2,3-triazol-1-yl)isophthalic acid and different transition metals have been reported [31–33]. A stable microporous framework with multiple accessible adsorption sites for high capacity adsorption and efficient separation of light hydrocarbons has been obtained starting from 3-hydroxy-4-(1,2,4-triazol-4-yl)benzoic acid and Ni(II) [34], the magnetic and dye adsorption properties of five coordination polymers based on three different triazole-based carboxylate ligands have been studied [35], while the versatility of 5-[[4-(4*H*-1,2,4-triazol-4-yl)phenoxy]methyl]isophthalic acid as a ligand in the formation of structurally diverse coordination properties has been investigated [36]. In order to contribute to this field, 1-(4-carboxyphenyl)-5-methyl-1*H*-1,2,3-triazole-4-carboxylic acid was employed as ligand for the preparation of four novel coordination polymers and two mononuclear complexes [37], and the reaction of the analogous tricarboxylate ligand 5-(4-carboxy-5-methyl-1*H*-1,2,3-triazol-1-yl)isophthalic acid with Zn^{2+} produced a tridimensional coordination polymer whose fluorescent properties were shown to be useful in the detection of nitroaromatics [38]. In a manner similar to Zn^{2+} , cadmium ions with a d^{10} electronic structure are also expected to afford coordination geometries ranging from tetrahedral, or trigonal-bipyramidal, or square-pyramidal to octahedral with the appropriate organic ligands, rendering them desirable for the synthesis of luminescent coordination complexes [39]. Cadmium (Cd) is a toxic non-essential transition metal that poses a health risk for both people and animals. Even cadmium has no known biological properties, it is used indirectly in industry (as a corrosive reagent, as well as its use as a stabilizer in PVC products, colour pigments, and Ni-Cd batteries).

The goal of this study presents the synthesis of a novel cadmium-based coordination polymer derived from 5-(4-carboxy-5-methyl-1*H*-1,2,3-triazol-1-yl)isophthalic acid, its structural characterization, along with its photoluminescence study in solid state.

Experimental

Materials

The used reagents ($\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, *N,N*-dimethylacetamide) were purchased from Sigma-Aldrich and used without further purification. The 5-(4-carboxy-5-methyl-1*H*-1,2,3-

triazol-1-yl)isophthalic acid (H_3L) was prepared through a Dimroth azide-enolate cycloaddition reaction involving dimethyl 5-azidoisophthalate and ethyl acetoacetate, as previously described [38].

Synthesis of the $\{[\text{Cd}_3\text{L}_2(\text{H}_2\text{O})_6] \cdot 2\text{H}_2\text{O}\}_n$ coordination polymer (1)

A solution of cadmium nitrate (0.06 g, 0.204 mmol) in 0.5 mL of distilled water was slowly added to a solution of the ligand H_3L (0.01 g, 0.034 mmol) in 2 mL of *N,N*-dimethylacetamide. The reaction mixture was placed into 8 mL Duran glass vial with a PTFE-coated PBT screw cap. The glass vial was heated at 80°C for 72 h, then the temperature in the oven was gradually lowered to room temperature. The resulting pale-beige crystals were isolated by filtration, washed successively with *N,N*-dimethylacetamide (2×2 mL), water (2×2 mL), and dried in air at room temperature. The crystalline product weighed 0.017 g, the yield (calculated based on cadmium nitrate) is 32%. Elemental analysis calculated for $\text{C}_{24}\text{H}_{28}\text{N}_6\text{O}_{20}\text{Cd}_3$ (1057.72 g/mol): C 27.25; H 2.66; N 7.94; found C 27.68; H 2.85; N 8.17%. IR (ν , cm^{-1}): 3382 s, 3238 sh, 3069 w, 1605 s, 1572 m, 1540 vs, 1474 vw, 1447 vw, 1421 m, 1371 vs, 1358 vs, 1308 m, 1280 m, 1256 m, 1130 s, 1074 w, 1046 w, 947 sh, 925 m, 822 s, 768 vs, 734 vs, 685 w, 665 w.

