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Catena-Poly[[[2,4-dichloro-6-[(2-hydroxyethyl)imino]methyl}phenolato- κ^3N,O,O']-copper(II)]- μ -chlorido]monohydrate]: Synthesis, structural, spectroscopic and luminescent properties

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ABSTRACT

A new chloride bridged polymeric Cu(II) complex, $[Cu(HL)(\mu-Cl).H_2O]_n$, **1**, ($H_2L = 2,4$ -dichloro-6-[(2-hydroxyethyl)imino]methyl}phenol) has been synthesized and characterized by elemental-spectral analysis, crystal structure analysis and photoluminescence measurements. The Copper(II) atom is five-coordinate in a slightly distorted square-pyramidal geometry ($\tau = 0.085$), with one N and two O atoms of the Schiff base ligand and one Cl atom defining the basal plane and a symmetry-related Cl atom occupying the apical position. The bridging Cl atom lies in apical position for one Cu(II) ion and basal for the other, and it makes this structural arrangement unusual. The linked moieties form polymeric zigzag chains running along the *c* axis. This zigzag chains connect each other with intramolecular and intermolecular O–H...O hydrogen bonds, which form 3D structure through $\pi-\pi$ interactions. Furthermore, the photoluminescence properties of **H₂L** and **1** were investigated, they exhibit unique bright green visible emissions in the solid state, under the excitation of 349 nm UV light. The strong luminescence emission of them makes **1** a potentially useful photoactive material in photo-physical chemistry.

KEYWORDS

Crystal structure analysis;
polymeric Cu(II) complex;
photoluminescence

1. Introduction

Transition metal complexes containing oxygen, nitrogen or sulfur donor ligands have been widely studied for decades, not only for their structural diversity, but also for their fascinating potential application in various fields such as nonlinear optic, catalysis, luminescent probes, sensors, magnetism, gas storage and separation *etc.* [1–10]. Especially, Schiff base ligands can serve as excellent candidates to construct the above-mentioned complexes [11]. Schiff base ligands are energetic chelators and can form stable molecular architectures with transition metal ions. Schiff base metal complexes with spectroscopic properties can be used in applications involving fabrications of new materials [12,13].

Moreover, the Schiff base copper complexes have received continual and considerable attention in many potential application fields [14]. In this context, luminescent organic

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materials with emission in the solid state are particularly attractive because of their widespread applications in biotechnology, optics, memory and laser systems. Especially green solid-state emitters are crucial for organic white lighting, fluorescence imaging and full color OLEDs [15].

In the last decade, our research group and others have reported the structural, magnetic and spectroscopic characterization of mono- and dinuclear copper(II) complexes containing Schiff base ligands [3,16–23]. In terms of the significance of Cu(II) complexes and our interest in the coordination chemistry of these complexes including Schiff bases as chelating ligands, we report here the synthesis of a new unusual chloride-bridged polynuclear Cu(II) complex along with its characterization by single crystal X-ray structure, UV and IR spectroscopy and photoluminescence study. Since the presence of Cu²⁺ drastically quenches the luminescence, the examples of copper(I, II) and copper(II) luminescent complexes are rather scarce [24–26]. However, the bridging chloride ion which lies in apical position for one Cu(II) ion and basal for the other has a rather specialized role in chain arrangement of this copper(II) complex, and it makes this structural arrangement unusual.

