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ORIGINAL PAPER

Photophysical Properties of Novel Ternary Lanthanide Complexes with Long Chain Mono-L Cis-Butene Dicarboxylate (L = Hexadecyl, Octadecyl and Eicosyl) and 2,2'-Bipyridyl (or 1,10-Phenanthroline)

Bing Yan · Hongxia Zhu

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Abstract Maleic anhydride was modified by long chain alcohol (1-hexadecanol, 1-octadecanol and 1-eicosanol) to a novel sort of corresponding long monoester mono-L cis-butene dicarboxylate (L = hexadecyl, octadecyl and eicosyl), i.e. monohexadecyl cis-butene dicarboxylate (MAH), monooctadecyl cis-butene dicarboxylate (MAO), and monoeicosyl cis-butene dicarboxylate (MAE), respectively. Then the some novel ternary lanthanide (Eu^{3+}, Tb^{3+}) complexes with the as-derived long chain monoester and assistant nitrogen-heterocyclic ligands (2,2'-bipyridyl (bipy) and 1,10-phenanthroline (phen)) were synthesized and characterized by elemental analysis and IR spectra. The photophysical properties of these complexes were studied in detail with ultraviolet absorption spectra, luminescent excitation and emission spectra and luminescent lifetimes, indicating that the intramolecular energy transfer mechanism runs smoothly within these ternary complexes in terms of sensitized functions of bipy and phen and strong characteristic red or green emissions of Eu^{3+} or Tb^{3+} have been achieved.

Keywords Lanthanide complexes · Long chain maleic monoester · Energy transfer · Photophysical properties

B. Yan (✉) · H. Zhu
Department of Chemistry, Tongji University,
Siping Road 1239, Shanghai 200092, China
e-mail: byan@tongji.edu.cn

B. Yan
State Key Lab of Rare Earth Materials Chemistry and Applications, Peking University,
Beijing, 100871, China

Introducion

Lanthanide complexes have received much attention because of their interesting photophysical properties which have potential applications in the luminescent probes or labels for chemical or biological macromolecules [1, 2] and the active center for luminescent materials [3, 4] or electroluminescent devices [5, 6]. Considerable studies have been focused on the design and assembly of lanthanide complexes with organic ligands such as aromatic carboxylic acids, β-diketones, cryptands, calixarenes and heterocyclic ligands, etc. These organic molecules possess strong absorption of ultraviolet light and then occur an effective intramolecular energy transfer process to central lanthanide ions [7–12]. For binary complexes, it was found that most β-diketone derivatives are more suitable for luminescence of Eu^{3+} while most aromatic carboxylic acids are more suitable for the luminescence of Tb^{3+} [7–15]. We also have studied the energy match and intramolecular energy transfer mechanism in ternary lanthanide complexes with aromatic carboxylic acids and 1,10-phenanthroline in details [16, 20], indicating there still exist intramolecular energy transfer mechanism between ligands (i.e. from aromatic carboxylic acids and 1,10-phenanthroline). On account of the energy match and energy transfer mechanism, it can be realized to predict the luminescent properties of lanthanide complexes. Phthalate or aminobenzoate derivative have been found to exhibit favorable properties of luminescence and film formation [21, 22].

In this context, using maleic anhydride as staring material, three kinds of long chain alcohol (1-hexadecanol, 1-octadecanol and 1-eicosanol) were grafted to achieve their long monoester mono-L cis-butene dicarboxylate (L = hexadecyl, octadecyl and eicosyl), i.e. monohexadecyl cis-butene dicarboxylate (MAH), monooctadecyl cis-butene

dicarboxylate (MAO), and monoeicosyl cis-butene dicarboxylate (MAE), respectively. The corresponding ternary lanthanide (Eu^{3+}, Tb^{3+}) complexes with these long chain monoester ligands and nitrogen heterocyclic ligands (2,2'-bipyridyl (bipy) and 1,10-phenanthroline (phen)) were synthesized and characterized. The photophysical properties for these long esters and their lanthanide complexes were studied in detail, which can be expected to have practical application to fabricate the Langmuir-Blodgett films.

