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Synthesis and spectroscopic behavior of highly luminescent trinuclear europium complexes with tris-β-diketone ligand

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ABSTRACT

A new tris-β-diketone ligand, 2-[4,6-bis-(1-benzoyl-2-oxo-2-phenyl-ethyl)-[1,3,5]triazin-2-yl]-1,3-diphenyl-propane-1,3-dione (**H_3L**), and its trinuclear europium complexes, $Eu_3(DBM)_6L$ (**C1**), $Eu_3(DBM)_6(Bipy)_3L$ (**C2**) and $Eu_3(DBM)_6(Phen)_3L$ (**C3**) were synthesized and their spectroscopic behaviors were studied by FT-IR, ^1H NMR, UV–vis and photoluminescence spectroscopic techniques. These europium complexes exhibited the characteristic emission bands that arise from the $^5D_0 \rightarrow {}^7F_J$ ($J = 0$–4) transitions of the europium ion in solid state. The Ω_2 and Ω_4 intensity parameters, lifetime (τ) and luminescence quantum yield (η) were calculated according to the emission spectra and luminescence decay curves in solid state. The results indicated that these trinuclear europium complexes displayed a longer lifetime (τ) and higher luminescence quantum efficiency (η), especially complexes **C2** ($\tau = 0.820$ ms, $\eta = 46.5\%$) and **C3** ($\tau = 0.804$ ms, $\eta = 47.4\%$), which due to the effect of two additional europium ion lumophors and the introduction of the third ligands, Bipy or Phen in trinuclear complexes. Their Ω_2 values demonstrated that the europium ion in these complexes is in a highly polarizable chemical environment.

1. Introduction

Since Tang and Van Slyke reported Organic electroluminescent diodes in 1987 [1], Organic light emitting diodes have been carrying out considerable research over the past three decades [2,3]. In particular, europium complexes have been attracted attention owing to their highly monochromatic red light and extremely sharp emission [4]. However, the quantum efficiency of most europium complexes is unfortunately still low because the Laporte-forbidden 4*f*–4*f* transition prevents direct excitation of europium luminescence [5]. Chemistry and materials scientists have realized that it is crucial to design and synthesize organic ligands, which have better energy transfer property to the europium ion.

To overcome the problem, organic ligands must have a high absorption coefficient, which can sensitize the europium ion better. Consequently, many europium complexes with the high absorption coefficient ligands, especially β-diketone ligands, have been designed and synthesized [6–8]. Recently, we also reported some new europium complexes with β-diketone ligands and investigated their photoluminescence properties [9,10].

Generally, one efficient method of improving the luminescent properties is the introduction of organic ligands with multiple binding sites in europium complexes [11]. Therefore, to further develop the luminescent properties of europium complexes, we designed and synthesized a new tris-β-diketone ligand, 2-[4, 6-bis-(1-benzoyl-2-oxo-2-phenyl–ethyl)-[1,3,5]triazin-2-yl]-1,3-diphenyl- propane-1,3-dione (**H_3L**). With this tris-β-diketone as the first ligand, dibenzoylmethane (DBM) as the second ligand and 2,2-dipyridine (Bipy) or 1,10-phenanthroline (Phen) as the third ligand, three new trinuclear europium complexes, $Eu_3(DBM)_6L$, $Eu_3(DBM)_6(Bipy)_3L$, $Eu_3(DBM)_6(Phen)_3L$ were synthesized and characterized. Their photoluminescence behaviors have also been studied by fluorescence spectroscopy. Furthermore, based on their photoluminescence spectra and luminescence decay curves, the Judd–Ofelt intensity parameters (Ω_λ), radiative (A_{rad}), nonradiative (A_{nrad}), and photoluminescence quantum yield (η) were calculated and analyzed.

2. Experimental

2.1. Materials and instrumentation

Dibenzoylmethane, 2,4,6-trichloro-1,3,5-triazine, potassium, europium oxide, 2,2-dipyridine and 1,10-phenanthroline were purchased from Shanghai Chemical Reagent Company Ltd. (Shanghai, China). Other reagents were used as received or were purified using standard procedures.

