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Scientific paper

Copper(II) and Nickel(II) Complexes Derived from Isostructural Bromo- and Fluoro-Containing Bis-Schiff Bases: Syntheses, Crystal Structures and Antimicrobial Activity

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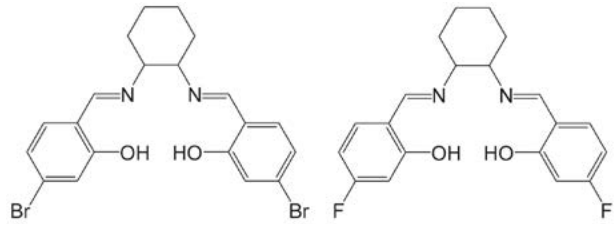
Abstract

A mononuclear copper(II) complex [CuL^a] (**1**), and three mononuclear nickel(II) complexes [NiL^a] (**2**), [NiL^a]·CH_3OH (**2**·CH_3OH) and [NiL^b] (**3**), where L^a and L^b are the dianionic form of *N,N*'-bis(4-bromosalicylidene)-1,2-cyclohexanediamine (H_2L^a) and *N,N*'-bis(4-fluorosalicylidene)-1,2-cyclohexanediamine (H_2L^b), respectively, were prepared and structurally characterized by spectroscopy method and elemental analyses. The detailed structures were determined by X-ray single crystal diffraction. All the copper and nickel complexes are mononuclear compounds. The metal ions in the complexes are in square planar coordination, with the two phenolate oxygens and two imine nitrogens of the Schiff base ligands. The biological effect of the four complexes were assayed on the bacteria strains *Staphylococcus aureus*, *Escherichia coli* and *Candida albicans*, which show that the substituent groups of the ligands and the metal ions can influence the antimicrobial activities. Complexes **1** and **3** have strong activity against *Staphylococcus aureus* and *Escherichia coli*, which are comparable to the reference drug tetracycline.

Keywords: Copper complex, Nickel complex, Schiff base, Crystal structure, Antimicrobial activity

1. Introduction

Schiff bases containing the functional group –C=N– have received much attention in the fields of inorganic chemistry because of their interesting biological and versatile coordination modes to metal ions.[1] Schiff bases have effective anti-cancer, anti-bacterial, anti-fungal, anti-convulsant, and antioxidant activities.[2] When coordinated to metal ions, the biological activities can be enhanced. A large number of Schiff base complexes have been reported to have remarkable biological activities like antifungal, anti-proliferative, antibacterial and antitumor.[3] Among the complexes of various types of Schiff bases, those with salen type Schiff bases have excellent antimicrobial activities.[4] Copper and nickel complexes have good antimicrobial potential.[5] Recent research indicated that the halide groups can severely increase the antimicrobial activities.[6] Our research group has reported some Schiff base complexes with antimicrobial properties.[7] To explore new antimicrobial agents, herein we report four mononuclear copper(II) and nickel(II) complexes, [CuL^a] (**1**), [NiL^a] (**2**), [NiL^a]·CH_3OH (**2**·CH_3OH) and [NiL^b] (**3**), where L^a and L^b are the dianionic form of *N,N*'-bis(4-bromosalicylidene)-1,2-cyclohexanediamine (H_2L^a) and *N,N*'-bis(4-fluorosalicylidene)-1,2-cyclohexanediamine (H_2L^b; Scheme 1).



Scheme 1. The Schiff base H_2L^a (left) and H_2L^b (right)

2. Experimental

2. 1. Material and Methods

4-Bromosalicylaldehyde, 4-fluorosalicylaldehyde and 1,2-diaminocyclohexane were obtained from Fluka.

The reagents and solvents with AR grade used in the experiment were used as received. The Schiff bases H_2L^a and H_2L^b were synthesized with the method as described in literature.[8] C, H and N elemental analyses were performed on a Perkin-Elmer 240B analyzer. Copper and nickel analyses were carried out by EDTA titration method. IR and UV-Vis spectra were recorded on IR-408 Shimadzu 568 and Lambda 900 spectrometers, respectively. Single crystal X-ray determination was done with a Bruker SMART 1000 CCD diffractometer.

