Synthesis, characterization, and fluorescent properties of two Pb(II) complexes: $\{[Pb(hca)_2 \cdot DMF] \cdot DMF\}_\infty$ and $[Pb(hca)_2(phen) \cdot DMF]_2$

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Abstract

Two novel Pb(II) complexes, $\{[Pb(hca)_2 \cdot DMF] \cdot DMF\}_\infty$ and $[Pb(hca)_2(phen) \cdot DMF]_2$ (hca = *trans*-4-hydroxycinnamic group), were obtained by solid-phase reactions of $PbAc_2$ and Hhca and $PbAc_2$, Hhca, and phen, respectively, and characterized by spectroscopy. X-ray crystallography analysis reveals that complex **1**, $\{[Pb(hca)_2 \cdot DMF] \cdot DMF\}_\infty$, adopts a 2-dimensional structure through the weak interactions of Pb and O atoms and that complex **2**, $[Pb(hca)_2(phen) \cdot DMF]_2$, shows a discrete dimeric structure, in which hydrogen bonds link the dimers into a 2D network. Both complexes **1** and **2** show visible fluorescence and the intensity is stronger than that of the ligand. More interestingly, the intensity of emission was increased at least fivefolds when the pH of the solution was adjusted to alkalinity. This can be attributed to that the deprotonization of phenolic group enhancing the conjugation of the ligand hca. These results indicate that this method may be an effective way to increase the emission intensity of similar complexes.
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1. Introduction

Metal–organic frameworks (MOFs) built from aromatic carboxylate building blocks have been attracted considerable attention because of their fascinating topologies, architectures, and potential applications as functional materials [1–5]. Many efforts have been devoted to the selection or design of suitable carboxylate-containing ligands with certain features, such as flexibility, versatile binding modes, and the ability to form hydrogen bonds [6–10]. Although dozens of carboxylate-containing ligands and hundreds of their coordination polymers have been intensively studied, constructing interesting architectures with various applications utilizing aromatic carboxylate-containing ligands is still very popular. Recently, *trans*-4-

hydroxylcinnamonic acid (Hhca), another aromatic acid, has been used as a potential carboxylate-containing ligand to synthesize coordination polymers. In previous researches [11–14], the structure of this aromatic acid has been reported in detail and two important features have been shown: (i) Three coordination sites (carboxylate group and hydroxyl group) may result in various coordination architectures such as 1D and 2D networks. The hydroxyl group can also easily form hydrogen bonds to stabilize the framework. (ii) It is well known that the derivates of cinnamic esters are potential luminescent materials because of their good π-conjugation [15]. Thus, we speculate that the similar π-conjugated structure of the ligand Hhca may introduce interesting optical properties into the metal complex. In this study, we synthesized and characterized two compounds utilizing the Hhca ligand as a block builder, a polynuclear complex **1**, $\{[Pb(hca)_2 \cdot DMF] \cdot DMF\}_\infty$ and its dinuclear derivate **2**, $[Pb(hca)_2(phen) \cdot DMF]_2$, by solid-phase reaction. Both of these complexes exhibit weak fluorescence in DMF solution, and **1** and **2** show much higher emission intensity

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than the ligand. However, it was found that the addition of NaOH to the DMF solution has a significant effect on the emission of all compounds. Here we report these results.

2. Experimental section

2.1. General methods

Reagents were from commercial sources and used as received without further purification, and the solvents were also commercially available and further purified before use. Elemental analyses of C, H, and N were performed on an Elemental Analysis Service of EA1110-CHNS elemental analyzer. Infrared spectra were measured with a KBr disk on a MagNa-550 FT-IR system. Emission and excitation spectra of complexes were determined in DMF solution using an Edinburgh-920 fluorescence spectrum photometer at room temperature. Thermal gravimetric (TG) analysis was carried out on a Universal V3.7A TA instrument in flowing N_2 with a heating rate of $10\,^\circ C/min$.