2. Experimental

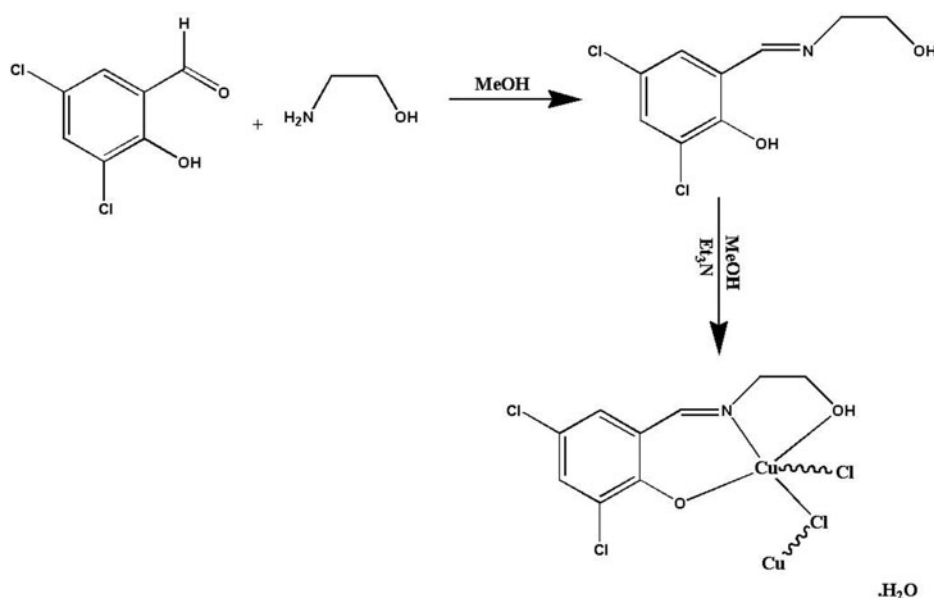
2.1. Materials and general methods

All chemical materials and solvents in the synthesis phase were reagent grade and used without any purification. C, H and N elemental analyses were realized by standard methods. Infrared spectra were measured in the range of 4000–550 cm^{−1} with a Perkin-Elmer Spectrum 65. The UV–visible spectra in solid state were determined by Ocean Optics Maya 2000 Pro Spectrometer (250–600 nm). The photoluminescence spectra in solid state were measured at ambient temperature with an ANDOR SR500i-BL Photoluminescence Spectrometer in the visible region. The measurements were performed by the use of excitation source (349 nm) of a Spectra-physics Nd:YLF laser with a 5 ns pulse width and 1.3 mJ of energy per pulse.

2.2. Synthesis of Schiff base ligand **H₂L** and **1**

The tridentate ONO type Schiff base ligand, **H₂L** [2,4-dichloro-6-[(2-hydroxyethyl)imino]methyl]phenol], was prepared by mixing 3,5-dichlorosalicylaldehyde (1 mmol, 0.191 g) and ethanolamine (1 mmol) with the equimolar ratio in hot methanol (40 cm³) as in a similar manner to related literature [27]. The resulting solution was stirred for 30 min at 65°C. The yellow products of the ligand have precipitated from cooling of the solution.

Complex **1** was prepared with the addition of CuCl₂ (1 mmol, 0.134 g) and Et₃N (0.5 mmol) in 40 cm³ of hot methanol to the **H₂L** ligand (1 mmol) in 40 cm³ of hot methanol. The mixture was stirred, and then cooled to room temperature to give a powder precipitate, which was collected by suction filtration. The powder product was dissolved in methanol for recrystallization and two weeks later prismatic stick crystals of **1** were found. The synthetic route of the metal complex is outlined in Scheme 1. **H₂L**: Yellow products of the ligand yield 68%. Anal. Calc. for **H₂L**: C₉H₉Cl₂NO₂; Calc.: C, 38.83.18; H, 11.24; N, 0.92%. Found: C, 38.81; H, 11.21; N, 0.96%. **1**: Green crystals yield 75%. Anal. Calc. for **1**: C₉H₁₀Cl₃CuNO₃; Calc.: C, 31.43; H, 3.69; N, 3.67%. Found: C, 31.41; H, 3.72; N, 3.69%.



Scheme 1. The synthetic route of complex 1.

2.3. Crystallography details

X-ray data for appropriate single crystals of **1** were collected on an Oxford Diffraction Xcalibur-3 diffractometer equipped with a CCD camera using a monochromatic $\text{MoK}\alpha$ source ($\lambda = 0.71073 \text{ \AA}$ at 296 K). The CrysAlis CCD Software system and CrysAlis RED Software system [28] were used for the data collections and reductions. Using Olex2 [29], the structure were solved using SHELXS [30] by direct methods and refined using SHELXL [31] by using full-matrix least-squares against $|F_{\text{obs}}|^2$. The details of the supramolecular π -interactions and hydrogen bond geometry were investigated with a Platon 1.17 program [32]. The crystallographic data and structure refinement details for **1** have been given in Table 1. All non-hydrogen atoms were refined anisotropically. Hydrogen atom positions were calculated

Table 1. Crystal data and refinement information for complex 1.

