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Syntheses, Crystal Structures, and Antimicrobial Activities of Copper(II) Complexes Derived from *N,N'*-Bis(5-Fluorosalicylidene)-1,3-Propanediamine

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Abstract—Two new Schiff base copper(II) complexes, $[Cu_2L_2]$ (**I**) and $[Cu_2L(NCNCN)_2]_n$ (**II**), where L is the dianionic form of *N*,*N'*-bis(5-fluorosalicylidene)-1,3-propanediamine (H_2L), were synthesized and characterized by elemental analyses, infrared and electronic spectroscopy, and single crystal X-ray determinations (CIF files CCDC. nos. 2000415 (**I**) and 2000416 (**II**)). Complex **I** is a phenolate bridged dinuclear copper(II) compound. Complex **II** is a phenolate and dicyanoamide bridged polynuclear copper(II) compound. In both complexes, the Cu atoms are in square pyramidal coordination. The Schiff base ligand coordinates to the Cu atoms through the phenolate O and imino N atoms. The Schiff base H_2L and the two complexes showed high selectivity and antimicrobial activities against a number of the bacteria and fungi.

Keywords: Schiff base, copper(II) complex, polynuclear complex, crystal structure, antimicrobial activity

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INTRODUCTION

Polynuclear complexes containing bridging ligands are of current interest because of their molecular topologies as well as the fact that they may be designed with specific functionalities [1–3]. The copper, cobalt, nickel, zinc, and manganese complexes with Schiff base ligands have interesting structures and biological activities, such as antibacterial, antifungal, and antitumor [4–9]. Due to the versatile coordination modes of the dicyanoamide ligand, it has become the interesting building block in the formation of polynuclear complexes [10, 11]. Recent research indicates that the compounds bearing halide groups, such as F and Cl, can severely increase the antimicrobial activities [12, 13]. This work is aimed at obtaining polynuclear complexes and investigating their antimicrobial activities. Based on the above considerations, we designed and synthesized a fluoro-containing Schiff base ligand, *N*,*N'*-bis(5-fluorosalicylidene)-1,3-propanediamine (H_2L). With the Schiff base ligand, a dinuclear copper(II) complex $[Cu_2L_2]$ (**I**) was prepared. Interestingly, the addition of dicyanoamide ligand in complex I can generate a novel polynuclear complex $[Cu_2L(NCNCN)_2]_n$ (**II**). The antimicrobial activities of the compounds are studied.

EXPERIMENTAL

Materials and measurements. 5-Fluorosalicylaldehyde and propane-1,3-diamine with AR grade were purchased from Sigma-Aldrich and used as received. Elemental analyses were performed on a Perkin-Elmer 240C elemental analyzer. The IR spectra were recorded on a Jasco FT/IR-4000 spectrometer with KBr pellets. UV–Vis spectra were recorded on a Lambdar 35 spectrometer. X-ray diffraction was carried out at a Bruker SMART 1000 CCD area diffractometer equipped with MoK_α radiation.

Synthesis of H_2L. 5-Fluorosalicylaldehyde (0.28 g, 2.0 mmol) and propane-1,3-diamine (0.074 g, 1.0 mmol) were dissolved in methanol (50 mL). The mixture was refluxed for 30 min and with the solvent removed by distillation. The yellow solid was recrystallized from methanol to give crystalline product. Yield was 92%.

For $C_{17}H_{16}N_2O_2F_2$			
Anal. calcd., %	C, 64.14	H, 5.07	N, 8.80
Found, %	C, 63.96	H, 5.16	N, 8.87

IR data (ν, cm^{-1}): 3438 w, 1635 s, 1468, 1387, 1245, 1102, 930, 638, 465. UV–Vis data (λ, nm (ε ×

10^4, L mol^{-1} cm^{-1})): 230 (2.23); 273 (1.15); 330 (5.53); 420 (3.06). ^{1}H NMR (126 MHz; d_6-DMSO; δ, ppm): 13.17 (s., 2H, OH), 8.56 (s., 2H, CH=N), 7.32 (s., 2H, ArH), 7.20 (d., 2H, ArH), 6.89 (d., 2H, ArH), 3.69 (t., 4H, CH_2), 2.03 (t., 2H, CH_2). ^{13}C NMR (126 MHz; d_6-DMSO; δ, ppm): 156.74, 155.49, 153.63, 119.19, 119.00, 118.70, 118.64, 117.65, 117.59, 116.56, 116.38, 56.10, 31.23.