2.2. Synthesis of complex [Pb(hca)$_2$(DMF)] · DMF (1)

A mixture of $PbAc_2 \cdot 3H_2O$ (0.2 mmol, 76.0 mg) and 4-hydroxylcinnamonic acid (0.4 mmol, 65.7 mg) was ground in an agate mortar for half an hour with an obvious smell of acetic acid. The mixture was dissolved in DMF solution and filtered. Colorless prisms were obtained often a few days by evaporation of the filtrate at room temperature. Yield: 85% (116.9 mg). *Anal.* Calc. for crystals: $C_{24}H_{28}N_2O_8Pb$ (**1**): C, 42.41; H, 4.15; N, 4.12%. Found for crystals (**1**): C, 42.19; H, 4.22; N, 4.31%. IR (1, crystals): 1652(s), 1606(s), 1506(s), 1447(m), 1384(m), 1279(w), 1167(m), 1094(m), 980(m), 828(w), 724(w), 661(w), 634(w) cm^{-1}.

2.3. Synthesis of complex [Pb(hca)$_2$(phen)(DMF)] (2)

Complex **1** was further reacted with phen (0.2 mmol, 36.0 mg) in solid state at room temperature. The obtained mixture was dissolved in DMF solution and filtered. The colorless blocks (**2**) were obtained by slowly evaporating the filtrate at room temperature. Yield: 90% (144.5 mg). *Anal.* Calc. for crystals: $C_{33}H_{29}N_3O_8Pb$ (**2**): C, 49.37; H, 3.64; N, 5.23%. Found for crystals: C, 49.54; H, 3.32; N, 5.59%. IR (crystals, **2**, KBr disk): 1648(s), 1601(s), 1549(s), 1493(m), 1449(m), 1385(w), 1222(m), 1068(m), 1027(m), 906(w), 756(m), 698(s) cm^{-1}.

2.4. X-ray diffraction crystallography

All measurements were made on a Rigaku mercury CCD X-ray diffractometer (3 kV, sealed tube) at $-80\,^\circ C$ using graphite monochromated Mo $K\alpha$ ($\lambda = 0.71070\,\text{Å}$). A colorless block of **1** with dimensions $0.36 \times 0.20 \times 0.13$ mm or a colorless block of **2** with dimensions $0.35 \times 0.31 \times 0.11$ mm was mounted at the top of a glass fiber. Diffraction data

were collected in ω mode in the ranges $3.08^\circ < \theta < 25.35^\circ$ for **1** and $3.75^\circ < \theta < 25.48^\circ$ for 2. The collected data were reduced using the program CrystalClear (Rigaku and MSC, Version 1.3, 2001), and a semiempirical absorption correction was applied, which resulted in transmission factors ranging from 0.1951 to 0.4743 for **1** and from 0.2533 to 0.5877 for **2**. The reflection data were also corrected for Lorentz and polarization effects.

The structure was solved by the SHELXS-97 program and refined using full-matrix least-squares on F^2 with the SHELXL-97 program [16]. For **1**, all nonhydrogen atoms were refined anisotropically. All hydrogen atoms were introduced at the calculated positions and included in the structure-factor calculations. For **2**, one 4-hydroxylcinnamonic group was twofold disordered and refined isotropically without placing hydrogen atoms, while other non-hydrogen atoms were refined anisotropically and hydrogen atoms were introduced at the calculated positions. All calculations were performed on a Dell workstation using the SHELXS software package. A summary of the key crystallographic information for **1** and **2** is given in Tables 1–3 (CCDC numbers: 291154 and 291155). The selected band lengths and band angles for **1** and **2** are given in Tables 2 and 3, respectively.

3. Results and discussion

3.1. Synthesis aspects

Grinding of ligand Hhca and $PbAc_2 \cdot 3H_2O$ can release an obvious smell of acetic acid. The mixture was dissolved in DMF solution and **1** was obtained in high yield. A similar procedure was applied to the synthesis of [Pb(hca)$_2$(phen)] by the reaction of $PbAc_2 \cdot 3H_2O$, Hhca, and phen as shown in Scheme 1, and **2** was obtained from DMF solution compounds. **1** and **2** are air-stable, only soluble in DMF and DMSO. The obtained crystals were characterized by IR spectra and elemental analysis. According to the IR spectra, the absence of characteristic bands at 1730–1690 cm^{-1} for the carboxylic acid indicates the complete deprotonation of hca in the reaction.