Experimental

Synthesis of monoester mono-L cis-butene dicarboxylate by the modification of maleic anhydride

Maleic anhydride(1.96 *g* / 20 mmol) was mixed with equimolar amount of long chain alcohols (1-hexadecanol (4.85 *g*), 1-octadecanol (5.41 *g*) and 1-eicosanol (5.96 *g*)) in a flask. Then the solid mixtures were placed in an oil bath at the temperature of 110–115°C under stirring for 16 hours. Finally the samples were recrystallized with *n*-hexane three times to afford the white powder mono-L cis-butene dicarboxylate. Anal. Calcd. for $C_{20}H_{36}O_4$ (monohexadecyl cis-butene dicarboxylate (MAH)): C, 70.55; H, 10.66; Found: C, 70.69; H, 10.39. Anal. Calcd. for $C_{22}H_{40}O_4$ (monooctadecyl cis-butene dicarboxylate (MAO)): C, 71.70; H, 10.94; Found: C, 71.47; H, 10.63. Anal. Calcd. for $C_{24}H_{44}O_4$ (monoeicosyl cis-butene dicarboxylate (MAE)): C, 72.68; H, 11.18; Found: C, 72.41; H, 10.79.

Synthesis of ternary lanthanide complexes with monoester mono-L cis-butene dicarboxylate and 2,2'-bipyridyl

The corresponding lanthanide complexes with this ligand were prepared by homogeneous precipitation. The lanthanide oxides (Eu_2O_3, Tb_4O_7) were converted to their nitrates by treatment with concentrated nitric acid. Maleic monoester (0.340 *g* for 16-maleic monoester, 0.368 *g* for 18-maleic monoester and 0.396 *g* for 20-maleic monoester; 1.0 mmol) was dissolved into the alcohol solutions. Then alcohol solutions of lanthanide nitrates ($Ln(NO_3)_3 \cdot 6H_2O$, L = Eu, Tb; 0.33 mmol) were added very slowly to the above mixed solution with vigorous stirring, then added a small quantity of $NH_3 \cdot H_2O$ into the above mixed solution, in order to adjust the *p*H at 7 or so. An hour later, 2,2'-bipyridyl (0.0521 *g* / 0.33 mmol) was introduced to the solutions. Then added a small quantity of $NH_3 \cdot H_2O$ into the above mixed solution, in order to adjust the *p*H at 7 or so. After that, white precipitates appeared and were filtered off, washed with ethanol, dried in a vacuum. Six ternary lanthanide (Eu, Tb) complexes were prepared (as shown in Fig. 1). The compositions of the complex systems were confirmed by elemental analysis: $Ln(L\text{-}MA)_3 \cdot bipy$, L = 16, 18, 20; Ln = Eu, Tb. Anal. Calcd. for $C_{70}H_{113}N_2O_{12}Eu$: C, 63.38; H, 8.59; N, 2.11; Found: C, 63.17; H, 8.35; N, 1.97. For $C_{70}H_{113}N_2O_{12}Tb$: C, 63.05; H, 8.54; N, 2.10; Found: C, 62.82; H, 8.20; N, 1.96. Anal. Calcd. for $C_{76}H_{125}N_2O_{12}Eu$: C, 64.70; H, 8.93; N, 1.99; Found: C, 64.51; H, 8.59; N, 1.84. For $C_{76}H_{125}N_2O_{12}Tb$: C, 64.39; H, 8.89; N, 1.98; Found: C, 64.56 H, 8.59; N, 1.82. Anal. Calcd. for $C_{82}H_{137}N_2O_{12}Eu$: C, 65.88; H, 9.24; N, 1.88; Found: C, 65.61; H, 8.98; N, 1.75. For $C_{82}H_{137}N_2O_{12}Tb$: C, 65.58; H, 9.19; N, 1.87; Found: C, 65.77; H, 8.99; N, 1.75.

