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Synthesis and photoluminescence properties of novel europium complexes of 2'-hydroxyacetophenone and 4,6-diacetylresorcinol

Liu Sheng-Li*, Wen Chun-Lin, Qi Su-Su, Liang En-Xiang

School of Chemistry and Chemical Engineering, Hunan University of Science and Technology, Xiangtan 411201, China Received 17 January 2007; received in revised form 7 May 2007; accepted 8 May 2007

Abstract

Novel europium (III) complexes of the formulae $Eu(OHAP)_3 \cdot 2H_2O$, $Eu(OHAP)_3Phen$, $Eu_2(DAR)_3 \cdot 4H_2O$ and $Eu_2(DAR)_3Phen_2$ (HOHAP = 2'hydroxyacetophenone, $H_2DAR = 4,6$ -diacetylresorcinol, Phen = 1,10-phenanthroline) have been designed and synthesized in this paper. These complexes were characterized by elemental analysis, FT-IR, and UV-vis. Based on these observations, the ligands are coordinated to Eu(III) via the acetyl and phenolic oxygens, and H_2DAR is concluded to be bis-bidentate donor. Photoluminescence studies showed that the several complexes emitted red luminescence. Thermo-gravimetric analysis showed that the complexes possess good thermal stability. Also, it was found that Phen as a synergic ligand, coordinated to Eu(III) in a composite system like 2'-hydroxyacetophenone and 4,6-diacetylresorcinol, could enhance the complexes luminescence intensity, quantum yield and lifetime.

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Keywords: Europium (III) complexes; Luminescence; Quantum yield; 2'-Hydroxyacetophenone; 4,6-Diacetylresorcinol; 1,10-Phenanthroline

1. Introduction

Lanthanide complexes have been studied by many researchers as potential emitting materials and typically have been found to possess narrow emission bands and high efficiency [1–4]. Since the emissions of lanthanide complexes originate from transitions between f levels, which are effectively field from the influence of external forces by filling $5s^2$ and $5p^6$ orbital, the various states arising from f^n configurations are splited by external fields only to the extent of 100 nm^{-1} . In particular, Eu(III) has five narrow emission bands corresponding to the ${}^5D_0 \rightarrow {}^7F_J$ transitions, where J=0, 1, 2, 3, 4. The strongest transition, ${}^5D_0 \rightarrow {}^7F_2$, usually occurs at approximately 613 nm with a narrow emission band which produces a red color in Eu(III) complexes [5,6]. In this manuscript, we find that 2'-hydroxyacetophenone and some of its derivatives have the same character.

Also, novel Eu(III) complexes of the formulae, Eu(OHAP)₃· 2H₂O, Eu(OHAP)₃Phen, Eu₂(DAR)₃·4H₂O and Eu₂(DAR)₃ Phen₂ have been designed and synthesized. The emission data shows narrow and strong bands from the ⁵D₀ \rightarrow ⁷F₂ transition,

1386-1425/\$ - see front matter © 2007 Elsevier B.V. All rights reserved. doi:10.1016/j.saa.2007.05.019 and specially the Phen can enhance the luminescence intensity, quantum yield and lifetime of the Eu(III) complexes.

2. Experimental

2.1. Materials and physical techniques

The 2'-hydroxyacetophenone (98%) and Eu₂O₃ (99.99%) were purchased from Aldrich and used as received, while the other reagents used were of analar grade. H₂DAR was prepared following the method reported in Ref. [7], and was characterized by C, H microanalysis, melting point, IR and UV–vis spectroscopic analysis.

Elemental analyses were measured using an Elementar vario EL III elemental analyzer, and europium ion contents were determined by EDTA complexometry. IR spectra were recorded over the range 4000–400 cm⁻¹ using Perkin-Elmer Spectrum One spectrophotometer by the KBr pellet technique. UV was performed with a Perkin-Elmer UV/Vis Lambda 35 spectrometer. Fluorescence excitation and emission spectra were recorded using an F-4500 FL spectrophotometer.

Fluorescence lifetime was measured using an EMG 201 MSC quasi -molecular 308 nm laser by monitoring ${}^{5}D_{0} \rightarrow {}^{7}F_{2}$ emission line of Eu(III) at room temperature.

^{*} Corresponding author. *E-mail address:* slliu@hnust.edu.cn (S.-L. Liu).