3.2. Crystal structure of 1

Crystal analysis reveals that **1** adopts a two-dimensional structure through the weak interaction of Pb and O atoms. The asymmetric unit of compound **1** consists of a crystallographically independent lead(II) motif [Pb(hca)$_2$(DMF)] and a free DMF molecule. For **1**, the lone pair of electrons on the lead atom causes the coordination geometry to be distorted and hemidirected [17–19]. The structural environment of the lead atom is shown in Fig. 1, with the lone pair directed approximately opposite to the short Pb–O (chelating) distances. Each lead atom is coordinated by two chelating hca ligands, a DMF molecule forming a [Pb(hca)$_2$(DMF)] unit. Each O atom of one of the hca (O(**1**) and O(**2**)) ligands is further coordinated to different

Table 1
Crystal data for 1···2DMF and 2·DMF

| Formula	$C_{48}H_{56}N_4O_{16}Pb_2$	$C_{66}H_{58}N_6O_{14}Pb_2$
Formula weight	1359.35	1573.56
Temperature	193(2) K	193(2) K
Wavelength	0.71070 Å	0.71070 Å
Crystal system	Monoclinic	Triclinic
Space group	$P2_1/n$	P–1bar
Unit cell dimensions	$a = 16.3427(14)$ Å	11.651(3) Å
	$b = 7.4169(5)$ Å	11.682(3) Å
	$c = 22.2080(19)$ Å	12.913(4) Å
	$\beta = 110.541(2)°$	$\alpha = 113.544(6)°$
		$\beta = 100.710(5)°$
		$\gamma = 91.011(4)°$
Volume	2520.7(3) Å^3	1574.9(7) Å^3
Z	2	1
Calculated density	1.791 g/cm^3	1.659 g/cm^3
Absorption coefficient	6.743 mm^{-1}	5.408 mm^{-1}
$F(0\,0\,0)$	1328	772
Crystal size	$0.36 \times 0.20 \times 0.13$	$0.35 \times 0.31 \times 0.11$
θ for data collection	3.05–27.48°	3.08–25.35°
Reflections collected/unique	27,116/5783 [R(int) = 0.0347] 15,450/5700 [R(int) = 0.0542]	
Parameters	321	389
Goodness–of–fit on F^2	1.151	1.119
Final R indices [$I > 2\sigma\,(I)$]	$R1 = 0.0287$, w$R2 = 0.0486$	$R1 = 0.0840$, w$R2 = 0.2355$
R indices (all data)	$R1 = 0.0413$, w$R2 = 0.0522$	$R1 = 0.0932$, w$R2 = 0.2408$
Largest diff. peak and hole	1.498 and -1.296 e Å^{-3}	6.271 and -3.407 e Å^{-3}

Table 2
Selected bond lengths (Å) and angles(°) of 1·2DMF

Pb(1)–O(4)	2.404(2)	Pb(1)–O(1)	2.524(2)
Pb(1)–O(2)	2.567(2)	Pb(1)–O(5)	2.583(2)
Pb(1)–O(1′)	2.620(2)	Pb(1)–O(7)	2.774(3)
Pb(1)–O(2′)	2.785		
O(4)–Pb(1)–O(1)	76.25(8)	O(4)–Pb(1)–O(2)	78.95(8)
O(1)–Pb(1)–O(2)	51.30(7)	O(4)–Pb(1)–O(5)	52.24(8)
O(1)–Pb(1)–O(5)	111.00(8)	O(2)–Pb(1)–O(5)	74.19(8)
O(4)–Pb(1)–O(1′)	75.11(8)	O(1)–Pb(1)–O(1′)	66.41(8)
O(2)–Pb(1)–O(1′)	116.40(7)	O(5)–Pb(1)–O(1′)	124.14(8)
O(4)–Pb(1)–O(7)	83.45(9)	O(1)–Pb(1)–O(7)	143.68(8)
O(2)–Pb(1)–O(7)	151.76(8)	O(5)–Pb(1)–O(7)	77.57(8)
O(1′)–Pb(1)–O(7)	79.49(8)		
O(3)–H(3)·O(4′)	2.618(3)		
O(6)–H(6)·O(8′)	2.629(4)		