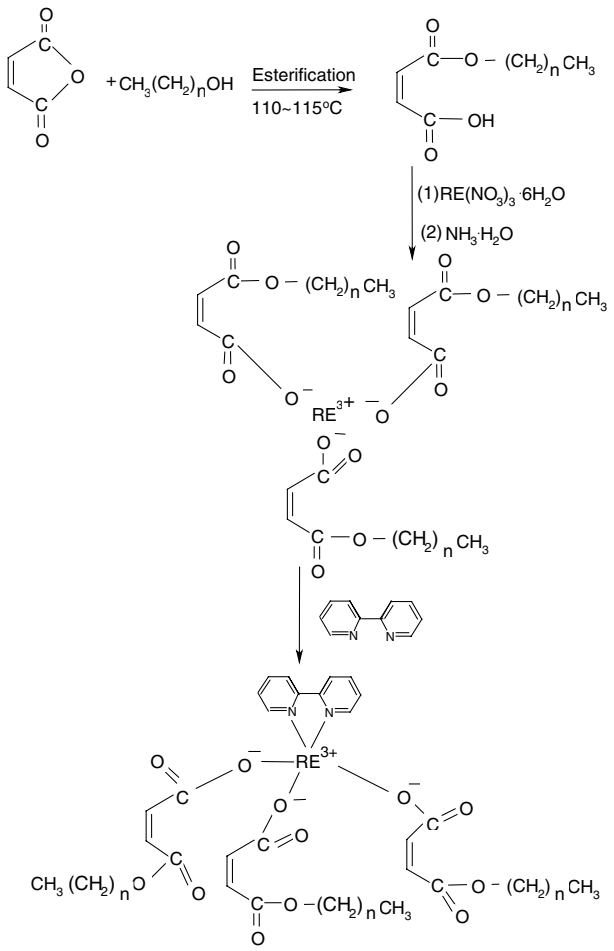


Fig. 1 Preparation scheme of both binary and ternary europium and terbium complexes with mono-L cis-butene dicarboxylate and organic base bipy, for phen, it is the similar scheme.

Synthesis of ternary lanthanide complexes with monoester mono-L cis-butene dicarboxylate and 1,10-phenanthroline

The lanthanide oxides (Eu_2O_3, Tb_4O_7) were converted to their nitrates by treatment with concentrated nitric acid. The corresponding lanthanide complexes with this ligand were prepared by homogeneous precipitation. Maleic monoester (0.340 *g* for 16-maleic monoester; 0.368 *g* for18-maleic

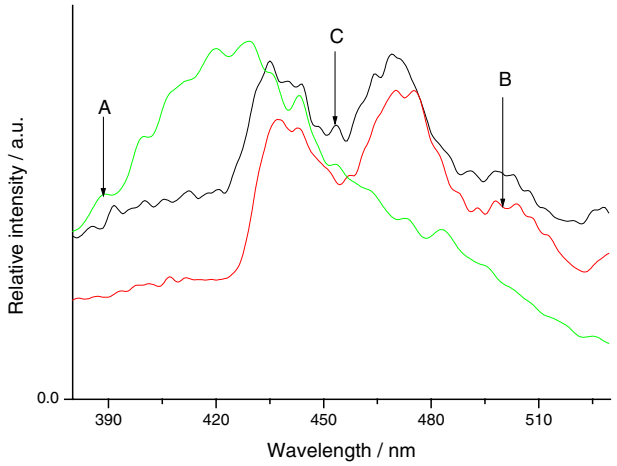


Fig. 2 The phosphorescence spectra of (A) Gd-MAO complex, (B) Gd-bipy complex and (C) bipy-Gd-MAO ternary complex. (5×10^{-5} mol · L^{-1} acetone solution)