Synthesis of complex I. To a methanol solution (10 mL) of $CuCl_2·2H_2O$ (35 mg, 0.20 mmol) was added a methanol solution (10 mL) of H_2L (32 mg, 0.10 mmol). The mixture was stirred for 30 min at room temperature and then filtered. Upon keeping the filtrate in air for a week, blue block-shaped single crystals of the complex, suitable for X-ray crystal determination, formed at the bottom of the vessel on slow evaporation of the solvent. The crystals were isolated, washed with cold methanol and dried in air. Yield was 37%.

For $C_{34}H_{28}N_4O_4F_4Cu_2$

Anal. calcd., %	C, 53.75	H, 3.71	N, 7.37
Found, %	C, 53.53	H, 3.82	N, 7.45

IR data (ν, cm^{-1}): 1626 s, 1546, 1469, 1397, 1318, 1271, 1245, 1210, 1188, 1147, 1081, 965, 866, 814, 781, 547, 519. UV–Vis data (λ, nm (ε × 10^4, L mol^{-1} cm^{-1})): 270 (1.87); 370 (1.10).

Synthesis of complex II. *Method* 1. To a methanol solution (10 mL) of $CuCl_2·2H_2O$ (35 mg, 0.20 mmol) was added a methanol solution (10 mL) of H_2L (32 mg, 0.10 mmol) and sodium dicyanoamide (20 mg, 0.20 mmol). The mixture was stirred for 30 min at room temperature and then filtered. Upon keeping the filtrate in air for 5 days, blue block-shaped single crystals of the complex, suitable for X-ray crystal determination, formed at the bottom of the vessel on slow evaporation of the solvent. The crystals were isolated, washed with cold methanol and dried in air. Yield was 26%.

Method 2. To a methanol solution (10 mL) of complex **I** (76 mg, 0.10 mmol) was added a methanol solution (10 mL) of sodium dicyanoamide (20 mg, 0.20 mmol). The mixture was stirred for 30 min at room temperature and then filtered. Upon keeping the filtrate in air for a week, blue block-shaped single crystals of the complex, suitable for X-ray crystal determination, were formed at the bottom of the vessel on slow evaporation of the solvent. The crystals were isolated, washed with cold methanol and dried in air. Yield was 43%.

For $C_{21}H_{14}N_8O_2F_2Cu_2$

Anal. calcd., %	C, 43.83	H, 2.45	N, 19.47
Found, %	C, 43.96	H, 2.37	N, 19.32

IR data (ν, cm^{-1}): 2180 s, 1626 s, 1555, 1437, 1356, 1307, 1232, 1158, 1115, 1061, 972, 853, 786, 631, 563, 507. UV–Vis data (λ, nm (ε × 10^4, L mol^{-1} cm^{-1})): 270 (1.79); 372 (9.81).

X-ray crystal structure determination. Diffraction intensities for the compounds were collected at 298(2) K using a Bruker SMART 1000 CCD area-detector diffractometer with MoK_α radiation (λ = 0.71073 Å). The collected data were reduced with the SAINT program [14], and multi-scan absorption correction was performed using the SADABS program [15]. The structures were solved by direct methods. The compounds were refined against F^2 by full-matrix least-squares method using the SHELXTL [16]. All of the non-hydrogen atoms were refined anisotropically. The hydrogen atoms were placed in calculated positions and constrained to ride on their parent atoms. The crystallographic data for the compounds are summarized in Table 1. Selected bond lengths and angles are listed in Table 2.

Supplementary material for structures has been deposited with the Cambridge Crystallographic Data Centre (CCDC nos. 2000415 (**I**) and 2000416 (**II**); deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/const/retrieving.html).