Table 3
Selected bond lengths (Å) and angles(°) of 2·DMF

Pb(1)–O(4B)	2.38(3)	Pb(1)–O(4A)	2.465(19)
Pb(1)–N(1)	2.576(16)	Pb(1)–O(1)	2.578(11)
Pb(1)–N(2)	2.594(14)	Pb(1)–O(2)	2.697(13)
Pb(1)–O(5B)	2.75(4)	Pb(1)–O(5A)	2.753(18)
Pb(1)–O(7)	2.893(16)	Pb(1)–O(1′)	2.830
O(4B)–Pb(1)–N(1)	80.7(8)	O(4A)–Pb(1)–N(1)	84.9(6)
O(4B)–Pb(1)–O(1)	79.6(7)	O(4A)–Pb(1)–O(1)	82.9(5)
N(1)–Pb(1)–O(1)	142.2(5)	O(4B)–Pb(1)–N(2)	66.2(8)
O(4A)–Pb(1)–N(2)	78.8(6)	N(1)–Pb(1)–N(2)	63.9(5)
O(1)–Pb(1)–N(2)	78.6(4)	O(4B)–Pb(1)–O(2)	84.4(8)
O(4A)–Pb(1)–O(2)	77.2(5)	N(1)–Pb(1)–O(2)	157.4(5)
O(1)–Pb(1)–O(2)	49.2(3)	N(2)–Pb(1)–O(2)	124.4(4)
O(4B)–Pb(1)–O(5B)	50.0(8)	O(4A)–Pb(1)–O(5B)	39.3(7)
N(1)–Pb(1)–O(5B)	75.9(9)	O(1)–Pb(1)–O(5B)	113.6(8)
N(2)–Pb(1)–O(5B)	108.3(9)	O(2)–Pb(1)–O(5B)	81.6(9)
O(4B)–Pb(1)–O(5A)	61.6(8)	O(4A)–Pb(1)–O(5A)	49.8(6)
N(1)–Pb(1)–O(5A)	83.8(9)	O(1)–Pb(1)–O(5A)	113.9(7)
N(2)–Pb(1)–O(5A)	121.8(7)	O(2)–Pb(1)–O(5A)	74.2(8)
O(5B)–Pb(1)–O(5A)	13.6(9)	O(4B)–Pb(1)–O(7)	140.8(8)
O(4A)–Pb(1)–O(7)	131.6(6)	N(1)–Pb(1)–O(7)	79.4(6)
O(1)–Pb(1)–O(7)	133.6(5)	N(2)–Pb(1)–O(7)	130.6(5)
O(2)–Pb(1)–O(7)	102.3(5)	O(5B)–Pb(1)–O(7)	92.4(8)
O(5A)–Pb(1)–O(7)	83.0(7)		
O(3)·O(4A′)	2.81(2)		
O(3)·O(4B′)	2.47(3)		

