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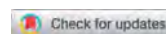
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# Synthesis, characterization and photoluminescence studies of new Cu(II) complex

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## ABSTRACT

In the current work, a new coordination complex,  $[\text{Cu}(\text{HL})_2]$ , **1** [ $\text{H}_2\text{L} = 2\text{--}((\text{E})\text{--}(2\text{--hydroxypropylimino)methyl)\text{--}4\text{--nitrophenol}]$ ] was successfully synthesized and characterized by IR, UV-vis and photoluminescence spectroscopic techniques, single crystal and powder X-ray diffraction measurements. In the crystalline structure of complex **1**, the aliphatic  $\text{--OH}$  group of the ligand is not coordinated and points away from the metal coordination zone, and also actively participates in intermolecular bifurcated  $\text{O--H}\cdots\text{O}$  hydrogen bonds which link the molecules to form hydrogen-bonded linear chains.  $\text{C--H}\cdots\pi$  and  $\pi\cdots\pi$  contacts also connect the molecules in the structure which form to 3D structure. This hydrogen bonded polymeric networks lie in the  $bc$ -plane and stacks along to the  $a$ -axis. Furthermore, complex **1** and its ligand  $\text{H}_2\text{L}$  display an intense navy-blue emission and blue emission in the solid state at room temperature, respectively, when they are excited under UV light.

## KEYWORDS

Copper(II) complex; crystal structure; photoluminescence

## 1. Introduction

Schiff bases are considered as a very important class of organic compounds because of their ability to form complexes with transition metal ions and widely used for industrial purposes [1–6]. Due to having great interesting properties of Salen-type schiff-bases metal complexes, they could be found various potential applications areas in the literature [7–10]. These application and usage areas can ordered as single-molecule magnets (SMMs), luminescent probes, catalyst in cleavage reactions for specific biological components as like DNA and RNA, non-linear optics and as a performance enhancer of absorber or buffer layer in photovoltaic devices [11–20].

The beneficial effect of transition metal compounds on the crystal structure and photoluminescence properties of the new designed Schiff bases derived from salicylaldehyde and its derivatives have been a subject of magnificent interest since its discovery [5]. It can be easily seen that Schiff base complexes is used as materials during growing OLEDs [21–23] and is used as fluorescent sensors for the detection of certain metal ions [24] due to the having

an interesting luminescence properties. One of the most promising candidates of this kind of complexes are Schiff-base copper complexes. Since copper(II) quenches luminescence, the examples of copper(I,II) and copper(II) luminescent complexes are rather scarce [25–27].

Over the last years, our research group and others have reported the synthesis, electronic-optic and magnetic properties of Cu(II) complexes containing ONNO, ONO and NNO type Schiff base ligands [28–32], but still there is need to explore the photoluminescence properties of these complexes and to synthesize new complexes with more properties. In view of these findings and in an effort to enlarge the library of such complexes, we report herein the synthesis, structural characterizations and photoluminescence studies of new Schiff base Cu(II) complex, [Cu(HL)<sub>2</sub>], **1**.

## 2. Experimental

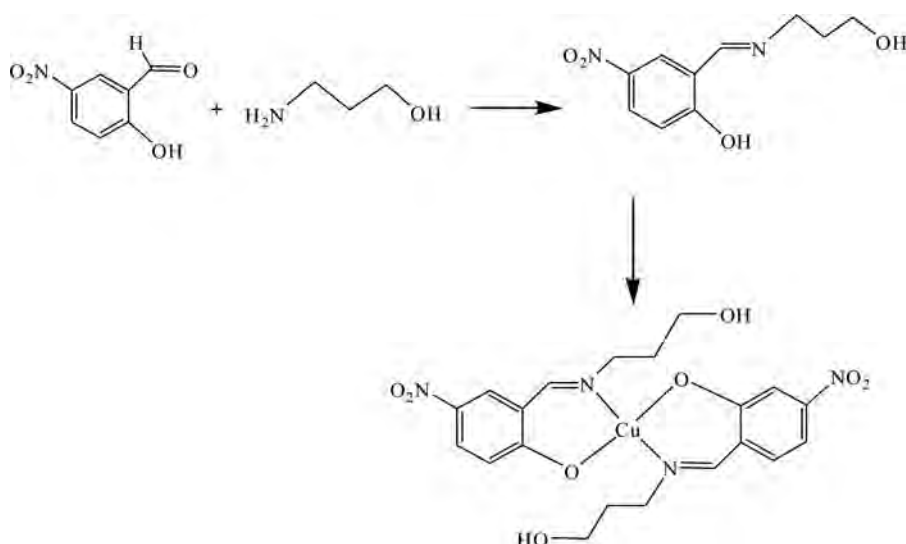
### 2.1. Materials and physical measurements