2. 2. Synthesis of [CuL^a] (1)

H_2L (0.48 g, 1.0 mmol) and copper chloride dihydrate (0.17 g, 1.0 mmol) were dissolved and mixed in 50 mL EtOH. The reaction mixture was heated to reflux and stirred for 30 min. Brown and block like single crystals were formed at the bottom of the vessel by slow evaporation in air for four days. Yield: 0.23 g (43%). Analysis Calcd. for $C_{20}H_{18}Br_2CuN_2O_2$ (%): C, 44.34; H, 3.35; N, 5.17; Cu, 11.73. Found (%): C, 44.22; H, 3.43; N, 5.05; Cu, 11.91. IR data (KBr, cm^{-1}): 1626s, 1585s, 1523s, 1455w, 1417m, 1383m, 1280w, 1191w, 1137m, 1092w, 1064m, 919m, 853w, 790w, 624w, 607m, 517w, 510w, 445w. UV-Vis in MeOH (λ, ε): 232 nm, 2.53×10^3 L mol^{-1} cm^{-1}; 248 nm, 2.61×10^3 L mol^{-1} cm^{-1}; 276 nm, 1.72×10^3 L mol^{-1} cm^{-1}; 355 nm, 6.72×10^2 L mol^{-1} cm^{-1}.

2.2. Synthesis of [NiL^a] (2)

According to the same method as described for the synthesis of complex **1**, this complex was synthesized from H_2L^a (0.48 g, 1.0 mmol) with nickel chloride hexahydrate (0.24 g, 1.0 mmol). The product was green single crystals. Yield: 0.29 g (54%). Analysis Calcd. for $C_{20}H_{18}Br_2N_2NiO_2$ (%): C, 44.74; H, 3.38; N, 5.22; Ni, 10.93. Found (%): C, 44.57; H, 3.32; N, 5.30; Ni, 11.12. IR data (KBr, cm^{-1}): 1617s, 1585s, 1517m, 1455m, 1431m, 1385m, 1338w, 1293w, 1245w, 1207w, 1132w, 1060w, 1037w, 1000w, 930m, 917m, 857w, 780m, 633w, 601m, 525w, 458w. UV-Vis in MeOH (λ, ε): 250 nm, 2.46×10^3 L mol^{-1} cm^{-1}; 260 nm, 2.72×10^3 L mol^{-1} cm^{-1}; 313 nm, 4.92×10^2 L mol^{-1} cm^{-1}; 405 nm, 3.32×10^2 L mol^{-1} cm^{-1}.

2. 3. Synthesis of [NiL^a]·CH$_3$OH (2·CH$_3$OH)

According to the same method as described for the synthesis of complex **2**, this complex was synthesized from MeOH, instead of EtOH. The product was green single crystals. Yield: 0.22 g (39%). Analysis Calcd. for $C_{21}H_{22}Br_2N_2NiO_3$ (%): C, 44.33; H, 3.90; N, 4.92; Ni, 10.32. Found (%): C, 44.41; H, 3.81; N, 4.84; Ni, 10.55. IR data (KBr, cm^{-1}): 3455w, 1618s, 1586s, 1520m, 1477w, 1453w, 1428m, 1383m, 1338w, 1295w, 1254w, 1213w, 1188m, 1131w, 1064w, 1032w, 1002w, 917m, 865w, 733w, 598w, 523w, 487w, 464w, 449w. UV-Vis in MeOH (λ, ε): 250 nm, 2.38×10^3 L mol^{-1} cm^{-1}; 260 nm, 2.88×10^3 L mol^{-1} cm^{-1}; 315 nm, 5.92×10^2 L mol^{-1} cm^{-1}; 405 nm, 2.75×10^2 L mol^{-1} cm^{-1}.