Chemical formula	$\text{C}_9\text{H}_8\text{Cl}_3\text{CuNO}_2 \cdot \text{H}_2\text{O}$
M_r	350.07
Crystal system	Orthorhombic
Space group	$Pca2_1$
Unit cell dimensions	$a = 9.9764 (9) \text{ \AA}$ $b = 17.019 (4) \text{ \AA}$ $c = 7.2553 (9) \text{ \AA}$ $\alpha = \beta = \gamma = 90^\circ$
$V/\text{\AA}^3$	1231.9 (4)
Z	4
$\rho_{\text{calc}}/\text{g.cm}^{-3}$	1.888
μ/mm^{-1}	2.416
Reflections collected	10417
Independent reflections	2750 [$R_{\text{int}} = 0.0561$]
Refinement method	Full-matrix least-squares on F^2
Goodness-of-fit on F^2	$S = 0.939$
R indices [$ I > 2\sigma(I)$]	$R_1 = 0.0324, wR_2 = 0.0732$

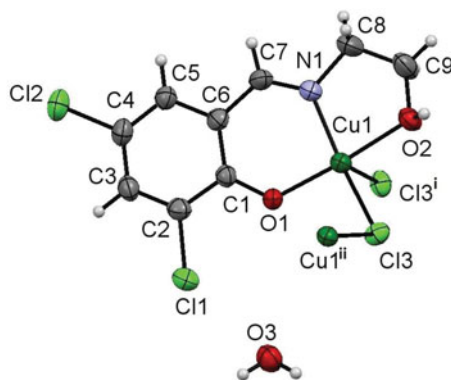


Figure 1. The molecular structure of **1**. Thermal ellipsoids have been drawn at 50% probability levels. [Symmetry code: (i) $-x + 1/2, y, z - 1/2$; (ii) $-x + 1/2, y, z + 1/2$].

geometrically and refined using the riding model. The absolute structure was determined on the basis of the Flack parameter $x = 0.010$ (12) for **1**. The Flack's parameter as a value which close to 0 is indicative of a non-centrosymmetric structure [33].

3. Results and discussions

3.1. Crystal structure determination

ORTEP diagrams of **1** are displayed in Fig. 1. The repeat unit of the **1** contains a $[\text{Cu}(\text{HL})\text{Cl}]$ molecule and one water molecule of crystallization. $[\text{Cu}(\text{HL})\text{Cl}]$ units connected to one another by a single bridging chloride anion, in which each copper atom is pentacoordinated as shown in Fig. 1. For a pentacoordinated metal center, the distortion of the coordination environment from a trigonal-bipyramidal (TBP) to a square-pyramidal (SP) can be evaluated by the Addison distortion index, τ defined as $\tau = (\alpha - \beta)/60$, where α and β are the two largest coordination angles. The coordination geometry of the metal atom is determined as $\tau = 0$ for perfect SP and 1 for ideal TBP [34,35]. In the present complex, the calculated τ value is 0.085, suggesting a slightly distorted square-pyramidal geometry for the Cu atom. The base of the pyramid of the copper atom (Cu1) is formed by the imine nitrogen atom (N1), the phenoxo and alkoxy oxygen atoms (O1 and O2) from the Schiff base ligand and a chloride ion (Cl3), while the apical position of the pyramid is occupied a chloride ion that is related to the symmetry of the Cl3 atom [Cl3ⁱ; symmetry code: (i) $-x + 1/2, y, z - 1/2$]. The Cu atom is displaced from the mean basal plane towards the apical Cl atom by 0.186 Å. The bond lengths between the metal and donor atoms in the base of the pyramid are as follows: $\text{Cu}-\text{N}_{\text{imine}} = 1.933$ (4) Å; $\text{Cu}-\text{O}_{\text{phenolic}} = 1.918$ (3) Å, $\text{Cu}-\text{O}_{\text{alkoxy}} = 2.031$ (3). The axial $\text{Cu}-\text{Cl3}^i$ bond length is 2.632 Å while the basal $\text{Cu}-\text{Cl3}$ bond length is 2.255 Å. The average $\text{Cl3}-\text{Cu1}-\text{Cl3}^i$ and $\text{Cu1}-\text{Cl}-\text{Cu1}^{ii}$ [symmetry code: (ii) $-x + 1/2, y, z + 1/2$] bond angles of 99.05 (4)° and 106.19 (5)°, respectively, and $\text{Cu1}\cdots\text{Cu1}^{ii}$ non-bonding distance is 3.915 Å. Selected geometric parameters are summarized in Table 2, which lie well within the range of reported values for corresponding bond lengths and angles of other mononuclear copper(II) complexes [36–38].