adjacent lead atoms to form a polymeric Pb_2O_2 backbone. Thus Pb atom is seven-coordinated. Such a polymeric chain is similar to that found in the compounds lead(II) crotonate [20] and [Pb(Phen)(O_2CCH_3)](ClO$_4$) [21], in which only one hca anion (carboxylic group) is involved in the construction of the infinite chain and the other acts as a chelating counteranion. Although the infinite double-stranded ladder is usual in carboxylic Pb(II) compounds, there are still some differences in **1**. For example, due to the stereochemistry of the 4-hydroxylcinnamonic group, the bond lengths of Pb–O (2.404−2.785 Å) are generally larger than those in the compound lead(II) crotonate (2.350−2.635 Å) and the O(1′)-Pb(1)-O(2″) bond angle is 160.63°, obviously sharper than 168.0(2)° in the compound
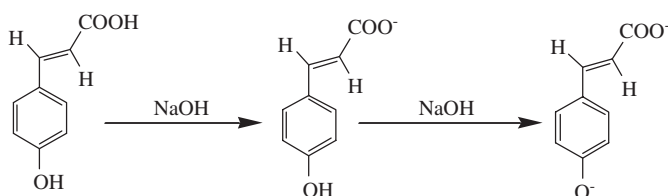
lead(II) crotonate. Furthermore, there is an obvious interaction (3.03 Å) between the Pb atom and the O(**3**) atom of the hydroxyl group in the hca anion from the adjacent ladder and the interaction makes the ladders extend to an infinite 2D network (Fig. 2). In addition, the other hydroxyl group from the chelating hca anion
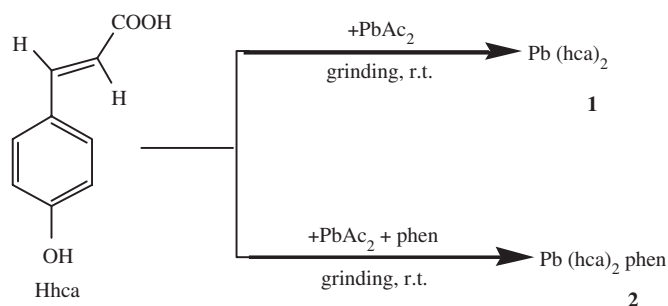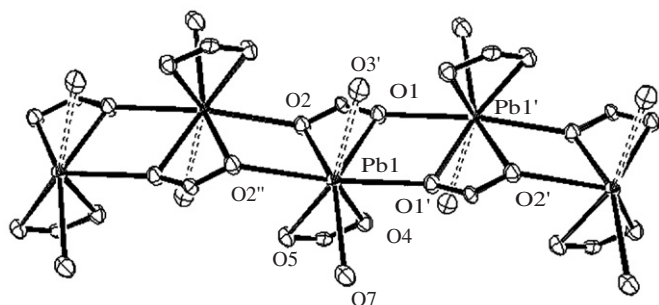
Scheme 1.

Scheme 2.

Fig. 1. Coordination backbone of complex **1** (hydrogen atoms and ligand groups and DMF molecule were omitted, except the oxygen atoms, for clarity).
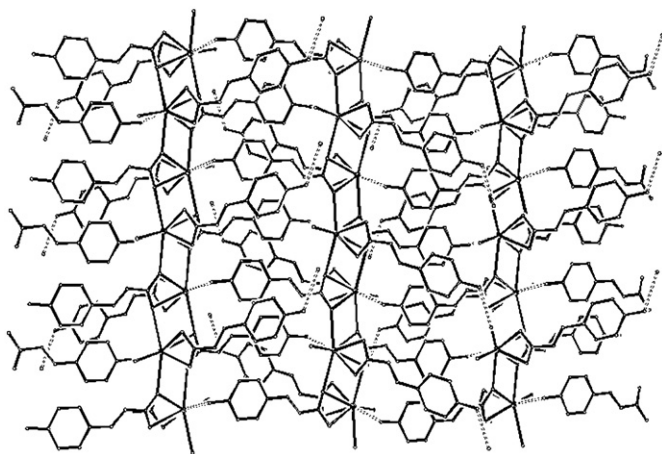

Fig. 2. View of a 2D network of complex **1** constructed by hydrogen bonds (hydrogen atoms were omitted; only oxygen atoms of the DMF molecule were preserved, for clarity).

interacts with the O(8) atom of the free DMF molecule via hydrogen bonds.

3.3. Crystal structure of 2

The addition of a strong donor ligand, phen, may snip the polymeric Pb–O chain. Crystal structure analysis verifies that the 1D backbone in **1** was cut off and that the new compound **2** shows a dimeric structure. The asymmetric unit of compound **2** consists of a crystallographically independent lead(II) motif [Pb(hca)$_2$ (phen)(DMF)] in which one of the hca anions is twofold disordered. Each lead atom is eight-coordinated with an N$_2$O$_6$ donor atom array: two N atoms from a phen molecule, four O atoms from two chelating hca ligands with Pb–O distances ranging from 2.38(3) to 2.75(2) Å, one μ_3-O atom from the adjacent [Pb(hca)$_2$(phen)(DMF)] unit, and one O atom from the DMF molecule, which has a bond distance (2.893(16) Å) longer than that in **1** (2.774(8) Å). Unlike the polymeric backbone of compound **1** and [Pb(phen)(O$_2$CCH$_3$)](ClO$_4$) [18], two [Pb(hca)$_2$ (phen)(DMF)] units in **2** interlink each other via a Pb$_2$O(1)$_2$ planar ring to form a dimer. The existence of the phen molecule hinders further connection with adjacent units. In addition, the hydrogen bond in this structure should be mentioned. The uncoordinated phenolic group has a strong interaction with one of the oxygen atoms from the coordinated carboxylate group via hydrogen bond. The dimers of [Pb(hca)$_2$(phen)(DMF)]$_2$ are thus connected to form a 2D hydrogen-bond network. See Figs 3 and 4.

