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Three multinuclear metal-organic coordination compounds based on 8-hydroxyquinoline derivative: syntheses, structures

and fluorescence properties

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Abstract: By taking advantage of the synthetic 8-hydroxyquinoline derivatives, (E)-2-[2- (4-chlorophenyl)vinyl]-8-hydroxyquinoline (HL), three new metal-organic coordination compounds $[Zn_3L_6]$ (1), $[Cd_3L_6]$ (2) and $[Cu_2L_4]$ (3) have been obtained by solvothermal or solution processing methods and structurally characterized. Single crystal X-ray diffraction analysis revealed that complexes 1 and 2 have similar 'V'-type trinuclear structures. Different from complexes 1 and 2 (trinuclear structure), complex 3 displays dinuclear structure. The effects of central metals and reaction solvents on the structures of complexes 1–3 have been discussed. Furthermore, thermogravimetric analysis and solid state fluorescence of ligand HL, 1–3 were investigated.

Keywords: 8-hydroxy quinoline derivatives; metal-organic coordination compounds; crystal structure; solid state fluorescence

1. Introduction

The construction of novel metal-organic coordination polymers (MOCPs) has received extensive research efforts not only for their intriguing architectures and topologies but also for their promising application in chemical separation [1], catalysis [2], selective adsorption [3], luminescence [4], magnetism [5], etc. The selection of optimal organic ligands plays an important role in the construction of desirable MOCPs. Since the researchers first reported aluminum tris-8-hydroxyquinoline (AlQ₃) could be used as an emissive material in organic light-emitting diodes (OLEDs) [6], the research of quinolinates-based MOCPs have resulted in a large number of functional materials. In particular, a lot of 8-Hydroxyquinoline derivatives could be fabricated through organic reactions [7], hence 8-hydroxyquinoline and its derivatives are usually selected as functional organic ligands to react with a wide range of metal ions, including main group, transition and rare earth metal ions, and leads to the formation of materials with light-emitting properties [8]. Previous reports have demonstrated that luminescent properties of 8-hydroxyquinolinate-based complexes are not only related to the composition of the materials, but also are heavily dependent on the molecular and supramolecular structure which fabricated via the intermolecular noncovalent interactions [9]. It is thus crucial to tune the structure and intermolecular packing at the molecular level to accomplish luminescence materials [10]. Specifically, to overcome the limitations of the MQ_n -type mononuclear complexes, Lewiński [11] and Yuan [12] recently have synthesized a series of multinuclear Zn(II) and Cd(II) coordination compounds involving 8-hydroxyquinoline derivative ligands aiming to obtain improved and controllable photoluminescence features. So it is anticipated that more quinolinates-based coordinaton compounds could be constructed consistently and insights into the relationship between the structural characteristics and properties could be elucidated and consequently, help to develop useful functional materials.

On the basis of the aforementioned points, in this contribution, we synthesized a 8-hydroxyquinoline derivative as ligand (Scheme 1), namely (E)-2-[2- (4-chlorophenyl)vinyl]-8-hydroxyquinoline (**HL**), to react with different

transition metal ions with the aim to understand the effect of the central metals on the construction and architectures of the target coordination compounds. As a result, three new coordination compounds: $[Zn_3L_6]$ (1), $[Cd_3L_6]$ (2) and $[Cu_2L_4]$ (3) have been achieved under solvothermal or solution processing conditions. The effect of the central metals on the assembly and structures of the title complexes has been discussed. Additionally, thermogravimetric analysis and solid state fluorescence of 1–3 were investigated.



Scheme 1 The synthetic routes of HL and complexes 1-3.

2. Experimental

2.1. Reagents and general techniques

All chemicals were reagent grade and used as supplied from Aladdin-Reagent without further purification. Elemental analyses of C, H and N were performed with a vario MICRO elemental analyzer. The IR (KBr pellet) spectrum was recorded (400–4000 cm⁻¹ region) on a VARIAN CARY5000 IR spectrometer. ¹H and ¹³C NMR experiments were carried out on an ADVENCE400M spectrometer operating at resonance frequencies of 400 MHz. Thermal gravimetric analysis (TGA) was performed using a STA-409PC Synchronous thermal analyzer with a heating rate of 0.5 °C min⁻¹. Luminescence spectra were measured on a Hitachi F4000 apparatus at

room temperature.