Each copper(II) moiety of complex **1** is nearly coplanar, with a mean deviation from the Cl1/Cl2/C1-C6/C7/N1/C8/O2 plane of 0.064 Å and with a dihedral angle of 7.35° between

Table 2. Selected geometric parameters ($\text{\AA}$, $^\circ$) for **1**.

<i>Bond lengths ($\text{\AA}$)</i>			
Cu1—Cl3	2.2547 (12)	Cu1—O1	1.917 (3)
Cu1—Cl3 ⁱ	2.6322 (14)	Cu1—O2	2.025 (3)
Cu1—N1	1.933 (4)		
<i>Bond angles ($^\circ$)</i>			
Cl3—Cu1—Cl3 ⁱ	99.07 (4)	O1—Cu1—N1	93.47 (15)
N1—Cu1—Cl3 ⁱ	100.12 (14)	O1—Cu1—O2	176.00 (13)
N1—Cu1—Cl3	158.75 (15)	O2—Cu1—Cl3	89.39 (10)
N1—Cu1—O2	82.55 (15)	O2—Cu1—Cl3 ⁱ	87.08 (11)
O1—Cu1—Cl3 ⁱ	93.96 (11)	Cu1—Cl3—Cu1 ⁱⁱ	106.20 (5)
O1—Cu1—Cl3	94.25 (10)		

[Symmetry code: (i) $-x + 1/2, y, z - 1/2$; (ii) $-x + 1/2, y, z + 1/2$]

these parallel planes. This planar arrangement can decrease the steric repulsion of the two near planar Cu(II) moieties, and also encourage a square-pyramidal geometry. In **1**, the bridging chloride ion lies in apical position for one Cu(II) ion and basal for the other, and it makes this structural arrangement unusual. The linked moieties form polymeric zigzag chains running along the c axis (Fig. 2(b)). These zigzag chains connect each other with intramolecular and intermolecular O—H $\cdots$ O and O—H $\cdots$ Cl hydrogen bonds, which form 3D structure (Fig. 2(a) and Table 3). This hydrogen-bonded polymeric networks lie in the ab plane and stacks along the c axis through π — π interactions with a separation of 3.825 and 3.896 $\text{\AA}$ (Fig. 3).

Before proceeding to the spectroscopic and photoluminescence studies we note that experimental X-ray powder diffraction traces for **1** are well compatible with those of simulated traces on the basis of single crystal structure of **1** (Fig. 4).