3.4. Fluorescent properties

We obtained the fluorescent spectra of the compounds in DMF solution because they could only be dissolved in DMF and DMSO. Figs. 5 and 6 shows the emission spectra of **1**, **2** and the free ligand in DMF solution measured at room temperature. The emission spectra of both compounds are obviously red-shifted and dominated by a broad band at $\lambda = 457$ nm for **1** and $\lambda = 461$ nm for **2** compared with that of the ligand ($\lambda = 434$ nm, $\lambda_{ex} = 360$ nm). Because PbAc$_2$ in DMF solution shows no emission under the same excitation wavelength according to previous research on luminescence of Pb(II) ion and its complexes [22–24], the emission band of **1** and **2** is ligand-centered ($p-\pi^*$) and the coordination of Pb and carboxylate in this case can change the fluorescent emission wavelength and the intensity is increased in some degree. On the other hand, the addition of the second ligand, phenanthroline, in **2** can increase the emission intensity slightly, but the effect is not obvious.

It can be seen from Fig. 5 that the free ligand Hhca in DMF emits a weak broad emission at 434 nm. However, the pattern of the emission band is sharpened ($\lambda_{ex} = 328$ nm) and the intensity is seven-fold increased after the pH value of the DMF solution of Hhca is adjusted alkalinity by NaOH. It is well known that the conjugation
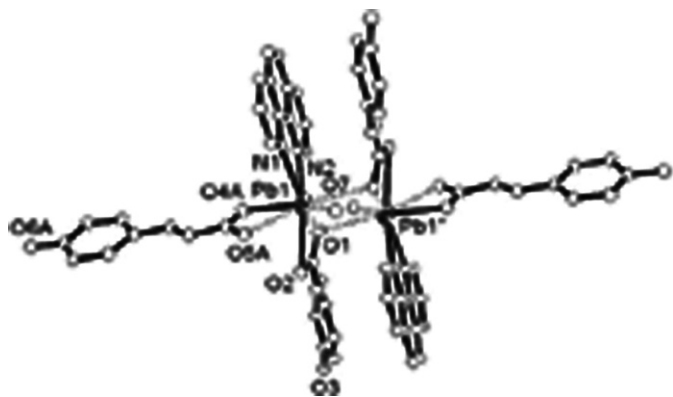
Fig. 3. The dimeric structure of complex **2** (hydrogen atoms were omitted, only oxygen atom of DMF molecule was preserved for clarity).
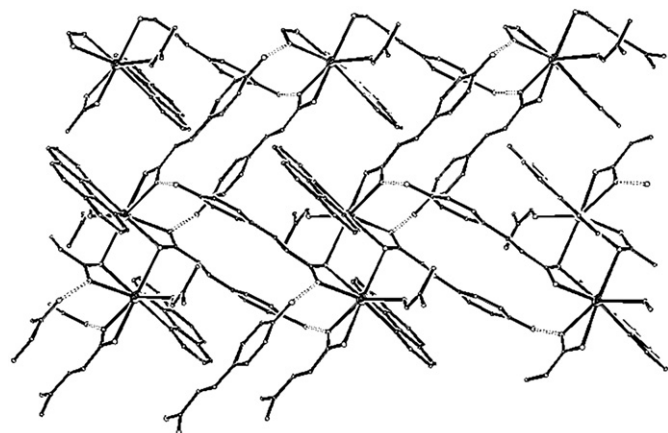