The emission quantum yield q for the Eu(OHAP)₃·2H₂O, Eu(OHAP)₃·Phen, Eu₂(DAR)₃·4H₂O and Eu₂(DAR)₃·2Phen were obtained following the procedure described in Ref. [8]. The q value is defined as the ratio between the number of photons emitted by the Eu(III) and the number of photons absorbed by the ligands. According to the method developed by Bril [9] at Philips Research Laboratories, the q value for a given sample can be determined by comparison with standard phosphors, whose quantum yields have been previously determined by absolute measurements. This method provides absolute yields while avoiding absolute measurements, which are in general complicated. The quantum yield q_x of a sample is thus determined as follows:

$$q_x = \left(\frac{1 - r_{\rm ST}}{1 - r_x}\right) \left(\frac{\Delta \phi_x}{\Delta \phi_{\rm ST}}\right) q_{\rm ST}$$

where r_{ST} and r_x are the amount of exciting radiation reflected by the standard and by the sample, respectively, and q_{ST} is the quantum yield of the standard phosphor. The terms $\Delta \Phi_x$ and $\Delta \Phi_{\text{ST}}$ give the integrated photon flux (photons s⁻¹) for the sample and the standard phosphor, respectively. The standard in our case was sodium salicylate (Aldrich), which has a broad emission band with a maximum at 450 nm and q = 60%at room temperature [9]. But our experiment show that the best excited wavelength of the complexes are not the same as that of sodium salicylate, so the q is not an absolutely quantum yield, it only can be used for making a comparison between each other.

2.2. Preparation of the complexes

Europium chloride solution was prepared as follows: 0.5 mmol of europium oxide was dissolved in 4 ml of concentrated hydrochloric acid (36%) in a beaker and digested on a steam bath until completely dissolved. Surplus HCl was removed by evaporation and the residue was diluted with 10 ml anhydrous alcohol.

Eu(OHAP)₃Phen was prepared as follows [10]: 3 mmol of HOHAP and 1 mmol of Phen were dissolved in 95% ethanol and a proper NaOH solution with ethanol (0.5 mol/l) was added in the solution above. The europium chloride solution (1 mmol of EuCl₃) was added dropwise to the mixed solution of HOHAP and 1,10-phen with stirring at 50–60 °C, and then stirred for another 4 h. The precipitation appeared immediately, then filtered and washed with 80% ethanol solution $(V_{(H_2O)}:V_{(EtOH)}=1:4)$ until no Cl⁻ was found in the water. The product was vacuum dried for 6 h at 80 °C. The other rare earth complexes with the ligands $(Eu_2(DAR)_3\cdot4H_2O)$.

Table 1
Elemental analysis results of complexes

 $Eu_2(DAR)_3Phen_2$, $Gd_2(DAR)_3\cdot 4H_2O$, $Gd(OHAP)_32H_2O$) were prepared in the same way.

3. Results and discussions

All the complexes are yellow, and slightly soluble in DMF and DMSO. The elemental analytical data (Table 1) agree well with their formula as expected. Thermal data of the complexes show that there is no endothermic peak before decompose, which indicates that the complexes have no melting point due to decomposed when the temperature was increased to some degree at atmospheric pressure, all these complexes start decomposing around 300 °C as indicated by the exotherms.

3.1. FT-IR analysis

In Eu(OHAP)₃·2H₂O and Eu₂(DAR)₃·4H₂O, the broad band in the region $3500-3100 \text{ cm}^{-1}$ is due to the coordination with water. The rocking mode of the coordinated water was observed around 800 cm^{-1} . The bands at 1312 and 1323 cm⁻¹ are assigned to the phenolic C-O stretching mode of the ligands. Compared with the ligand, these shift to higher frequencies in the spectra of the chelated complexes is due to its coordination to europium ion through the oxygen atom of the phenolic group and it appeared as a singlet or a doublet [11]. Furthermore, the absorption in the region 1182 and 1160 cm⁻¹ due to O-H was not observed in the infrared spectra of the complexes, suggesting that the deprotonation of the ligands occurs before their coordination to europium metal. Likewise, carbonyls' stretching frequencies in the ligands appearing at 1659 and $1643 \,\mathrm{cm}^{-1}$ have a small decrease (by 30 and 40 cm^{-1} , respectively) in all the complexes. This indicates that the carbonyl oxygen is involved in the coordination with the metal ion. A characteristic feature of the IR spectra of the complexes is the appearance of a prominent band in the region $480-530 \text{ cm}^{-1}$ for all the complexes. From a comparison with the ligand spectra, we have tentatively attributed this prominent band to the M-O vibrational mode. Such bands have been observed in acetylacetonate complexes [12] and other o-hydroxyarylcarbonyl compounds [13]. Based on these observations the ligands are chelated to the europium ions via the acetyl oxygen and the phenolic oxygen, H2DAR is concluded to be bis-bidentate donor, the similar structure that coordinated with metal ions had been confirmed by Shyamala [14] and Takano [15]. The C-H bending vibration peaks of Phen shifted from 854 and 738 cm⁻¹ to 845 and 729 cm⁻¹, respectively in Eu₂(DAR)₃Phen₂, which indicates that the bonds are formed between europium ions and nitrogen atoms of Phen [16–19].

