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Syntheses, structures, and properties of supramolecular complexes with bipyridine and amino-Schiff-base derived from salicylaldehyde with glycine

Feng Ying Bai^{ab}; Xiao Tian Li^a; Hai Yan Zhao^c; Jing Han^c; Yongheng Xing^c

^a Department of Material Science and Engineering, Key Laboratory of Automobile Materials of Ministry of Education, Jilin University, Changchun, 130012, P.R. China ^b College of Life Science, Liaoning Normal University, Dalian, 116029, P.R. China ^c College of Chemistry and Chemical Engineering, Liaoning Normal University, Dalian 116029, P.R. China

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Syntheses, structures, and properties of supramolecular complexes with bipyridine and amino-Schiff-base derived from salicylaldehyde with glycine

FENG YING BAI^{†‡}, XIAO TIAN LI^{*†}, HAI YAN ZHAO[§],
JING HAN[§] and YONGHENG XING[§]

[†]Department of Material Science and Engineering, Key Laboratory of Automobile Materials of Ministry of Education, Jilin University, Changchun, 130012, P.R. China

[‡]College of Life Science, Liaoning Normal University, Huanghe Road 850#, Dalian, 116029, P.R. China

[§]College of Chemistry and Chemical Engineering, Liaoning Normal University, Huanghe Road 850#, Dalian 116029, P.R. China

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The reactions of stoichiometric amounts of $\text{Cu}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ or $\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ with KHL (potassium salt of salicylidene-glycine) and 4,4'-bipy or 2,2'-bipy ethanol afforded $[\text{Cu}_2(\text{L})_2(\mu\text{-}4,4'\text{-bipy})(4,4'\text{-bipy})_2] \cdot 2\text{H}_2\text{O}$ (**1**) (L = salicylidene-glycine) and $\text{Ni}(\text{OAc})_2(2,2'\text{-bipy})(\text{H}_2\text{O})_2$ (**2**), respectively. The structure of **1** contains two Cu(II)'s, bridged by 4,4'-bipy; each copper is square pyramidal. Complex **2** contains a Ni(II) in highly distorted octahedral geometry. The two complexes were constructed into 3-D interwoven networks by typical H-bonding (i.e., $\text{O}-\text{H} \cdots \text{O}$ and $\text{N}-\text{H} \cdots \text{O}$) and some other intra- or intermolecular weak interactions (i.e., $\text{C}-\text{H} \cdots \text{O}$).

Keywords: Supramolecular complex; Crystal structure; Spectroscopic properties; Amino-Schiff-base

1. Introduction

Helical structures and superstructures have received considerable attention in biomimetic and self-assembled supramolecular systems because they are key structural features of proteins, nucleic acids, and oligosaccharides, especially helices from coordination compounds [1–5]. Our research involves transition metal complexes with Schiff-base ligands (L = salicylidene-glycine) derived from salicylaldehyde, with abundant H-bonding and some weak interactions, such as π - π stacking. Transition metal complexes of the Schiff-base derived from condensation of salicylaldehyde yield mono-, bi-, 1-D, 2-D, and 3-D complexes with other ligands such as azide, carboxylate, and bipyridine either bridging or terminal [6–10]. Hydrogen-bonded assemblies of coordination complexes that lead to helical structures have not been explored, even though

*Corresponding author. Email: yhxing2000@yahoo.com

supramolecular architecture takes advantage of noncovalent (e.g., hydrogen bonding) interactions [11, 12]. We have synthesized two complexes using Schiff-base and bipyridine as a spacer, $[\text{Cu}_2(\text{L})_2(\mu\text{-}4,4'\text{-bipy})(4,4'\text{-bipy})_2] \cdot 2\text{H}_2\text{O}$ (**1**) and $\text{Ni}(\text{OAc})_2(2,2'\text{-bipy})(\text{H}_2\text{O})_2$ (**2**), in order to investigate the potential application of complexes of Schiff-base ligands with helical structures and superstructures as enzyme inhibitors against superoxide dismutase (SOD).

