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Cu(II) coordination polymer bearing diazenyl-benzoic ligand: Synthesis, physico-chemical and XRD/HSA-interactions

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ABSTRACT

New binuclear copper(II) complex $[Cu_2L_2]$ zig-zag coordination polymer (CCP) have been synthesized *via* one-pot copper(II) acetate/(E)-2-((2-hydroxynaphthalen-1-yl)diazenyl)benzoic dye through an in situ aqua doubly deprotonated ligand substitution reaction. The coordination polymer complex was characterized by spectroscopic and CHN-elemental analysis. The crystal structure **has** been specified by the XRD-method exhibiting that the Cu(II) center geometry is with five-coordinated distorted tetragonal pyramidal. The desired CCP structure chains are connected into a 2D-Supramolecular assembly *via* one C-H…O H-bond and enhanced by a $\pi-\pi$ stacking interactions. These interconnections strengthened the lattice of CCP that originated the formation of super thermal CCP complexes. The fundamental vibrational wavenumbers and electron transfer in the free ligand are combined with the complex before and after coordination to demonstrate the spectral behavior of L^{-2} in the desired zig-zag CCP. The 2D-fingerprint (2D-FP) and Hirshfeld surface analysis (HSA) computations were served to prove the 2D-network packed crystal lattice interactions.

1. Introduction

Azo ligands are very important in the field of pigments, dyes and advanced material. These compounds demonstrate one of the most broadly applied organic materials that are appealing to scientists due their different applications [1-3]. They can coordinate to transition metal in a wide coordination mode and employ the N as a ligating donor [4-6]. In both applied and basic research, azo-metallic chelates compounds are of a great importance due to their peculiar electronic and structural characteristics and their use in different crucial fields [7-10]. All these features create the possibility of obtaining new compounds with various biological activities [11,12], like inhibition of DNA, RNA, carcinogenesis, protein synthesis and nitrogen fixation [13]. They are important dyes for synthetic vinyl polymers and leather. Metal-azo pigments are also used in the DVD-R recording layer since they are with high thermal stability [14-16].

Currently, azo-metallic complexes have exhibiting photochromic activity [17,18], interacting strongly with CT DNA [19] and found application in (C–C) coupling reactions [20-21].

Over the last decades, the coordination polymers had an interesting attention because of their potential applications and diverse structural properties [22-25].

The presence of carboxyl functional group in ligands is an acceptable candidate for the building of a metal coordination polymer due to its multi coordination modes [26-29]. Synthesis and design of metallic coordination compounds which contain carboxyl ligands is an active area of research due to its potential applications in the fields of molecular magnetism [30,31]. Thus, many chemists have developed strategies to prepare and design suitable polydentate ligands to develop new coordination polymers applications [32-34]. In general, copper coordination complexes have

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many industrial applications like catalysis [35-38], photo-catalysis, nonlinear optics, magnetism dye adsorption, gas sorption, conductivity, luminescence and chirality [39-40]. Where most of their electronic and photochromic efficiency have been explained by DFT calculations.

2. Experimental

2.1. Measurements and materials

All the reagents and chemicals were obtained through commercial sources and used without further purification. UV spectra were recorded on an Agilent UV-VIS spectrophotometer 8453 (spectroscopy system) with G1120A multicell transport and a computer with ChemStation (G Visible Ultra Violet 1120 A). Infrared spectra were recorded with a Fourier transform infrared spectrometer (ALPHA) FTIR from the brand BRUKER controlled by Opus 6.5 software and fitted with and Attenuated Total Reflectance (ATR) accessory in diamond crystal.

2.2. XRD-analysis

A crystal of dimension [0.040 × 0.045 × 0.050 mm] was selected and mounted on a Bruker Apex II [41], CCD area detector diffractometer with a graphite monochromated Mo-Kα radiation source (0.71073 Å), intensities were collected at 293 (2) °K. The resulting set of hkl was used for structure solution and refinement. Structure was solved by direct methods with SIR 2002 [42], to locate all the non-H atoms which were refined anisotropically with SHELXL97 [43] using full-matrix least-squares on F2 procedure within the WinGX [44] suite of software used to prepare the material for publication. Absorption corrections were performed with the MULABS program [45]. The H atoms were included in calculated positions and treated as riding atoms: C-H = 0.93 Å with Uiso(H) = 1.2 Ueq(C). The Mercury 3.8 [46] for Windows program were used for generating figures of structures. Structure refinement details, crystal data and data collection are summarized in Table 1. Some selected bond distances and angles are listed in Table 2.