monoester and 0.396 *g* for 20-maleic monoester; 1.0 mmol) was dissolvedinto the alcohol solutions. Then alcohol solutions of lanthanide nitrate ($Ln(NO_3)_3 \cdot 6H_2O$,L = Eu, Tb; 0.33 mmol) was added very slowly to the above mixed solution with vigorous stirring, then added a small quantity of $NH_3 \cdot H_2O$ into the above mixed solution in order to adjust the *p*H at 7 or so. After that, white precipitates appeared, an hour later, 1,10-phenanthroline(0.0661 *g* / 0.33 mmol) was introduced to the solutions. Then, white precipitates were filtered off, washed with ethanol, dried in a vacuum. Four ternary lanthanide (Eu, Tb) complexes were prepared(as shown in Fig. 2). The compositions of the complex systems were confirmed by elemental analysis: $Ln(L\text{-}MA)_3 \cdot phen$, L = 16, 18, 20; Ln = Eu, Tb. Anal. Calcd. for $C_{72}H_{113}N_2O_{12}Eu$: C, 64.02; H, 8.44; N, 2.08; Found: C, 63.83; H, 8.15; N, 1.95. For $C_{72}H_{113}N_2O_{12}Tb$: C, 63.70; H, 8.39; N, 2.06; Found: C, 63.92; H, 8.15; N, 1.94. Anal. Calcd. for $C_{78}H_{125}N_2O_{12}Eu$: C, 65.28; H, 8.78; N, 1.95; Found: C, 65.59; H, 8.59; N, 1.84. For $C_{78}H_{125}N_2O_{12}Tb$: C, 64.98; H, 8.74; N, 1.94; Found: C, 64.76; H, 8.57; N, 1.84. Anal. Calcd. for $C_{84}H_{137}N_2O_{12}Eu$: C, 66.42; H, 9.09; N, 1.85; Found: C, 66.59; H, 8.97; N, 1.73. For $C_{84}H_{137}N_2O_{12}Tb$: C, 66.12; H, 9.05; N, 1.84; Found: C, 65.83; H, 8.86; N, 1.72.

Physical measurements

Elemental analyses (C, H, N) were carried out by the Elementar Cario EL elemental analyzer. Infrared spectroscopy with KBr pellets was performed on a Nicolet Nexus 912 AO446 model spectrophotometer in the $4000 \sim 400$ cm^{-1}. Ultraviolet absorption spectra (5×10^{-5} mol · L^{-1} acetone solution) were obtained with an Agilent 8453 spectrophotometer. Low temperature phosphorescence spectrum (5×10^{-5} mol · L^{-1} acetone solution) was determined by Perkin-Elmer LS-55 spectrophotometer under 77 K. The fluorescence (excitation and emission) spectra of solid complexes were measured with Perkin-Elmer LS-55 spectrophotometer: excitation slit width = 10 nm, emission slit width = 5 nm. Luminescence lifetime measurements for solid complexes were carried out on a Ediburgh LFS920 phosphorimeter.

Results and discussion

Table 2 shows the main bands data and their assignments of the FT-IR spectra of the ligands and their complexes. Compared the IR spectra of cis-butene dicarboxyl (Maleic anhydride, MA). The peak at 1783 cm^{-1} of MA presents the stretching vibration of C=O and band at 3130 cm^{-1} corresponds to stretching vibration of C—H which belong to the backbone of maleic anhydride. While the the IR spectra of the long chain monoester cis-butene dicarboxylate appear the vibration peaks of methyl group (—CH_3) and the $\nu_{(C-H)}$ of the long carbon chains changes to 2918 cm^{-1} and proves that the three kinds of long chain alcohols were successfully grafted to maleic anhydride. Additionally, the peak at 1726 cm^{-1} substantiates that the absorption of C=O was affected by the long chain group –OR. Besides, so far as the ternary complexes are concerned, the occurrence of complexation between Tb^{3+} and MA is evidenced by a new narrow band located at 1383 to 1387 cm^{-1} and 1596 to 1604 cm^{-1} appeared to prove that terbium ions may coordinate to two oxygen atoms of the free carboxylic groups. The twisting bending vibrations at 896, 761 cm^{-1} and 857, 743 cm^{-1} which correspond to absorption of hydrogen atoms belong to heterocycle of bipy and phen have shift to lower frequency of 854, 727 cm^{-1}, and the facts firmly prove bipy or phen can coordinate to lanthanide ions effectively.