2. 4. Synthesis of [NiL^b] (3)

According to the same method as described for the synthesis of complex **2**, this complex was synthesized from H_2L^b (0.36 g, 1.0 mmol), instead of H2L^a. The product was green single crystals. Yield: 0.28 g (67%). Analysis Calcd. for $C_{20}H_{18}F_2N_2NiO_2$ (%): C, 57.87; H, 4.37; N, 6.75; Ni, 14.14. Found (%): C, 57.63; H, 4.45; N, 6.82; Ni, 14.33. IR data (KBr, cm^{-1}): 1628s, 1544s, 1479w, 1442m, 1388w, 1312w, 1222m, 1150w, 1115m, 987w, 845w, 778w, 639w, 609w, 526w, 466w, 438w. UV-Vis in MeOH (λ, ε): 240 nm, 2.23×10^3 L mol^{-1} cm^{-1}; 252 nm, 2.45×10^3 L mol^{-1} cm^{-1}; 302 nm, 4.25×10^2 L mol^{-1} cm^{-1}; 390 nm, 2.87×10^2 L mol^{-1} cm^{-1}.

2.5. X-Ray Diffraction

Single crystal X-ray data were measured with MoK_α radiation (λ = 0.71073 Å) at 298(2) K using a Bruker SMART 1000 CCD diffractometer. The data were reduced with SAINT[9] and corrected for absorption with SADABS.[10] Multi-scan absorption correction was performed with ψ-scans.[11] Crystal structures of both complexes were solved with SHELXS-97 by direct method and refined by full-matrix least-squares technique on F^2 using anisotropic displacement parameters.[12] All hydrogens of the complexes were placed at the calculated positions. Idealized H atoms were refined with isotropic displacement parameters set to 1.2 (1.5 for O and methyl group) times the equivalent isotropic *U* values of the parent carbon atoms. The crystallographic data for the complexes are presented in Table 1.

Supplementary material has been deposited with the Cambridge Crystallographic Data Centre (nos. 2286356 (1), 2286358 (2), 2286595 (3), 2286596 (4)); deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

2. 6. Antimicrobial Assay

The assay of antimicrobial activity was performed with the disk diffusion method.[13] The antibacterial activity was tested against *E. coli*, *B. subtilis*, *S. aureus* and *P. fluorescence* using Mueller-Hinton (MH) medium. The minimum inhibitory concentrations (MICs) of the assayed compounds were measured by a colorimetric method using the dye 3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide (MTT). A stock solution of the assayed compound at the concentration of 50 μg mL^{-1} in DMSO was prepared and quantities of the compound were incorporated in specified quantity of sterilized liquid MH medium. A specified quantity of the medium containing the compound was poured into micro-titration plates. A

Table 1. Crystallographic data and experimental details for the complexes

	1	**2**	**2·CH$_3$OH**	**3**
Molecular formula	$C_{20}H_{18}Br_2CuN_2O_2$	$C_{20}H_{18}Br_2N_2NiO_2$	$C_{21}H_{22}Br_2N_2NiO_3$	$C_{20}H_{18}F_2N_2NiO_2$
Formula weight	541.72	536.89	568.94	415.07
Crystal system	Monoclinic	Orthorhombic	Monoclinic	Monoclinic
Space group	$P2_1/n$	$Pca2_1$	$P2_1/n$	$P2_1/c$
a, Å	11.2186(12)	13.7957(12)	12.0963(12)	12.0131(12)
b, Å	9.0649(11)	16.7158(13)	14.6857(10)	24.4839(15)
c, Å	19.7670(13)	8.6151(10)	12.8546(11)	12.6104(12)
α, °	90	90	90	90
β, °	105.379(1)	90	108.561(1)	107.583(1)
γ, °	90	90	90	90
V, Å^3	1938.2(3)	1986.7(3)	2164.7(3)	3535.8(5)
Z	4	4	4	8
ρ_{calcd}, g cm^{-3}	1.856	1.795	1.746	1.559
F(000)	1068	1064	1136	1712
Absorption coefficient, mm^{-1}	5.268	5.016	4.612	1.135
Reflections collected	10047	10187	10617	20485
Independent reflections (R_{int})	3608 (0.0409)	3538 (0.0631)	3942 (0.0627)	6565 (0.0803)
Reflections [$I > 2\sigma(I)$]	2469	2419	2029	3629
Data/parameters	3608/244	3538/244	3942/262	6565/487
Restraints	0	1	49	0
GooF on F^2	1.039	1.048	0.996	1.013
R_1, wR_2 ($I > 2\sigma(I)$)	0.0414, 0.0814	0.0444, 0.0883	0.0748, 0.2021	0.0531, 0.1072
R_1, wR_2 (all data)	0.0754, 0.0926	0.0869, 0.1214	0.1424, 0.2449	0.1197, 0.1326
$\Delta\rho_{max}/\Delta\rho_{min}$, e/A^3	0.727, –0.472	0.540, –0.332	1.059, –0.847	0.578, –0.416