2. Experimental

2.1. Physical measurements

Elemental analyses were performed on a P.E. 240C automatic analyzer. IR spectra were determined on a JASCO FT/IR-480 PLUS Fourier Transform spectrometer ($200\text{--}4000\text{ cm}^{-1}$, with pressed KBr pellets); UV-vis spectra were determined by a JASCO V-570 UV-Vis spectrometer (as a solid). All chemicals were of analytical grade and used without purification. The amino-Schiff-base was prepared by modified literature method [13] and the other reactants were used as purchased. All reactions were carried out at room temperature.

2.2. Preparation of KHL (potassium salt of salicylideneglycine)

A solution of potassium hydroxide (10 mmol, 0.56 g) in 30 mL absolute alcohol was added to glycine (10 mmol, 0.75 g) [13]. The mixture was stirred until complete dissolution. Then, ethanolic solution (100 mL) of salicylaldehyde (10 mmol, 1.22 g) was added dropwise. The reaction was precipitated by diffusion of excess absolute diethyl ether into the solution. The product was filtered and dried in a vacuum desiccator. Yield: 1.59 g (63.0%), Anal. Calcd for $\text{C}_9\text{H}_8\text{NO}_3\text{K}$ (%): C, 49.77; H, 3.69; N, 5.45. Found(%): C, 48.88; H, 3.58; N, 5.33%. m.p.: 207°C .

2.3. Preparation of complexes

2.3.1. $[\text{Cu}_2(\text{L})_2(\mu\text{-}4,4'\text{-bipy})(4,4'\text{-bipy})_2] \cdot 2\text{H}_2\text{O}$ (1**).** To a solution of $\text{Cu}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ (0.10 g, 0.5 mmol) in ethanol (10 mL) was added a solution of KHL (0.11 g, 0.5 mmol) and 4,4'-bipy ($\text{C}_{10}\text{H}_8\text{N}_2$, 0.16 g, 1.0 mmol) in ethanol (10 mL). The dark green solution was stirred for 3 h and there was no precipitation. After about 35 days, some dark green crystals obtained were dried under vacuum to afford 0.16 g of product, yield 65% (based on $\text{Cu}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$). Anal. Calcd for $\text{C}_{48}\text{H}_{44}\text{Cu}_2\text{N}_8\text{O}_8$ (%): C, 58.30; H, 4.45; N, 11.34. Found(%): C, 57.97; H, 4.15; N, 11.20.

2.3.2. $\text{Ni}(\text{OAc})_2(2,2'\text{-bipy})(\text{H}_2\text{O})_2$ (2**).** Preparation of **2** is the same as that of **1** with 1 : 1 : 2 ratio of $\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$, KHL and 2,2'-bipy. After 40 days, green crystals were obtained and dried under vacuum to afford 0.12 g of product, yield 63% (based on $\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$). Anal. Calcd for $\text{C}_{14}\text{H}_{18}\text{N}_2\text{O}_6\text{Ni}$ (%): C, 45.53; H, 4.88; N, 7.59. Found(%): C, 45.66; H, 4.78; N, 7.64.

Table 1. Crystallographic data for **1**.

Formula	C ₄₈ H ₄₄ N ₈ O ₈ Cu ₂
M (g mol ⁻¹)	987.99
Crystal system	Monoclinic
Space group	C2/c
Unit cell dimensions (Å, °)	
<i>a</i>	36.519(7)
<i>b</i>	13.251(3)
<i>c</i>	9.5398(19)
α	90
β	103.49(3)
γ	90
<i>V</i> (Å ³)	4488.9(16)
<i>Z</i>	4
<i>D</i> _{Calcd}	1.462
Crystal size (mm ³)	0.23 × 0.17 × 0.26
<i>F</i> (000)	2040
μ (Mo-K α) (cm ⁻¹)	1.012
θ (°)	3.07–27.45
Reflections collected	21,600
Independent reflections (<i>I</i> > 2 σ (<i>I</i>))	3885
Parameters	306
$\Delta(\rho)$ (e Å ⁻³)	0.374 and -0.407
Goodness-of-fit	1.050
<i>R</i> ^a	0.0422 (0.0618) ^b
<i>wR</i> ₂ ^a	0.0922 (0.0998) ^b

^a $R = \sum ||F_o| - |F_c|| / \sum |F_o|$, $wR_2 = [\sum (w(F_o^2 - F_c^2))^2 / \sum (w(F_o^2))^2]^{1/2}$; [$F_o > 4\sigma(F_o)$].