From the ultraviolet absorption spectra of three long chain long monoester mono-L cis-butene dicarboxylate (L = hexadecyl, octadecyl and eicosyl), it can be observed the main absorption bands of them are all similar at 245 nm, suggesting that the introduction of different long chain alcohol hardly has influence on the absorption property for the long aliphatic (hydrocarbon) chain has no delocolized structure for ultraviolet absorption activity. It is observed that there are nearly no difference between maleic anhydride is similar to that of MAO (around 245 nm), because they have the same chromophore C=C and similar π-$\pi*$ transitions influenced by conjugating effect of both C=C and C=O groups. Which indicates that the aborption band can be ascribed to MA fragment for its unsaturated delocalized structure and the modification of long chain can not change the absorption. As for ultraviolet spectrum of bipy-Tb-MAO, the obvious band at 266 nm shows characteristic absorption of bipy, which can be ascribed as the absorption of bipy for free bipy appears one absorption peak at 265 nm [19]. The small red-shift of

Table 1 The main band assignments of IR spectra for ligands and their complexes

Compounds	νs (CH_3) (cm^{-1})	νs (C–H) (cm^{-1})	ν (C=O) (cm^{-1})	νs (COO–) (cm^{-1})	νas (COO–) (cm^{-1})	νs (C–O–C) (cm^{-1})	νas (C–O–C) (cm^{-1})	ν (CN) (cm^{-1})	π (CH) (cm^{-1})
MA			1783			1070	1243		
MAH	2852	2918	1726			1165	1261		
MAO	2848	2918	1726			1162	1267		
MAE	2848	2922	1726			1162	1274		
bipy								1417	896, 761
phen								1422	857 743
Eu-MAH-bipy	2848	2917	1717	1387	1600	1173	1226	1435	852, 722
Tb-MAH-bipy	2852	2921	1717.39	1383	1604	1174	1226	1435	856, 731
Eu-MAO-bipy	2854	2923	1721	1383	1602	1183	1228	1435	848, 722
Tb-MAO-bipy	2854	2917	1721	1387	1602	1183	1228	1435	856, 731
Eu-MAE-bipy	2857	2917	1721	1387	1596	1183	1267	1413	857, 722
Tb-MAE-bipy	2852	2917	1722	1386	1066	1183	1265	1421	843, 724
Eu-MAH-phen	2854	2924	1721	1386	1602	1174	1218	1431	847, 727
Tb-MAH-phen	2852	2922	1721	1383	1600	1174	1218	1432	852, 727
Eu-MAO-phen	2864	2924	1721	1386	1602	1183	1218	1427	852, 727
Tb-MAO-phen	2852	2917	1717	1383	1604	1183	1226	1426	848, 727
Eu-MAE-phen	2854	2924	1728	1386	1602	1181	1218	1430	854, 727
Tb-MAE-phen	2848	2922	1726	1387	1600	1181	1222	1424	852, 727

1 nm is due to the more extensive conjugated system of electronic distribution form because the coordination between lanthanide ions and bipy. For ultraviolet absorption spectra of maleic anhydride, MAO and phen-Tb-MAO complex, it shows the similar feature. Therefore in the ternary complex systems, 2, 2′-bipyridy and 1,10-phenanthroline play the major energy donor role and will transfer it to Tb ions. We can found the further evidences concerning the above predictions from the ultraviolet absorption spectra of ternary complexes bipy-Eu-L-MA, bipy-Tb-L-MA, phen-Eu-L-MA and phen-Tb-L-MA ($L = 16, 18, 20$), it can be found there exist the main absorption bands for bipy or phen and L-MA can not develop a dominating function in binary systems and its π-π^* transition corresponds to the strongest absorption.