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Melting points were determined on X-4 digital melting-point apparatus and uncorrected. Infrared spectra were recorded on a Nicolet FTIR 5700 spectrophotometer in KBr pellets. ^1H NMR spectra were performed on an Avance III™ 300 MHz NB Digital NMR spectrometer using TMS as internal standard and $CDCl_3$ and DMSO-d_6 as the solvent. The UV–Vis spectra were measured on a Hitachi U-3010 spectrometer. Electrospray ionization mass spectroscopy (ESI-MS) was performed using a Finnigan LCQ Advantage Max spectrometer. Fluorescence spectra and lifetimes were recorded on a Varian Cary Eclipse fluorescence spectrometer. Elemental analysis (C, H, N) was measured using a Perkin–Elmer 2400 elemental analyzer. The percentage of europium (III) was determined by the complexometric titration with EDTA.

2.2. Synthesis of tris-β-diketone (**H_3L**)

2-[4,6-bis-(1-benzoyl-2-oxo-2-phenyl–ethyl)-[1,3,5]triazin-2-yl]-1,3-diphenyl-propane-1,3-dione (**H_3L**): The freshly cut metal potassium (1.95 g, 50 mmol) was added in part to 40 mL of tert-butanol with stirring. The suspension was heated to 80 °C until the potassium was completely exhausted. The excess tert-butanol was evaporated under reduced pressure and the white residue was dissolved in 20 mL of benzene. To this mixture, a solution of dibenzoylmethane (11.20 g, 50 mmol) in benzene (50 mL) was added and stirred at 60 °C for 4 h. Then 2,4,6-trichloro-1,3,5-triazine (1.85 g, 10 mmol) was added to the reaction mixture and kept to reflux for 8 h. The reaction was monitored by TLC using ethyl acetate/petroleum ether (1:3) as eluent. After cooling to the room temperature, the mixture was poured into 50 mL ice-water; then acidified with dilute hydrochloric acid and extracted with chloroform. The organic layer was washed with a saturated $NaHCO_3$ solution, dried over anhydrous $MgSO_4$. After solvent removal, the product was recrystallized from ethanol to obtain the tris-β-diketone (**H_3L**). Yellow crystals, yield 38%, Mp 110–112 °C; IR (KBr): ν 3058 (w), 2893 (w), 1638 (s), 1565 (s), 1502 (s), 1315 (s), 1246 (s), 1178 (m), 1091 (m), 965 (m), 878 (m), 748(s), 695(s) cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 1.69 (s, 3H, CH), 6.58–7.86 (m, 30H) ppm; ESI–MS: m/z 748.43 $[M+1]^+$; Anal. Calcd. for $C_{48}H_{33}N_3O_6$: C, 77.10; H, 4.45; N, 5.62; Found C, 77.42; H, 4.43; N, 5.65.

2.3. Synthesis of trinuclear europium complexes

$Eu_3(DBM)_6L$ (**C1**): The tris-β-diketone (**H_3L**) (0.75 g, 1 mmol), DBM (1.35 g, 6 mmol) and sodium ethoxide (0.62 g, 9 mmol) were dissolved in 40 ml anhydrous ethanol and stirred at 60 °C for 30 min. To this solution, an ethanol solution containing 3 mmol $EuCl_3$, which was obtained by reaction of Eu_2O_3 and concentrated HCl, was added dropwise and reacted at this temperature for 12 h. The reaction solution was cooled to the room temperature and a yellow solid was precipitated. The precipitate was separated by suction filtration, purified by washing for several times with ethanol and deionized water to give the trinuclear europium complex **C1**. Yield: 68%. IR ν (KBr): 3073(m), 1601(s), 1554(s), 1520(s), 1499(s), 1309(s), 1289(s), 1239(s), 1219(s), 1184(m), 1032(m), 865(m), 752(s), 696(s), 468(m) cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6): δ 4.58 (s, 6H, C=CH), 6.21–7.93 (br, m, 90H) ppm; Anal. Calcd. for $Eu_3C_{138}H_{96}N_3O_{18}$: C, 65.25; H, 3.81; N, 1.65; Eu, 17.95; Found C, 65.61; H, 3.75; N, 1.68; Eu, 17.88.