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2.3. Computational details

The Cif file crystallographic data was taken as reference for the calculation when HAS was perfomed using the CRYSTAL EXPLORER 3.1 [47]

2.4. Synthesis

((E)-2-((2-hydroxynaphthalen-1-yl)diazenyl)benzo)Copper(II), was obtained by mixing 2 mmol of (E)-2-((2-hydroxynaphthalen-1-yl)diazenyl)benzoic acid and 1 mmol of $Cu(OAc)_2.H_2O$ dissolved in a mixture of THF/MeOH (10/10ml), that is was stirred at 298 °K for 48 h. Volatile materials were removed under vacuum and the residue was washed twice from hexane solution to give red solid. The resulting solid was crystallized from CH_2Cl_2/hexane (1: 5) to finally lead to the formation of dark red crystals, Yield: 86%. Elementary analysis was carried out to confirm the formula of the complex. Anal. Calc. for $C_{17}H_{10}CuN_2O_3$, C, 57.71%, N, 7.92%, and H, 2.85%, found C, 57.02%, N, 7.671%, and H, 2.95%, TOF-MS = 354.8 $[M+1]^+$ m/z.

Table 1
Crystallographic data and structure refinement details of the coordination polymer.

Empirical formula	$C_{17}H_{10}CuN_2O_3$
CCDC	2040435
Formula weight, g/mol	707.62
Space group	$P2_1$ /c
Crystal system	Monoclinic
Dimensions du monocristal (mm^3)	0.045 × 0.050 × 0.040
F(000)	716
Refinement method	full-matrixleast-squareson *F2*
a (Å)	13.3284 (13)
b (Å)	5.4998(3)
c (Å)	18.5250(19)
α(°)	90
β(°)	90.971 (4)
γ(°)	90
V($Å^3$)	1357.8(2)
Z	2
Temperature/K	293(2)
θ Range for data collection (°)	2.699 - 27.423
Radiation	Mo Kα (λ= 0.71073)
Dcalcd (g/cm^{-3})	1.731
Range/indices	-17≤ h ≤17, -7≤ k ≤7
(h,	0≤ l ≤20
Ref Nmb of reflections measured	4875
Independent reflections	2881
Reflections with I > 2σ(I)	1414
Number of parameters	208
Absorption coefficient (mm−1)	1.626
Goodness-of-fit (GOF)	0.831
wR(F2)	0.0712
Rint	0.0471

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

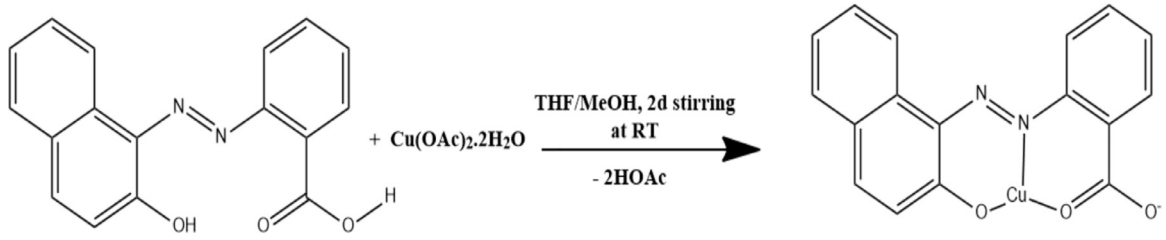
Bond	Bond lengths (Å)	Bond Angle	Angle (°)
Cu1-O1	1.882(2)	O2-Cu1-O1	171.29(11)
Cu1-O2	1.878(2)	O2-Cu1-N1	94.17(12)
Cu1-O3	2.045(2)	O1-Cu1-N1	94.34(12)
Cu1-O2	2.454(26)	O2-Cu1-O3	85.45(11)
Cu1- N1	1.955(3)	O1-Cu1-O3	85.99(10)
C1-N1	1.442(5)	N1-Cu1-O3	178.45(12)
N1-N2	1.294(4)	C8 O1 Cu1	126.3(3)
N2-C7	1.345(5)	N1 N2 C7 . . .	124.2(3)
O1-C8	1.284(4)	N2 N1 C1	111.6(3)
		N2 N1 Cu1	124.1(2)
		C1 N1 Cu1	124.2(2)
		C17 O2 Cu1	130.4(3)