As shown in Fig. 2 for the phosphorescence spectra of (A) Gd-MAO complex, (B) Gd-bipy complex and (C) bipy-Gd-MAO ternary complex, ti can be observed that B and C are rather similar in terms of the same organic ligand 2, 2′-bipyridyl responsible for the emissions. According to the energy transfer and intramolecular energy mechanism [16–20], intramolecular energy transfer efficiency depends chiefly on two energy transfer processes: the first one leads from the triplet level of ligands to the emissive energy level of the Eu^{3+} and Tb^{3+} ion by Dexter's resonant exchange interaction [23]; the second one is just an inverse energy transfer by a thermal deactivation mechanism [24]. Established on this theory, the conclusion can be drawn that energy differences is of opposite influence on the two energy transfer processes and an optimal value can be assumed to exist. The peak at 429 nm of A shows MAO's triplet state energy level is approximately 23310 cm^{-1} which is higher than phen (437 nm, 22880 cm^{-1}), therefore, we suppose that energy transfer process will occur from T-A1 to phen, substantiating that the heterocyclic ligand will become main energy donor and have the possibility to sensitize Eu^{3+} and Tb^{3+} ions. For the systems with phen, they show the similar feature because the intramolecular energy transfer between

Table 2 Luminescent properties of ternary lanthanide complexes (solid samples)

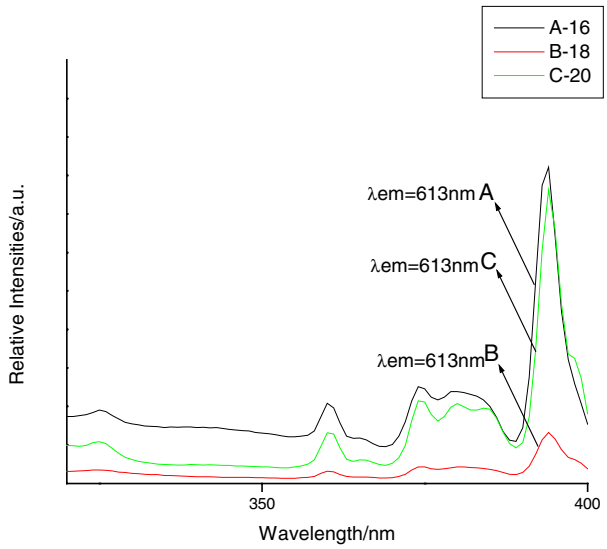
Ternary lanthanide solid complexes	Emission bands (nm)*	Relative intensiteis (a.u.)	Lifetimes (μs)**
bipy-Eu-MAH	590, 615, 648, 701	841	
bipy-Eu-MAO	590, 614, 649, 700	792	875
bipy-Eu-MAE	591, 616, 650, 701	166	
bipy-Tb-MAH	488, 544, 582, 619	557	
bipy-Tb-MAO	488, 544, 582, 619	345	1260
bipy-Tb-MAE	488, 544, 582, 619	571	
phen-Eu-MAH	591, 614.5, 649, 700	760	
phen-Eu-MAO	591, 615, 649, 700	1245	848
phen-Eu-MAE	591, 615, 648, 700	640	
Phen-Tb-MAH	489, 543, 584, 619.5	221	
phen-Tb-MAO	489, 543, 584.5, 619.5	214	1394
phen-Tb-MAE	489, 543, 584.5, 619.5	246	

*For Eu^{3+}: $^5D_0$–^7F_J (J = 1, 2, 3, 4); For Tb^{3+}: $^5D_4$–^7F_J (J = 6, 5, 4, 3).