$Eu_3(DBM)_6(Bipy)_3L$ (**C2**): Same procedure as for $Eu_3(DBM)_6L$ (**C1**), but the reaction mixture with **H_3L** (0.75 g, 1 mmol), DBM (1.35 g, 6 mmol), sodium ethoxide (0.62 g, 9 mmol), Bipy (0.47 g, 3 mmol) and 3 mmol $EuCl_3$. The trinuclear europium complex **C2** was obtained as a yellow power, yield 63%. IR ν (KBr): 3067(m), 1598(s), 1552(s), 1522(s), 1496(s), 1309(s), 1296(s), 1243(s), 1218(s), 1190(m), 1050(m), 882(m), 748(s), 699(s), 512(m), 471(m) cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6): δ 4.66 (s, 6H, C=CH), 6.35–8.23 (br, m, 90H), 8.35 (br, 6H, Bipy–H), 9.63 (br, 6H, Bipy–H), 10.48 (d, 6H, Bipy–H, J = 7.2 Hz), 12.06 (d, 6H, Bipy–H, J = 7.8 Hz) ppm; Anal. Calcd. for $Eu_3C_{168}H_{120}N_9O_{18}$: C, 67.07; H, 4.02; N, 4.19; Eu, 15.15; Found C, 67.36; H, 3.99; N, 4.23; Eu, 15.25.

$Eu_3(DBM)_6(Phen)_3L$ (**C3**): Same procedure as for $Eu_3(DBM)_6L$ (**C1**), but the reaction mixture with **H_3L** (0.75 g, 1 mmol), DBM (1.35 g, 6 mmol), sodium ethoxide (0.62 g, 9 mmol), Phen (0.55 g, 3 mmol) and 1 mmol $EuCl_3$. The trinuclear europium complex **C3** was obtained as a yellow power, yield 66%. IR ν (KBr): 3071(m), 1597(s), 1549(s), 1523(s), 1497(s), 1316(s), 1293(s), 1245(s), 1227(s), 1209(s), 1195(m), 1045(m), 875(s), 798(m), 757(s), 701(s), 510(m), 472(m) cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6): δ 4.73 (s, 6H, C=CH), 6.41–8.42 (br, m, 90H), 9.05 (br, 6H, Phen–H), 10.33 (br, 6H, Phen–H), 10.75 (br, 6H, Phen–H), 11.02 (br, 6H, Phen–H) ppm; Anal. Calcd. for $Eu_3C_{174}H_{120}N_9O_{18}$: C, 67.84; H, 3.93; N,4.09; Eu, 14.80; Found C, 67.58; H, 3.98; N, 4.13; Eu, 14.77

3. Results and discussion

3.1. Synthesis

The synthetic procedures of the tris-β-diketone (**H_3L**) and trinuclear europium complexes are summarized in Scheme 1. The tris-β-diketone was synthesized by nucleophilic substitution between dibenzoylmethane and 2,4,6-trichloro-1,3,5-triazine by refluxing in benzene in the presence of potassium tert-butanolate. The trinuclear europium complexes **C1**–**C3** were prepared by enolization of the tris-β-diketone and dibenzoylmethane ligands in the presence of sodium ethoxide at first, and then complexation using Bipy or Phen as the third ligand with europium chloride in ethanol solution according to the method of Ref. [11].

