Scientific paper

Syntheses, Crystal Structures, Antimicrobial Activity and Thermal Behavior of Copper(II) Complexes Derived from 1-Naphthylacetic Acid and Diamines

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Received: 06-15-2019

Abstract

Two copper(II) complexes, $[CuL_2(EA)]$ (**1**) and $[Cu_2L_4(PA)_2] \cdot 2H_2O$ (**2** · $2H_2O$), where L is 1-naphthylacetate, EA is *N,N*-diethylethane-1,2-diamine, PA is propane-1,3-diamine, have been prepared and characterized. Structures of the complexes have been characterized by single-cyrstal X-ray diffraction. Complex **1** is a mononuclear copper(II) compound, with the Cu atoms coordinated in square planar geometry. Complex **2** · $2H_2O$ is a centrosymmetric dinuclear copper(II) compound, with the Cu atoms coordinated in square pyramidal geometry. Single crystals of the complexes are stabilized by hydrogen bonds. The effects of the compounds on the antimicrobial activity against *Staphylococcus aureus*, *Escherichia coli*, and *Candida albicans*, as well as thermal behavior of the complexes were studied.

Keywords: Copper complex; crystal structure; hydrogen bonding; antimicrobial activity; thermal property

1. Introduction

Carboxylic acids are a kind of interesting ligands for the preparation of metal complexes.[1] Copper is a very important element in biological systems, functions as the active site of hydrolytic enzymes, such as Cu-Zn superoxide dismutase.[2] Copper complexes with carboxylate ligands have been widely reported for their versatile structures and biological applications.[3] Intriguingly, the copper(II) complexes were shown to be significantly more potent than the metal free chelate, leading to the suggestion that the metal complex was the biologically active species.[4] Copper(II) complexes are known to play a significant role either in naturally occurring biological systems or as pharmacological agents.[5] 1-Naphthylacetic acid is structurally similar to carboxylic acids. However, very few copper complexes derived from 1-naphthylacetic acid have been reported so far. In the present paper, two new copper(II) complexes, $[CuL_2(EA)]$ (**1**) and $[Cu_2L_4(PA)_2] \cdot 2H_2O$ (**2** · $2H_2O$), where L is 1-naphthylacetate, EA is *N,N*-diethylethane-1,2-diamine, PA is propane-1,3-diamine, are reported.

2. Experimental

2. 1. Materials and Measurements

Commercially available 1-naphthylacetic acid, *N,N*-diethylethane-1,2-diamine, and propane-1,3-diamine were purchased from Aldrich and used without further purification. Other solvents and reagents were made in China and used as obtained. C, H and N elemental analyses were performed with a Perkin-Elmer elemental analyser. Infrared spectra were recorded on a Nicolet AVATAR 360 spectrometer as KBr pellets in the 4000–400 cm^{-1} region. Thermal stability analysis was performed on a Perkin-Elmer Pyris Diamond TG-DTA thermal analyses system.

2. 2. Synthesis of $[CuL_2(EA)]$ (1)

An aqueous solution (10 mL) of $CuSO_4 \cdot 5H_2O$ (0.10 mmol, 24.9 mg) was added to an aqueous solution (10 mL) of 1-naphthylacetic acid (0.20 mmol, 37.2 mg) and *N,N*-diethylethane-1,2-diamine (0.20 mmol, 23.2 mg) with stirring. The mixture was stirred for 1 h at ambient condition

to give a clear blue solution. The resulting solution was allowed to stand in air for several days. Blue block-shaped crystals suitable for X-ray single crystal analysis were formed at the bottom of the vessel. The isolated product was washed three times with water, and dried in a vacuum over anhydrous $CaCl_2$. For $C_{30}H_{34}CuN_2O_4$: anal. calcd., %: C, 65.60; H, 6.23; N, 5.09. Found, %: C, 65.71; H, 6.32; N, 4.98. IR data (ν, cm^{-1}): 3267, 1590, 1460, 1388, 1258, 1119, 1043, 782, 718, 630, 537, 465.

