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Scientific paper

Synthesis, Crystal Structure and Antimicrobial Activity of a Linear Trinuclear Nickel(II) Complex with Schiff Base Ligand

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Abstract

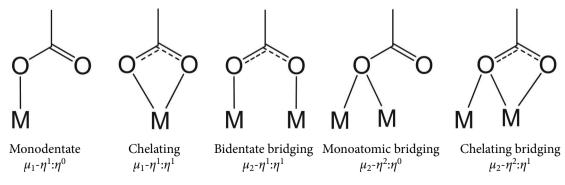
A new linear trinuclear Schiff base nickel(II) complex, $[Ni\{NiL(\mu_2-\eta^1:\eta^1-OAc)(OH_2)\}_2]\cdot H_2O$, where L is the dianionic form of N,N'-bis(5-chloro-2-hydroxybenzylidene)-1,3-propanediamine (H_2L), was synthesized and characterized by elemental analyses, IR spectroscopy, and X-ray single-crystal determination. There are three bridges across the Ni-Ni atom pairs, involving two phenolate O atoms of a Schiff base ligand, and an O-C-O moiety of a $\mu_2-\eta^1:\eta^1$ -OAc group. The Ni atoms have octahedral coordination. The acetate bridges linking the central and terminal nickel atoms are mutually *trans*. The adjacent Ni···Ni distances are 3.047(1) Å. The complex was evaluated for its antibacterial (*Bacillus subtilis, Staphylococcus aureus, Escherichia coli*, and *Pseudomonas aeruginosa*) and antifungal (*Candida albicans* and *Aspergillus niger*) activities by MTT (3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide) method.

Keywords: Nickel(II) complex; Trinuclear complex; Schiff base; Crystal structure; Antibacterial activity

1. Introduction

Schiff bases are biological active compounds bearing the –N=CH– functional groups, which can be prepared by the condensation reactions of carbonyl-containing compounds with primary amines. The compounds have been attracted considerable attention for their wide range of biological activities, such as antibacterial, antifungal, antitumor, anti-inflammatory, and cytotoxic. Metal-organic

complexes containing Schiff bases and bridging ligands are of current interest because of their interesting molecular topologies as well as the fact that they may be designed with specific functionalities.⁶ Among the bridging groups, acetate anion is highly flexible, and can coordinate to metal atoms in a variety of coordination modes such as monodentate, chelating, bidentate bridging, monoatomic bridging, and chelating bridging (Scheme 1).⁷ Bis-Schiff bases



Scheme 1. Various coordination modes of acetate anion

derived from salicylaldehyde and its derivatives with various diamines usually coordinate to metal atoms through NNOO donor atoms,⁸ thus, it is possible for the introduction of bridging groups. It was reported that Schiff bases bearing electron-withdrawing groups can improve their antimicrobial activities.⁹ Rai and co-workers reported a series of fluoro, chloro, bromo, and iodo-substituted compounds, and found that they have significant antimicrobial activities.¹⁰ A number of metal complexes have been reported with the bis-Schiff base *N*,*N*'-bis(5-chloro-2-hydroxybenzylidene)-1,3-propanediamine (H₂L; Scheme 2).¹¹ As a continuation of work on the exploration of new antimicrobial agents, in this paper, a new trinuclear nickel complex was obtained.

Scheme 2. H₂L

2. Experimental

2. 1. General Methods and Materials

5-Chlorosalicylaldehyde and 1,3-propanediamine with AR grade were purchased from Sigma-Aldrich. All other chemicals (reagent grade) used were commercially available. Elemental analyses for C, H and N were performed on a Perkin-Elmer 240C elemental analyzer. IR spectra were recorded on a Nicolet AVATAR 360 spectrometer as KBr pellets in the 4000–400 cm⁻¹ region. ¹H NMR and ¹³C NMR spectra were recorded on a Bruker Avance 300 spectrometer operating at 300 MHz for ¹H NMR and 75.5 MHz for ¹³C NMR. Molar conductivity value was measured with a Shanghai DDS-11A conductometer.