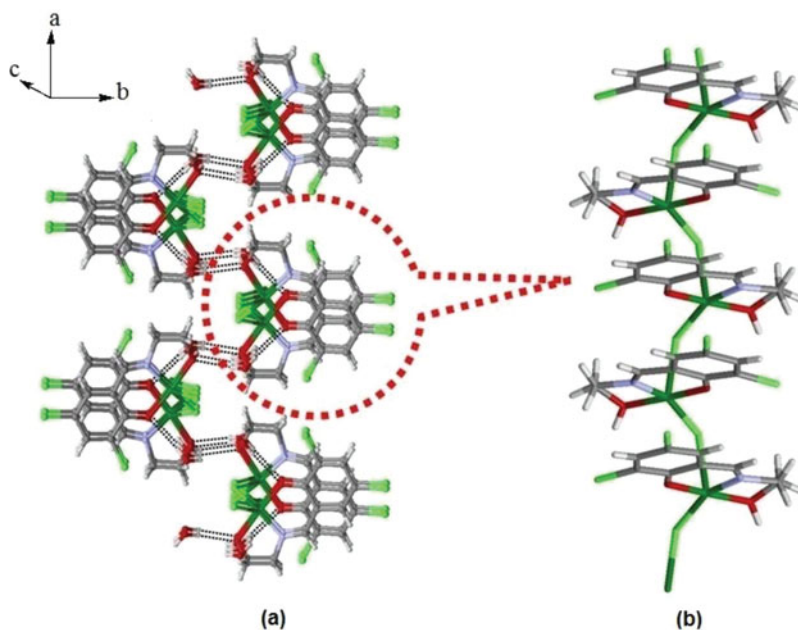
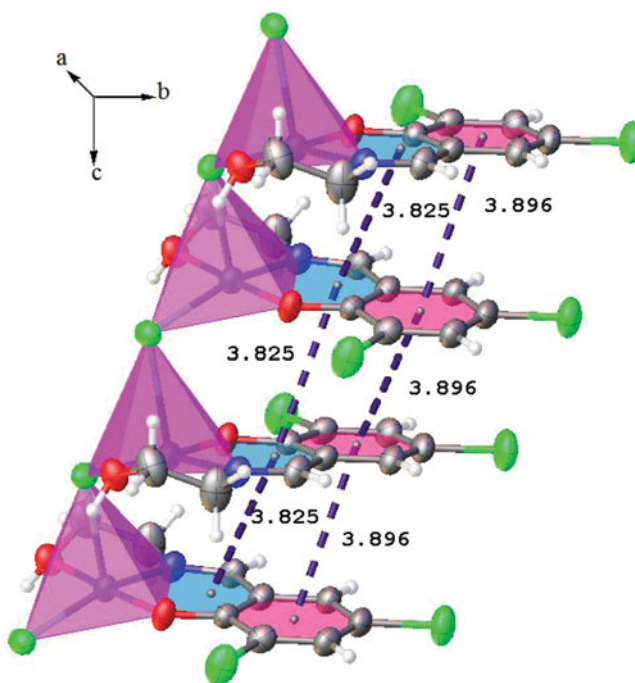
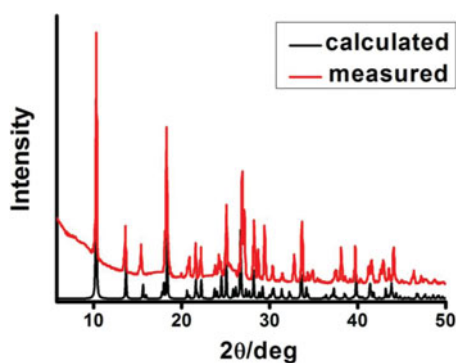


Figure 2. (a) Hydrogen-bonding network in complex **1**. (b) A view of polymeric zigzag chains along the c axis.

Table 3. Short contact and hydrogen bond geometry ($\text{\AA},^\circ$) for **1**.

D—H...A*	D—H	H...A	D...A	D—H...A	Symmetry
O2—H2...O3	0.87	1.82	2.623(6)	153	$-1+x, y, z$
O3—H3A...O1	0.83	2.30	3.082(5)	157	$3/2-x, y, 1/2+z$
O3—H3B...O2	0.83	2.21	3.012(5)	161	$1-x, 1-y, 1/2+z$
O3—H3A...Cl3	0.83	2.80	3.339(4)	124	$3/2-x, y, 1/2+z$
Cg(I)...Cg(J)	Cg(I)...Cg(J)				
Cg(2)...Cg(2)	3.825(3)				$1/2-x, y, -1/2+z$
Cg(3)...Cg(3)	3.896(3)				$1/2-x, y, -1/2+z$

*D: Donor, A: Acceptor, M: Metal, Cg(I): Plane number I (ring number in () above), Cg–Cg: Distance between ring Centroids ($\text{\AA}$), Cg(2): Cu1—O1—C1—C6—C7—N1, Cg(3): C1—C2—C3—C4—C5—C6.