Compound	Exp _C (Cal) (%)	Exp _H (Cal) (%)	Exp_{N} (Cal) (%)	Exp _{Eu} (Cal) (%)
Eu(OHAP)3·2H2O	47.95 (48.58)	4.20 (4.25)	_	25.48 (25.61)
Eu(OHAP) ₃ Phen	58.60 (58.62)	3.89 (3.96)	3.82 (3.80)	20.70 (20.60)
$Eu_2(DAR)_3 \cdot 4H_2O$	37.80 (37.83)	3.19 (3.39)	_	31.86 (31.91)
$Eu_2(DAR)_3Phen_2$	52.50 (52.27)	3.60 (3.25)	4.58 (4.52)	24.50 (24.49)



 $Fig. \ 1. \ The \ chemical \ structure \ of \ the \ complexes \ (a): Eu(OHAP)_3 \cdot 2H_2O; \ (b): Eu(OHAP)_3Phen; \ 2a: Eu_2(DAR)_3 \cdot 4H_2O; \ 2b: Eu_2(DAR)_3Phen_2).$

Table 2				
Infrared	spectral	bands	in	cm ⁻¹

Compound	$V_{\rm H-O-H}$	V _{O-H}	V _{C=O}	V _{C-O} phenolic	$\delta_{\rm C-H}$ phen	$\delta_{\mathrm{M-O}}$
Phen	_	_	_	_	854, 739	-
HOHAP	-	3011, 3061	1643	1323	-	_
Eu(OHAP)3·2H2O	3500-3200	-	1610	1338	-	525
Eu(OHAP) ₃ Phen	-	-	1612	1340	846, 732	521
H ₂ DAR	-	2850, 2920	1659	1312	-	_
Eu ₂ (DAR) ₃ ·4H ₂ O	3450-3200	-	1621	1325	-	482
Eu ₂ (DAR) ₃ Phen ₂	-	-	1620	1326	845, 729	478

The possible chemical structure of the complexes is given in Fig. 1.

The main absorption peaks of different complexes are listed in Table 2.

3.2. UV absorption spectra

The UV-vis spectra of synthesized compounds also confirmed the formation of the europium complexes. Fig. 2(1) and

Table 3 Electronic absorption data for Eu (III) complexes

Compound	UV			
	$\lambda_{abs.}$ (nm)	$\varepsilon_{\rm max.}~(\times 10^4)$		
НОНАР	322.8, 266.5	1.2623		
Eu(OHAP)3.2H2O	332.8, 269.0	1.7051		
H ₂ DAR	321.7, 288.6	0.9725		
Eu2(DAR)3·4H2O	324.9, 293.3	1.2803		

(2) give the absorption spectra of the ligands H_2DAR , HOHAP and their europium complexes in dimethylsulfoxide (DMSO, 10^{-5} mol/l) and in the 250–500 nm range.

For example, it can be seen that the main absorption peaks of H₂DAR are at 288.6 and 321.7 nm. The weak peak at 288.6 nm are attributed to $\pi \rightarrow \pi^*$ transitions in the aromatic rings, and the peak at 321.0 nm is the maximum absorption (kmax), which can be attributed to $n \rightarrow \pi^*$ transitions of the carbonyl oxygen [18–20]. The main absorption spectra of H₂DAR in Eu₂(DAR)₃·4H₂O are red-shifted to around 293.3 and 324.9 nm. These also indicate that the ligands are chelated to the europium ions via the carbonyl and the phenolic oxygens (Table 3).

3.3. Luminescence properties

The excitation spectrum of the Eu(OHAP)₃·2H₂O complex in the solid was recorded in the spectral range from 250 to 550 nm with the emission monitored on the hypersensitive ${}^{5}D_{0} \rightarrow {}^{7}F_{2}$



Fig. 2. (1) Electronic absorption spectra of (a) $Eu_2(DAR)_3 \cdot 4H_2O$ (solid line) and (b) H_2DAR (dotted line); 1×10^{-5} M (DMSO); (2) electronic absorption spectra of (a) $Eu(OHAP)_3 \cdot 2H_2O$ (solid line) and(b) HOHAP (dotted line); 1×10^{-5} M (DMSO).

transition at 612 nm (Fig. 3(1) and (2)). This spectrum displays a strong band from 300 to 450 nm that is attributed to the HOHAP. In addition, several narrow bands are also observed which are assigned to $4f^6$ -intraconfigurational transitions from the ground state 7F_0 to the following excited state of the europium ion (in nm): 5D_2 (464) and 5D_1 (532).