2. 3. Synthesis of $[Cu_2L_4(PA)_2] \cdot 2H_2O$ ($2 \cdot 2H_2O$)

This complex was prepared by the same method as that described for **1**, but with *N,N*-diethylethane-1,2-diamine replaced by propane-1,3-diamine (0.20 mmol, 14.2 mg). For $C_{54}H_{60}Cu_2N_4O_{10}$: anal. calcd., %: C, 61.64; H, 5.75; N, 5.32. Found, %: C, 61.50; H, 5.82; N, 5.23. IR data (ν, cm^{-1}): 3309, 3229, 1616, 1367, 1270, 1173, 1110, 1026, 912, 781, 710, 634, 546, 486.

2. 4. X-Ray Diffraction

Diffraction intensities for the complexes were collected at 298(2) K using a Bruker D8 VENTURE PHOTON diffractometer with Mo*Ka* radiation (l = 0.71073 Å). The collected data were reduced using SAINT,[6] and multi-scan absorption corrections were performed using SADABS.[7] The structures were solved by direct methods and refined against F^2 by full-matrix least-squares methods using SHELXTL.[8] All of the non-hydrogen atoms were refined anisotropically. The water H atoms in $\mathbf{2} \cdot 2H_2O$ were located from a difference Fourier map and refined isotropically, with O–H and H···H distances restainred to 0.85(1) and 1.37(2) Å, respectively. The remaining H atoms were placed in idealized positions and constrained to ride on their parent atoms. The low data completeness of complex **1** (76.2%) is due to the poor-quality data of the crystal. The crystallographic data for the complexes are summarized in Table 1.

2. 5. Antimicrobial Assay

Qualitative determination of antimicrobial activity was done using the disk diffusion method as described in the literature.[9] Suspensions in sterile peptone water from 24-h cultures of microorganisms were adjusted to 0.5 McFarland. Muller-Hinton Petri dishes of 90 mm were inoculated using these suspensions. Paper disks (6 mm in diameter) containing 10 μL of the substance to be tested (at a concentration of 2048 μg mL^{-1} in DMSO) were placed in a circular pattern in each inoculated plate. Incubation of the plates was done at 37 °C for 18–24 h. Reading of the results was done by measuring the diameters of the inhibition zones generated by the test substance. Tetracycline was used as a reference substance. Determination of MIC was done using the serial dilutions in liquid broth method. The materials used were 96-well plates, suspensions of microorganism (0.5 McFarland), Muller-Hinton broth (Merck) and stock solutions of each substance to be tested (2048 μg mL^{-1} in DMSO). The following concentrations of the substances to be tested were obtained in the 96-well plates: 1024, 512, 256, 128, 64, 32, 16, 8, 4 and 2 μg mL^{-1}. After incubation at 37 °C for 18–24 h, the MIC for each tested substance was determined by microscopic observation of

Table 1. Crystallographic and experimental data for complexes **1** and $\mathbf{2} \cdot 2H_2O$

	1	$\mathbf{2 \cdot 2H_2O}$
Formula	$C_{30}H_{34}CuN_2O_4$	$C_{54}H_{60}Cu_2N_4O_{10}$
FW	550.1	1052.1
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/c$	$P2_1/c$
a/Å	13.496(2)	11.474(2)
b/Å	9.111(1)	27.474(2)
c/Å	22.411(2)	8.409(1)
β/°	97.331(2)	101.937(2)
V/Å^3	2733.2(6)	2593.3(6)
Z	4	2
μ/mm^{-1}(Mo-Kα)	0.836	0.881
D_c/g cm^{-3}	1.337	1.347
Reflections/parameters	3873/336	4493/322
Independent reflections	1669	2316
Restraints	0	3
F(000)	1156	1100
Goodness of fit on F^2	1.007	1.011
R_1, wR_2 [$I \geq 2\sigma(I)$][a]	0.0648, 0.1068	0.0647, 0.1298
R_1, wR_2 (all data)[a]	0.2245, 0.1547	0.1625, 0.1599
Largest diff. peak and hole/e Å^{-3}	0.409, -0.728	0.845, –0.500

[a] $R_1 = \sum||F_o| - |F_c||/\sum|F_o|$, $wR_2 = [\sum w(F_o^2 - F_c^2)^2/\sum w(F_o^2)^2]^{1/2}$.

microbial growth. It corresponds to the well with the lowest concentration of the tested substance where microbial growth was clearly inhibited.