All chemical reagents and solvents were purchased from Sigma Aldrich and used without further purification. Elemental (C, H, N) analyses were carried out by standard methods with a LECO, CHNS–932 analyzer. Solid state UV-visible spectra were measured at room temperature with an Ocean Optics Maya 2000Pro Spectrophotometer. FT-IR spectra were measured with a Perkin-Elmer Spectrum 65 instrument in the range of 4000–500 cm<sup>-1</sup>. The solid state UV-vis spectra were determined by Ocean Optics Maya 2000Pro Spectrometer (230–550 nm). Solid state photoluminescence spectra in the visible region were measured at room temperature with an ANDOR SR500i-BL Photoluminescence Spectrometer, equipped with a triple grating and an air-cooled CCD camera as detector. The measurements were done using the 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 as the source. Powder x-ray diffraction (PXRD) patterns were recorded on a Bruker-AXS D8-Advance diffractometer by using Cu-K $\alpha$  radiation ( $\lambda = 1.5418 \text{ \AA}$ ) in the range  $5^\circ < 2\theta < 50^\circ$  in  $\theta$ - $\theta$  mode with a step ns ( $5 \text{ s} < n < 10 \text{ s}$ ) and step width of  $0.03^\circ$ . Comparison between experimental and calculated (from CIF's) PXRD patterns was performed with Mercury 3.8 [33].

X-ray single crystal data for complex **1** was collected on an Oxford Diffraction Gemini a Ultra diffractometer at 150 K using  $MoK\alpha$  radiation ( $\lambda = 0.71073 \text{ \AA}$ ). The data were collected for Lorentz, polarization and absorption effects using the analytical numeric absorption correction technique [34]. The structures were solved by direct methods using SHELXS [35] and refined by full-matrix least-squares based on  $|F_{\text{obs}}|^2$  using SHELXL [35]. The non-hydrogen atoms were refined anisotropically, while the hydrogen atoms, generated using idealized geometry, were made to “ride” on their parent atoms and used in the structure factor calculations. Details of the supramolecular  $\pi$ -interactions were calculated PLATON 1.17 program [36].

### 2.2. Synthesis of Schiff base ligand, H<sub>2</sub>L

The Schiff base ligand, **H<sub>2</sub>L** was synthesized by the reaction of 2-hydroxy-5-nitrobenzaldehyde (1 mmol, 0.167 g) with 3-amino-1-propanol (1 mmol, 0.075 g) in hot methanol (50 ml). The solution was stirred at 65°C for 30 min. The yellow product of the ligand precipitated from the solution on cooling. **H<sub>2</sub>L**: Yellow compounds, yield 80%. Anal. Calcd. for C<sub>10</sub>H<sub>12</sub>N<sub>2</sub>O<sub>4</sub> (%): C, 53.57; H, 5.39; N, 12.49. Found (%): C, 53.54; H, 5.37; N, 12.46.



**Scheme 1.** The synthetic route of complex **1** evaluated in this study.

### 2.3. Synthesis of Complex 1

Complex **1** was prepared by addition of copper(II) acetate monohydrate (0.199 g, 1 mmol) in hot methanol (30 cm<sup>3</sup>) to the ligand (**H<sub>2</sub>L**) (0.224 g, 1 mmol) in hot ethanol (30 cm<sup>3</sup>). The resulting solution was filtered rapidly and then allowed to stand at room temperature. Several weeks of standing led to the growth of clear green crystals of complex **1** suitable for the X-ray analysis. The synthetic route of the complex **1** is outlined in [Scheme 1](#). Complex **1**: Clear green crystals, yield 70%. Anal. Calcd. for C<sub>20</sub>H<sub>22</sub>CuN<sub>4</sub>O<sub>8</sub> (%): C, 47.10; H, 4.35; N, 10.99. Found (%): C, 47.20; H, 4.39; N, 10.96.