Electrochemistry property was investigated in a three-electrode arrangement with a glassy carbon ($\Phi = 1 \text{ mm}$) working electrode, a Pt wire as the counter electrode, and Ag / AgCl as the reference electrode. Ferrocene was added as an internal standard and all potentials are quoted against ferrocenium / ferrocene couple (Fc⁺ / Fc).

Safety consideration: the $Zn(ClO_4)_2GH_2O$ is potential explosive and it is recommended that care be taken when handling this substance and only minimum amount be used.

2.2. Synthesis of ligand (HL)

Step one: The p-chlorobenzaldehyde (0.01 mol, 1.41 g) was added to a suspension of 2-methyl-8-hydroxyquinoline (0.01 mol, 1.59 g) in acetic anhydride (10 mL) at the room temperature. Then the mixture was magnetically stirred and heated at reflux for 20 hours. When the mixture cooled to room temperature, the reaction solution was poured into 800 mL ice water, and the yellow-brown solid has been obtained. Then the solid was extracted with dichloromethane. The organic phase was washed by distilled water and saline solution, and dried by anhydrous sodium sulfate over 0.5 hour. The solvent of the organic phase was removed by reduced pressure to give the crude product, and the crude product was purified by silica gel column chromatography to give (E)-2-[2-(4-chlorophenyl) ethenyl]-8-acetoxyquinoline (2.17 g, 70%).

Step two: (E)-2-[2-(4-chlorophenyl)ethenyl]-8-acetoxyquinoline (6.7 mmol, 2.17 g) was dissolved in pyridine (30 mL) and stirred for 20 minutes, then distilled water (10 mL) was added under magnetic stirring, the mixture was reflux for 12 hours. Next let it cool to room temperature, the pyridine was removed under reduced pressure. After added 200 mL distilled water, the mixture was stirred for 0.5 hour at room temperature, and yellow solid was got after filtration. Finally, the yellow solid was purified by column chromatography to give the desired product (E)-2-[2-(4-chlorophenyl)ethenyl]-8-hydroxyquinoline (HL) (1.89 g, 90%). ¹H-NMR(CDCl₃) δ : 7.14-7.16(d, J = 8.4 Hz, 1H), 7.29-7.41(m, 4H), 7.53-7.67(m, 4H), $8.09-8.11(d, J = 8.8 Hz, 1H); {}^{13}C-NMR(CDCl_3) \delta: 110.0, 117.7, 120.4, 127.4, 128.4,$

129.0, 132.8, 134.4, 134.8, 136.5, 137.9, 151.9, 135.1. Elemental analysis (%): calcd for (C₁₇H₁₂ONCl) C: 72.46, H: 4.26, N: 4.97. Found C: 72.33, H: 4.41, N: 4.86.

2.3. Synthesis of complex 1 (Zn_3L_6):

A solvothermal reaction of $Zn(ClO_4)_2$ $^{\circ}6H_2O$ (7.4 mg), and HL (1.2 mg) in a mixture solvent system of DMF-ethanol-acetonitrile (0.2 mL/0.5 mL/3 mL) was performed at 100 °C for 24h. The orange-red crystals of **1** were obtained in 20% yield (based on ligand). Elemental analysis (%): Calcd for $C_{102}H_{66}Cl_6N_6O_6Zn_3$: C: 65.15, H: 3.54, N: 4.47. Found: C: 62.95, H: 3.73, N: 4.92. IR (KBr pellet): 410.8(m), 449.8(w), 488.9(m), 528.0(m), 710.2(m), 749.2(m), 827.8(s), 974.8(w), 1004.3(w), 1088.2(m), 1151.8(m), 1194.7(s), 1235.2(m), 1322.9(m), 1401.5(m), 1436.2(m), 1460.8(s), 1514.8(s), 1563.5(s), 1587.6(m), 3039.8(w), 3373.4(s).