Physical measurements

Elemental analysis. The complexes were analysed for C, H, and N on a Vario EL (III) Elemental Analyzer. The *infrared spectrum* (4000–600 cm^{-1}) was recorded on a Perkin-Elmer 100 FT-IR spectrometer at room temperature using ATR techniques. Intensities are given as: vs - very strong, s - strong, m - medium, w - weak, vw - very weak, and sh - shoulder.

Thermogravimetric analysis was performed with a STA 449F1 Jupiter instrument. Approximately 10 mg of the solid sample was weighed and placed in an aluminium pan, and the thermogravimetric experiment was run within a temperature range of 30 to 700°C, with a heating rate of 5°C/min, under an atmosphere of dry nitrogen at a flow rate of 50 mL/min. The data was processed with Netzsch Proteus 4.2 software.

X-ray diffraction data for single crystals was collected on a Rigaku Oxford-Diffraction XCALIBUR E CCD diffractometer equipped with graphite-monochromated $\text{MoK}\alpha$ radiation. The unit cell determination and data integration were carried out using the CrysAlis package of Oxford Diffraction [40]. The structure was solved by Intrinsic Phasing using Olex2 [41] software

with the SHELXT [42] structure solution program and refined by full-matrix least-squares on F^2 with SHELXL-2015 [43] applying an anisotropic model for non-hydrogen atoms. All H atoms attached to carbon were introduced in idealized positions ($d_{\text{CH}} = 0.96 \text{ \AA}$) and refined using the riding model. The hydrogen atoms of the OH groups were located from difference Fourier maps and their positions were refined in agreement with the H-bonds parameters. The molecular plots were obtained using the Olex2 program. The crystal data, conditions of data collection and refinement are reported in Table 1. The supplementary crystallographic data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44-1223-336-033; or deposit@ccdc.cam.ac.uk).

Table 1

Crystallographic data, details of data collection and structure refinement parameters for compound 1.

Parameters	Value
Empirical formula	$\text{C}_{24}\text{H}_{28}\text{Cd}_3\text{N}_6\text{O}_{20}$
Formula weight (g/mol)	1057.72
Space group	$P-1$
a (Å)	9.0286(6)
b (Å)	10.1274(7)
c (Å)	10.5441(7)
α (°)	117.448(7)
β (°)	93.767(6)
γ (°)	99.033(6)
Volume (Å ³)	834.42(11)
Z	1
λ (Å)	0.71073
ρ_{calc} (g/cm ³)	2.105
Crystal size (mm ³),	$0.15 \times 0.15 \times 0.02$
Temperature (K)	295
μ (mm ⁻¹)	1.990
Reflections collected	6590
Independent reflections	2916, [$R_{\text{int}} = 0.0355$]
Data/ restraints/ parameters	2916/0/250
R_1	0.0500
wR_2	0.1348
GOF	1.062
CCDC no.	2464761

Luminescence was generated using a pulsed nitrogen laser (337.1 nm) at 300°C. The excitation pulse duration was 15 ns, the frequency repetition was 50 Hz, and the pulse energy was 0.3 mJ. Radiation was recorded using an FEU-79 (multi-alkali photocathode, Sb{Na₂K} with an

adsorbed caesium layer on the surface. The recording equipment used was a boxcar integrator SR-250. The spectral region of study was 350–750 nm.

Results and discussion

The synthesis of the new Cd(II) coordination polymer based on of 5-(4-carboxy-5-methyl-1H-1,2,3-triazol-1-yl) isophthalic acid (H_3L) was carried out using the reaction of H_3L with cadmium nitrate tetrahydrate under solvothermal conditions in a mixture of solvents *N*, *N*-dimethylacetamide and water.