## 3. Results and discussion

### 3.1. X-ray structure of 1

The crystal data and structure refinement details for complex **1** are listed in [Table 1](#). Selected bond lengths and angles for the complex **1** are given in [Table 2](#). A perspective ORTEP view with the atom labelling scheme of complex **1** is shown in [Fig. 1](#) while packing diagrams are given in [Fig. 2](#) and [3](#).

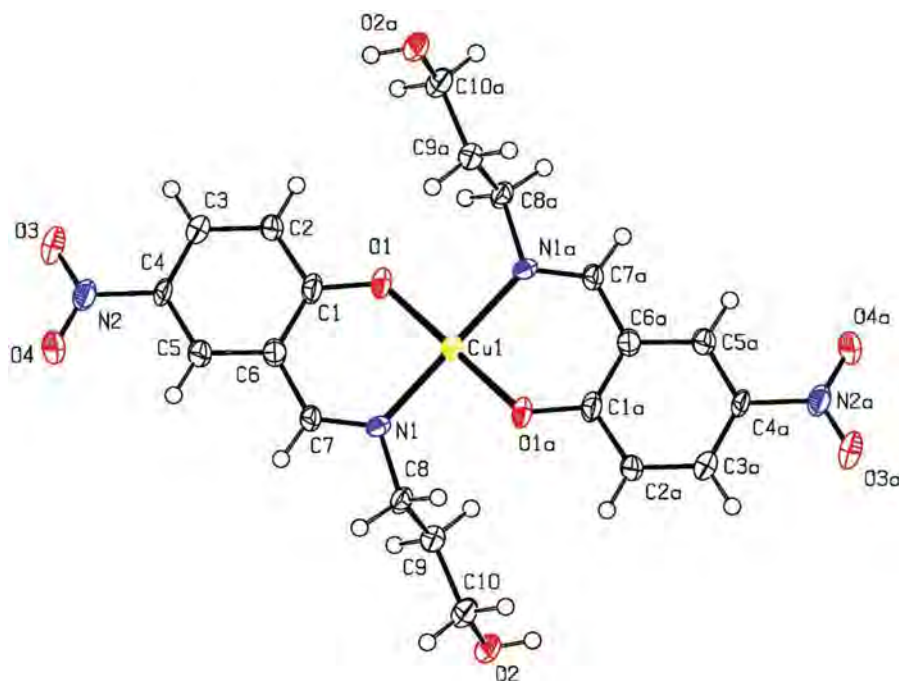
The complex **1** crystallizes in the monoclinic space group *P2<sub>1</sub>/c* and its asymmetric unit consists of a half of the monomeric [Cu(HL)<sub>2</sub>] unit. The Cu<sup>II</sup> atom is chelated by two bidentate Schiff base ligands by using two imine nitrogen and two phenolate oxygen atoms in a mutual *trans* disposition. In the equatorial plane, the bond lengths are Cu–O<sub>alk</sub> = 1.908 (5) Å and Cu–N<sub>imi</sub> = 2.009 (5) Å. As usually observed for four coordinate Cu<sup>II</sup> ion will include additional weak interactions with axial atoms, Cu1–O1<sup>i</sup><sub>alk</sub> = 3.322 (5) Å and Cu1–O1<sup>ii</sup><sub>alk</sub> = 3.322 (5) Å [*i* = 1 + *x*, *y*, *z*, *ii* = 1 – *x*, 1 – *y*, 1 – *z*]. If this weak Cu⋯O interaction are considered as part of the coordination geometry of the Cu<sup>II</sup> ion, the geometry of Cu<sup>II</sup> atom is best described as octahedral coordination with Jahn-Teller distortion. The bond lengths and angles lie well within the range of corresponding values reported for other mononuclear copper(II) complexes [28–32, 37].