2. 2. Synthesis of H₂L

5-Chlorosalicylaldehyde (2.0 mmol, 0.31 g) was dissolved in methanol (20 mL), to which a methanol solution (10 mL) of 1,3-propanediamine (1.0 mmol, 0.074 g) was added dropwise with stirring at room temperature. The mixture was stirred at room temperature for 30 min, and most of the solvent was removed by distillation. The yellow crystalline product was obtained by filtration. Yield: 93% (0.33 g). IR data (cm⁻¹, KBr): 3353w, 1645s, 1585s, 1487s, 1382w, 1329w, 1258s, 1213m, 1127w, 1033m, 985w, 865m, 831m, 772m. ¹H NMR (300 MHz, DMSO) δ 12.33 (s, 2H, OH), 8.54 (s, 2H, CH=N), 7.58 (s, 1H, ArH), 7.37 (d, 1H,

Ar*H*), 6.92 (d, 1H, Ar*H*), 3.73 (t, 4H, C*H*₂), 1.99 (m, 2H, C*H*₂). 13 C NMR (75 MHz, DMSO) δ 158.77, 156.32, 134.57, 131.26, 124.81, 121.05, 117.73, 61.78, 32.50. Elemental analysis found: C, 58.32; H, 4.67; N, 7.91%, $C_{17}H_{16}Cl_2N_2O_2$ calcd: C, 58.13; H, 4.59; N, 7.98%.

2. 3. Synthesis of the Complex

 H_2L (0.2 mmol, 0.70 g) was dissolved in methanol (20 mL), to which a methanolic solution (20 mL) of nickel acetate tetrahydrate (0.3 mmol, 0.75 g) was added with stirring. The mixture was stirred at room temperature for 30 min to give a green solution, which was kept still to slow evaporate of the solvents. Green block-like single-crystals suitable for X-ray diffraction were formed. Yield: 45% (0.16 g). IR data (cm $^{-1}$, KBr): 3451, 2910, 2862, 1637, 1580, 1464, 1416, 1385, 1306, 1164, 1075, 948, 859, 712, 549, 517, 465. Elemental analysis found: C, 43.77; H, 3.98; N, 5.27%, $C_{38}H_{40}Cl_4N_4Ni_3O_{11}$ calcd: C, 43.61; H, 3.85; N, 5.35%.

2. 4. X-ray Crystallography

X-ray diffraction was carried out at a Bruker SMART 1000 CCD area diffractometer equipped with MoK α radiation ($\lambda = 0.71073$ Å). The collected data were collected with SMART and reduced with SAINT,¹² and multi-scan absorption correction was performed using SADABS.¹³ The structure of the complex was solved by direct method, and refined against F^2 by full-matrix least-squares method using SHELXTL.¹⁴ All of the non-hydrogen atoms were refined anisotropically. The H5A and H5B atoms attached to O5 was located in a difference Fourier map and refined

Table 1. Crystallographic information for the complex

Parameter	Value		
Formula	C ₃₈ H ₄₀ Cl ₄ N ₄ Ni ₃ O ₁₁		
Fw	1046.67		
<i>T</i> , K	298(2)		
Crystal system	Monoclinic		
Space group	$P2_1/c$		
a, Å	11.049(2)		
b, Å	13.148(2)		
c, Å	14.988(2)		
b, °	90.971(1)		
<i>V</i> , Å ³	2177.0(6)		
Z	2		
D_c , g cm ⁻³	1.597		
F(000)	1072		
Measured reflections	11692		
Observed reflections $[I^3 2s(I)]$	4052		
Data/restraints/parameters	2008/3/284		
Goodness-of-fit on F^2	1.049		
$R_1, wR_2 [I^3 2s(I)]^a$	0.0642, 0.1684		
R_1 , wR_2 (all data) ^a	0.1429, 0.2024		

 $^{{}^}aR_1 = \mathring{a} ||Fo| - |Fc||/\mathring{a}|Fo|, \ wR_2 = [\mathring{a}w(Fo^2 - Fc^2)^2/\mathring{a}w(Fo^2)^2]^{1/2}, \ w = [\sigma^2(Fo)^2 + (0.091(Fo^2 + 2Fc^2)/3)^2 + 0.5732(Fo^2 + 2Fc^2)/3]^{-1}.$

isotropically, with O–H and H···H distances restrained to 0.85(1) and 1.37(2) Å, respectively. The remaining H atoms were placed in geometrically ideal positions and constrained to ride on their parent atoms. The O6 atom is disordered and its hydrogen atoms cannot be added reasonably. The crystallographic data for the complex are summarized in Table 1. Selected bond lengths and angles are given in Table 2.