Fig. 4. View of a 2D network of complex **2** constructed by hydrogen bonds (hydrogen atoms were omitted; only oxygen atoms of the DMF molecule were preserved, for clarity).
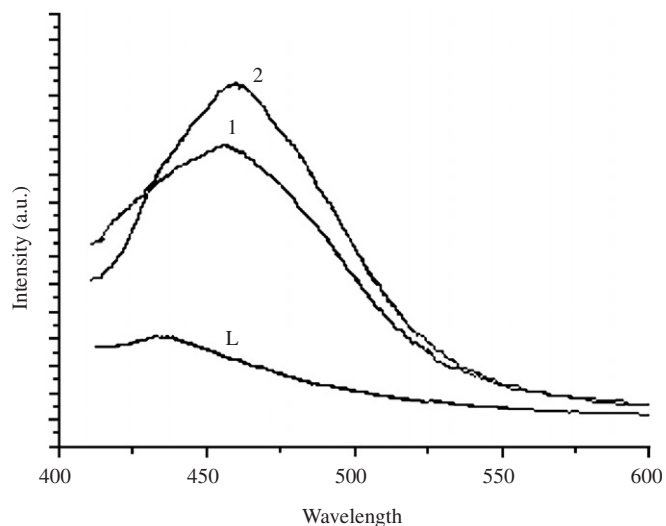

Fig. 5. Emission spectra of complexes **1** and **2** and the ligand in DMF solution (concentration: 10^{-4} mol dm^{-3}; λ_{ex}: 360 nm; λ_{em}, L: 434 nm; 1: 457 nm; 2: 461 nm).

($p-\pi^*$ and $\pi-\pi^*$) is strengthened by deprotonization (Scheme 2), which should cause on increase of the fluorescence. The $\pi-\pi$ conjugation exists because the
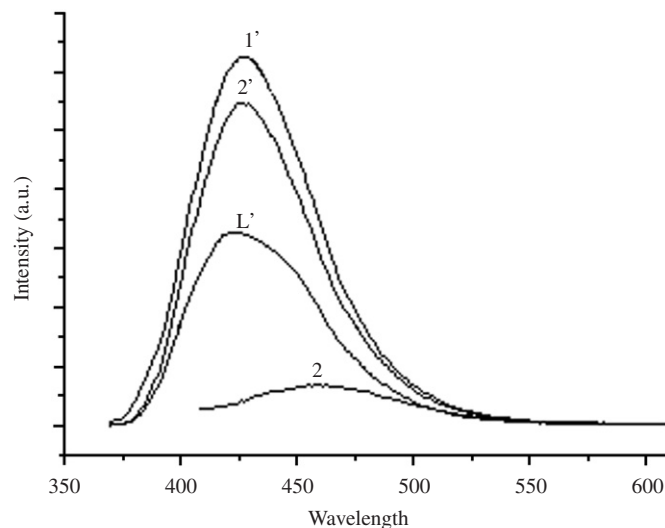

Fig. 6. Emission spectra of complexes **1** and **2** and the ligand in DMF solution (pH value at 8; concentration: 10^{-4} mol dm^{-3}; λ_{ex}: 360 nm; λ_{em}, L': 434 nm; 1': 428 nm; 2': 423 nm).

double bond in the *trans*-cinnamonic acid and the benzene ring is coplanar and the $p-\pi$ conjugation exists between the hydroxyl and the benzene ring. So the conjugation between the oxygen anion and arene ring can be strengthened by deprotonization of the hydroxyl group. The conjugation system exists not only in the ligand but also in the compound. So what will happen when the solution of the compounds is adjusted to alkalinity by NaOH? As shown in Fig. 6, the emission bands of **1** and **2** are blue-shifted to 428 and 423 nm, respectively, close to that of the ligand, and as expected, the emission intensity increases distinctly (almost fivefold). These results revealed that the deprotonization of the phenolic group may be an effective way to increase the emission intensity of similar complexes.

3.5. Thermal analysis

The thermal behavior of **1** and **2** was studied. The TGA curve of 1 shows that the first gradual weight loss is about 9.4%, corresponding to the loss of a free DMF molecule (calculated as 10.7%). The decomposition begins at 130 °C with the weight loss of the coordinated DMF molecule and hca, and the residual weight of 34.0% at 694 °C is PbO (calculated as 32.8%) and carbon. The thermogravimetric analysis (TGA) of **2** shows that the first step is ascribed to the gradual loss of the coordinated DMF, starting around 100 °C, and a corresponding weight loss of 10.7% (calculated as 9.1%). The decomposition of the compound begins at 207 °C, the main remnant is PbO at 700 °C, and the corresponding weight is 29.6% (calculated as 27.7%). See Figs 7 and 8.