^bBased on all data.

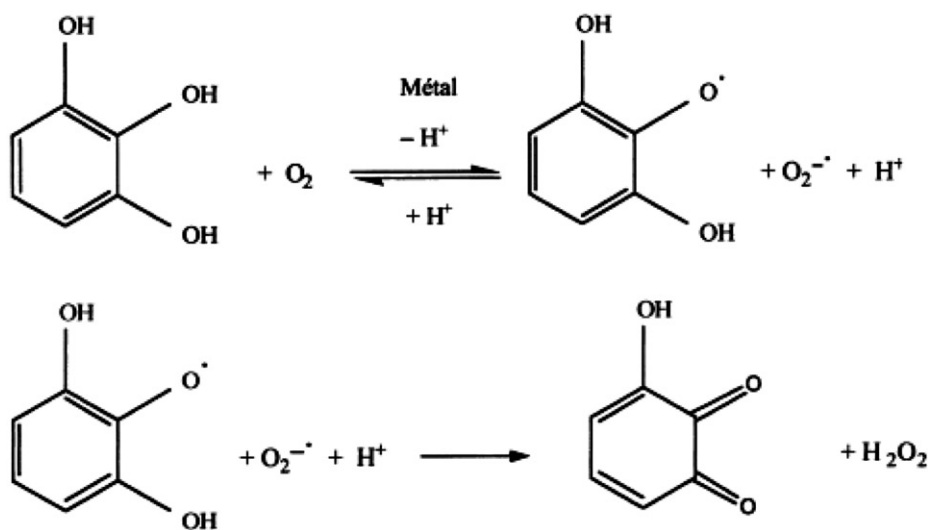
2.4. X-ray data collection and refinement of crystal structures

The crystals of **1** were mounted on a glass fiber for X-ray measurement. Reflection data were collected at room temperature on an APEX II CCD diffractometer with graphite monochromated Mo-K α radiation ($\lambda = 0.7107 \text{ \AA}$) and ψ scan mode. All measured independent reflections were used in the structural analysis and semi-empirical absorption corrections were applied using SADABS [14]. The crystal data, data collection and refinement details are summarized in table 1.

The structure was solved by direct methods using SHELXS-86 and refined using SHELXTL-97 [15]. All nonhydrogen atoms were refined anisotropically and the contributions of hydrogens were included in calculated positions, constrained to ride on their carbon atoms with group U_{iso} values assigned. Atomic scattering factors and anomalous dispersion terms were as in SHELXTL-97. The drawings were made with Diamond and all calculations were performed on a Pentium 4 computer.

2.5. Thermogravimetric assay of **1** and **2**

Thermogravimetric analyses (TGA) were carried out on a Perkin Elmer Diamond TG/DTA instrument. The samples are initially heated for 1 h at 50°C to remove air. During the simple ramping experiment, weight changes were recorded as a function of temperature for 10°C min⁻¹ temperature ramp between 50 and 1000°C in nitrogen. The masses of **1** and **2** are 2.106 and 2.361 mg, respectively.



Scheme 1. The process of pyrogallol autodidactic.

2.6. SOD activity for 1 and 2

Free radical scavenging activity was determined using the Marklund method. All solutions were freshly prepared before experiments and used immediately. The oxidation substrate was pyrogallol, which first became a semiquinone radical reacting with O₂⁻ to give a quinone (scheme 1).

A solution of 100 mL 0.2 mol L⁻¹ Tris [tris(hydroxymethyl)aminomethane], 3 mmol L⁻¹ pyrogallol solution, 0.1 mL L⁻¹ and 10 mmol L⁻¹ hydrochloric acid and then Tris-HCl buffer was prepared (pH=8.8). Cells containing solution, which was prepared by adding 0.3 mL of pyrogallol solution to Tris-HCl buffer or various concentrations (2–50 μg mL⁻¹) of tested samples of **1** and **2**, were illuminated by ultraviolet lamps. Reduction of pyrogallol was by increased absorbance at 318 nm on a Perkin Elmer Lambda 35 UV/Vis Spectrometer. All photoinduced reactions were performed at 25°C with 0.1 mol L⁻¹ hydrochloric acid as the reference solution. Homogenization for exactly 30 s after addition of pyrogallol gave an absorbance change of 1 nm s⁻¹; measurement was made for a further 5 min exactly [16, 17].