Table 2. Selected bond lengths (Å) and angles (°) for the complexes with estimated standard deviations (e.s.d.s) in parentheses

Ni1-O1	1.985(5)	Ni1-O2	2.018(4)
Ni1-N1	2.009(5)	Ni1-N2	2.000(6)
Ni1-O3	2.051(6)	Ni1-O5	2.248(8)
Ni2-O2	2.092(5)	Ni2-O1	2.117(4)
Ni2-O4	2.121(7)		
O1-Ni1-N2	170.4(2)	O1-Ni1-N1	91.2(2)
N2-Ni1-N1	96.1(2)	O1-Ni1-O2	83.13(18)
N2-Ni1-O2	89.0(2)	N1-Ni1-O2	172.0(2)
O1-Ni1-O3	92.2(3)	N2-Ni1-O3	94.0(2)
N1-Ni1-O3	90.3(2)	O2-Ni1-O3	95.5(2)
O1-Ni1-O5	82.8(3)	N2-Ni1-O5	91.5(3)
N1-Ni1-O5	85.0(3)	O2-Ni1-O5	88.7(3)
O3-Ni1-O5	173.1(2)	O2-Ni2-O1A	101.73(18)
O2-Ni2-O1	78.27(17)	O2-Ni2-O4A	92.0(2)
O1-Ni2-O4A	90.4(2)	O2-Ni2-O4	88.0(2)
O1-Ni2-O4	89.6(2)		

Symmetry code for A: 1 - x, - y, - z.

2. 5. Antimicrobial Assay

The antibacterial activity of the compounds was tested against Bacillus subtilis, Staphylococcus aureus, Escherichia coli, and Pseudomonas aeruginosa using LB medium. The antifungal activities of the compounds were tested against Candida albicans and Aspergillus niger using RPMI-1640 medium. The IC₅₀ (half inhibitory concentration) of the test compounds were determined by a colorimetric method using the dye MTT (3-(4,5-di-methylth-iazol-2-yl)-2,5-diphenyltetrazolium bromide). stock solution of the synthesized compound (1000 µg mL⁻¹) in DMSO was prepared and graded quantities of the test compounds were incorporated in specified quantity of sterilized liquid LB medium. Suspension of the microorganism was prepared and applied to 96-well assay plate with serially diluted compounds to be tested. 10 µL of tested samples at pre-set concentrations were added to wells with penicillin G as a positive reference, the solvent control (5% DMSO) in medium and incubated at 37 °C for 24 h. After 24 h exposure, 10 µL of PBS (phosphate buffered saline 0.01 mol L^{-1} , pH = 7.4) containing 4 mg m L^{-1} of MTT was added to each well. After 4 h, the medium was replaced by 150 µL DMSO to dissolve the purple formazan crystals produced. The absorbance at 492 nm of each well was measured with an ELISA plate reader. The IC₅₀ value

was defined as the concentration at which 50% of the bacterial strain could survive.

3. Results and Discussion

The Schiff base ligand H₂L was readily prepared by the condensation reaction of 5-chlorosalicylaldehyde with 1,3-propanediamine in 2:1 molar ratio. Recently, two mononuclear nickel complexes were prepared in a 1:1 molar ratio of nickel acetate and H₂L under reflux in methanol. 15 And also an acetate and phenolate bridged trinuclear nickel complex was prepared by nickel acetate with H₂L under solvothermal condition.¹⁶ Interestingly, the present complex was prepared by the reaction of the Schiff base ligand with nickel acetate in a 2:3 molar ratio under ambient condition. Crystals of the complex are stable in air at room temperature, soluble in DMF, DMSO, MeOH, EtOH and MeCN, insoluble in water. The elemental analyses of the complex agree well with the component determined by X-ray analysis. The molar conductivity of the complex measured in DMSO/water at concentration of 10⁻³ mol L⁻¹ is $25 \Omega^{-1} \text{ cm}^2 \text{ mol}^{-1}$, indicating the non-electrolytic nature of the complex in solution.¹⁷

3. 1. Crystal Structure Description of the Complex

The molecular structure of the complex is shown in Fig. 1. The asymmetric unit of the compound contains half of a trinuclear complex. The molecule of the complex possesses crystallographic inversion center symmetry, with the inversion center located at the site of Ni2 atom. There are three bridges across the Ni···Ni atom pairs, involving two phenolate O atoms of a Schiff base ligand, and an O–C–O moiety of a μ_2 - η^1 : η^1 -OAc group. The acetate bridges linking the central and terminal nickel atoms are mutually *trans*. The trinuclear nickel complex molecule consists of two NiL units connected to each other by a completely encapsulated third metal atom, Ni2. The adjacent Ni1···Ni2 distance is 3.047(1) Å.