3.2. Spectroscopic characterization

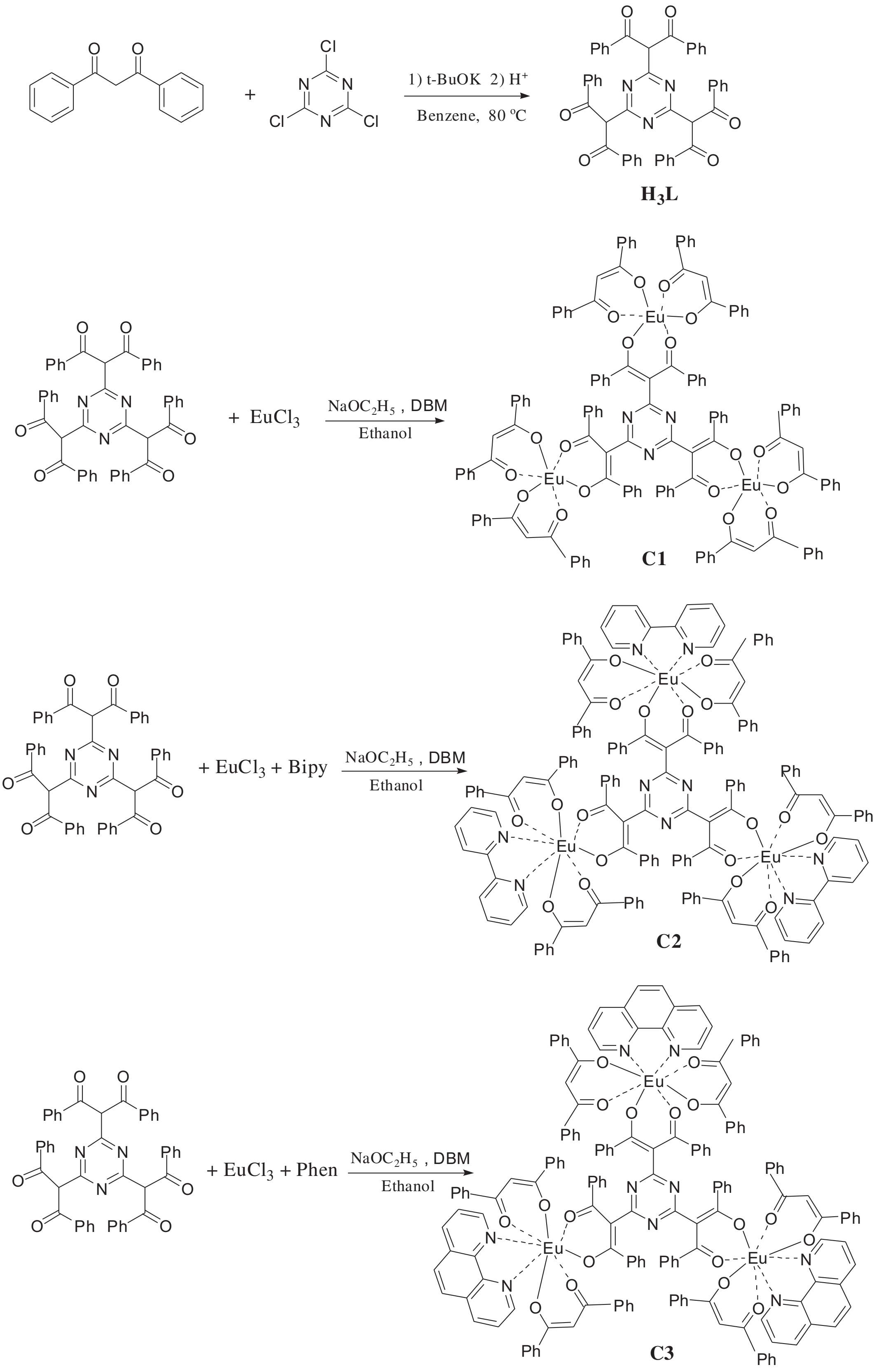
In the IR spectra, trinuclear europium complexes exhibited noticeable changes in comparison with those of the tris-β-diketone ligand. Their characteristic absorption peaks were summarized in Table 1. Firstly, the IR spectra of the tris-β-diketone showed a weak peak at 2893 cm^{-1} due to the saturated C—H stretching vibration, but which is nonexistent in the complexes. Secondly, In complexes, the strong C=O stretching vibrations in the regions of 1601–1597 cm^{-1} were red-shifted 37–41 cm^{-1} with respect to those of the tris-β-diketone, and new absorption peaks were observed at the region of 1523–1520 cm^{-1}, which ascribed to the enolic C=C stretching vibrations of trinuclear europium complexes [12,13]. Thirdly, the medium absorptions of complexes in the region of 510–512 cm^{-1} and 468–472 cm^{-1}, which attributed to the Eu–N and Eu–O stretching vibrations [14,15], respectively. These results indicated that the formation of trinuclear europium complexes **C1**–**C3**.

In the ^1H NMR spectra, the keto-CH proton of the tris-β-diketone exhibited a singlet of at δ 1.69 ppm and the enolic proton signal was not observed, which indicated that the tris-β-diketone ligand existed only one single tautomer as the keto form. But in complexes **C1**–**C3**, the ^1H NMR spectra did not show the presence of the keto-CH proton signals, which attributed to the enolization of the tris-β-diketone in its complexes. The results also confirmed that the formation of these trinuclear europium complexes. In addition, the proton signals of chelated Bipy or Phen were found to shift notably to lower fields in comparison with those of the free Bipy or Phen, which due to the electron-withdrawing inductive effect of complexation [16].