3. Results and discussion

3.1. Synthesis, MS, X-ray and CHN analysis

Desired coordination polymer complex was prepared *via* one pot addition reaction, mixing two equivalent amount of the ligand with one equivalent of $Cu(OAc)_2.2H_2O$ in a mixture of THF/MeOH, under vigorous stirring condition for 48h, at room temperature, as seen in Scheme 1. The presence of acetate anions, easily exchangeable, allows the complexation of the organic molecule. The product was isolated as solid brown powder, soluble in DMF, DMSO and CH_2Cl_2, the compound was invented by IR, MS, UV-Vis, spectroscopy, CHN-EA, and XRD-analysis. The spectra of UV–vis. And IR of the ligand and the corresponding complex were recorded in CH_2Cl_2.

Crystals of complex grown in CH_2Cl_2/hexane solution through slow evaporation, then suitable crystal was collected and analyzed through X-rays diffraction analysis. The complex $[Cu_2L_2]$ crystallizes in a monoclinic system with a $P2_1/c$ space group and we note that is binuclear, the ligand engages in a doubly deprotonated



Scheme 1. Synthesis of the compound.

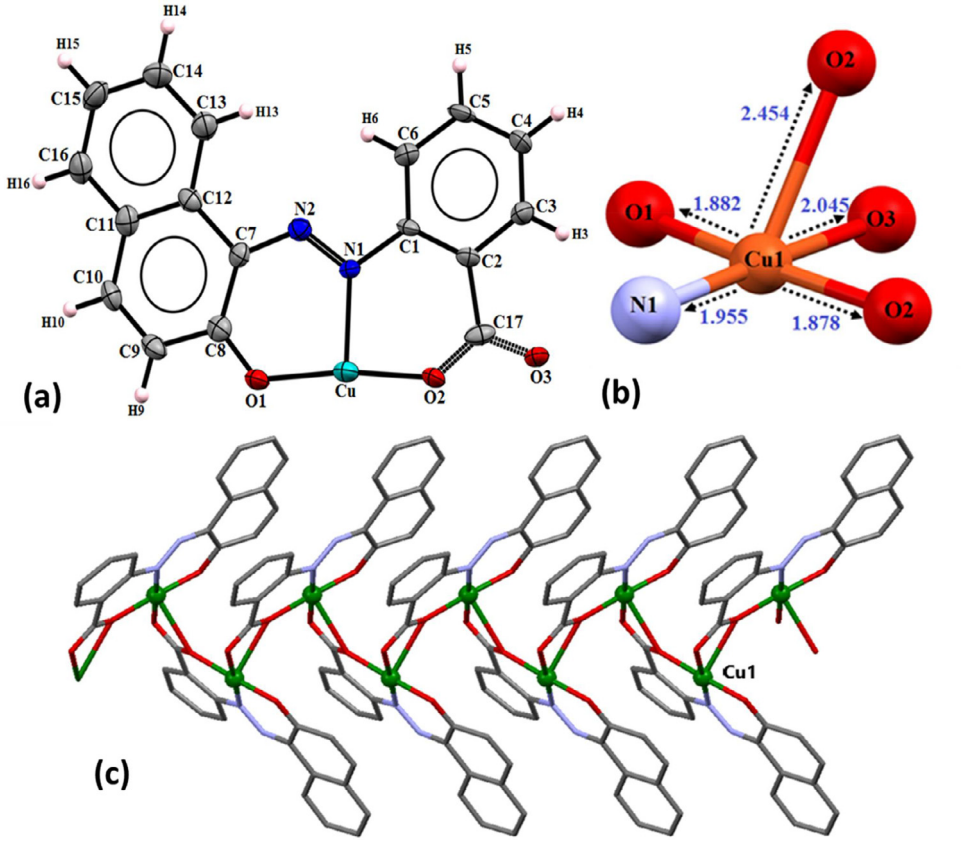


Fig. 1. Views of CCP $[Cu_2L_2]$ showing: (a) the asymmetric unit, (b) the metal environments, (c) the coordination modes of the ligand, hydrogen atoms here have been omitted for clarity.

manner, involving an anionic ligand. The asymmetric unit (Fig. 1a) consists of one copper (II) atom (localized on a general position) coordinated by nitrogen atom in α of the benzene ring and by O1 oxygen of naphthol, and an O2 oxygen atom of the benzoic. The coordination sphere is completed by two more oxygen atoms O2 and O3 of the benzoic from another ligand witch, O2 occupied axial position, the others atoms forming the equatorial plane, exhibiting a distorted tetragonal pyramid geometry (4+1) coordination (Fig. 1b). In fact, the oxygen atom O2 (in axial position), acts as a bridging atom between the two Cu(II) centers, thus forming a dimeric structure in the form of two rings with four and six members fused at the edges. The propagation of this fused-ring motif generates a coordination polymer chain running parallel to the [010] direction, so the ligand is doubly bridged (Fig .1c).