The cage of Ni2 is formed by phenolate bridges, O1 and O2, from the Schiff base ligands, and by two O atoms from two μ_2 - η^1 : η^1 -OAc ligands that furthermore connect the central metal with the two outer metal atoms resulting in an octahedral environment. The coordination around Ni2 atom displays only slight distortion. The bond distances Ni–O are relatively similar and range from 1.985(5) to 2.121(7) Å. The greatest deviation of the bond angles from those expected for an ideal octahedral geometry is found for O1–Ni2–O2 with 78.3(2)°, and O1–Ni2–O2A with 101.7(2)°. The remaining bond angles are close to the ideal values for the octahedral coordination.

The coordination around the inversion-related terminal Ni atoms is also octahedral, with two imino N and two phenolate O atoms from a Schiff base ligand defining

the equatorial plane, and with two O atoms respectively from a methanol and a μ_2 - η^1 : η^1 -OAc ligands occupying the axial positions. The coordination around the terminal metal atoms also displays slight distortion. The greatest deviation of the bond angles from those expected for an ideal octahedral geometry is O1–Ni1–O2 (81.9(2)°), which is

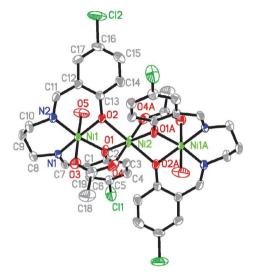


Fig. 1. Molecular structure of the complex. Displacement ellipsoids are drawn at the 30% probability level and H atoms are omitted for clarity. Atoms labeled with the suffix A or unlabeled are related to the symmetry position 1 - x, - y, - z.

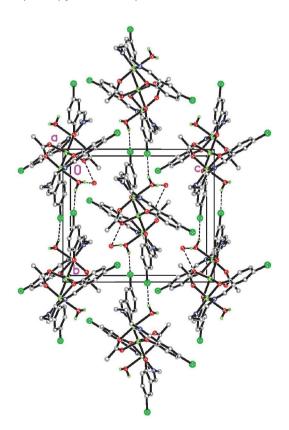


Fig. 2. Molecular packing diagram of the complex, viewed along the *a* axis. Hydrogen bonds are drawn as dashed lines.

caused by the strain created by the four-membered chelate ring Ni1–O1–Ni2–O2.

The NiL units in the complex are butterfly-shaped, with the dihedral angles formed by the two benzene rings of the Schiff base ligands of 53.2(5)°. In the crystal structure of the complex, the water hydrate molecules are linked to the nickel complex molecules through O5–H5A···O6ⁱ, O5–H5B···Cl2ⁱⁱ and C18–H18B···O6ⁱⁱⁱ hydrogen bonds [O5–H5A = 0.86 Å, H5A···O6ⁱ = 1.66 Å, O5···O6ⁱ = 2.518(5) Å, O5–H5A···O6ⁱ = 173°; O5–H5B = 0.85 Å, H5B···Cl2ⁱⁱ = 2.48 Å, O5···Cl2ⁱⁱ = 3.251(5) Å, O5–H5B···Cl2ⁱⁱ = 151°; C18–H18B = 0.96 Å, H18B···O6ⁱⁱⁱ = 2.36 Å, C18···O6ⁱⁱⁱ = 3.207(5) Å, C18–H18B···O6ⁱⁱⁱ = 147°; symmetry codes: i: x, $\frac{1}{2}$ – y, $\frac{1}{2}$ + z; ii: 1 – x, -y, 1 – z; iii: 1 – x, $\frac{1}{2}$ + y, $\frac{1}{2}$ – z], forming chains along the b axis (Fig. 2).