3. Results and discussion

3.1. Synthesis

Although we prepared complexes **1** and **2** by reactions of amino-Schiff-base and Cu(CH₃COO)₂·2H₂O or Ni(CH₃COO)₂·2H₂O in the same reaction conditions, two kinds of crystals, blue and green, were formed in the reaction of **2**. We can confirm the existence of the amino-Schiff-base in the green crystals by IR spectra. But unfortunately, we did not obtain the crystal structure of the green crystal, only blue crystal without amino-Schiff-base ligand. The crystal structure of the complex has been

previously published [18]. Both complexes were soluble in DMF, CHCl_3 , CH_2Cl_2 , DMSO, pyridine, CH_3CN , and THF and insoluble in hexane and ether.

3.2. Spectroscopic characterization

3.2.1. IR spectra. Complex **1** has a broad band due to water centered at 3441 cm^{-1} . The binding pattern of amino-Schiff-base to metal ion (Cu) is evident from the IR spectra where a single strong C=N bond centered at 1635 cm^{-1} is observed, suggesting the existence of amino-Schiff-base. Two strong and sharp bands of **1** at 1598 and 1356 cm^{-1} are attributed to asymmetric and symmetric stretching vibrations of the coordination carboxylate [13]. The fact that $\Delta\nu$ [$\Delta\nu = \nu_{\text{as}}(\text{CO}_2^-) - \nu_{\text{s}}(\text{CO}_2^-)$] is greater than 200 cm^{-1} demonstrates that the carboxylate is monodentate [19]. Absorptions with variable intensity in the frequency range of 1408 – 1598 cm^{-1} correspond to ring vibrations of the “py” of the 4,4'-bipy. The spectra have bands at 1535 and 761 cm^{-1} for **1**, assigned to $\nu_{(\text{C}=\text{C})\text{aromatic}}$ and $\nu_{(\text{C}-\text{H})\text{aromatic}}$ from amino-Schiff-base, coordination to metal, confirmed by three bands at 517 cm^{-1} for $\nu_{(\text{M}-\text{N})}$, 462 cm^{-1} for $\nu_{(\text{M}-\text{O})\text{hydroxyl}}$, and 420 cm^{-1} for $\nu_{(\text{M}-\text{O})\text{C}=\text{O}}$.

For **2**, the IR spectrum exhibits broad absorption at 3385 – 3031 cm^{-1} , assignable to coordinated water. The 1556 and 1341 cm^{-1} bands are assigned to $\nu_{\text{asym}}(\text{COO})$ and $\nu_{\text{sym}}(\text{COO})$, respectively, indicating monodentate carboxylate [19]. Peaks at 1471 , 1417 , and 774 cm^{-1} are attributed to stretching vibrations of 2,2'-bipy. Sharp absorptions at 661 and 417 cm^{-1} can be attributed to $\nu_{(\text{M}-\text{O})\text{carboxyl}}$ and $\nu_{(\text{M}-\text{N})}$.

3.2.2. UV-Vis absorption spectroscopy. The electronic absorption spectra of **1** and **2** are recorded as solids. Bands at 256 nm for **1** and **2** are assigned as π - π^* transitions of the bipy or the amino-Schiff-base ligand. Bands at 626 – 804 nm and 588 – 938 nm for **1** and **2** are attributed to the d-d* transitions [13].

3.3. Structural description

The molecular structure of $[\text{Cu}_2(\text{L})_2(\mu\text{-}4,4'\text{-bipy})(4,4'\text{-bipy})_2] \cdot 2\text{H}_2\text{O}$ with the atom-numbering scheme is illustrated in figure 1 and selected bond distances and angles are listed in table 2. Each copper is bonded to two oxygens and three nitrogens. Copper is five-coordinate and the structure can be evaluated by the Addison distortion index τ [20]: $\tau = |\beta - \alpha|/60^\circ$, α and β being the two largest angles around the central atom. In a penta-coordinated system, values for τ are 1.0 for trigonal bipyramidal and 0.0 for square pyramidal. The value of τ is 0.01, for Cu1, indicating slightly distorted square pyramidal. The average deviation of N1, O1, O2, and N2 from the square plane is $0.0000(1)\text{ \AA}$; N3 occupies the axial position. The square plane for Cu1 consists of two nitrogens (N1 and N2) from two different 4,4'-bipy and two oxygens (O1 and O2) from the amino-Schiff-base ligand. The Cu1 center lies $0.0007(1)\text{ \AA}$ above the N1–O1–N2–O2 coordination plane.