3. Results and Discussion

3. 1. Chemistry

The two copper complexes were prepared by the reaction of $CuSO_4 \cdot 5H_2O$, 1-naphthylacetic acid and *N,N*-diethylethane-1,2-diamine in a molar ratio of 1:2:2 in aqueous solution. Single crystals of the complexes are stable in air at room temperature. The complexes are soluble in water, DMSO, methanol and ethanol, but insoluble in dichloroform and chloroform. 1-Naphthylacetate ligand usually adopt bidentate coordination mode with copper ion,[10] with only two reported examples of monodentate coordination mode.[11]

3. 2. IR Spectra

The infrared spectra of the complexes were consistent with their structures as determined by X-ray diffraction. Features corresponding to naphthyl and benzene ring puckering exist in the region between 800 and 600 cm^{-1}. Asymmetric and symmetric C–O stretching modes of the fully deprotonated, ligated carboxylate groups were substantiated by strong, broadened bands at 1590 and 1389 cm^{-1} for complex **1**, and 1618 and 1369 cm^{-1} for complex **2** · $2H_2O$.[12] The lack of bands in the region of about 1710 cm^{-1} is indicative of complete deprotonation of all carboxylate groups in both complexes.

3. 3. Structure Description of the Complexes

The molecular structures of complexes are shown in Figures 1 and 2. Selected bond lengths and angles are listed in Table 2. Complex **1** is a mononuclear copper(II) compound. The Cu atom in the complex is coordinated by two N atoms of the diamines, and two carboxylate O atoms from two L, forming square planar geometry. Complex **2** · $2H_2O$ is a centrosymmetric dinuclear copper(II) compound, and contains two water molecules of crystallization. The Cu atoms in the complex are coordinated by the N atoms of the diamines, and the carboxylate O atoms from L, forming square pyramidal geometry. The bond lengths related to the Cu atoms in both complexes are similar to those observed in comparative copper complexes with carboxylate and amine ligands.[11,13] The square planar coordination of complex **1** and the basal plane of the square pyramidal coordination of complex **2** · $2H_2O$ are somewhat distorted, as evidenced from the coordinate bond angles, ranging from 85.3(2) to 93.6(2)° for the *cis* angles and from 167.3(3) to 169.0(3)° for the *trans* angles for **1**, and from 88.7(2) to 93.7(2)° for the *cis* angles and from 162.4(2) to 168.9(2)° for the *trans* angles for **2**.

In the crystal structures of the complexes (Figure 3 for **1** and Figure 4 for **2**), the molecules are linked through intermolecular N–H···O and O–H···O hydrogen bonds (Table 3), to form 1D chains.

Table 2. Selected bond lengths (Å) and angles (°) for **1** and **2** · $2H_2O$

1			
Cu1–O2	2.000(5)	Cu1–O3	1.965(5)
Cu1–N1	2.046(5)	Cu1–N2	1.984(5)
O2–Cu1–N1	93.74(17)	O2–Cu1–N2	168.90(18)
N1–Cu1–N2	90.6(2)	O2–Cu1–O3	88.72(15)
N1–Cu1–O3	162.43(18)	N2–Cu1–O3	90.19(17)
2 · $2H_2O$			
Cu1–O2	1.955(3)	Cu1–O3	2.002(4)
Cu1–N1	1.983(5)	Cu1–N2	1.984(4)
Cu1–O3$^{\#1}$	2.467(5)		
O3–Cu1–N2	167.3(3)	O3–Cu1–O2	90.6(2)
N2–Cu1–O2	93.6(2)	O3–Cu1–N1	92.8(2)
N2–Cu1–N1	85.3(2)	O2–Cu1–N1	169.0(3)
O3$^{\#1}$–Cu1–N1	115.0(3)	O3$^{\#1}$–Cu1–N2	84.5(3)
O3$^{\#1}$–Cu1–O2	84.4(3)	O3$^{\#1}$–Cu1–O3	82.6(3)