**For E Eu^{3+}: $^5D_0$–$^7F_2$ transition; For Tb^{3+}: $^5D_4$–$^7F_5$ transition.

ligands becomes more easily for the phen is lower (453 nm, 22075 cm^{-1}) [16–20].

Figure 3 wears the excitation for four series of ternary lanthanide complexes. It can be found the all of them are similar with the maximum absorption at around 266 nm, corresponding to the absorption of nitrogen heterocycle ligands (bipy or phen) and taking agreement with the results from ultraviolet and phosphorescent spectra. Which further indicate that in the ternary complex systems, bipy or phen behave as main energy donor and play the major role on the energy transfer to europium and terbium ions. The corresponding emission spectra of the resulting complexes are shown in Figs. 4 and 5.



(A)

Fig. 3 The excitation spectra of both bipy-Eu-L-MA (L = 1, 18, 20)

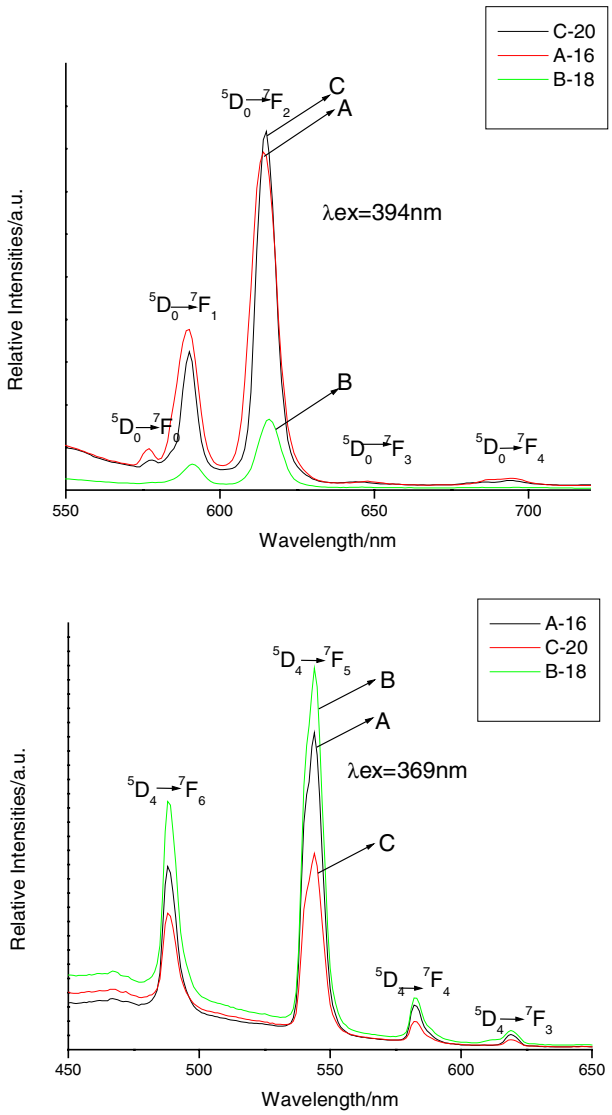


Fig. 4 The emission spectra of (A) bipy-Eu-L-MA and (B) bipy-Tb-L-MA

As described in Fig. 4 (A) for bipy-Eu-L-MA; the $^5D_0 \rightarrow {}^7F_1$, $^5D_0 \rightarrow {}^7F_2$, $^5D_0 \rightarrow {}^7F_3$, $^5D_0 \rightarrow {}^7F_4$ transitions at 590, 614, 650 and 700 nm of Eu^{3+} were obviously obtained and strong red emission ($^5D_0 \rightarrow {}^7F_2$) was dominating. In regard to Fig. 4 (B) for bipy-Tb-Eu-L-MA, there are four assigned transitions from the $^5D_4 \rightarrow {}^7F_J$ (J = 6, 5, 4, 3) transitions at 490, 544, 587 and 622 nm of terbium ions and a striking green luminescence ($^5D_4 \rightarrow {}^7F_5$) was accomplished. Fig. 5 presents the similar features. We further determined the luminescent relative intensities and lifetimes for the ternary complexes and binary complexes of phen, the detailed data have been shown in Table 2. The relative emission intensities of ternary complexes show the similar order to those of corresponding binary ones of phen, indicating that the effective energy transfer and sensitization take place between phen and lanthanide ions in the ternary systems. Especially