The emission spectrum of the Eu(OHAP)₃·2H₂O complex in solid state was recorded in the range of 500–750 nm. The emission data show intense narrow bands from the ${}^{5}D_{0} \rightarrow {}^{7}F_{J}$ transitions (where J = 0–4) and are dominated by the hypersensitive ${}^{5}D_{0} \rightarrow {}^{7}F_{2}$ transition around 610 nm. The presence of the ${}^{5}D_{0} \rightarrow {}^{7}F_{0}$ of Eu(III) transition indicates that the europium ion is located in a symmetry site of the type C_s, C_n or C_{nv} [21]. The most outstanding feature in the emission spectra is the enhancement of the Eu(III) emissions in Eu(OHAP)₃Phen excited at 392 nm. The excitation and emission spectrum of others complexes were measured as the same way, the result indicates that HOHAP and H_2DAR are good sensitizer for the Eu(III) luminescence and Phen as a synergic ligand can enhance the complexes luminescence intensity.

The lifetimes of ${}^{5}D_{0}$ excited state for the complexes were measured by time-resolved spectroscopy, the lifetime valves were calculated through the mono-exponential decay method [22], and the results are given in Table 4, too.

 Gd^{3+} complex was selected as model complex for determination of the triplet state energies of the organic ligands owing to their high phosphorescence–fluorescence ratio compared to those of other Ln³⁺ complexes and Gd³⁺ can sensitize the phosphorescence emission of ligands. The phosphorescence spectra of the gadolinium complexes in ethanol with HOHAP and H₂DAR were measured (Fig. 4(1) and (2)), and the triplet



Fig. 3. (1) Emission spectra of (a) Eu(OHAP)₃Phen (dotted line, $\lambda_{ex} = 376.5$ nm) and (b) Eu(OHAP)₃·2H₂O (dotted line, $\lambda_{ex} = 376.0$ nm) in the solid state; (2) emission spectra of (a) Eu₂(DAR)₃Phen₂ (solid line, $\lambda_{ex} = 392.1$ nm) and (b) Eu₂(DAR)₃·4H₂O (solid line, $\lambda_{ex} = 391.5$ nm) in the solid state.

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Table 4

Complexes	$\lambda_{max.}(ex) (nm)$	$\lambda_{max.}(em) (nm)$	Luminescence intensity (a.u.)			Lifetimes (7/ms)	q (%)	
			$^{5}D_{0}\rightarrow ^{7}F_{0}$	$^5D_0 \rightarrow {}^7F_1$	$^5D_0 \rightarrow {}^7F_2$	$^5D_0 \rightarrow {}^7F_3$		
Eu(OHAP) ₃ ·2H ₂ O	391.5	610.6	84.5	85.5	847.5	73.2	0.954	5.0
Eu(OHAP) ₃ Phen	392.1	610.0	247.5	362.5	4354	60.8	1.012	7.5
$Eu_2(DAR)_3 \cdot 4H_2O$	376.0	610.4	30.0	152.6	1837.0	34.41	0.269	4.8
Eu ₂ (DAR) ₃ Phen ₂	376.5	611.6	95.9	412.3	7780.5	71.3	0.482	5.4

The luminescence properties of the Eu (III) complexes in the solid state

state of HOHAP(H₂DAR) can be determined to be $23,095 \text{ cm}^{-1}$ (21,739 cm⁻¹) based on the both maximum phosphorescence band at 433 nm(460 nm).

The luminescence properties of the europium complexes in the solid state are summarized in Table 4.

Luminescence in rare earth complexes is produced by the f-f transitions of the metal ion, and the complexes display a bright and narrow lanthanide ion emission. For Eu(III) complexes, the emission spectra are dominated by the ${}^{5}D_{0} \rightarrow {}^{7}F_{2}$ transition around 610 nm. During luminescence, the excited energy of the organic ligand undergoes an intramolecular energy transfer from the triplet state of the ligand to an excited state of Eu(III), and then the emission occurs when the energy transfer from the lowest excited state $({}^{5}D_{0})$ to the ground state $({}^{7}F_{2})$ of Eu(III). In order for energy transfer to be effective, the triplet state of the ligand must be higher in energy than the emissive excited state of the metal ion, and for Eu(III) complexes the most efficient ligands of energy transfer are those in which the triplet state is a little higher (too much and too small should decrease the luminescence intensity) in energy than the ${}^{5}D_{0}$ level of Eu(III). Thus, although the emitting color of a rare earth complex depends on the metal ion, its luminescence properties are affected by the ligands, especially their energy transfer properties. Thus it can be seen that HOHAP and H2DAR are two ligands that can transfer the energy to the Eu(III) efficiently and emit characteristic red luminescence emission of Eu³⁺ strongly.