suspension of the micro-organism was prepared to contain 10^5 cfu·mL^{-1} and applied to micro-titration plates with serially diluted compounds in DMSO to be tested and incubated at 37 °C for 24 h. After the MICs visually determined on each micro-titration plate, 50 μL of PBS containing 2 mg MTT per milli-litre was added to each well. Incubation was continued at room temperature for 4–5 hours. The content of each well was removed and 100 μL of isopropanol containing hydrochloric acid was added to extract the dye. After 12 hours of incubation at room temperature, the optical density (OD) was measured with a micro-plate reader at 550 nm.

3. Results and Discussion

3. 1. Chemistry

The two isostructural Schiff bases were readily synthesized from the reaction of 1:2 molar ratio of 1,2-diaminocyclohexane with 4-bromosalicylaldehyde and 4-fluorosalicylaldehyde, respectively in MeOH. The four complexes were prepared by the reaction of 1:1 molar ratio of the Schiff bases with copper chloride and nickel chloride. Complexes **1**, **2** and **3** were prepared with ethanol as solvent, while complex **2**·CH$_3$OH was synthesized and crystallized from methanol. When methanol was used for the synthesis and crystallization of complexes **1** and **3**, the same structures can be obtained as those with ethanol. All the complexes are soluble in EtOH, MeOH, DMSO and DMF.

3. 2. IR and Electronic Spectra

In the infrared spectra of the complexes, the intense bands at 1617–1628 cm^{-1} can be assigned to $\nu_{C=N}$.[14] There is a weak and broad absorption centered at 3455 cm^{-1} in the spectrum of complex **2**·CH$_3$OH, while no such band in the spectrum of **2**, indicates the presence of O–H bond in **2**·CH$_3$OH.

The electronic spectra of the complexes were recorded in MeOH. The charge transfer bands at 230–250 nm and 260–280 nm can be assigned to $\pi\rightarrow\pi^*$ and $n\rightarrow\pi^*$ transitions of the Schiff base ligands. The bands at 350–410 nm can be assigned to the metal to ligand charge transfer (MLCT) transition.[15]

3. 3. Crystal Structure Description of the Complexes

The bond lengths and bond angles related to the metal ions are given in Table 2. The Schiff bases L^a and L^b act as tetradentate ligands. Compound **1** is a mononuclear copper(II) complex with the bis-Schiff base ligand L^a (Fig. 1). Compounds **2** and **3** are mononuclear nickel(II) complexes with the bis-Schiff base ligand L^a (Figs. 2 and 3). The difference between the two structures is the presence of methanol solvent in **3**, while absence for **2**. Compound **3** is a mononuclear nickel(II) complex with the bis-Schiff base L^b (Fig. 4). The metal atoms in the complexes are coordinated by two phenolate oxygens (O1 and O2), and two imine nitrogen atoms (N1 and N2), forming square planar

coordination. The metal atoms deviate from the least-squares planes defined by the N_2O_2 donor atoms by 0.028(2) Å (**1**), 0.015(2) Å (**2**), 0.008(2) Å (**2**·CH_3OH), and 0.001(2) Å for Ni1 and 0.007(2) Å for Ni2 (**3**). The dihedral angles between the C1-C6 and C15-C20 benzene rings are 11.0(4)° for **1** and **2**, 4.2(5)° for **2**·CH_3OH, and 7.2(4) and 11.2(4)° for **3**. The Cu–O and Cu–N bond distances of complex **1** are longer than those of the Ni–O and Ni–N bonds in complexes **2**, **3** and **4**. The bond lengths in the three nickel complexes are similar to each other. All the M–O and M–N bond distances are comparable to those observed in similar copper(II) and nickel(II) complexes with Schiff bases.[12d,16]