Intra- or intermolecular weak interactions (C–H \cdots O, weak H-bonding) and two types of hydrogen bonds in **1** are listed in table 3: (i) O–H \cdots O formed between uncoordinated carboxyl oxygen of the amino-Schiff-base ligand and the oxygen of water; (ii) N–H \cdots O formed between uncoordinated nitrogen of terminal 4,4'-bipy and water.

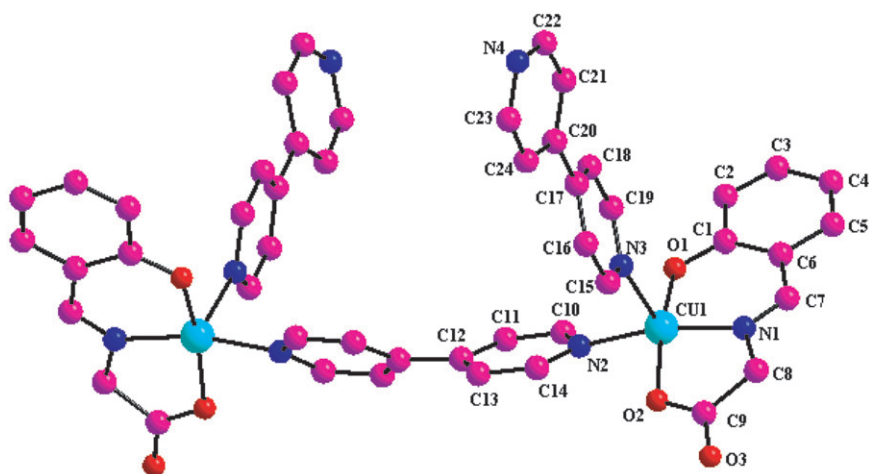


Figure 1. The molecular structure of **1** (all hydrogen atoms and the lattice waters are omitted for clarity).

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

Cu1–O1	1.9191(2)	C9–O2	1.281(3)	O1–Cu1–N1	92.91(8)
Cu1–N1	1.938(2)	C10–N2	1.323(3)	O1–Cu1–O2	167.27(8)
Cu1–O2	1.9803(2)	C12–C12 ^{#1}	1.480(4)	N1–Cu1–O2	83.39(7)
Cu1–N2	2.042(2)	C14–N2	1.340(3)	O1–Cu1–N2	91.00(8)
Cu1–N3	2.281(2)	C15–N3	1.334(3)	N1–Cu1–N2	166.56(8)
C1–O1	1.308(3)	C19–N3	1.338(3)	O2–Cu1–N2	90.01(8)
C7–N1	1.279(3)	C22–N4	1.319(4)	O1–Cu1–N3	97.08(9)
C8–N1	1.464(3)	C23–N4	1.323(4)	N1–Cu1–N3	100.70(8)
C9–O3	1.222(3)	N2–Cu1–N3	91.57(8)	O2–Cu1–N3	95.57(8)

Symmetry transformations used to generate equivalent atoms: (^{#1}) $-x+1, y, -z-1/2$.

Table 3. Intermolecular hydrogen bond distances (Å) and angles (°) for **1**.

D–H...A	D(D–H) (Å)	d(H...A) (Å)	D–H–A (°)	d(D...A) (Å)
N4–H4B...O4a	0.860	2.127	2.930	155.17
O4–H21...N4a	0.750	2.195	2.930	166.36
O4–H22...O3	0.934	1.909	2.822	165.44
C7–H7A...O3b	0.9300	2.5841	3.4228	150.26
C8–H8B...O2b	0.9700	2.5740	3.4213	145.97
C10–H10A...O1	0.9300	2.4878	2.9627	111.87
C14–H14A...O2	0.9300	2.5146	2.9956	112.45
C24–H24A...O4	0.9300	2.3704	3.2848	167.61

Symmetry transformation used to generate equivalent atoms: a = $-x+3/2, -y+1/2, -z$; b = $x, -y, 1/2+z$.