Note: Our attempts to use other Zn salts such as $Zn(OAc)_2^{-2}H_2O$, $ZnSO_4^{-7}H_2O$, $Zn(NO_3)_2^{-6}H_2O$ etc were unsuccessful (to avoid using $Zn(ClO_4)_2^{-6}H_2O$ due to the safety concern), this may indicate that careful selecting the starting material is crucial for the experiment.

2.4. Synthesis of complex $2(Cd_3L_6)$

Complex 2 was synthesized following the procedure similar to that of complex 1, except that $Cd(OAc)_2 H_2O$ (2.3 mg) was added instead of $Zn(ClO_4)_2 H_2O$. The red crystals of 2 were obtained in 20% yield (based on ligand). Elemental analysis (%): Calcd for $C_{102}H_{66}Cd_3Cl_6N_6O_6$, C: 60.60, H: 3.29, N: 4.16. Found: C: 59.81, H: 3.63, N: 4.25. IR (KBr pellet): 404.5(m), 484.5(m), 717.9(m), 746.9(s), 839.4(m), 973.9(m), 1009.1(m), 1089.1(s), 1153.2(s), 1199.5(m), 1234.7(w), 1316.2(w), 1432.9(s), 1508.1(s), 1560.6(m), 1595.8(s), 3043.2(w), 3357.0(m), 3374.4(m).

2.5. Synthesis of complex $3(Cu_2L_4)$

A mixture of CuSO₄·5H₂O (2.5 mg), and HL (1.2 mg) in a solvent system of DMF-THF (3 mL/2 mL) was performed at room temperature. Then the solvent was evaporated at room temperature to give the black crystals **3** in 35% yield (based on the ligand) after three days. Elemental analysis (%): Calcd for C₆₈H₄₄Cl₄Cu₂N₄O₄, C: 65.34, H: 3.55, N: 4.48. Found: C: 65.17, H: 3.70, N: 4.38. IR (KBr pellet): 409.3(m),

537.1(w), 740.5(m), 828.3(s), 967.6(m), 1009.1(m), 1106.5(s), 1270.0(m), 1333.5(m), 1375.5(w), 1455.0(s), 1497.0(s), 1549.5(m), 1598.6(s), 1629.6, 2362.0(w), 3048.0(m), 3449.6(m).

2.6. X-ray crystallography

Suitable crystals of **1-3** were collected on a Xcalibur, Eos, Gemini diffractometer. The crystal was kept at 293 K during data collection. Using Olex2 [13], the structure was solved with the Superflip [14] structure solution program using Charge Flipping and refined with the olex2.refine refinement package using Gauss-Newton minimisation. Crystallographic data and structure refinements for **1-3** are summarized in Table 1 and structural diagrams are shown in supporting information.

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Compound	$Zn_{3}L_{6}(1)$	$\operatorname{Cd}_{3}\operatorname{L}_{6}(2)$	$Cu_2L_4(3)$	
Empirical formula	$C_{102}H_{66}Cl_6N_6O_6Zn_3$	$C_{102}H_{66}Cl_6N_6O_6Cd_3$	$C_{68}H_{44}Cl_4N_4O_4Cu_2$	
Formula weight	1880.41	2021.50	1249.95	
Crystal system	Monoclinic	Monoclinic	Monoclinic	
Space group	P2 ₁	<i>I</i> 2/c	<i>P</i> 2 ₁ /c	
a(Å)	16.0776(9)	13.9862(6)	13.2890(1)	
b(Å)	21.6907(9)	20.3381(1)	14.6353(1)	
c(Å)	24.8601(1)	29.9964(1)	14.2812(10)	
a(deg)	90	90	90	
β (deg)	101.029(5)	91.454(4)	104.948(7)	
γ(deg)	90	90	90	
$V(\text{\AA}^3), Z$	8509.4(7), 4	8529.8(7), 4	2683.5(4), 2	
$D_c(\mathbf{g}\cdot\mathbf{cm}^{-3})$	1.468	1.574	1.574	
<i>F</i> (000)	3840	4056	1276	
GOOF on F^2	1.025	1.020	0.995	
μ(mm ⁻¹)	1.089	0.991	1.050	
Reflns collected/unique	42002/31275	19037/9835	12652/6160	
R _{int}	0.0505	0.0641	0.0832	
R_1 , wR_2 ($I > 2\sigma(I)$)	0.0934/0.1922	0.0924/0.1919	0.0995/0.2046	
Flack parameter	0.605(5)			
$R_{l} = \Sigma \mid F_{0} \mid - F_{c} \mid /\Sigma$	$ F_0 \cdot wR_2 = \{\Sigma[w($	$(F_0^2 - F_c)^2] / \Sigma [w(F_0^2)^2] \}^{1/2}$	/2	