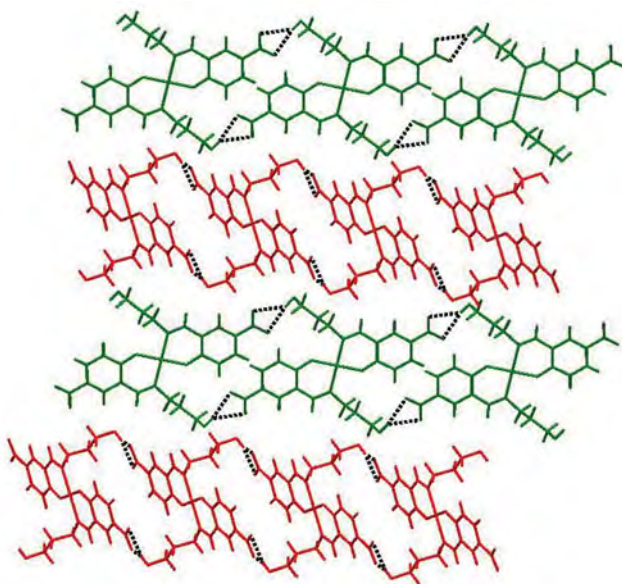
**Table 1.** Crystal data and structure refinement for complex **1**.

Chemical Formula	$C_{20}H_{22}CuN_4O_8$
Formula weight ( $\text{g mol}^{-1}$ )	509.95
Crystal system	Monoclinic
Space group	$P2_1/c$
Unit cell dimensions	$a = 4.2926 (7) \text{ \AA}$ $\alpha = 90^\circ$ $b = 11.7836 (15) \text{ \AA}$ $\beta = 103.088 (14)^\circ$ $c = 20.195 (2) \text{ \AA}$ $\gamma = 90^\circ$
$V / \text{\AA}^3$	995.0 (2)
$Z$	2
$\rho_{\text{calc}} / \text{g cm}^{-3}$	1.702
$\mu / \text{mm}^{-1}$	1.158
Temperature (K)	150.15
Reflections collected	6515
Independent reflections	2170
Goodness-of-fit on $F^2$	1.177
$R$ indices [ $I > 2\sigma(I)$ ]	$R_1 = 0.0959, wR_2 = 0.2491$

**Table 2.** Some selected bond lengths [ $\text{\AA}$ ] and angles [ $^\circ$ ] for complex **1**.

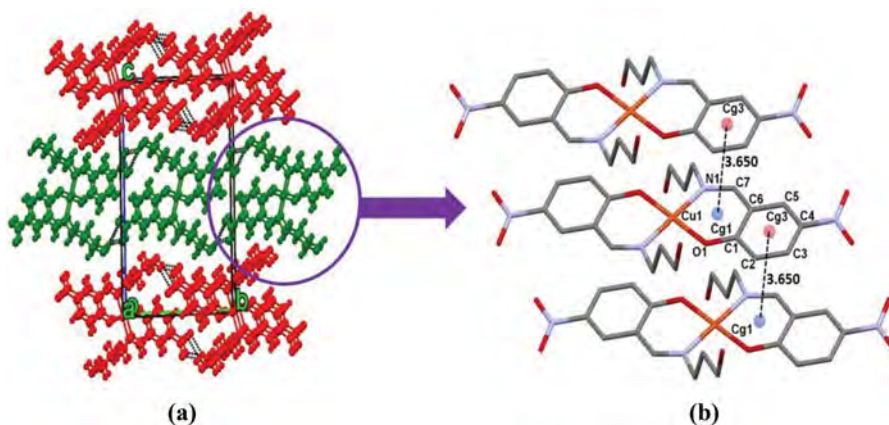
Cu1—O1	1.908 (5)	Cu1—N1	2.009 (5)
Cu1—O1 <sup>ii</sup>	3.322 (5)	O1 <sup>i</sup> —Cu1—N1	88.0 (2)
O1—Cu1—N1	92.0 (2)	O1 <sup>ii</sup> —Cu1—N1	81.9 (2)
O1 <sup>ii</sup> —Cu1—O1	107.2 (2)		

Symmetry code: (i)  $-x + 2, -y + 1, -z + 1$ , (ii)  $1 + x, y, z$ **Figure 1.** ORTEP drawing of complex **1** with atom labelling. Thermal ellipsoids have been drawn at 50% probability level [Symmetry code: (i)  $-x + 2, -y + 1, -z + 1$ ].