Table 3. Geometrical parameters for hydrogen bonds

Hydrogen bonds	***D*–H (Å)**	**H···*A* (Å)**	***D*···*A* (Å)**	***D*–H···*A* (°)**
1				
N2–H2B···O1$^{\#1}$	0.90	2.14	2.991(10)	157
N2–H2A···O4$^{\#2}$	0.90	2.09	2.946(8)	158
2 · $2H_2O$				
O5–H5B···O4$^{\#3}$	0.85(1)	2.03(3)	2.780(8)	148(5)
N2–H1B···O2$^{\#4}$	0.90	2.25	3.080(6)	153
N1–H2B···O1$^{\#3}$	0.90	2.16	2.971(6)	149
N1–H2A···O5	0.90	2.25	3.119(9)	162

$^{\#1}$ 1 – *x*, ½ + *y*, ½ – *z*; $^{\#2}$ 1 – *x*, –½ + *y*, ½ – *z*; $^{\#3}$ 1 – *x*, –*y*, 1 – *z*; $^{\#4}$ 1 – *x*, –*y*, –*z*.

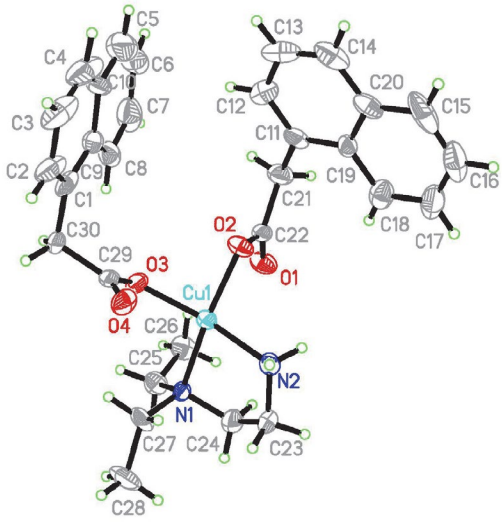

Figure 1. Molecular structure of **1** at 30% probability displacement.

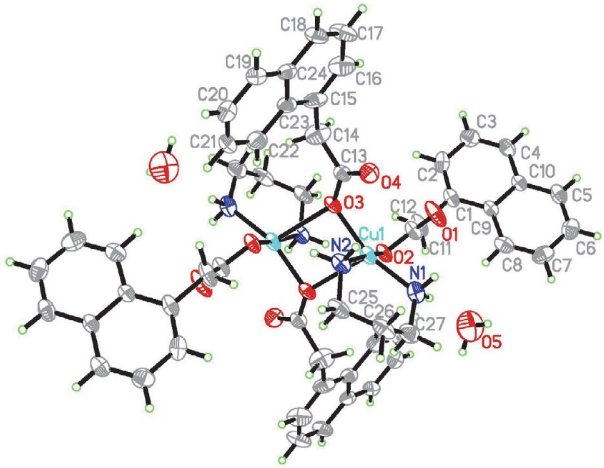

Figure 2. Molecular structure of **2** · $2H_2O$ at 30% probability displacement.