Antimicrobial assay. The antibacterial activities of the compounds were tested against *B. subtilis*, *S. aureus*, *E. coli*, and *P. fluorescence* using MH (Mueller–Hinton) medium. The antifungal activities of the compounds were tested against *C. albicans* and *A. niger* using RPMI-1640 medium. The MIC values of the tested compounds were determined by a colorimetric method using the dye MTT [17]. A stock solution of the aroylhydrazone compound (150 μM) in DMSO was prepared and graded quantities (75, 37.5, 18.8, 9.4, 4.7, 2.3, 1.2, 0.59 and 0.30 μM) of the tested compounds were incorporated in specified quantity of the corresponding sterilized liquid medium. A specified quantity of the medium containing the compound was poured into microtitration plates. Suspension of the microorganism was prepared to contain approximately 10^5 cfu/mL and applied to microtitration plates with serially diluted compounds in DMSO to be tested and incubated at 37°C for 24 h and 48 h for bacteria and fungi, respectively. Then the MIC values were visually determined on each of the microtitration plates, 50 μL of PBS (phosphate buffered saline 0.01 M, pH 7.4) containing 2 mg of MTT/mL was added to each well. Incubation was continued at room temperature for 4–5 h. The content of each well was removed, and 100 μL of isopropanol containing 5% 1 M HCl was added to extract the dye. After 12 h of incubation at room temperature, the optical density was measured with a microplate reaer at 550 nm.

Table 1. Crystallographic data and structural refinement parameters for **I** and **II**

Parameter	Value	
	I	**II**
Formula	$C_{34}H_{28}N_4O_4F_4Cu_2$	$C_{21}H_{14}N_8O_2F_2Cu_2$
Mr	759.68	575.48
Crystal shape/color	Block/blue	Block/blue
Crystal size, mm	0.23 × 0.20 × 0.20	0.22 × 0.18 × 0.17
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/c$	$P2_1/c$
a, Å	13.514(1)	8.800(1)
b, Å	6.8189(5)	18.668(1)
c, Å	17.101(1)	13.934(1)
β, deg	109.014(8)	99.954(2)
V, Å^3	1489.9(2)	2254.7(3)
Z	2	4
ρ_c, g/cm^{-3}	1.693	1.695
μ, mm^{-1}	2.405	1.940
F(000)	772	1152
θ range, deg	3.46–66.94	2.78–25.49
Reflections collected	5450	10999
Unique reflections	2634	3948
Observed reflections ($I \geq 2\sigma(I)$)	2132	2709
Parameters	218	316
Min and max transmission	0.6077, 0.6448	0.6749, 0.7339
Goodness-of-fit on F^2	1.202	1.033
R_1, wR_2 ($I \geq 2\sigma(I)$)	0.0619, 0.2151	0.0784, 0.1658
R_1, wR_2 (all data)	0.0724, 0.2237	0.1161, 0.1863
Large diff. peak and hole, e Å^{-3}	0.948 and –0.473	0.955 and –0.552