**Figure 2.** A perspective view of one-dimensional chain structures, showing the intermolecular O—H...O hydrogen bonds (dashed lines).

In the crystal structure of complex **1**, the aliphatic —OH group of the Schiff base ligand is not coordinated and points away from the metal coordination zone, and actively participates in intermolecular bifurcated O—H...O hydrogen bonds which link the molecules to form hydrogen-bonded linear chains (Fig. 2). The intermolecular  $\text{Cu}^{\text{II}} \dots \text{Cu}^{\text{II}}$  distance is 12.541 Å in this chain structure. Besides that,  $\text{Cu}^{\text{II}}$  atom is further linked into a discrete monomeric unit by a pair of long weak  $\text{Cu} \dots \text{O}$  interactions which will contribute a molecular stack along the *a* axis. The  $\text{Cu}^{\text{II}} \dots \text{Cu}^{\text{II}}$  distance within this stacking structure is 4.293 Å. Besides that intermolecular C—H... $\pi$  and  $\pi \dots \pi$  contacts also connect the molecules in the crystal structure which form to 3D structures (Table 3). This hydrogen bonded polymeric networks lie in the *bc*-plane and stacks along to the *a*-axis (Fig. 3).



**Figure 3.** (a) The crystal packing view of the complex **1** in the *bc* plane (b) Showing the distance between ring centroids for complex **1**.

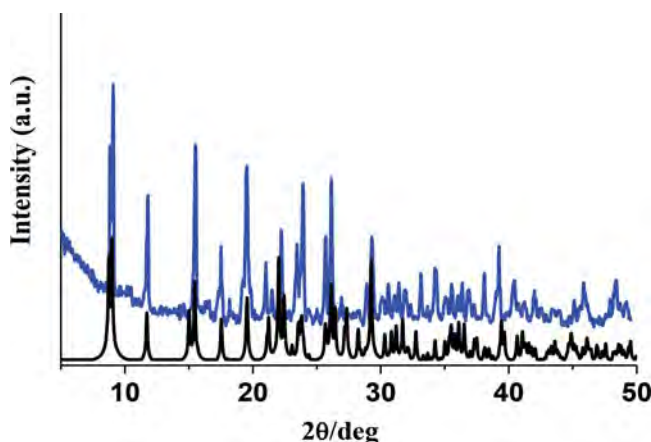
**Table 3.** Hydrogen bond geometry ( $\text{\AA}$ ,  $^\circ$ ) and distance between ring centroids [ $\text{\AA}$ ] for complex **1**.

D—H...A*	D—H	H...A	D...A	D—H...A	Symmetry
O2—H2...O3	0.85(6)	2.29(6)	3.109(7)	161(6)	1 + x, 1 + y, z
O2—H2...O4	0.85(6)	2.43(7)	3.137(8)	141(6)	1 + x, 1 + y, z
<b>C—H...Cg(I)</b>					
C8—H8B...Cg(1)	0.99	2.98	3.568	119	1 + x, y, z
C8—H8B...Cg(2)	0.99	2.98	3.568	119	3 - x, 1 - y, 1 - z
	<b>Cg(I)...Cg(J)</b>	<b>Cg(I)_Perp</b>	<b>Cg(J)_Perp</b>		
Cg(1)...Cg(3)	3.650(4)	-3.339(2)	3.407(3)		1 + x, y, z
Cg(3)...Cg(1)	3.650(4)	3.406(3)	-3.339(2)		-1 + x, y, z

\*D: Donor, A: Acceptor, Cg(I): Plane number I (= ring number in () above), Cg(I)-Cg(J) = Distance between ring Centroids, Cg(I)\_Perp = Perpendicular distance of Cg(I) on ring J, Cg(J)\_Perp = Perpendicular distance of Cg(J) on ring I, Cg(1): Cu1-O1-C1-C6-C7-N1, Cg(3): C1-C2-C3-C4-C5-C6

### 3.2. X-ray powder diffraction pattern of **1**

Before proceeding to the spectroscopic and photoluminescence characterization, we note that experimental powder X-ray pattern for complex **1** are well in position with those of simulated patterns on the basis of single crystal structure of complex **1** which is in agreement of their single phase and purity (Fig. 4).