IR spectroscopic characterization

In the IR spectrum of the coordination polymer $\{[\text{Cd}_3\text{L}_2(\text{H}_2\text{O})_6] \cdot 2\text{H}_2\text{O}\}_n$, the bands between 3382 and 3238 cm^{-1} correspond to O–H stretching vibrations of coordinated water molecules. The characteristic vibration modes $\nu(\text{C–H})$ of the methyl group appear at 3069 cm^{-1} . The peaks at 1605 and 1371 cm^{-1} are the characteristic absorptions for the asymmetric $\nu_{\text{as}}(\text{COO}^-)$ and symmetric stretching $\nu_{\text{s}}(\text{COO}^-)$ vibrations of C=O bond. The absorption bands appearing at 1308, 1280, 1256, 1130 cm^{-1} confirms the presence of the triazole ring in the structure of the polymeric compound. The peak at 1074 cm^{-1} belongs to the stretching vibration of $\nu(\text{C–N})$ and the peak at 1046 cm^{-1} belongs to the stretching vibration of $\nu(\text{C–O})$. In the low-frequency regions of the spectrum, the strong bands at 768 and 734 cm^{-1} could be attributed to the $\pi(\text{CH})$ vibrations of the aromatic hydrogen atoms [44].

Thermal analysis

The thermogravimetry (TG) and derivative thermogravimetry (DTG) curves of $\{[\text{Cd}_3\text{L}_2(\text{H}_2\text{O})_6] \cdot 2\text{H}_2\text{O}\}_n$ polymer complex are presented in Figure 1. According to the information from the TG data, the decomposition process initiates at 32°C and takes place in four steps. According to the DTG analysis, the first stage of decomposition occurs with a weight loss of 12.93% from 32°C to 158°C, and corresponds to the loss of eight molecules of water: two lattice water molecules and six coordinated aqua molecules (calcd. 13.61%). In the second step, the weight loss of 11.21% recorded between 158°C to 270°C is associated with decomposition of the organic ligand molecules (calcd. 11.25%) [37]. There is no weight loss observed in the temperature range 270–325°C. But at the interval 325–570°C occurs the most significant mass change recorded during the TGA of coordination polymer. Like this, the third and fourth overlapping steps, at 341–425°C and 425–570°C are associated with decomposition of organic ligand (weight loss of

58.41%, calcd. 57.76%). In the temperature range 528–699°C range likely to cause residue formed by organic and inorganic cadmium-compounds up to a final solid residue mass representing 16.80% (calcd. 36.42% for 3CdO molecules) of the initial amount of the sample. Similar Cd(II) coordination polymers with the composition $\{[\text{Cd}_3\text{L}_2(\text{H}_2\text{O})_6] \cdot 2\text{H}_2\text{O}\}_n$ described in [37] manifest a similar thermal behaviour. The significant difference in the obtained residue mass and the calculated value was observed as well for other Cd(II) compounds, which is explained by the formation of volatile organic and inorganic derivatives at high temperatures [37].

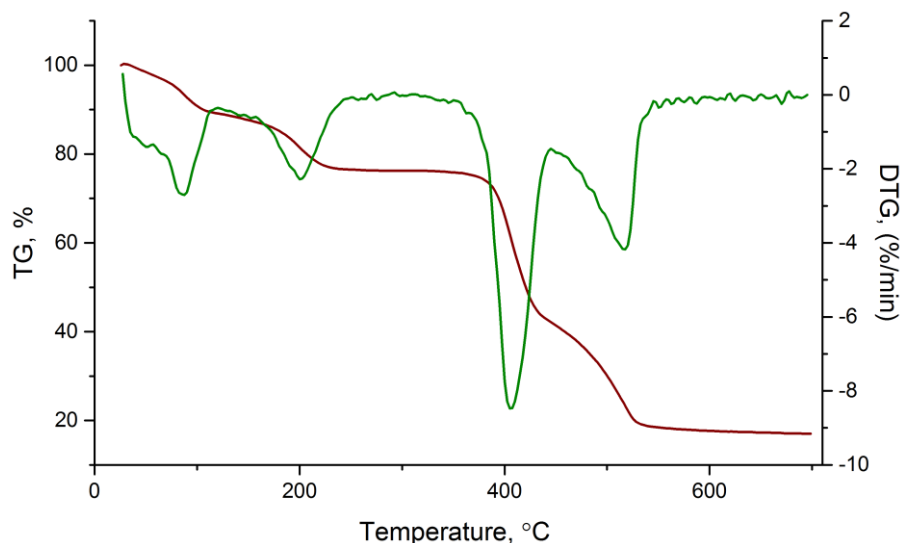
Single crystal X-ray diffraction analysis and crystal structure description