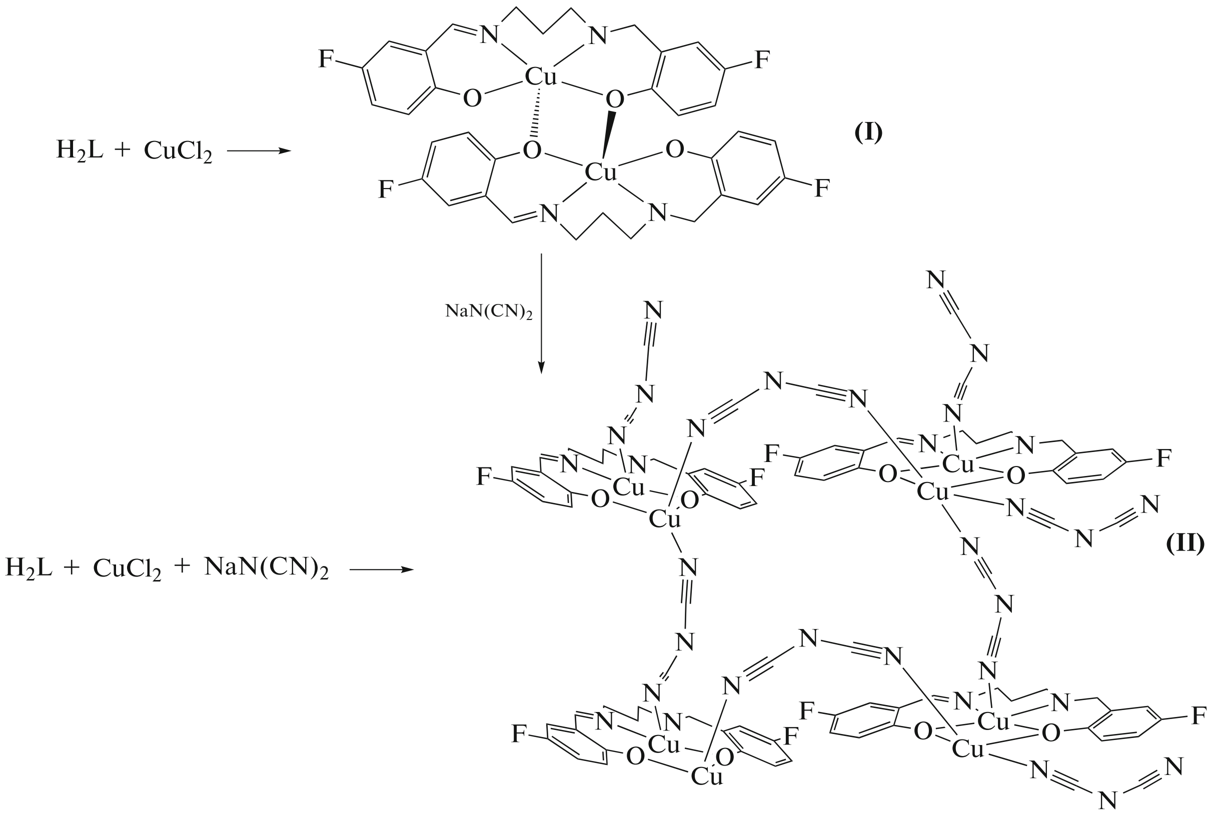
RESULTS AND DISCUSSION

The Schiff base H_2L was prepared by the condensation reaction of 5-fluorosalicylaldehyde with propane-1,3-diamine in a good yield (over 90%) in methanol. The compound is yellow crystalline solid. It is soluble in common polar organic solvents, such as DMSO, DMF, MeOH, EtOH, $CHCl_3$ and MeCN, but insoluble in water and Et_2O. The elemental analysis is in good agreement with the chemical formula proposed for the compound. Complex **I** was prepared by the reaction of H_2L with copper chloride in methanol. Complex **II** was prepared by the reaction of H_2L with copper chloride and sodium dicyanoamide in methanol. Notably, complex **II** can be obtained by the reaction of complex **I** with dicyanoamide in methanol (Scheme 1). Both complexes are stable in air at room temperature, soluble in DMF, DMSO, MeOH, EtOH and MeCN; insoluble in water and Et_2O.

Table 2. Selected bond lengths (Å) and bond angles (deg) for complexes **I** and **II***