3. 2. Infrared Spectra

The IR spectra of the free Schiff base ligand and the nickel complex provide information about the metal-ligand bonding. The assignments are based on the typical group frequencies. The broad absorptions centered at 3353 cm⁻¹ for H₂L and 3451 cm⁻¹ for the complex are generated by the ν (O–H) of the hydroxy groups or methanol molecules. The strong absorption band at 1645 cm⁻¹ for H₂L is assigned to the azomethine groups, ν (C=N). The band is shifted to 1637 cm⁻¹ in the spectrum of the complex, what can be attributed to the coordination of the nitrogen atoms of the azomethine groups to the metal atoms. The phenolic v(Ar-O) for the free ligand exhibits medium band at 1213 cm⁻¹. 18 However, in the complex, the band appears at 1164 cm⁻¹. This may be assigned to the skeletal vibrations related to the phenolic oxygen of the Schiff base ligands, and the bands are known to shift to lower frequency when the phenolic oxygen coordinates to metal atoms.¹⁹ There exhibit typical acetate vibrations $v_{\text{asym}}(\text{OAc})$ at 1580 cm⁻¹ and $v_{\text{sym}}(\text{OAc})$ at 1464 cm⁻¹.²⁰

3. 3. Antibacterial Activity

The complex and the free Schiff base were screened for antibacterial activity against two Gram (+) bacterial strains (B. subtilis and S. aureus) and two Gram (-) bacterial strains (E. coli and P. aeruginosa) by MTT method. The IC₅₀ values of the compounds against four bacteria are listed in Table 3. Penicillin G was used as the standard drug. The Schiff base H₂L showed medium activity against the bacteria B. subtilis, S. aureus, and E. coli, while no activity against P. aeruginosa. The complex has strong activity against B. subtilis, medium activity against S. aureus, while no activity against the other two bacteria. From the results, it is difficult to give a definite conclusion about which one is good for the antibacterial activities of the free Schiff base and the complex. For example, the complex has stronger activities against B. subtilis and S. aureus than the Schiff base. However, as for E. coli, the Schiff base has stronger activity than the complex. The particular interest is that

Tested material B. subtilis S. aureus E. coli P. aeruginosa C. albicans A. niger 15.50 29.11 12.35 H_2L > 50 > 50 > 50 Complex 2.16 13 33 > 50 > 50 1772 > 50 Penicillin G 2.38 0.71 18.23 16.31 > 50 > 50

> 50

> 50

Table 3. IC_{50} values (µg mL⁻¹) of the tested material

> 50

the complex showed the most effective activity against *B. subtilis*, which is even more effective than penicillin G.

Ketoconazole

The antifungal activities of the complex and the free Schiff base were also evaluated against two fungal strains (C. albicans and A. niger) by MTT method. Ketoconazole was used as a reference drug. It is interesting that the complex has effective activity against C. albicans, with IC₅₀ value of 17.72 µg mL⁻¹.

4. Conclusion

In summary, a new linear trinuclear Schiff base nickel(II) complex derived from *N*,*N*'-bis(5-chloro-2-hydroxy-benzylidene)-1,3-propanediamine was presented. Structure of the complex was confirmed by single-crystal X-ray determination. The Ni atoms are in octahedral coordination. The complex has interesting antibacterial activities against *B. subtilis* and *S. aureus*, and antifungal activity against *C. albicans*.

5. Supplementary Material

CCDC-1507164 contain the crystallographic data for the complex. The data can be obtained at https://www.ccdc.cam.ac.uk or from the Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336033 or e-mail: deposit@ccdc.cam.ac.uk.

6. Acknowledgments

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Povzetek

Sintetizirali smo nov linearni trijedrni nikljev(II) kompleks s Schiffovo bazo, [Ni{NiL(μ_2 - η^1 : η^1 -OAc)(OH₂)}₂]-H₂O, kjer je L dianionska oblika *N*,*N*'-bis(5-kloro-2-hidroksibenziliden)-1,3-propandiamina (H₂L), in ga okarakterizirali z elementno analizo, IR spektroskopijo in rentgensko monokristalno analizo. Trije mostovi povezujejo Ni-Ni atomske pare, in sicer preko dveh fenolatnih O atomov Schiffove baze in O-C-O skupine μ_2 - η^1 : η^1 -OAc liganda. Nikljevi atomi imajo oktaedrično koordinacijo. Acetatna mostova, ki povezujeta sredinski in terminalna nikljeva atoma, sta v medsebojnem *trans* položaju. Ni-Ni razdalja je 3.047(1) Å. Kompleksu smo določili antibakterijske (*Bacillus subtilis*, *Staphylococcus aureus*, *Escherichia coli* in *Pseudomonas aeruginosa*) in antimikotične (*Candida albicans* in *Aspergillus niger*) lastnosti z uporabo MTT (3-(4,5-dimetiltiazol-2-il)-2,5-difeniltetrazolijev bromid) metode.