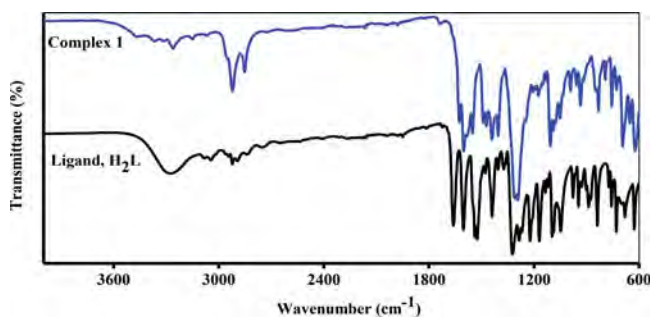


**Figure 4.** X-ray powder diffraction pattern of complex **1** (Black- simulated from CIFs, Blue Experimental).

### 3.3. IR and UV-Vis analysis

The IR and solid state UV-Vis spectra of complex **1** were shown in comparison with that of its free ligand  $\text{H}_2\text{L}$  which are in agreement with its single crystal structure analysis (Figs. 5 and 6).

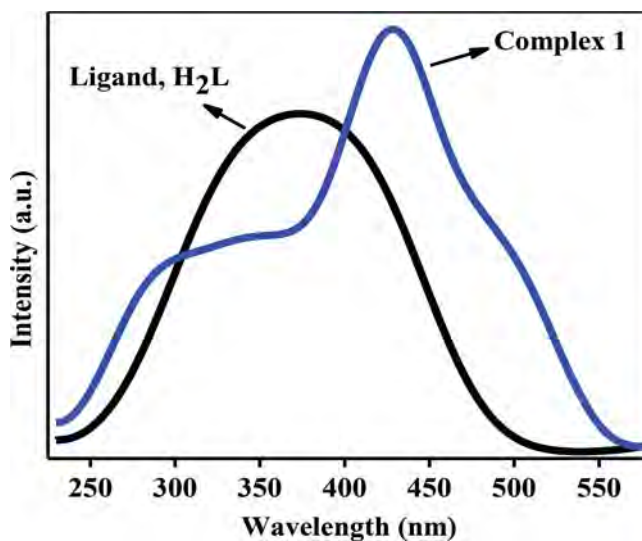
As seen from Figure 5, the IR spectra of the free ligand and its complex **1** depicts peaks in nearly similar region. However, some significant variations have been illustrated in the spectra of the complex **1** and its free ligand  $\text{H}_2\text{L}$ . The IR spectra of the free ligand  $\text{H}_2\text{L}$  exhibited the prominent broad band at  $3274\text{ cm}^{-1}$  which could be attributed to (O-H) stretching vibrations. This band is disappeared in complex **1** indicating deprotonation of the phenolic hydroxyl group and coordination of phenolic oxygen to the metal ion [38]. The characteristic stretching vibrations for  $\nu(\text{C}=\text{N})$  bond is shifted from  $1659\text{ cm}^{-1}$  (for ligand  $\text{H}_2\text{L}$ ) to  $1626\text{ cm}^{-1}$  (for complex **1**), which indicates the coordination of the imine nitrogen atom to



**Figure 5.** IR spectra of the free ligand  $\text{H}_2\text{L}$  and its complex **1**.

the Cu(II) center [39]. The symmetric and asymmetric stretching bands appeared at the range of  $1550\text{--}1405\text{ cm}^{-1}$  which attributed to the  $\nu(\text{C--NO}_2)$  for the free ligand and complex **1** [40].

As seen from Figure 6, the UV-Vis spectrum for the ligand  $\text{H}_2\text{L}$ , a prominent broad absorption band appears at  $\lambda_{\text{max}} = 374\text{ nm}$  which may be assigned to the  $\pi\text{-}\pi^*$  transitions [41]. The absorption spectra of the complex **1** displayed different absorption pattern as compared to the ligand  $\text{H}_2\text{L}$ . The absorption peak was observed with the maxima at  $428\text{ nm}$  in the spectrum of complex **1**, could be assigned to  $\pi\text{-}\pi^*$  or  $n\text{-}\pi^*$  transition of the its ligand  $\text{H}_2\text{L}$  [42]. The shifting of absorption bands in the spectra of the complex **1** signifies the copper(II) ion coordination with the ligand  $\text{H}_2\text{L}$  [7].