Bond	*d*, Å	Bond	*d*, Å
I			
Cu(1)–O(1)	1.928(4)	Cu(1)–O(2)	1.910(4)
Cu(1)–N(1)	2.006(5)	Cu(1)–N(2)	1.984(5)
Cu(1)–O(1A)	2.712(5)		
II			
Cu(1)–O(1)	1.947(5)	Cu(1)–O(2)	1.959(5)
Cu(1)–N(1)	1.951(7)	Cu(1)–N(2)	1.970(7)
Cu(1)–N(3)	2.339(8)	Cu(2)–O(1)	2.019(6)
Cu(2)–O(2)	1.996(5)	Cu(2)–N(5)	1.961(7)
Cu(2)–N(7A)	1.978(8)	Cu(2)–N(8B)	2.111(7)
Angles	ω, deg	Angles	ω, deg
I			
O(2)Cu(1)O(1)	81.9(2)	O(2)Cu(1)N(2)	91.2(2)
O(1)Cu(1)N2	172.4(2)	O(2)Cu(1)N(1)	168.0(2)
O(1)Cu(1)N(1)	90.8(2)	N(2)Cu(1)N(1)	96.5(2)
O(1)Cu(1)O(1A)	91.9(2)	O(2)Cu(1)O(1A)	94.8(2)
N(1)Cu(1)O(1A)	95.0(2)	N(2)Cu(1)O(1A)	85.5(2)
II			
O(1)Cu(1)N(1)	91.9(3)	O(1)Cu(1)O(2)	76.7(2)
N(1)Cu(1)O(2)	168.3(3)	O(1)Cu(1)N(2)	163.7(3)
N(1)Cu(1)N(2)	98.7(3)	O(2)Cu(1)N(2)	91.9(3)
O(1)Cu(1)N(3)	91.5(3)	N(1)Cu(1)N(3)	96.1(3)
O(2)Cu(1)N(3)	86.9(3)	N(2)Cu(1)N(3)	99.6(3)
N(5)Cu(2)N(7A)	91.9(3)	N(5)Cu(2)O(2)	158.3(3)
N(7A) Cu(2)O(2)	92.3(3)	N(5)(Cu(2)O(1)	92.4(3)
N(7A) Cu(2)O(1)	151.6(3)	O(2)Cu(2)O(1)	74.2(2)
N(5)Cu(2)N(8B)	102.1(3)	O(2)(Cu(2)N(8B)	96.9(3)
O(1)Cu(2)N(8B)	98.8(3)		

* Symmetry codes: (A) x, $2 + y$, $2 + z$ (**I**). (A) x, $1/2 - y$, $1/2 + z$; (B) $1 + x$, y, z (**II**).



Scheme 1.

The molecular structure of complex **I** is shown in Fig. 1a. The molecule of the complex possesses crystallographic inversion center symmetry. The two Cu atoms are bridged by phenolate O atoms. Each Cu atom is in square pyramidal coordination with the two phenolate O and two imino N atoms of the Schiff base ligand defining the basal plane, and with the phenolate O atom of the other Schiff base ligand occupying the apical position. The Cu–O and Cu–N bond lengths are comparable to those observed in copper(II) complexes with Schiff bases [18–20]. The distortion of the square pyramid is revealed by the bond angles between the apical and basal donor atoms, ranging from 85.5(2)° to 95.0(2)°. The two benzene rings of the Schiff base ligand form a dihedral angle of 5.6(3)°. The molecules of the complex in the crystal are stabilized by π–π stacking interactions (Fig. 2a).

The molecular structure of complex **II** is shown in Fig. 1b. The asymmetric unit of the complex is $[Cu_2L(NCNCN)_2]$. The Cu(1) and Cu(2) atoms are bridged by phenolate O atoms. The Cu(1) atom is in square pyramidal coordination with the two phenolate O and two imino N atoms of the Schiff base ligand defining the basal plane, and with the N atom of the dicyanoamide ligand occupying the apical position. The Cu–O and Cu–N bond lengths are comparable to those observed in complex **I** and the copper(II) complexes with Schiff bases [18–20], as well as with dicyanoamide bridges [21, 22]. The distortion of the square pyramid is revealed by the bond angles between the apical and basal donor atoms, ranging from 76.7(2)° to 99.6(3)°. The two benzene rings of the Schiff base ligand form a dihedral angle of 23.3(5)°. The Cu(2) atom is also in square pyramidal coordination with the two phenolate O atoms of the Schiff base ligand and two N atoms of the dicyanoamide ligands defining the basal plane, and with the N atom of another dicyanoamide ligand occupying the apical position. The Cu–O and Cu–N bond lengths are comparable to those observed in the above mentioned copper(II) complexes. The distortion of the square pyramid is revealed by the bond angles between the apical and basal donor atoms, ranging from 74.2(2)° to 102.1(3)°. The $[Cu_2L(NCNCN)_2]$ units are linked by dicyanoamide ligands to form two-dimensional sheet along the *bc* plane. There are π–π stacking interactions along the *a* axis direction (Fig. 2b).