A hydrogen bond C-H… O (Table 3) serves as a link between the chains and leads to edge-fused rings R22(18) motifs [48], augmented by π …π interaction, involving between benzene rings helps to stabilizing the crystal structure (Fig. 2a). Hence, the formation of infinite layers unfolds in zig-zag parallel to the (bc) plane [49] (Fig. 2b). Ring-centroid separation is 3.955 (10) Å

Table 3
Distances (Å) and angles (°) of hydrogen bond for complex.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···O1^{i}	0.95	2.76	3.564 (15)	143

and the distance from the centroid of one ring to the plane of the other is 3.981 (10) Å, corresponding to a ring-centroid offset of *ca* 0.21 Å. In the dimer, the Cu/Cu distance is of 4.085 (3) Å, which is similar to than those distances found in the bis(salicylaldehydato) Cu(II) complex (4.05 (4) Å) [50,51]. Indeed, this Cu/Cu distance is shorter than those found in many dinuclear Cu(II) oxygen bridged complexes [52–58]. The dihedral angle between the plane of the C7–C13 naphthalene ring system and the benzene ring is 14.15° (2). the packing of the unit cell shows that two molecules of the dimer form a dihedral angle of 79.48° (3). While the three equatorial bonds to Cu1-O1, Cu1-O2 and Cu1-O3 are significantly different in length, the Cu1-O3 bond length (2.045 (2) Å) is longer than the other distances of (1.878 (2) and 1.882 (2) Å) respectively (Table 2), this difference is due to the