**Figure 3.** The solid state ordering of **1** showing $\pi \cdots \pi$ stacking interactions.**Figure 4.** Experimental (red) and calculated (black) PXRD traces of **1**.

3.2. Spectroscopic characterization

The IR spectral analysis of **H₂L** ligand and complex **1** in the region of 4000–550 cm^{-1} (Fig. 5) has been summarized in Table 4. The IR spectra of the ligand and the complex contain absorption bands in a similar region. However, some significant variations are seen in the spectra of the metal complex and its free ligand. The IR spectra of **1** shows a broad band in the region 3486–3370 cm^{-1} due to the $\nu_{(\text{O}-\text{H})}$ stretching mode of OH^- chelating group [39,40]. Several absorption bands occurred in the range 2976–2895 cm^{-1} are described to the aromatic and aliphatic $\nu_{(\text{C}-\text{H})}$ stretches [41]. The spectrum shows that the azomethine group $\nu_{(\text{C}=\text{N})}$ stretching mode of the coordinated ligand is observed at 1634 cm^{-1} as compared to 1644 cm^{-1} observed for the free ligand [42]. As expected, the negative shift of this band upon complexation indicates coordination of the imine group through nitrogen atom. The phenolic C–O group of free ligand indicates a strong band 1224–1199 cm^{-1} , whereas in the **1**, this band is observed in the higher wavenumber region 1241–1209 cm^{-1} . This band has shifted to higher wavenumber, proving coordination of the phenolic O atom to metal centre.

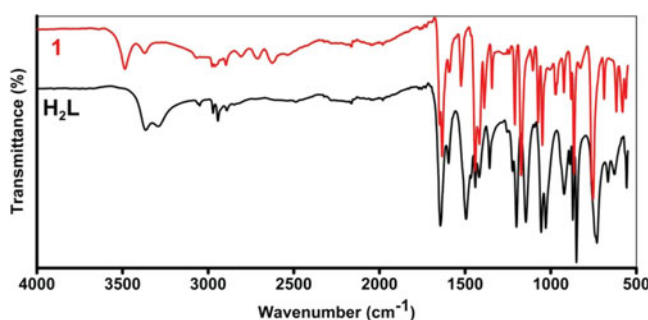


Figure 5. IR spectra of the Schiff base ligand **H₂L** and **1**.

Table 4. Major infrared bands (cm^{-1}) of **H₂L** and **1**.

	$\nu_{(\text{O}-\text{H})}$	$\nu_{(\text{C}-\text{H})}$	$\nu_{(\text{C}=\text{N})}$	$\nu_{(\text{C}=\text{C})}$	$\nu_{(\text{C}-\text{O})}$
H₂L	3365–3291	2971–2891	1644	1494	1224–1199
1	3486–3370	2976–2895	1634	1523	1241–1209

3.3. Solid state UV-Vis spectra

The solid state UV-Vis spectra of complex **1** and its free ligand **H₂L** were recorded and display three well resolved absorption bands (Fig. 6). The electronic spectrum of free ligand **H₂L** displays a prominent absorption band (λ) appears in the 300–600 nm region with maxima at 455 nm, which can be attributed to the ligand absorption band and imine-to-copper ligand-to-metal electronic charge transfer band (LMCT) [2,5,43,44]. The spectra of copper complex also indicates shoulder at 310 nm due to $\text{OH}^- \rightarrow \text{Cu(II)}$ or phenoxo-to- Cu(II) LMCT transition. In addition, the shifting of the absorption band in the spectra of the complex towards shorter wavelength compared to that of ligand, which is signified the metal ion coordination with the ligand [5,13,17,42].