The IR spectra of H_2L and the complexes provide information about the metal–ligand bonding. The assignments are based on the typical group frequencies. The weak and broad absorption centered at 3438 cm^{-1} prove the presence of the phenol groups in H_2L. The strong absorption band at 1635 cm^{-1} in the spectrum of H_2L is assigned to the azomethine group, ν(C=N) [23]. The bands are shifted to lower wave numbers in the complexes, 1626 cm^{-1}, what can be

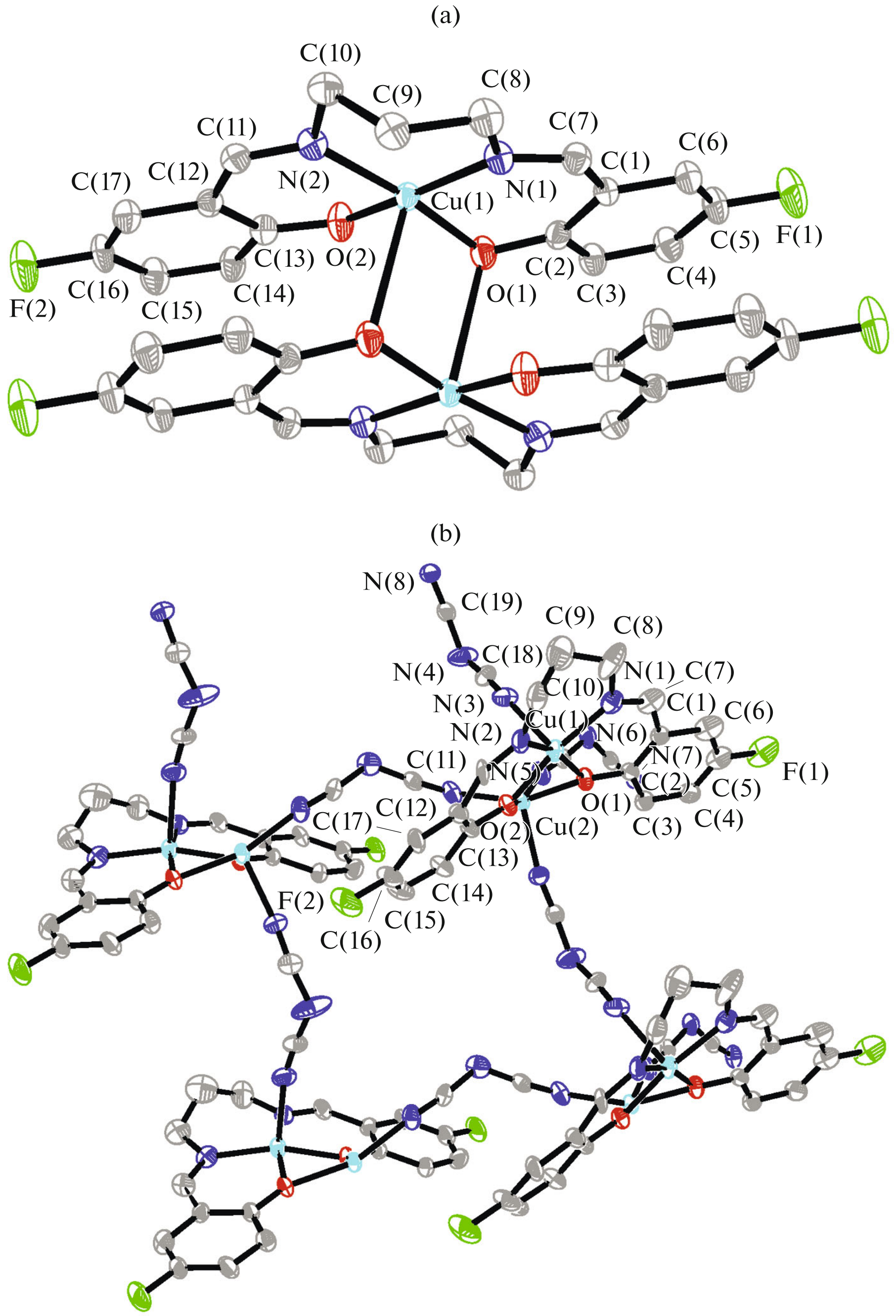


Fig. 1. Molecular structure of complexes **I** (a) and **II** (b). Displacement ellipsoids are drawn at the 30% probability level. H atoms are omitted for clarity.