Fig. 1 shows the UV–vis absorption spectra for the β-diketone ligands and europium complexes in chloroform solution (1×10^{-5} mol/L). The λ_{max} of the europium complex **C1** is 363 nm, which is red-shifted 21 and 45 nm, respectively, compared with those of the DBM ligand (λ_{max} = 342 nm) and tris-β-diketone ligand (λ_{max} = 318 nm). However, the λ_{max} values for europium complexes **C2** and **C3** are 374 and 377 nm, respectively, which caused a large red shift in comparison with those of their β-diketone ligands. This is because the introduction of the third ligand Bipy or Phen resulted in the bigger conjugated system in complexes **C2** and **C3**. The results further indicated that the formation of trinuclear europium complexes **C1**–**C3**.

3.3. Photoluminescence properties

The photoluminescence spectra of trinuclear europium complexes **C1**–**C3** in the solid state at room temperature are shown in Fig. 2. The principal features appearing in the emission spectra were the presence of the narrow bands in the 560–720 nm region that were attributed to the $^5D_0 \rightarrow {}^7F_J$ transitions (J = 0–4) of the europium (III) ion. The five expected Stark components were well resolved and the hypersensitive $^5D_0 \rightarrow {}^7F_2$ transition was very strong. The results indicated a highly polarizable chemical environment around the europium (III) ion, and were responsible for the brilliant-red emission color of these complexes. The presence of only one peak corresponding to $^5D_0 \rightarrow {}^7F_0$ transition around 580 nm suggested the existence of one local site of symmetry for the europium complexes [17]. Additionally, the emission intensity



Scheme 1. The synthetic routes of tris-β-diketone and europium complexes.

of complexes **C2** and **C3** are much stronger than that of complex **C1** due to the coordination of the "antenna" ligands to the europium ion, which brings about an efficient energy transfer from the ligands to central the Eu^{3+} ion [18]. This is because that the third ligands, Bipy and Phen, have high affinity toward various lanthanide ions, especially toward Eu^{3+} ion. The asymmetry of the com-

Table 1
Comparison of the characteristic IR data (cm^{-1}) of the tris-β-diketone ligand and trinuclear europium complexes.

Compound	ν(C—H)	ν(C=O)	ν(C=C)	ν(Eu—N)	ν(Eu—O)
H_3L	2893 (w)	1638 (s)	–	–	–
C1	–	1601 (s)	1520 (s)	–	468 (m)
C2	–	1598 (s)	1522 (s)	512 (m)	471 (m)
C3	–	1597 (s)	1523 (s)	510 (m)	472 (m)

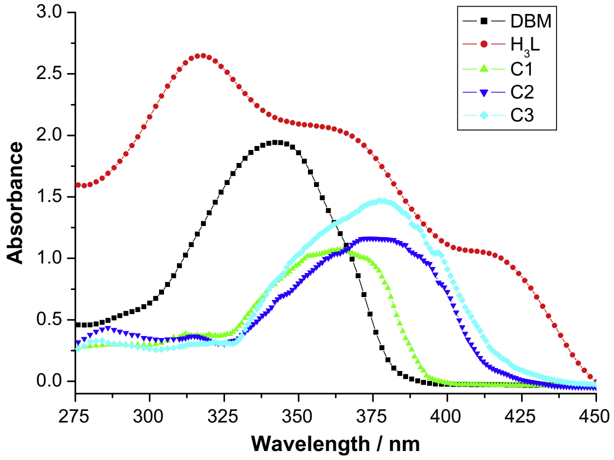


Fig. 1. UV–vis spectra of ligands and trinuclear europium complexes in chloroform solution (1×10^{-5} mol/L).