Table 1 Crystal data and structure refinements for 1–3.

3. Result and discussion

3.1. Structural description of complexes 1–3

3.1.1. Crystal structure of complex 1

Single-crystal X-ray diffraction analysis reveals that complex 1 crystallizes in the monoclinic $P2_1$ space group. Complex 1 consists of three Zn(II) ions and six L⁻ anions. As shown in Fig. 1, there are three crystallographically independent Zn(II) ions (Zn1, Zn2 and Zn3), which show different coordination geometries (Scheme 2).

Zn1 is five-coordinated by the N atom from quinoline ring as the vertice, three hydroxyl O atoms from three quinoline rings and one N atom from one quinoline ring as the basal plane, forming a distorted rectangular pyramid geometry. Similar to Zn1, Zn3 is also five-coordinated but in a trigonal bipyramid geometry by the hydroxyl O atom as the vertice, two hydroxyl O atoms from two quinoline rings and two N atoms from quinoline rings as the basal plane. While Zn2 is six-coordinated in a distorted octahedral geometry, which is defined by four hydroxyl O atoms from four quinoline rings and two N atoms from two quinoline rings. Three Zn (II) ions (Zn1, Zn2 and Zn3) are bridged by two hydroxyl O atoms of ligands L^{*} to construct a 'V'-type trinuclear cluster [Zn₃O₄] with the angle of 115.28° (Scheme 3), in which the Zn...Zn distances are 3.293 and 3.222 Å. The adjacent 'V'-type trinuclear clusters are further connected by the intermolecular hydrogen bonds [C(71)–H(71)^{...}Cl(5) = 3.406 Å] into a 1D supramolecular chain (Fig. 2).



Fig. 1 Coordination environment of Zn(II) ions (In order to clearly show the coordination environment of Zn, only show the quinoline ring of the ligand L^{-} .)



Fig. 2 1D supramolecular chain formed by the hydrogen bonding weak interactions in 1 [C(71)-H(71)-Cl(5) = 3.406 Å].

3.1.2. Crystal structure of complex 2

X-ray diffraction analysis reveals that complex **2** crystallizes in the monoclinic *I*2/c space group. The structure of complex **2** is similar with complex **1**, as shown in Fig. 3, there are two crystallographically independent Cd(II) ions (Cd1 and Cd2) (Scheme 2). Cd1 is five-coordinated, resulting in a distorted rectangular pyramid geometry with the equatorial plane occupied by the NO3 donors of three **L**⁻ ligands and the apical position by the N atom from quinoline ring. The coordination environment of Cd2 is a distorted octahedron and the equatorial plane is occupied by the NO3 donors of three **L**⁻ ligands, while the apical position by one hydroxyl O atom and a pyridine atom. Each two adjacent Cd(II) ions are bridged by two hydroxyl O atoms to form a 'V'-type trinuclear cluster [Cd₃O₄], in which the angle is 102.80° and the Cd⁻⁻Cd distance is 3.604 Å (Scheme 3).



Fig. 3 Coordination environment of Cd(II) (In order to clearly show the coordination environment of Cd, only show the quinoline ring of the ligand \mathbf{L}^2 .) Symmetry code: #1 1 - x, y, 0.5 – z.