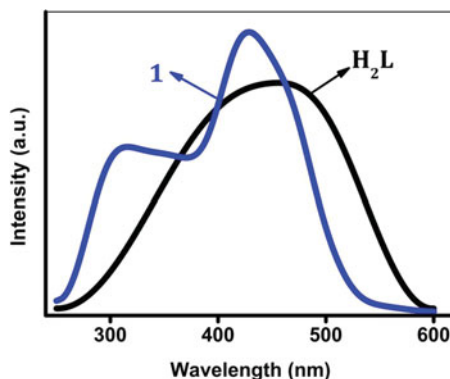


Figure 6. The solid state UV-Vis spectra of the free ligand H_2L and **1**.

3.4. Photo-physical study

The solid-state photoluminescence properties of the ligand H_2L and **1** were compared and recorded at ambient temperature in the visible regions upon excitation at $\lambda_{\text{exc}} = 349 \text{ nm}$ (Fig. 7). As obvious from the Fig. 7, the Schiff base ligand indicates a broad green emission band at $\lambda_{\text{max}} = 530 \text{ nm}$, which may be assigned to the $n \rightarrow \pi^*$ or $\pi \rightarrow \pi^*$ electron transition (ILCT) [17,45–50]. When free ligand H_2L is combined with Cu(II) in **1**, more intense green emission peak is indicated at 502 nm [39,51]. The maximum peak of **1** is slightly blue shift when compared with that of its free ligand, which is mainly due to originate from the influence of the coordination of the metal to the ligand [17]. As a result, the emission intensity of **1** is stronger than that of its ligand [51]. Thus, the enhanced luminescence efficiency of **1** is attributed to the coordination of the ligands to the Cu^{II} ions [52]. And also, complex **1** may have potential as a luminescent material in green light-emitting materials [53].

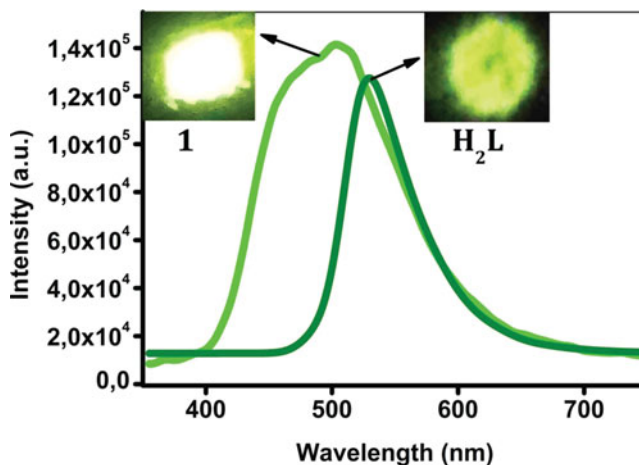


Figure 7. The emission spectra of the H_2L and **1** in the solid samples at ambient temperature ($\lambda_{\text{exc.}} = 349 \text{ nm}$).

4. Conclusions

The synthesis and structural characterization of a new chloride-bridged Cu(II) complex have been presented together with an investigation into its photoluminescence properties.

Single-crystal X-ray diffraction studies confirm that the metal center in **1** has slightly distorted square-pyramidal. In the crystalline architecture of **1**, the intermolecular O—H...O hydrogen bonds link the molecules which form two dimensional polymeric chains and $\pi \cdots \pi$ contacts also connect the molecules which form to 3D structure. In addition, structural arrangement of this copper(II) complex is unusual due to the bridging chloride ion, which has a rather specialized role in chain arrangement, lies in apical position for one Cu(II) ion and basal for the other. The photoluminescence studies show a blue shift compared with free ligand **H₂L** and the emission intensity of **1** is stronger than that of its free ligand. The photoluminescence studies demonstrate that **1** and its ligand are viable options for potential use as emitting materials in organic electroluminescent devices (OLEDs).

Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC No. **1574774** for (**1**). Copy of the data can be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44-1223-336-033; e-mail: deposit@ccdc.cam.ac.uk or www: <http://www.ccdc.cam.ac.uk>).

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