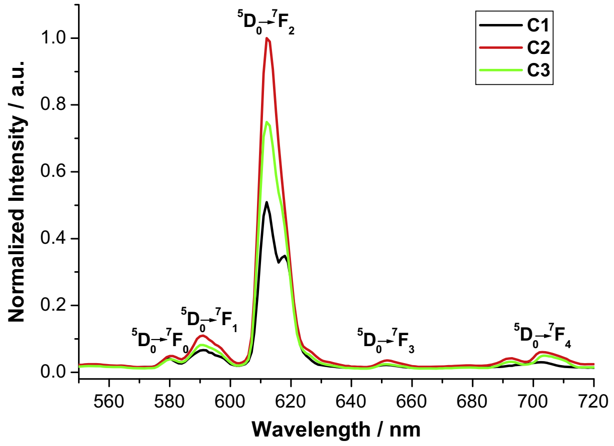


Fig. 2. Room-temperature emission spectra for complexes **C1**, **C2** and **C3** in solid state, excited at 397, 401 and 402 nm, respectively.

plexes **C2** and **C3** is increased and prevented the 4*f*–4*f* transition ascribed to the ligands, Bipy and Phen, participating in the coordination. This is much helpful to the emission intensity of europium complexes.

Fig. 3 presents the luminescence decay curves of $^5D_0$ level for complexes **C1**–**C3** by monitoring the emission at 612 nm at room temperature. The lifetime values (τ) (Table 2) for the emission of these complexes were obtained from fittings of the decay curves. It was found that the decay curves for these europium complexes exhibited a mono-exponential behavior, which was in agreement with one chemical environment around where the Eu^{3+} ion existed [19]. The much longer lifetime values had been observed for complexes **C2** (τ = 0.820 ms) and **C3** (τ = 0.804 ms) as compared to the complex **C1** (τ = 0.279 ms). This may be due to the introduction of the third ligands, Bipy or Phen, resulting in the photoluminescence stability of the trinuclear europium complex.

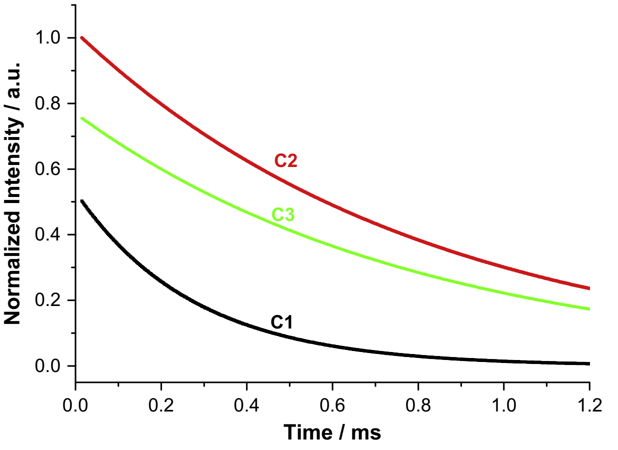


Fig. 3. Luminescence decay curves for complexes **C1**–**C3** in the powder, at room temperature, monitored at 612 nm.

The lifetime, non-radiative (A_{nrad}) and radiative (A_{rad}) rates are related through the equation $A_{tot} = 1/\tau = A_{rad} + A_{nrad}$. The radiative ($A_{rad}$) rates were obtained by summing over the radiative rates A_{0J} for each $^5D_0 \rightarrow {}^7F_J$ transition ($\sum_J A_{0J}$) of the europium ion. And A_{0J} is obtained according to the following equation [20]:

$$A_{0J} = A_{01}\frac{I_{0J}}{I_{01}}\frac{\nu_{01}}{\nu_{0J}} \tag{1}$$

where I_{0J} is the area under the emission curve related to the $^5D_0 \rightarrow {}^7F_J$ transitions, ν_{0J} is energy barycenter of transition. The $^5D_0 \rightarrow {}^7F_1$ transition does not depend on the local ligand field seen by europium ion and, thus, may be used as a reference, the value of A_{01} is estimated to be $\sim$50 s^{-1} [5].

According to the emission spectra and lifetimes of complexes **C1**–**C3**, the luminescent quantum efficiency (η) for the emitting $^5D_0$ level can be obtained from the equation [21]:

$$\eta = \frac{A_{rad}}{A_{rad} + A_{nrad}} \tag{2}$$

The calculated radiative decay rates A_{rad}, the non-radiative decay rates A_{nrad} and the quantum efficiency η are shown in Table 2. The results indicated these trinuclear europium complexes presented the higher luminescence quantum efficiency, especially complexes **C2** (η = 46.5%) and **C3** (η = 47.4%), which was in agreement with the results from their luminescence emission intensity.