The molecules of complex **1** are linked through C–H···O hydrogen bonds (Table 3) to form dimers, which are further linked through weak Br···O interactions to form two dimensional sheets parallel to the *bc* plane (Fig. 5). The molecules of complex **2** are linked through C–H···O hydrogen bonds (Table 3) to form one dimensional chains running along the *c* axis (Fig. 6). The molecules of complex **2·CH$_3$OH** are linked through C–H···O, O–H···O and C–H···Br hydrogen bonds (Table 3) to form two dimensional planes parallel to the *ab* plane (Fig. 7). The molecules of complex **3** are linked through C–H···O hydrogen bonds (Table 3) to form dimmers (Fig. 8).

3. 4. Antimicrobial Activity

The MIC values of the antimicrobial assay are listed in Table 4. In general, all the copper and nickel complexes have higher activity against *Staphylococcus aureus*, *Escherichia coli* and *Candida albicans* than the free Schiff bases H_2L^a and H_2L^b. This is caused by the greater lipophilic nature of the complexes than the Schiff base ligands, which can be explained with the chelating theory.[21] The fluoro-containing Schiff base H_2L^b has better activities

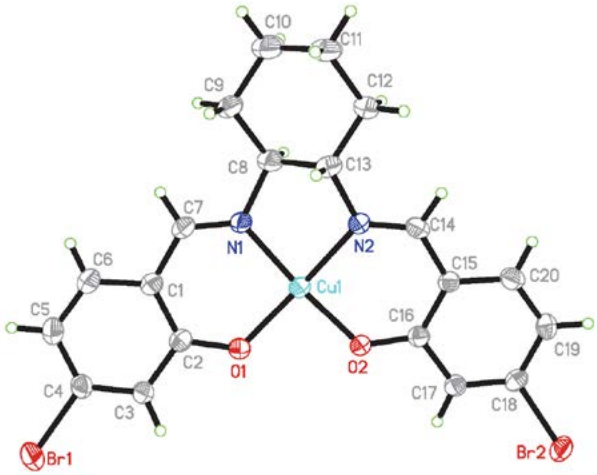


Fig. 1. Molecular structure of complex **1**. Thermal ellipsoid is shown at the 30% probability level.

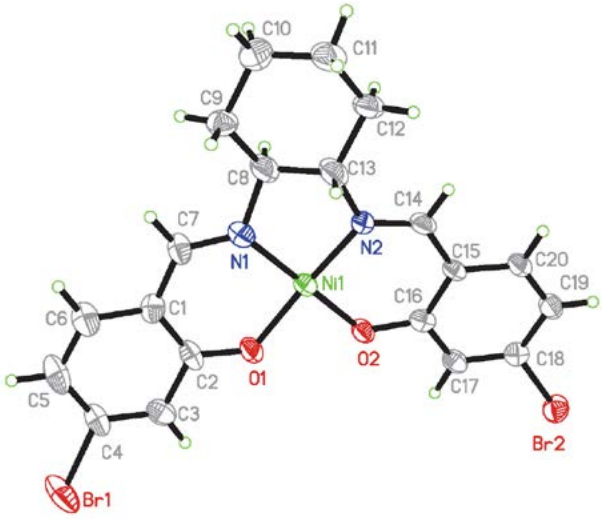


Fig. 2. Molecular structure of complex **2**. Thermal ellipsoid is shown at the 30% probability level.