An extended helix is formed through the hydrogen bonds O4–H21...N4a (2.930 Å, 166.36°) and O4–H22...O3 (2.822 Å, 165.44°) along *x*-axis, as shown in figure 2. Adjacent 1-D helix chains are packed through interaction of π -overlap at 3.823(7) Å between 4,4'-bipy from neighboring molecules, resulting in a 2-D supramolecular

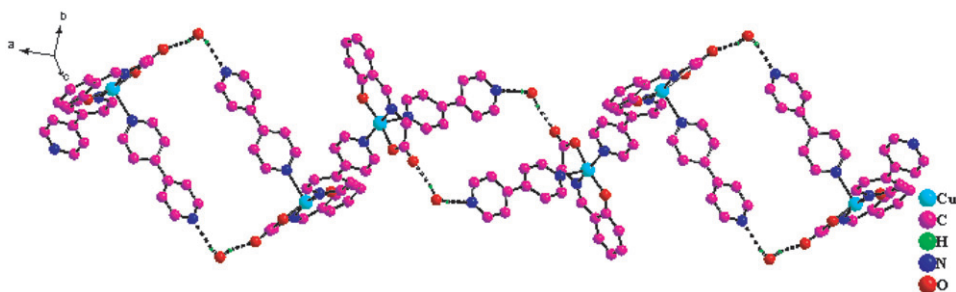


Figure 2. Fragment of 1-D network of hydrogen bonding found in **1** along the *a*-axis (all hydrogen atoms except for the hydrogen bonds are omitted for clarity).

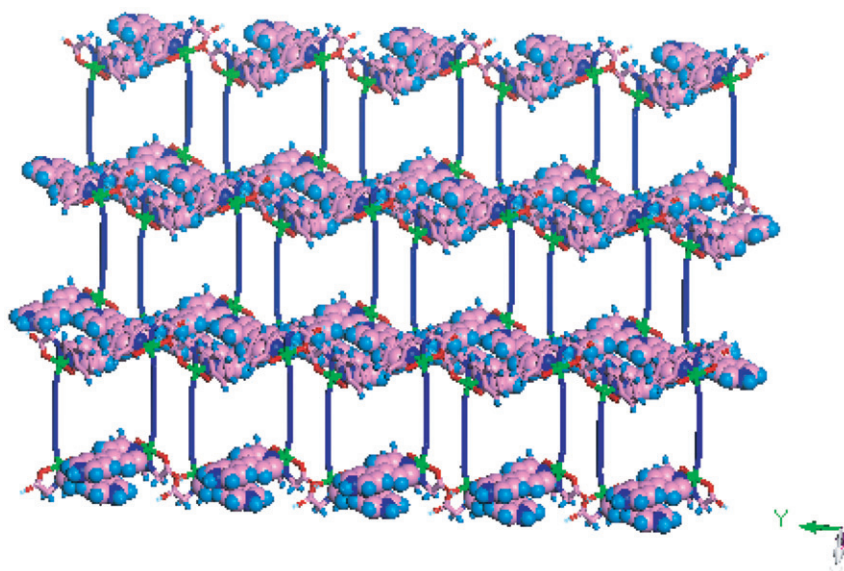


Figure 3. A view of the 4,4'-bipy as bridges for **1**; 4,4'-bipy are expressed as lines for clarity.

network with 4,4'-bipy as the bridge (figure 3). The 2-D supramolecular network of **1** forms a 3-D infinite interwoven framework with hydrogen bonds and π - π stacking interaction shown in figure 4.

3.4. Thermogravimetric properties

There are two steps for **1** in the TG-DTG-DTA curves. The first at 77–124°C has a weight loss of 3.65%, due to loss of two waters (calculated value 3.68%). The second step at 124–266°C has a weight loss of 56.49%, due to loss of two CO₂ molecules from two Schiff bases, one bridging 4,4'-bipy and two terminal 4,4'-bipy (calculated value 56.52%). The DTA curve of **1** indicates the two stages are endothermic-exothermic processes.