3.1.3. Crystal structure of complex 3

Complex 3 crystallizes in the monoclinic $P2_1/c$ space group and it consists of two Cu(II) ions and four L⁻ anions. As shown in Fig. 4, there is only one crystallographically independent Cu(II) ion (Cu1) (Scheme 2). Cu1 adopts a slightly distorted rectangular pyramid geometry with the equatorial plane occupied by two chelating NO donor sets from two L⁻ ligands and the apical position by one hydroxyl O atom of another L⁻ ligand. Two Cu(II) ions also are linked by two hydroxyl O

atoms to form a dinuclear cluster $[Cu_2O_2]$ with the Cu...Cu distances of 3.190 Å (Scheme 3).



Fig.4 Coordination environment of Cu(II) (In order to clearly show the coordination environment of Cu, only show the quinoline ring of the ligand L^{*}.) Symmetry code: #1 1 - x, 1 - y, 1 - z.



Scheme 2 Coordination geometries of all crystallographically independent metal ions $(Zn^{2+}, Cd^{2+} \text{ and } Cu^{2+})$ in complexes 1–3.

3.2. Effect of the central metals and reaction solvents on the structures of the title complexes

It is well known that the central metal ions play an important role in the formation of the final frameworks and various structures [15]. In this work, three coordination compounds based on 8-hydroxyquinoline derivative ligand and different metal centers (Zn(II), Cd(II), Cu(II)) have been obtained by solvothermal or solution

processing methods. When choosing DMF/C₂H₅OH/CH₃CN as mixed solvents, crystals **1** and **2** have formed with similar 'V'-type trinuclear structure, but there are subtle differences for the angles of 'V'-type structure and coordination environments (115.28° for complex **1**, and 102.80° for complex **2**) (Scheme 3). We hypothesized that it may be derived from different ionic radii of the metal ions ($r_{Zn(II)} = 74$ pm, $r_{Cd(II)} = 95$ pm). Under the similar reaction condition (T = 100°C), when we selected the same solvent system (DMF/C₂H₅OH/CH₃CN) for complexes **1** and **2**, and just changed the metal center into Cu(II), unfortunately, we could not obtain any suitable crystals. Therefore, we tried other solvent system in the synthesis. Only when the mixed solvents were replaced by DMF/THF, crystal **3** could be obtained at room temperature with different dinuclear structure, whereas the solvothermal experiment (DMF/THF system) carried at 100 °C could not achieve **3**. Above results indicate that the central metals, reaction solvents and temperature play important roles in the formation of the multinuclear structures of complexes **1**–**3**.



Scheme 3 The difference of the cluster structures of complexes 1–3.

3.3. Power X-ray diffraction (PXRD) and themogravimetric analysis (TGA)

In order to confirm the crystal structure of complexes 1-3 are truly representative of their bulk materials, the PXRD experiments have been carried out on the as-synthesized sample at room temperature. The PXRD experimental patterns of 1-3match well with the patterns simulated from the single-crystal structure (Fig. 5), confirming the high purity of bulky phase of those samples. The preferred orientation of the powder sample may cause the differences in intensity [16].



Fig. 5 PXRD patterns of complexes 1–3: (a) calculated (b) experimental.

The thermal stability of ligand **HL**, the complexes 1–3 was examined by thermogravimetric analysis (TGA) under a N₂ atmosphere from 17 to 800 °C with a heating rate of 0.5 °C min⁻¹ (Fig. 6). Thermogravimetric analyses indicate all compounds exhibit only one weight loss step which is attributed to the decomposition of organic ligand. For ligand **HL**, the weight loss process is slow before 235 °C, then fell rapidly, and finished the degradation at 500 °C. For complex 1, it didn't lose weight fast until 400 °C. Similarly, complex 2 and complex 3 started to lose weight fast until 450 °C and 300 °C, respectively. This indicates that combination of the metal ions and ligand **HL** enhances their thermal stability, and the strength of the thermal stability is **HL**<complex 3</p>



Fig. 6 TGA curves for HL and complexes 1–3.