attributed to the coordination of the nitrogen atom of the azomethine group to the metal ions [24]. The weak bands in the region 450–600 cm^{-1} for the complexes can be assigned to ν(Cu–O) and ν(Cu–N) [25], and provides further evidence for coordination through the deprotonated phenolic oxygen atoms. For complex **II**, the bands at 2305, 2235 and 2180 cm^{-1} can be assigned to the vibration of the dicyanoamide ligands [26].

In the electronic spectra of H_2L and the complexes, the absorption frequencies ascribed to the aromatic π–π* and *n*–π* transitions are located in the region 230–280 nm [25]. In the electronic spectra of the complexes, the absorptions centered at 370 nm are assigned to ligand-to-metal charge transfer [27] (see Figs. S1–S3).

The Schiff base H_2L and the copper complexes were screened for antibacterial activity against two Gram (+) bacterial strains (*Bacillus subtilis* and *Staphylococcus aureus*) and two Gram (–) bacterial strains (*Escherichia coli* and *Pseudomonas fluorescence*) by

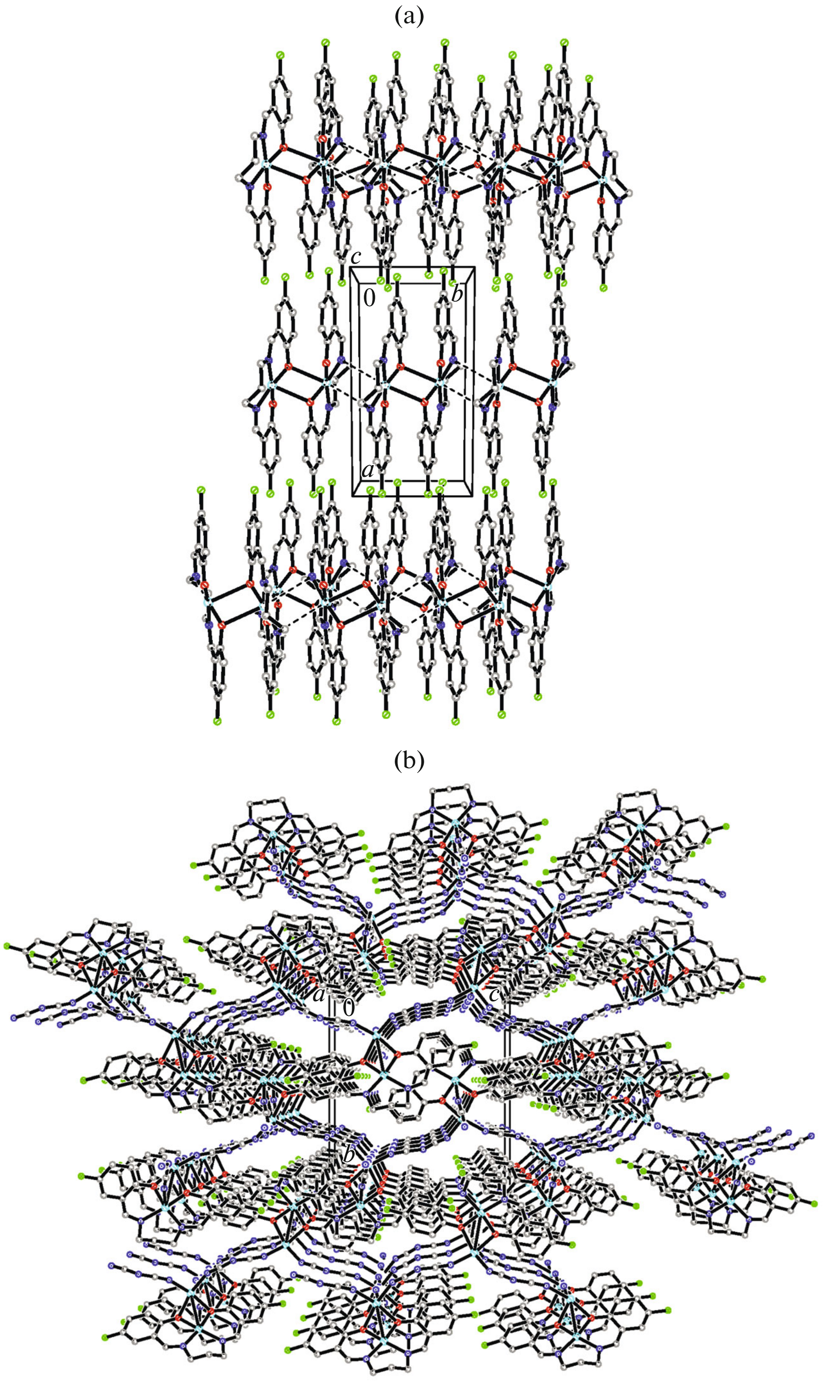


Fig. 2. The crystal packing of complexes **I**, viewed along the *c* axis (a) and **II**, viewed along the *a* axis (b).

MTT method. The MIC (minimum inhibitory concentration) values of the compounds against the bacteria are listed in Table 3. Kanamycin and Penicillin G were used as the standard materials. The free Schiff base H_2L has weak activities against *B. subtilis*, *S. aureus* and *E. coli*, and no activity against *P. fluorescence*, *C. albicans* and *A. niger*. Obviously, the two complexes have stronger activities against the bacteria than the free Schiff base. The trends are in accordance with that reported in [28, 29]. Complex **I** showed strong activity against *B. subtilis* and medium activities against the remaining bacteria. Complex **II** showed the most effective antibacterial activity against *B. subtilis*, good activity against *S. aureus*, and medium activities against the remaining bacteria. Notably, the two copper complexes have similar or even stronger activities against *B. subtilis* than Kanamycin and Penicillin G. Both complexes have stronger activities