4. Conclusion

Two novel Pb(II) complexes, $[Pb(hca)_2 \cdot 2DMF]_\infty$ and $[Pb(hca)_2(phen) \cdot DMF]_2$ (hca = *trans*-4-hydroxycinnamic
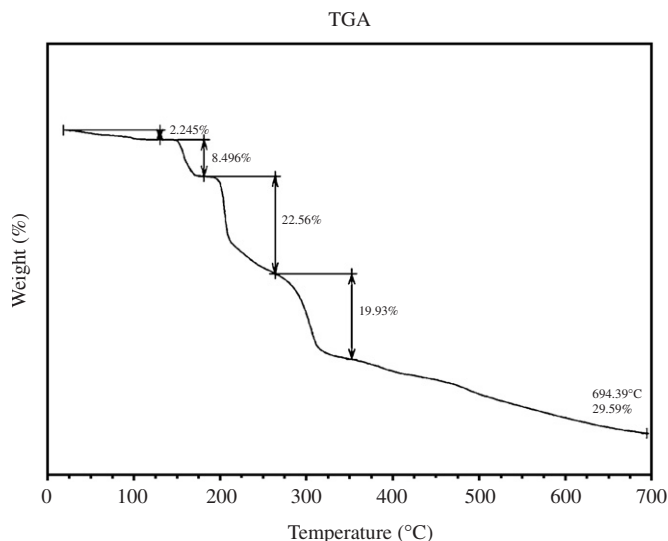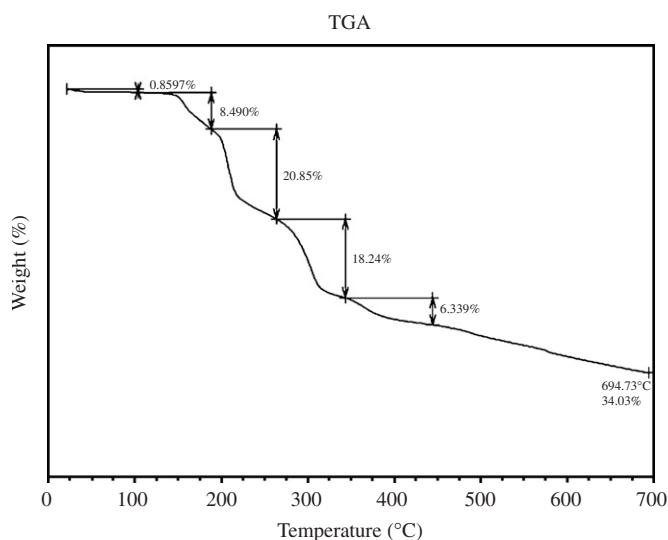
TGA

Fig. 7. TGA curves of crystal **1**.

TGA

Fig. 8. TGA curve of crystal **2**.

group), were obtained by solid-phase reaction and characterized by spectroscopy. X-ray crystallography analysis reveals that complex 1, $[Pb(hca)_2 \cdot 2DMF]_\infty$, adopts a 2-dimensional structure through the weak interactions of Pb and O atoms and that complex **2**, $[Pb(hca)_2 (phen) \cdot DMF]_2$, shows a dimeric structure. Both complexes show visible fluorescence which is stronger than that of the ligand, while the intensity of the emission is dramatically increased when the pH value of the solution is adjusted to alkalinity. The deprotonization of the phenolic group may be an effective way to increase the emission intensity of similar complexes. Whether the pH value influences the inner structure of the complexes is being investigated in our lab.

Appendix A. Supplementary data

Additional crystallographic data have been deposited at the Cambridge Crystallographic Data Centre, CCDC reference numbers 291154 (1), 291155(2). The data can be obtained free of charge from www.ccdc.cam.ac.uk/conts/retrieving.html(or from CCDC, 12 Union Road, Cambridge CB2 1EZ, UK; fax:+ 44 1223 336 033; e-mail: deposit@ccdc. cam.ac.uk.)

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