3.4. Luminescent Properties

Luminescent complexes are of great interest due to their various application in chemical sensors, photochemistry, and light-emitting diode (LED) [17]. Therefore, the soild-state luminescent properties of pure **HL** and complexes **1–3** are tested at room temperature (Fig.7). When excited with 419 nm light, the free HL ligand exhibited a emission peak at 585 nm, which originated from internal charge transfer [12c]. Upon excitation at 420 and 390 nm, complexes **1** and **2** displays intense photoluminescence, with emission maxima at 594 and 583 nm, respectively. Different from complexes **1** and **2**, complex **3** displays fluorescence quenching. Compared with the free **HL** ligand, the introduction of Zn(II) and Cd(II) to ligand **HL** led to a red-shift of 9 nm for complex **1** and a blue-shift of 2 nm for complex **2**, this may originate from $\pi^{m}\pi$ transition [12a].



Fig. 7 Emission spectra of HL and complexes 1–3 in the solid state.

3.5. Electrochemical property of complex 3

Fig. 8 shows cyclic voltammograms of complex **3** at different scan rates, and reduction and oxidation potentials for this dinuclear copper complex are summarized in Table 2. It can be seen that two irreversible redox couples were observed in the range of -1.5 to -0.5 V which could be attributed to the stepwise reduction of the Cu(II) center, namely, the redox process is $Cu^{II}Cu^{II} \rightarrow Cu^{I}Cu^{II} \rightarrow Cu^{I}Cu^{I}$ [18].



Fig. 8 Cyclic voltammograms of complex **3** in DMF ($c = 2.0 \times 10^{-4}$ M) (0.4 M Bu₄N⁺BF₄⁻ as supporting electrolyte).

Table 2 Electrochemical Data for 3 at the scan rate of 300 mV s⁻¹.[E(V) vs Fc⁺ / Fc]

Complex	$E^{1}_{ m pc}$	E^2_{pc}	E^{1}_{pa}	${E^2}_{ m pa}$
3	-1.025	-1.296	-0.684	-1.108

4. Conclusion

In summary, we have successfully prepared three metal-organic coordination compounds, namely, trinuclear structures for complexes 1 and 2, dinuclear structure which for based 8-hydroxyquinoline complex on derivative (E)-2-[2-(4-chlorophenyl)vinyl]-8-hydroxyquinoline (HL). The central metals, reaction solvents and temperature play important roles in the formation of complexes 1-3. Themogravimetric analysis indicates complexes 1-3 have good thermal stabilities. The fluorescent properties show that complexes 1 and 2 may be potential fluorescence materials. We believe this study demonstrates the effectiveness of general solvothermal and solution processing methods which are influenced by the nature of the solvent and reaction temperature. More variable parameters such as pH values, stoichiometries and reaction concentrations, kinetic and thermodynamic factors controlling the sequence of reaction steps will lead to diverse functional crystalline materials. Future work will use different transition and rare earth metal ions anticipating more specific coordination mode and systematic studies on the different experimental parameters are actively pursued in the laboratory.

Appendix A.

CCDC 1415160, 1415158, 1415159 contains the supplementary crystallographic data for **1**, **2** and **3**. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

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Graphical abstract

Three multinuclear metal-organic coordination compounds based on 8-hydroxyquinoline derivative: syntheses, structures and fluorescence properties



Synopsis

Three multinuclear metal-organic coordination compounds based on 8-hydroxyquinoline derivative: syntheses, structures and fluorescence properties

Three new metal-organic coordination polymers $[Zn_3L_6]$ (1), $[Cd_3L_6]$ (2) and $[Cu_2L_4]$ (3) based on 8-hydroxyquinoline derivative (E)-2-[2- (4-chlorophenyl)vinyl]-8-hydroxyquinoline (HL) have been obtained. 1 and 2 have similar 'V'-type trinuclear structures whereas complex 3 displays dinuclear structure. The effects of central metals and reaction solvents on the structures of 1–3 have been discussed.