Table 3. The MIC values (μM) of H_2L and complexes **I** and **II***

Tested material	*B. subtilis*	*S. aureus*	*E. coli*	*P. fluorescence*	*C. albicans*	*A. niger*
H_2L	18.8	75	37.5	>150	>150	>150
I	0.59	18.8	9.4	37.5	37.5	75
II	0.30	4.7	9.4	18.8	37.5	18.8
Kanamycin	0.59	2.3	4.7	4.7	>150	>150
Penicillin G	2.3	4.7	>150	>150	>150	>150
Ketoconazole	>150	>150	>150	>150	4.7	18.8

against *B. subtilis* than the copper(II) complexes with mono-Schiff base ligands [19]. The antifungal activities of the compounds were also evaluated against two fungal strains (*Candida albicans* and *Aspergillus niger*) by MTT method. Ketoconazole was used as a reference material. As a result, H_2L has no activity, and the two complexes have medium activities.

In the present paper, a new phenolate bridged dinuclear copper(II) complex and a new phenolate and dicyanoamide bridged polynuclear copper(II) complexes have been synthesized from the fluoro-containing Schiff base ligand *N*,*N*'-bis(5-fluorosalicylidene)-1,3-propanediamine. The Cu atoms in the complexes are in square pyramidal coordination with the Schiff base ligand coordinates by the phenolate O and imino N atoms. The two copper complexes showed interesting antimicrobial activities against *B. subtilis*, *S. aureus*, *E. coli*, *P. fluorescence*, *C. albicans* and *A. niger.* Both complexes showed the most effective antibacterial activities against *B. subtilis*. The compounds could be useful as a template for future development through modification to explore more effective antimicrobial materials.

SUPPLEMENTARY INFORMATION

The online version contains supplementary material available at https://doi.org/10.1134/S1070328421030076.

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