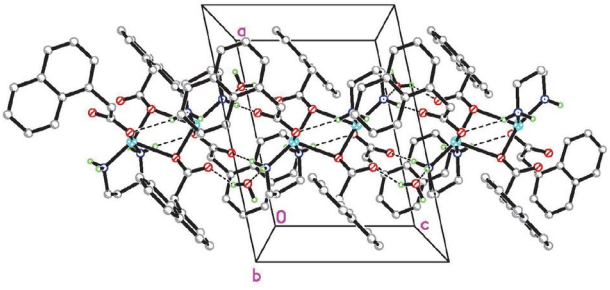

Figure 3. Molecular packing of **1**, viewed along the *a* axis. Hydrogen bonds are drawn as dashed lines. Hydrogen atoms not related to hydrogen bonds are omitted for clarity.

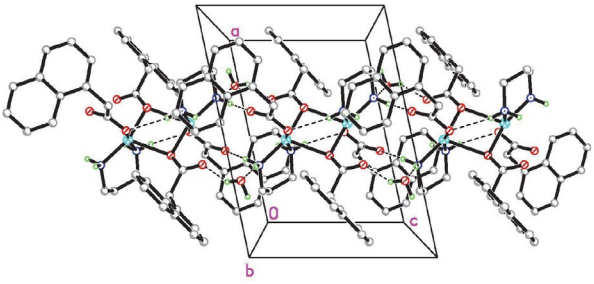

Figure 4. Molecular packing of **2** · $2H_2O$, viewed along the *b* axis. Hydrogen bonds are drawn as dashed lines. Hydrogen atoms not related to hydrogen bonds are omitted for clarity.

3. 4. Thermal Stability

Differential thermal (DT) and thermal gravimetric analyses (TGA) were conducted to examine the stability of the complexes. For **1** (Figure 5), the first step started at 166 °C and completed at 320 °C, corresponding to the loss of the *N,N*-diethylethane-1,2-diamine ligand and one 1-naphthylacetate ligand. The observed weight loss of 53.8% is close to the calculated value of 54.5%. The second step, from 320 °C to 526 °C, corresponds to the loss of the other 1-naphthylacetate ligand and the formation of the final product (CuO). The observed weight loss of 32.1% in this step is close to the calculated value of 30.7%. For **2** · $2H_2O$ (Figure 6), the first step slowly started at about 50 °C and was completed at 128 °C, corresponding to the loss of the lattice water molecule. The observed weight loss of 1.8% is close to the calculated value of 1.7%. The second step started at about 128 °C and was completed at 350 °C, corresponding to the loss of the propane-1,3-diamine ligand and one 1-naphthylacetate ligand. The observed weight loss of 49.8% is close to the calculated value of 49.2%. The third step, from 350 °C to 575 °C, corresponds to the loss of the other 1-naphthylacetate ligand and the formation of the final product (CuO). The observed weight loss of 33.9% in this step is close to the calculated value of 32.1%.

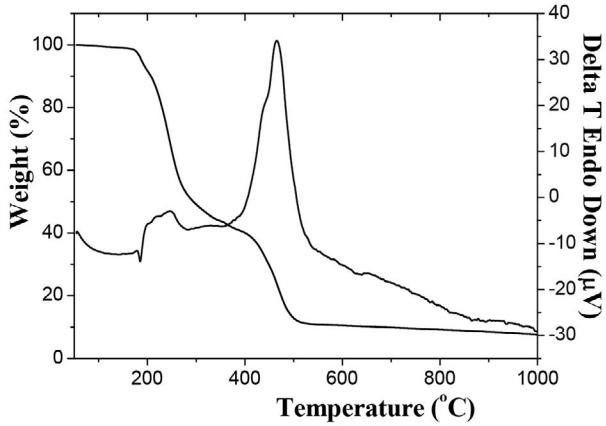

Figure 5. DT-TGA curve of **1**.