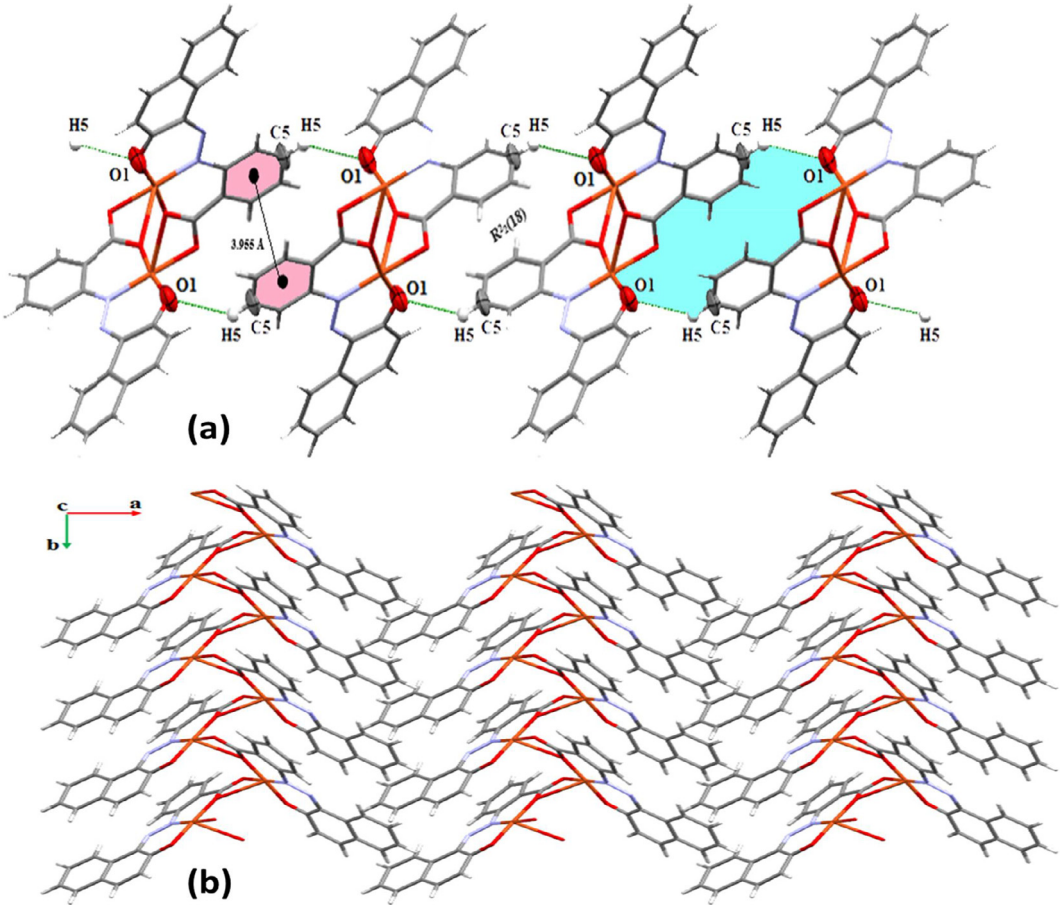


Fig. 2. (a) View of part of the crystal structure, showing the formation of a hydrogen-bonded chain running parallel to the [010] direction, comprising cage-like aggregates containing $R^2_2(18)$ rings and $\pi...\pi$ interaction. Dashed lines indicate hydrogen bonds, and (b) Packing arrangement of complex viewed along the (ab) plane.

Jahn–Teller distortion for Cu(II). the distance between nitrogen and metal center is similar to the value found in the Bis{(E)- 1-((2,4,6-tribromophenyl)diazenyl)naphthalen-2-olate}Copper(II) [57], but shorter than that observed at $[Cu_2(C_{17}H_{13}N_2O_2)_4]$ [56].

3.2. HSA and 2D-FP

To gain more information about the interactions in lattice of the crystal of the desired coordination Cu(II) complex, HSA analysis is performed using the original CIF file. The 3D-Hirshfeld map d_{norm} and shape index were performed in the range -0.654 to 1.464 a. u. Since the complex contains many heteroatoms that have lone pairs of electrons like 2N and 4O atoms and which act as protons acceptors, ~~therefore,~~ several of short contacts will be established between the computed molecule and its neighbor's molecules [59–62]. The d_{norm} of the computed complex reflects the surface with 6 red-holes (Fig. 3a), some of these points are large which reflects the strongest and shortest bonding like O...H or N...H hydrogen bonds and others smaller point reflect the weak and long contractions like Van der Waal forces. Interestingly, it is noticed the presence of the red points in two different directions with a straight angle 180° consistent with consisting of chain of hydrogen bonds in the lattice of the coordination polymer Cu(II) complex as seen in Fig. 3a. The shape index of the complex revealed the presence of electrophilic polar proton (blue) and several red nucleophilic functional groups on the complex surface (Fig. 3b). Moreover, the fingerprint (FP) of H-to-each atom percentage interactions were figured as in Fig. 3c. The result evidenced of the H····H interactions is the largest percentage participation and its complex surface with 47.7%, meanwhile, the contribution of H....N and H...Cu 0.1% is the smallest. The total 2D-FP H-to-toms ratios found to be as [H···H>C····H>O····H>N····H = Cu····H] order.

3.3. FT-IR investigations

The FT-IR of the CCP was matched to the free ligand as illustrated in Fig. 4. The absence of stretching vibration of O—H in the free ligand ~ 3500 cm^{-1} is because of the existence of intra-H-bond as O-H...N [35]. Meanwhile, the bending of the same bond appeared at 1750 cm^{-1}. The C_{Ph}-H stretching vibrations in the free ligand and the complex of phenyl groups appeared at ~ 3100 cm^{-1}. The important changes that distinguished the CCP spectrum over the ligand one are: a) the totally disappearance of O-H bending peak at 1700 cm^{-1}, b) shifting of carbonyl functional group vibration in free ligand from 1660 cm^{-1} to 1615 cm^{-1}in CCP, and c) the appearing of new peaks at ~ 550 due to the Cu-O new bonds in the CCP (Fig. 4b).