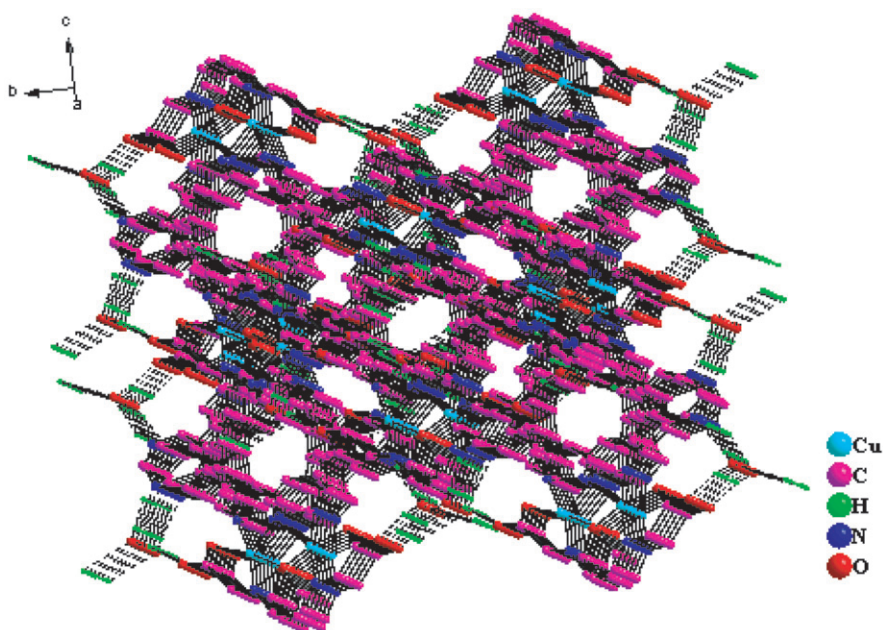


Figure 4. A view of infinite 3-D framework formed through hydrogen bond and π - π in **1** (all phenyl carbons of Schiff bases and all hydrogen atoms except for the hydrogen bonds are omitted for clarity).

In the TG-DTG-DTA curves of thermal decomposition of **2**, the first stage 83–257°C, has mass loss of 4.84% corresponding to coordinated water (calculated value 4.87%). The DTA curve of **2** indicates that it is an endothermic process. The second stage occurs within the range 257–790°C, with mass loss of 58.72% due to loss of water, a carboxyl group, and 2,2'-bipy; the thermal event agrees with the TG curve (calculated value 59.07%). The DTA curve of **2** shows an exothermic process.

3.5. SOD activity assay of **1** and **2**

Each % inhibition measured is translated to millimole per liter SOD equivalent. The absorbance change of the blank (K_0) and the sample (K_i) are used to calculate the % inhibition (η):

$$\eta = (1 - K_i/K_0) \times 100\%$$

The lower the concentration that corresponds to 50% inhibition of pyrogallol autoxidation, the more efficient the complex is; this concentration is termed IC_{50} for comparative purposes. Complex **1** possesses SOD-like activity showing 50% inhibition at 16.89 μ M. We did not get results for **2** at the same conditions as **1**. Complex **1** has better SOD-like activity than **2** and corresponds to some reported mononuclear copper complexes, which have high SOD-like activity [18, 21, 22]. The distorted geometry of **1** may favor the geometrical change, which is essential for catalysis as the geometry of copper in the SOD enzyme changes from distorted square pyramidal [for Cu(II)] to octahedral [for Ni(II)] during catalysis [23, 24]. The higher activity of **1** may indicate

favorable response of π -electrons of the aromatic side chain from the Schiff-base in stabilizing the Cu(II)–O₂⁻ interaction [23].

4. Conclusion

The copper complex shows abundant hydrogen bonds and different coordination modes for 4,4'-bipy. The distorted structure of **1** is responsible for the good superoxide dismutase activity. This study demonstrates that it is possible to further enhance the SOD activity of Cu(II) complex with an amino-Schiff-base ligand by modifying the coordination geometry.

Supplementary material

Tables of atomic coordinates, an isotropic thermal parameters, and complete bond distances and angles have been deposited with the Cambridge Crystallographic Data Center. Copies of this information may be obtained free of charge, by quoting the publication citation and deposition numbers CCDC: 665293 for **1** from the Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (Fax: +44-1223-336033; E-mail: deposit@ccdc.cam.ac.uk or <http://www.ccdc.cam.ac.uk>).

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