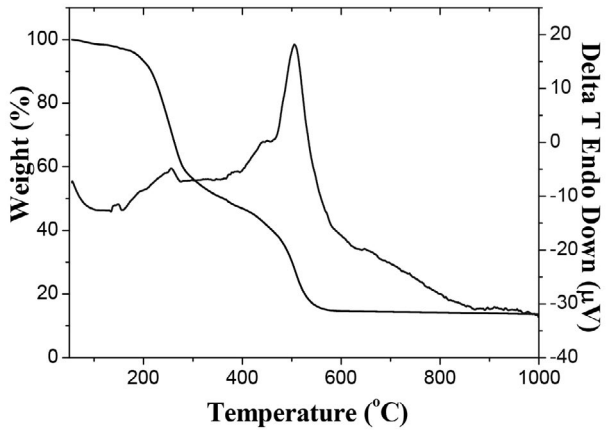

Figure 6. DT-TGA curve of **2**·$2H_2O$.

3. 5. Antimicrobial Activity

The antimicrobial activity results are summarized in Table 4. A comparative study of minimum inhibitory concentration (MIC) values of 1-naphthylacetic acid, *N,N*-diethylethane-1,2-diamine, propane-1,3-diamine, and the complexes indicates that the copper complexes have better activity than the free ligands. Generally, this is caused by the greater lipophilic nature of the complexes than the ligand.

Table 4. MIC values (μg mL^{-1}) for the antimicrobial activities

Compounds	*Staphylococcus aureus*	*Escherichia coli*	*Candida albicans*
1-naphthylacetic acid	64	128	>512
N,N-diethylethane-1,2-diamine	128	>512	>512
propane-1,3-diamine	128	>512	>512
1	0.5	2.0	32
2 · $2H_2O$	0.5	2.0	32
Tetracycline	0.32	2.12	>1024

Such increased activity of the metal chelates can be explained on the basis of chelating theory.[14] On chelating, the polarity of the metal atoms will be reduced to a greater extent due to the overlap of the ligand orbital and partial sharing of positive charge of the metal atoms with donor atoms. Further, it increases the delocalization of *p*-electrons over the whole chelate ring and enhances the lipophilicity of the complexes. This increased lipophilicity enhances the penetration of the complexes into lipid membrane and blocks the metal binding sites on enzymes of microorganisms.

From the results, it is obvious that the two complexes have higher antibacterial and antifungal activities against *Staphylococcus aureus*, *Escherichia coli*, and *Candida albicans* when compared to the free ligands. The two complexes have in general the same activities against all the bacteria and fungi strains. The best inhibition on *Staphylococcus aureus* is indicated by the MIC values of 0.5 μg mL^{-1}, which is similar to tetracycline. The complexes have strong activity against *Escherichia coli*, which is even comparable to tetracycline. It is interesting that both complexes have medium activity against *Candida albicans*, which is rarely seen in metal complexes.

4. Supplementary Material

CCDC–957485 (**1**) and 957486 (**2**·$2H_2O$) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge at http://www.ccdc.cam.ac.uk/const/retrieving.html 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.

Acknowledgments

This research was supported by the Top-class foundation of Pingdingshan University (no. PXY-BSQD-2018006 and PXY-PYJJ-2018002).

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Povzetek

Sintetizirali in okarakterizirali smo dva nova bakrova(II) kompleksa, $[CuL_2(EA)]$ (**1**) in $[Cu_2L_4(PA)_2] \cdot 2H_2O$ (**2** $\cdot 2H_2O$), kjer je L 1-naftilacetat, EA je *N,N*-dietiletan-1,2-diamin in PA je propan-1,3-diamin. Strukturi kompleksov smo določili z rentgensko monokristalno analizo. Kompleks **1** je enojedrna bakrova(II) spojina z Cu atomom koordiniranim s kvadratno planarno geometrijo. Kompleks **2** $\cdot 2H_2O$ je centrosimetrična dvojedrna bakrova(II) spojina z Cu atomom koordiniranim s kvadratno piramidalno geometrijo. Kristalna struktura je stabilizirana z vodikovimi vezmi. Antimikrobne aktivnosti obeh spojin smo testirali na *Staphylococcus aureus*, *Escherichia coli* in *Candida albicans*. Proučili smo tudi termične lastnosti spojin.