The results of single crystal X-ray diffraction study for **1** are illustrated in Figure 2. Compound **1** is a two-dimensional coordination polymer constructed by tri-nuclear $\{\text{Cd}_3\}$ metal nodes and deprotonated tricarboxylate ligands L^{3-} as linkers in 1:2 ratio. There are two interstitial water molecules in the asymmetric part, so that the charge balance and chemical composition are in agreement with the

formation of species $\{[\text{Cd}_3\text{L}_2(\text{H}_2\text{O})_6] \cdot 2\text{H}_2\text{O}\}_n$. The asymmetric part of the unit cell (Figure 2(a)) comprises one deprotonated ligand L^{3-} and two Cd(II) atoms. Cd1 is located on general position, while Cd2 occupies a special position on the inversion centre with site occupancy factor of 0.5. Cd2 atom exhibits a slightly distorted octahedral coordination, while in the case of Cd1 eight coordinated atoms (NO_7) are arranged to define the vertices of a strongly distorted bicapped trigonal prism (Figure 2(a)). In the crystal, asymmetric units are assembled through coordination bonds to form a two-dimensional network with (3×6) topology. A view of two-dimensional coordination polymer $\{[\text{Cd}_3\text{L}_2(\text{H}_2\text{O})_6]\}_n$, is illustrated in Figure 2(b). Further analysis of the structure has revealed the presence π – π stacking interactions with centroid-to-centroid distance of 3.610 Å between benzene rings of adjacent 2D coordination polymers (Figure 2(c)), which, along with numerous O–H \cdots O hydrogen bonds (Table 2), determine a 3D supramolecular assembly resulting from the parallel packing of 2D layers, as shown in Figure 2(d).

Table 2

Hydrogen bond distances [Å] and angles [°] for 1 .					
<i>D</i> – <i>H</i> ··· <i>A</i>	<i>d</i> (<i>D</i> – <i>H</i>)	<i>d</i> (<i>H</i> ··· <i>A</i>)	<i>d</i> (<i>D</i> ··· <i>A</i>)	\angle (<i>DHA</i>)	Symmetry code
O1w–H···O4	0.85	2.30	2.914(9)	128.6	1+x,+y,1+z
O1w–H···O5	0.85	2.12	2.867(8)	145.1	1–x,2–y,1–z
O1w–H···N2	0.85	2.32	2.909(8)	126.4	+x,+y,1+z
O2w–H···O6	0.86	2.48	2.987(8)	117.9	1–x,2–y,–z
O2w–H···O4	0.86	2.06	2.898(9)	163.1	1+x,+y,1+z
O3w–H···O5w	0.85	1.89	2.64(2)	145.6	x,–1+y,+z
O3w–H···O7	0.85	2.01	2.80(4)	153.7	x,–1+y,+z
O3w–H···O6	0.85	1.90	2.724(8)	162.1	x,–1+y,+z

Figure 1. TG and DTG analysis of complex $\{[\text{Cd}_3\text{L}_2(\text{H}_2\text{O})_6] \cdot 2\text{H}_2\text{O}\}_n$.