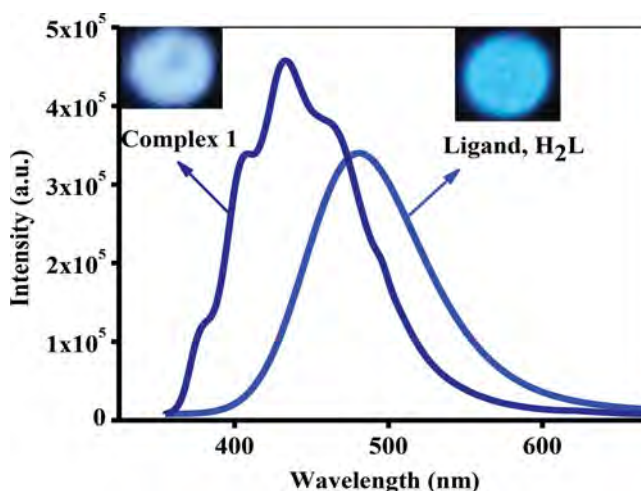


**Figure 6.** The solid state UV-Visible spectra of the free ligand  $\text{H}_2\text{L}$  and its complex **1**.

### 3.4. Photoluminescence properties

Figure 7 depicts the solid-state photoluminescence emission spectra of the free ligand and its complex **1** at ambient temperature in the visible regions. As seen from the Figure 7, the emission spectrum of the free ligand displayed a navy-blue emission peak at  $\lambda_{\text{max}} = 480\text{ nm}$  which may be assigned to the  $n\text{-}\pi^*$  or  $\pi\text{-}\pi^*$  electronic transition (ILCT) [38, 43, 44] whereas the emission spectrum of complex **1** revealed intense blue emission peak at  $\lambda_{\text{max}} = 433\text{ nm}$  upon excitation at  $349\text{ nm}$ . The observed blue shift of the emission maximum between the complex





**Figure 7.** Room temperature solid state photoluminescence spectrums of the free ligand  $\text{H}_2\text{L}$  and complex **1** (Upper-left photo is photoluminescent image of complex **1** and upper-right photo is photoluminescent image of ligand  $\text{H}_2\text{L}$ , while excited at 349 nm).

**1** and the free ligand  $\text{H}_2\text{L}$  was considered to mainly originate from the influence of the coordination of metal atom to the ligand. In addition, the increase of the luminescence intensity of complex **1** as compared to its free ligand  $\text{H}_2\text{L}$ , could be due to the fact that upon the formation of metal complex, which effectively increases the “rigidity” of the ligand and thus reduces the loss of energy via radiationless thermal vibrations [45–48].

#### 4. Conclusions

The synthesis and structural characterization of a new Schiff base  $\text{Cu}^{\text{II}}$  complex, **1**, has been presented together with an investigation into its photoluminescence properties. Spectroscopic techniques confirmed the metal ligand coordination and single x-ray results show that the  $\text{Cu}^{\text{II}}$  atom is coordinated by two singly deprotonated Schiff base ligands ( $\text{H}_2\text{L}$ ) using ON donors. The aliphatic  $-\text{OH}$  group of the ligand is not coordinated and points away from the metal coordination zone. The intermolecular  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds link the molecules which form 1D chains along  $b$  axis. And these chains are further interlinked via  $\text{C}-\text{H}\cdots\pi$  and  $\pi\cdots\pi$  interactions resulting in 3D networks. Additionally, photoluminescence studies show that under the excitation of UV light, intense navy-blue emission for complex **1** and blue emission for its free ligand  $\text{H}_2\text{L}$  are exhibited in the visible regions. The luminescent performances making this complex may be good candidates for potential luminescence materials.

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#### Supplementary data

Crystallographic data for the structure reported in this article have been deposited with the Cambridge Crystallographic Data Centre (The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK;

deposit@ccdc.cam.ac.uk; www: <http://www.ccdc.cam.ac.uk>; and are available free of charge on request, quoting the Deposition No. CCDC 1513023.

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