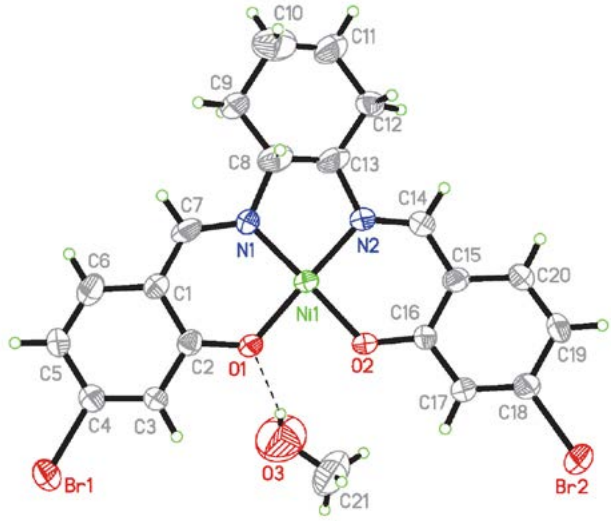


Fig. 3. Molecular structure of complex **2**·CH_3OH. Thermal ellipsoid is shown at the 30% probability level.

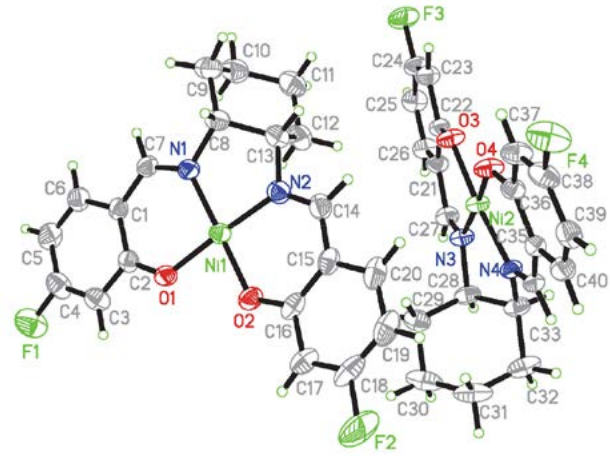


Fig. 4. Molecular structure of complex **3**. Thermal ellipsoid is shown at the 30% probability level.

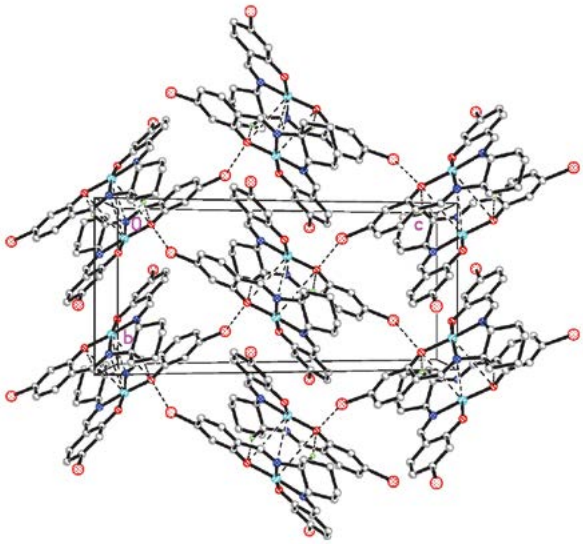


Fig. 5. Molecular packing structure of complex **1**, viewed along the *b* axis. Hydrogen bonds are shown as dashed lines.

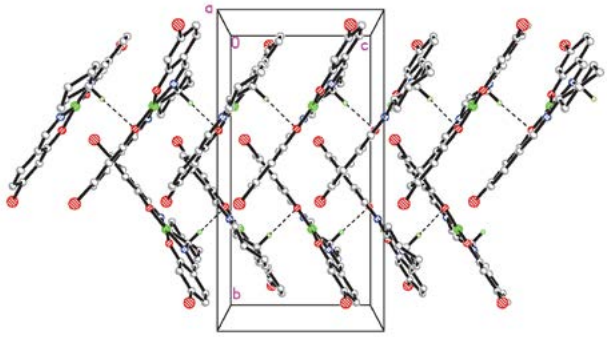


Fig. 6. Molecular packing structure of complex **2**, viewed along the *b* axis. Hydrogen bonds are shown as dashed lines.