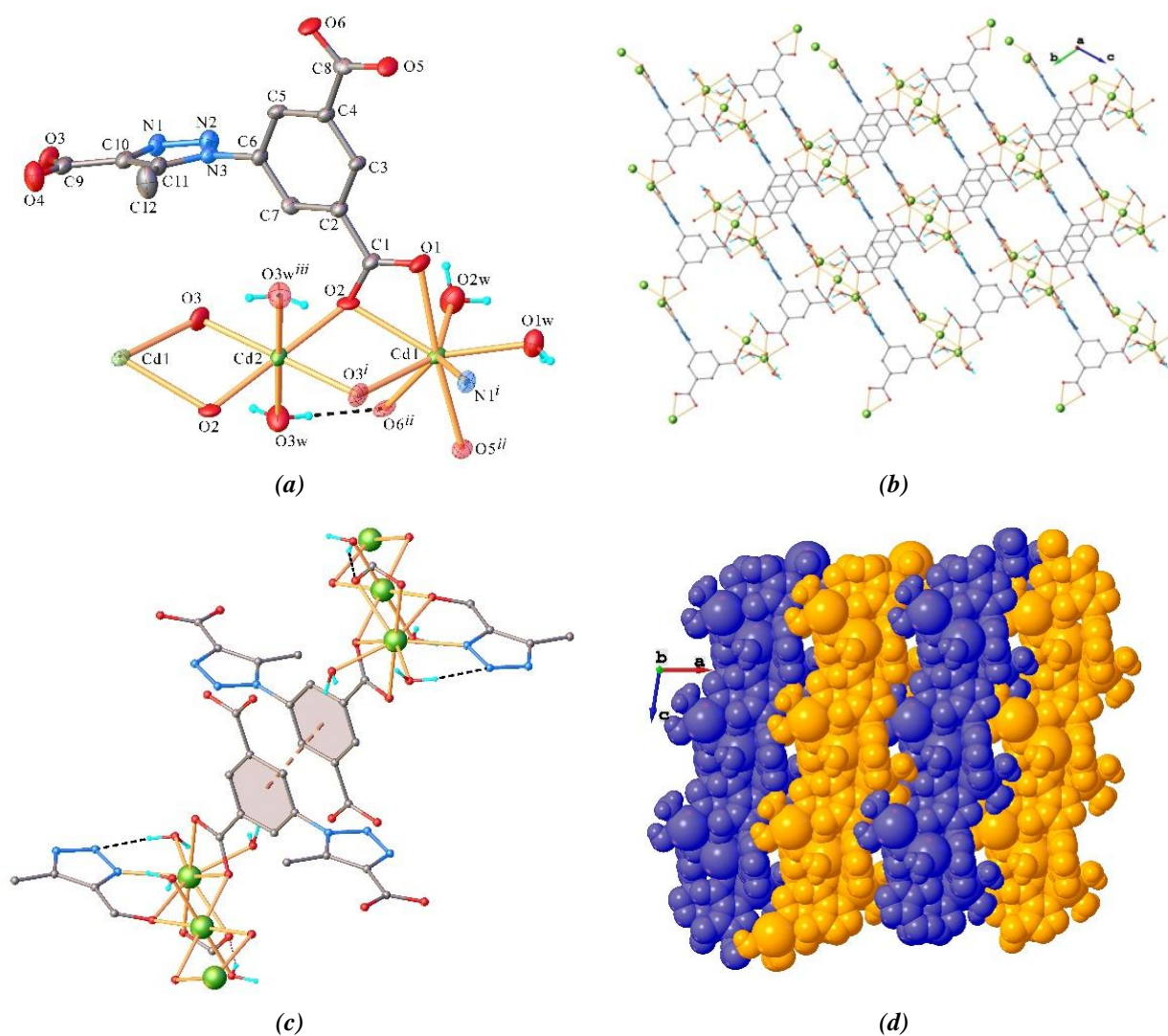


Figure 2. View of the asymmetric part in the crystal structure of 1 with thermal ellipsoids at 40% probability level showing the coordination environment of the Cd atoms. The co-crystallized water molecules and H atoms attached to carbon are not shown. Symmetry generated atoms are shown with faded colours. Symmetry codes: (i) $x, y, 1+z$; (ii) $x, y-1, z$; (iii) $-x, 1-y, -z$ (a); the two-dimensional coordination polymer viewed along *a* axis (b); a fragment of the crystal packing showing π - π stacking interaction between benzene rings of adjacent 2D coordination networks (c); partial view of the crystal packing along *b* axis illustrating the parallel arrangement of 2D coordination polymers driven by interlayer H-bonds and π - π stacking (d).