3.4. UV-Vis

With the goal of understanding ligand coordination behavior, electronic absorption spectra of the free ligand before and after reaction with Cu(II) complex to form the desired CCP were carried out in CH_2Cl_2 (10^{-4} M). The ligand showed a complex spectrum, several absorptions bands at 236 nm attributed to π to π^*, broad beak with two maxims 292 and 304 nm attributed to π to n, and

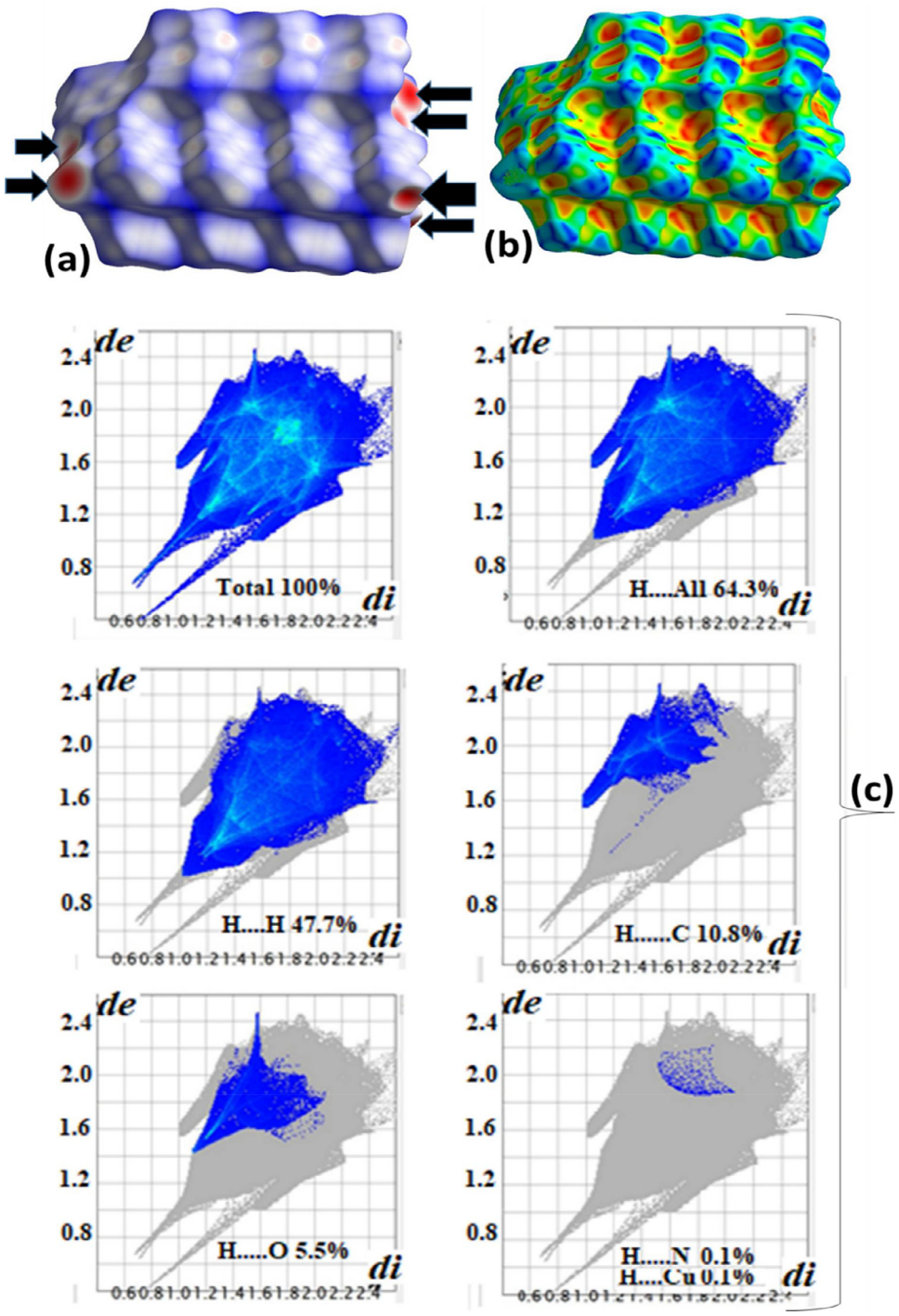


Fig. 3. (a) d_{norm}, (b) shape index., and (d) inside/outside 2-D-FP percentage plots of CCP.

another broad peak with two maxims at 490 and 500 nm which can be sited at n to π^* e-transfer (Fig. 5a). The significant band in the CCP solution is the broad new band at 675 nm that clearly attributed to d-d distorted tetragonal pyramidal geometry electron transfer. The copper coordination polymer solution showed similar absorption behavior but with hypsochromic and bathochromic shifts in relation with the free ligand in solution (Fig. 5).