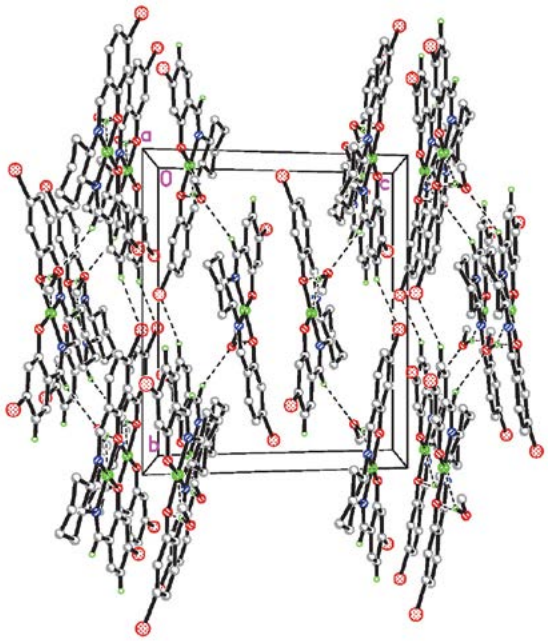


Fig. 7. Molecular packing structure of complex **2**·CH_3OH, viewed along the *b* axis. Hydrogen bonds are shown as dashed lines.

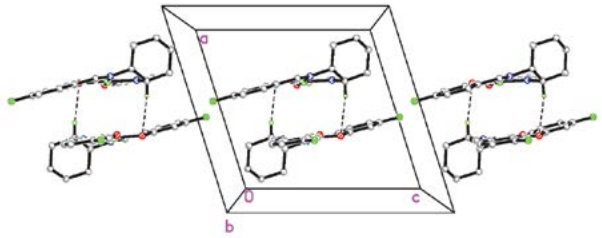


Fig. 8. Molecular packing structure of complex **3**, viewed along the *b* axis. Hydrogen bonds are shown as dashed lines.

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

1			
Cu1–O1	1.916(3)	Cu1–O2	1.898(3)
Cu1–N1	1.934(4)	Cu1–N2	1.957(4)
O2–Cu1–O1	89.81(14)	O2–Cu1–N1	173.53(16)
O1–Cu1–N1	93.98(15)	O2–Cu1–N2	93.20(15)
O1–Cu1–N2	170.86(16)	N1–Cu1–N2	83.85(16)
2			
Ni1–O1	1.851(5)	Ni1–O2	1.825(5)
Ni1–N1	1.855(7)	Ni1–N2	1.838(6)
O2–Ni1–N2	95.7(3)	O2–Ni1–O1	84.1(2)
N2–Ni1–O1	174.2(3)	O2–Ni1–N1	175.8(3)
N2–Ni1–N1	86.0(3)	O1–Ni1–N1	94.6(3)
2·CH_3OH			
Ni1–O1	1.843(6)	Ni1–O2	1.855(6)
Ni1–N1	1.821(8)	Ni1–N2	1.841(8)
N1–Ni1–N2	86.2(3)	N1–Ni1–O1	94.9(3)
N2–Ni1–O1	178.2(3)	N1–Ni1–O2	177.2(4)
N2–Ni1–O2	95.3(3)	O1–Ni1–O2	83.6(3)
3			
Ni1–O1	1.831(3)	Ni1–O2	1.830(3)
Ni1–N1	1.856(4)	Ni1–N2	1.843(4)
Ni2–O3	1.847(3)	Ni2–O4	1.841(3)
Ni2–N3	1.829(4)	Ni2–N4	1.846(4)
O2–Ni1–O1	84.69(14)	O2–Ni1–N2	93.71(17)
O1–Ni1–N2	176.94(16)	O2–Ni1–N1	178.60(15)
O1–Ni1–N1	94.95(17)	N2–Ni1–N1	86.71(19)
N3–Ni2–O4	175.70(16)	N3–Ni2–N4	85.81(16)
O4–Ni2–N4	95.12(15)	N3–Ni2–O3	94.25(15)
O4–Ni2–O3	85.19(13)	N4–Ni2–O3	174.91(15)

against *Staphylococcus aureus* and *Escherichia coli* than the bromo-containing Schiff base H_2L^a. The same pattern can be observed for the complexes **2** and **3**. Complex **3** with the fluoro-containing Schiff base ligand has stronger antimicrobial activities than complex **2** with the bromo-containing Schiff base ligand. Interestingly, the copper complex **1** has better activities than the nickel complex **2**. Thus, the substituent groups of the ligands and the metal ions can influence the antimicrobial activities. The copper complex **1** and the nickel complex **3** have strong activity against *Staphylococcus aureus* and *Escherichia coli*, and weak activ-