From these results, the trinuclear europium complexes **C2** and **C3** have much longer lifetime values and much higher quantum efficiency as compared to their corresponding dibenzoylmethane (DBM) mononuclear europium complexes, such as $Eu(DBM)_3$·Phen (τ = 0.503 ms, η = 31.3%) [22], Eu(4-methoxy-$DBM)_3$·Bipy (τ = 0.25 ms, η = 25.2%) [10], Eu(alkoxy-$DBM)_3$·Bipy (τ = 0.24 ms, η = 20–23%) [23], which due to the effect of two additional europium (III) ion lumophors in trinuclear complexes. Therefore, the introduction of ligands with multiple binding sites in europium complexes could effectively improve the luminescent properties of europium complexes.

3.4. Judd–Ofelt analysis

It is very important to obtain the Judd–Ofelt intensity parameters because it is possible to gain information about the chemical environment around the europium ion. The Judd–Ofelt intensity parameters Ω_t (t = 2, 4) can be calculated from the emission spectra (Fig. 2) using the $^5D_0 \rightarrow {}^7F_2$ and $^5D_0 \rightarrow {}^7F_4$ electronic transitions of the europium ion according to the following equation [4]:

Table 2
The photoluminescence data of europium complexes in solid state.

Complex	τ (ms)	A_{tot} (s^{-1})	A_{rad} (s^{-1})	A_{nrad} (s^{-1})	Ω_2 (10^{-20} cm^2)	Ω_4 (10^{-20} cm^2)	η (%)
C1	0.279	3584	536	3048	13.5	1.1	15.0
C2	0.820	1220	567	653	13.7	2.1	46.5
C3	0.804	1244	590	654	14.5	2.0	47.4

$$\Omega_t = \frac{3\hbar c^3 A_{0J}}{4e^2\omega^3\chi\langle {}^5D_0\|U^{(t)}\|{}^7F_J\rangle^2} \tag{3}$$

where $J = 2$ and 4, χ is a Lorentz local field correction that is given by $\chi = n_0^2(n_0^2+2)^2/9$, with a refraction index, n_0 of 1.5, and $\langle {}^5D_0\|U^{(2)}\|{}^7F_2\rangle^2$ are the squared reduced matrix elements whose values are 0.0032 and 0.0023 for $J = 2$ and 4, respectively.

The Ω_2 and Ω_4 intensity parameters of trinuclear europium complexes **C1**–**C3** are listed in Table 2. It was found that these complexes had high values of the Ω_2 intensity parameter, which reflected the hypersensitive character of the ${}^5D_0 \rightarrow {}^7F_2$ transition and indicated that the europium ion is in a highly polarizable chemical environment. On the other hand, complexes **C2** and **C3** exhibited the higher values for the Ω_4 parameter as compared to the complex **C1**, which suggested that steric effect prevent the third ligands, Bipy or Phen, from getting closer to the europium ion.

4. Conclusions

In summary, we designed and synthesized three new trinuclear europium complexes, $Eu_3(DBM)_6L$ (**C1**), $Eu_3(DBM)_6(Bipy)_3L$ (**C2**) and $Eu_3(DBM)_6(Phen)_3L$ (**C3**). Their spectroscopic behaviors were investigated by FT-IR, ${}^1H$ NMR, UV–vis and photoluminescence spectroscopic techniques. These europium complexes exhibited the characteristic emission lines for europium ion in solid state. The only one peak corresponding to ${}^5D_0 \rightarrow {}^7F_0$ transition and the mono-exponential behavior for their decay curves indicated the presence of only one europium (III) site in the chemical environment. In addition, the Ω_2 and Ω_4 intensity parameters, the radiative decay rate A_{rad}, the nonradiative rates A_{nrad}, the lifetime (τ) and the quantum yield (η) were calculated according to the emission spectra and the lifetime of the ${}^5D_0$ emitting level in solid state. The trinuclear europium complexes displayed a longer lifetime (τ) and higher luminescence quantum efficiency (η), especially complexes **C2** ($\tau = 0.820$ ms, $\eta = 46.5\%$) and **C3** ($\tau = 0.804$ ms, $\eta = 47.4\%$). These results indicated that the introduction of ligands with multiple binding sites in europium complexes could effectively improve the luminescence properties of europium complexes.

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