The energy differences between the triplet state of HOHAP (H₂DAR) and the resonance energy level of Eu^{3+} (⁵D₀,

 $17,265 \text{ cm}^{-1}$) can be calculated to be 5830 cm^{-1} (4474 cm⁻¹), they are all indeed sufficient to efficiently populate the ${}^{5}D_{0}$ luminescent state of Eu³⁺. Theoretically, if the differences between the triplet state of A (for example: HOAHP) and the resonance energy level of Eu^{3+} is larger than that of B (the other), the luminescent intensity of A should be smaller than B, but the luminescence intensity of Eu(OHAP)₃·2H₂O is larger than that of $Eu_2(DAR)_3 \cdot 4H_2O$. There may be two reasons. Firstly, compared with HOHAP, the difference between the triple state of H₂DAR and the excited states of Eu(III) is too small to transfer the energy efficiently from the ligand to europium ion, so HOHAP is more suitable than H₂DAR. Secondly, it should be an explanation that the complex $Eu(OHAP)_3 \cdot 2H_2O$ contains three chromophores/Eu, and the Eu₂(DAR)₃·4H₂O only contains 1.5/Eu (i.e. about one half of the more luminescent complex).

Phen as a synergic ligand enhances the complexes luminescence intensity. The main reason should be replaced the quenching water molecules in the coordination sphere of Eu(III). According to the theory of nonradiative transition in lanthanide complexes, the nonradiative relaxation between various *J* states may occur by interaction of the electronic levels of the lanthanide ion with suitable vibrational modes of the environment. The efficiency of these processes depends on the energy gap between the group and the excited states of the lanthanide ions and the vibrational energy of the oscillators. When solvents containing O–H groups are coordinated to lanthanide ions, efficient nonradiative deactivations take place via vibronic coupling with the



Fig. 4. (1) Phosphorescence spectra of Gd(OHAP)₃·2H₂O (λ_{em} = 433 nm, λ_{ex} = 310 nm); (2) phosphorescence spectra of Gd₂(DAR)₃·4H₂O (λ_{em} = 460 nm, λ_{ex} = 320 nm).

vibrational states of the O–H oscillators. To reduce the number of O–H oscillators (water molecular) in the coordination sphere and increase the intensity of the lanthanide luminescence exploiting the antenna effect, complexes with strongly absorbing synergic ligands have been designed, for example, some synergic ligands with aromatic segment, e.g. 1,10-phenanthroline, which show intense absorption bands in the UV region due $\pi \rightarrow \pi^*$ transitions. And if the coordination sphere is supplemented with 1,10-phenanthroline as donor atom, quenching water molecules in the coordination sphere of Eu(III) can be replaced and the emissions of Eu (III) complexes should be increased [23].

The difference of decay time and quantum yield between the complexes is explained mainly due to the coordinated water molecules in the complex, too. Donor atom of Phen occupies some coordination site around the central ion and simultaneously replace remaining water molecules-effective quenches-coordinated to Eu (III) in the inner sphere. Phen can then act as an antenna. These two effects should enhance the lifetime and the quantum yield as the example of Eu(OHAP)₃Phen shows in comparison with Eu(OHAP)₃·2H₂O.

4. Conclusions

Novel europium (III) complexes of formulae, Eu(OHAP)₃· 2H₂O, Eu(OHAP)₃Phen, Eu₂(DAR)₃·4H₂O and Eu₂(DAR)₃ Phen₂ (HOHAP = 2'-hydroxyacetophenone, H₂DAR = 4,6diacetylresorcinol, Phen = 1,10-phenanthroline) have been designed and synthesized. The characterizations of the complexes indicate that the ligands are chelated to Eu (III) via the carbonyl and the phenolic oxygens. The emission data show intense narrow and strong bands from the ⁵D₀ \rightarrow ⁷F₂ transition around 610 nm, the second ligand (Phen) as efficacious antenna of Eu (III) ions can enhance the Eu (III) luminescence intensity, quantum yield and lifetime.

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