Table 3. Hydrogen bonding parameters for the complexes

D*–H···*A	***d*(*D*–H) (Å)**	***d*(H···*A*) (Å)**	***d*(*D*···*A*) (Å)**	**∠(*D*–H···*A*) (°)**
1				
C13–H13···O2^{i}	0.98	2.49(3)	3.318(5)	143(5)
2				
C8–H8···O2^{ii}	0.98	2.46(4)	1.372(5)	154(5)
2·CH$_3$OH				
O3–H3A···O1^{iii}	0.82	1.98(3)	2.767(5)	160(4)
C14–H14···O3	0.93	2.48(4)	3.288(6)	145(5)
C19–H19···Br1^{iv}	0.93	2.92(5)	3.640(6)	135(6)
3				
C8–H8···O2^{v}	0.98	2.57(3)	3.507(5)	160(7)

Symmetry transformation used to generate equivalent atoms: (i) – *x*, 2 – *y*, – *z*; (ii) 3/2 – *x*, *y*, ½ + *z*; (iii) ½ – *x*, –½ + *y*, ½ – *z*; (iv) *x*, –1 + *y*, *z*; (v) 1 – *x*, – *y*, – *z*.

ity against *Candida albicans*. The activities of both complexes are comparable to the reference drug tetracycline. While for *Candida albicans*, all the three complexes have stronger activities than tetracycline.

Table 4. Antimicrobial activities with MIC values (μg mL^{-1})

Compound	***Staphylococcus aureus***	***Escherichia coli***	***Candida albicans***
H_2L^a	64	128	> 1024
H_2L^b	32	32	> 1024
1	0.5	4.0	64
2	4.0	16	256
3	1.0	4.0	32
tetracycline	0.25	2.0	> 1024

4. Conclusion

Four new mononuclear copper(II) and nickel(II) complexes derived from the tetradentate Schiff base ligands *N,N*'-bis(4-bromosalicylidene)-1,2-cyclohexanediamine and *N,N*'-bis(4-fluorosalicylidene)-1,2-cyclohexanediamine have been synthesized. All the complexes were characterized by physical-chemical methods. The detailed structures of the complexes were determined by X-ray single crystal diffraction. All the metal ions in the complexes are in square planar geometry. The complexes show effective antibacterial activities on *Staphylococcus aureus* and *Escherichia coli*. The copper complex has the most activity against *Staphylococcus aureus* with MIC value of 0.5 μg mL^{-1}.

Acknowledgments

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5. References

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Povzetek

Sintetizirali smo enojedrni bakrov(II) kompleks [CuL^a] (**1**) in tri enojedrne nikljeve(II) komplekse [NiL^a] (**2**), [NiL^a]·CH_3OH (**2**·CH_3OH) in [NiL^b] (**3**), kjer sta L^a in L^b dianionski obliki *N*,*N*'-bis(4-bromosaliciliden)-1,2-cikloheksandiamina (H_2L^a) in *N*,*N*'-bis(4-fluorosaliciliden)-1,2-cikloheksandiamina (H_2L^b). Spojine smo okarakterizirali s spektroskopskimi metodami, elementno analizo in rentgensko strukturno analizo. Bakrov in vsi nikljevi kompleksi so enojedrne spojine. Kovinski ioni v kompleksih so v kvadratno planarni koordinaciji z dvema fenolatnima kisikovima atomoma in dvema iminskima dušikovima atomoma Schiffovih ligandov. Biološki učinek štirih kompleksov je bil preverjen na sevih bakterij *Staphylococcus aureus*, *Escherichia coli* in *Candida albicans*. Substituente na ligandu in kovinski ion vplivajo na protimikrobno delovanje. Kompleksa **1** in **3** imata močno aktivnost proti *Staphylococcus aureus* in *Escherichia coli*, ki je primerljiva z referenčnim zdravilom tetraciklinom.