3.5. Thermal stability

Thermogravimetric analyses TGA of the desired CCP was performed in between 0-800 °C in an open atmosphere (Fig. 6) with heat rate ~ 10 °C/min. The Cu2L2 complex revealed an excellent super thermal behavior, it is stable up to ~ 400 °C without having uncoordinated or coordinated H2O molecules, weight lost in between 70-170 °C, and this corresponds to the XRD-result and FT-IR. The CCP mass was declining in a wide exothermic step started from 390 °C and ended at 650 °C with TDTA = 480 °C. The CCP decomposed in one-step thermal mechanism process, where 81% of the weight was lost since the organic ligand was de-structured or decomposed to light gasses at ~ 400 °C leaving 18.4 % CuO as the final inorganic product.

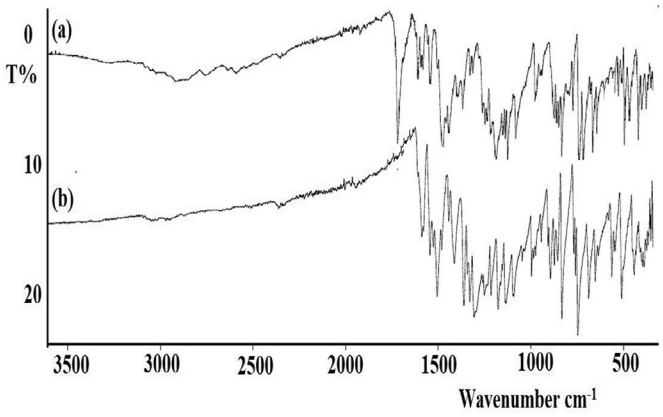


Fig. 4. FT-IR: (a) the free ligand, and (b) CCP.

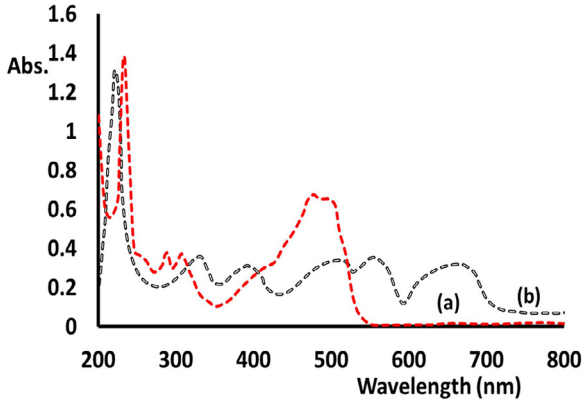


Fig. 5. (a) UV-Vis in CH_2Cl_2 (a) free ligand and (b) CCP.

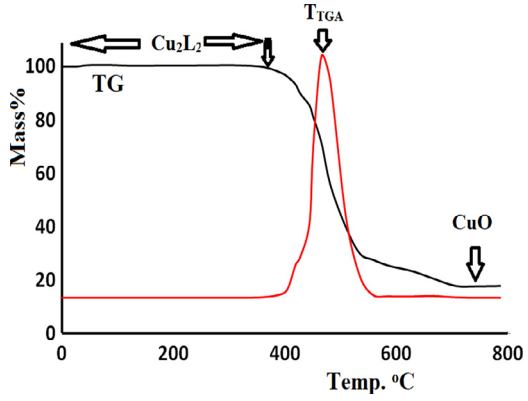


Fig. 6. TGA of the desired CCP.

4. Conclusion

The Cu_2L_2dimer zig-zag CCP complex was successfully synthesized via one-pot substitution reaction of (E)-2-((2-hydroxynaphthalen-1-yl)diazenyl)benzoic acid L^{-2} dye to Cu(II). The 2D-self-assembly CCP was successfully prepared in good yield at RT reaction. The molecular interactions are furthermore expanded to the 2D-supramolecular framework via $\pi-\pi$ stacking weak interactions and C-H...O strong H-bond. Interestingly, it is noticed that the CCP is super thermal stabile in high open atmosphere owing to the formation of 2D-supramolecular zig-zag framework dimer with five-coordinated distorted tetragonal pyramidal CCP rigid Cu(II) matrix. Such zig-zag interactions were computed via HSA to prove the 180° 2D-strong interaction patterns. The coordination mode of the L^{-2} ligand were successfully monitored via UV-vis spectroscopy as well as FT-IR.

Declaration of Competing Interest

The authors declare that they have no conflicts of interest.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.molstruc.2020.129604.

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