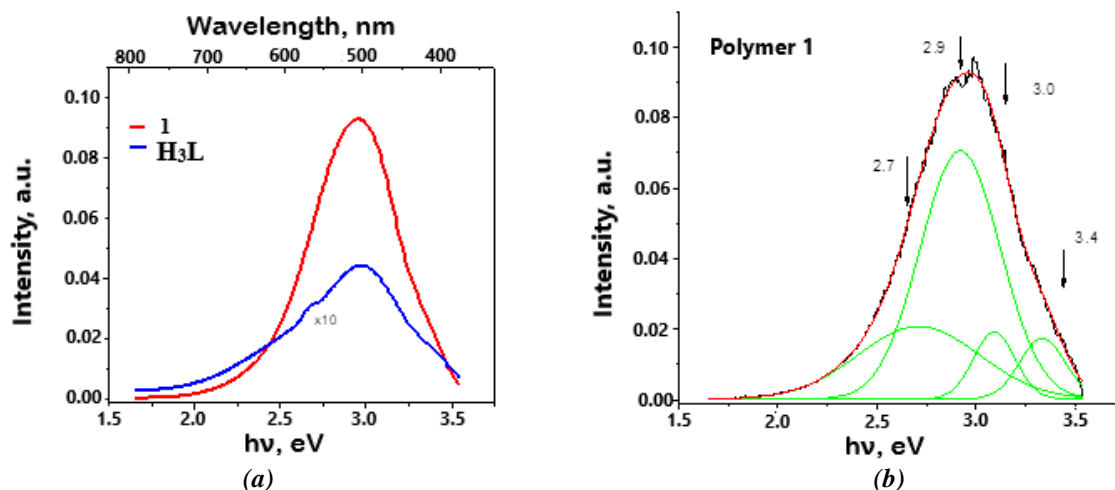


Figure 3. Photoluminescence spectra of coordination polymer 1 and Ligand H₃L (a); deconvolution of spectra using the Gaussian function (b).

Photoluminescence properties of ligand H_3L and its corresponding coordination polymer $\{[Cd_3L_2(H_2O)_6] \cdot 2H_2O\}_n$

The investigated complex was obtained from H_3L as the ligand. The photoluminescence (PL) spectra for both the ligand and the coordination polymer are a superposition of several peaks. The Gaussian function was used to decompose each spectrum into individual bands (Figure 3).

The luminescence studies reported by Zheng *et al.* for Zn(II) and Cd(II) coordination compounds show that the nature of the metal centres and ligands are the main factors that generate the luminescent properties [39]. For the Cd complex under investigation, photoluminescence was observed in the spectral region of 400–650 nm (Figure 3 (a)). The main bands that form the emission spectrum of the sample are observed at 2.7 eV (560 nm), 2.9 eV (530 nm), 3.0 eV (513 nm) and 3.4 eV (464 nm) (Figure 3(b)).

An intense photoluminescence emission is observed in the blue-violet region of the spectrum (410–430 nm). The intensity ratio of the peaks and the shape of the photoluminescence spectrum of the sample matches the shape of the PL spectrum of the ligand. In the photoluminescence spectrum of the ligand, an additional weak peak is observed in the yellow region of the spectrum at 2.3 eV (540 nm). The intensity of photoluminescence in the sample exceeds the PL of the ligand by more than one order of magnitude (almost 25 times). Thus, conversion of the ligand to its Cd(II) complex can be associated with a strong enhancement of photoluminescence. An increase in the photoluminescence of the coordination compound compared to the photoluminescence of the corresponding ligand may be attributed to complex formation, but could also be due to π – π^* type interactions in the coordination compound.

Conclusions

A new coordination polymer based on a 1,2,4-triazole-containing polycarboxylate ligand has been synthesized and characterized by single-crystal X-ray diffraction, elemental analysis, IR, photoluminescent spectra and TGA. The two-dimensional coordination polymer $\{[Cd_3L_2(H_2O)_6]\}_n$ presents π – π stacking interactions between benzene rings of adjacent 2D coordination polymers, which contribute along with numerous O–H \cdots O hydrogen bonds to the assembly of a 3D supramolecular assembly. The polymeric complex of cadmium(II) has a

pronounced photoluminescent activity, exhibiting an intense emission in the blue-violet region of the spectrum. The intensity of photoluminescence in the sample exceeds the PL of the ligand by more than one order of magnitude (almost 25 times). The complex can be proposed as a fluorescent material, such as light-emitting materials (*e.g.*, light-emitting diodes (LEDs)), or for other applications in various fields, such as in analytical chemistry.

Acknowledgments

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