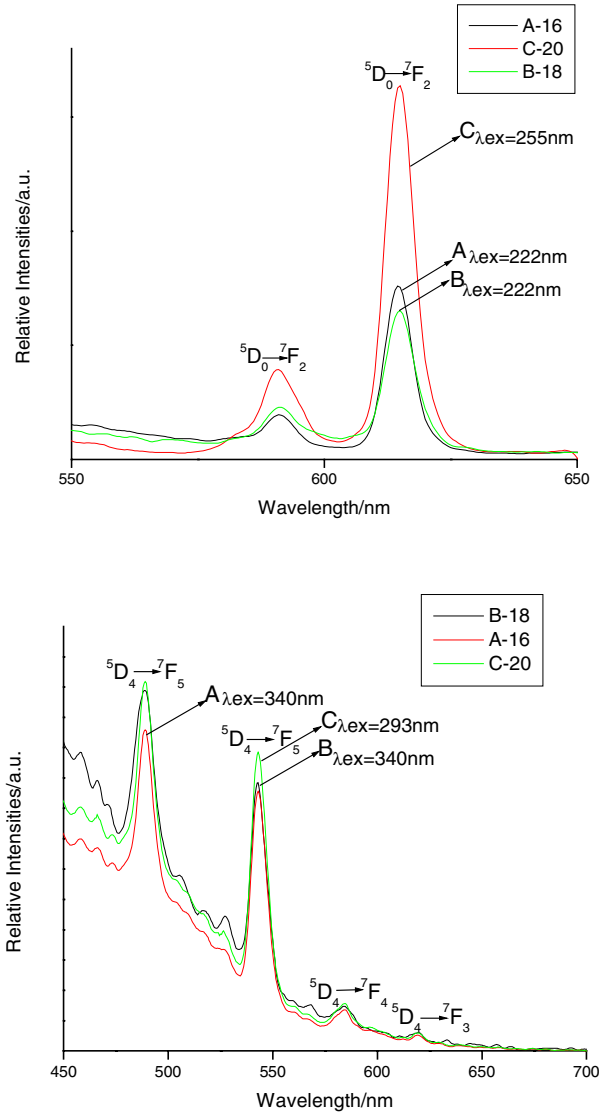


Fig. 5 The emission spectra of (A) phen-Eu-L-MA and (B) phen-Tb-L-MA

the luminescent lifetimes of ternary complexes are higher than those of binary one with phen, which suggest that the introduction of long chain monoester in the ternary molecular systems enhances the rigidity and stability of the whole complex and reduces the non-radiative energy loss compared the binary systems of phen.

Conclusion

Twelve ternary lanthanide (Eu, Tb) complexes with mono-L cis-butene dicarboxylate and bipy (or phen) have been synthesized and characterized. The photophysical properties of them have been studied with ultraviolet spectra, phosphorescence spectra, excitation and emission spectra, and luminescent lifetimes. The energy transfer process initiated from mono-eicosyl cis-butene dicarboxylate to 2,2′-bipyridyl (or 1,10-phenanthroline) then to lanthanide ions (Eu^{3+}, Tb^{3+}) has been discussed to predict that the optimum energy transfer exists between the heterocyclic ligand and Ln^{3+}. The emission spectra for the ternary complexes were described in detail and the results indicate that lanthanide complex show the extraordinarily strong luminescence (red and green) especially compared to binary complex molecules with low radiate efficiency.

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