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The Co-luminescence Effect of Eu–Gd–Ofloxacin–SDBS System and its Analytical Application

Feng Wang · Wei Huang · Yong Hou · Zhenni Xu

Abstract A fluorescence enhancement phenomenon in the europium (Eu)–Ofloxacin (OF)–Sodium Dodecyl Benzene Sulfonate (SDBS) fluorescence system was observed when Gd^{3+} was added. The fluorescence intensity of the systems was measured ($\lambda_{ex}/\lambda_{em} = 280/612$ nm) at pH 7.8. Under optimum conditions, a linear relationship between the enhanced fluorescence intensity and the Eu^{3+} concentration in the range of $5.0 \times 10^{-10} \sim 2.0 \times 10^{-7}$ mol·L^{-1} was observed. The detection limit of Eu^{3+} was 1.46×10^{-10} mol·L^{-1} (S/N = 3). This method was used for the determination of trace amounts of europium in synthetic rare earth samples with satisfactory results. In addition, the interaction mechanism is also studied.

Keywords Co-luminescence · Europium · Ofloxacin · Energy transfers

Introduction

Rare earth (RE) elements play a significant role in material science, catalysis, medicine, and life science. It is important to determine traceamounts of rare earth elements. Owing to the fluorimetry of rare earth elements they possess high sensitivity and good selectivity. Studying on a fluorescence enhancement or co-luminescence the effects of their multi-complex systems have been of interest for a long time [1–3]. This enhancement effect has been used for the determination of ultratrace amounts of RE ions; the fluorescence intensity of the systems could be increased by several orders of magnitude. Previously, studies on fluorescence enhancement systems focused on β-diketone complexes [4–8], only a few non-β-diketone [9, 10] has been studied.

In this paper, we found a new fluorescence enhancement system of Eu^{3+}–Gd^{3+}–Ofloxacin(OF)–SDBS which caused by a non-β-diketone ligand ofloxacin. The structure of ofloxacin {9-fluoro-2, 3-dihydro-3-methyl-10-(-methyl-1-piperzinyl)-7-oxo-7H-pyrido-[1,2,3-de]1,4-bonzoxazine-6-carboxylic acid} is shown as following:
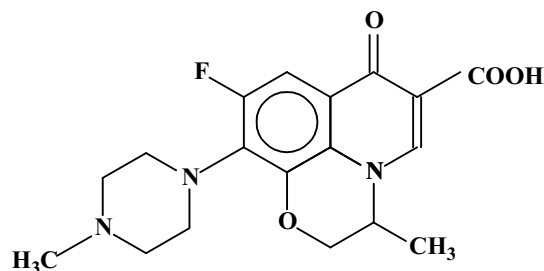
Ofloxacin is a synthetic fluoroquinolone derivative, which has demonstrated broad-spectrum activity against many pathogenic gram-negative and gram-positive bacteria. The bactericidal action of OF results from interference with enzyme DNA gyrase that is needed for the synthesis of bacterial DNA [11, 12].

In the system of Eu^{3+}–Gd^{3+}–Ofloxacin–SDBS, the Eu^{3+} could combine with OF to form complexes in the presence of SDBS and character the fluorescence of Eu^{3+} which can be increased by the addition of Gd^{3+}. The formation condition and the factors affecting the Eu–Gd–Ofloxacin–SDBS system were studied. This is a newly found fluorescence enhancement system of RE ion and can be used for the determination of ultra trace amounts of Eu^{3+}. Furthermore, this method is simple, high sensitive and good stability. In

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addition, the luminescence mechanism of the system was discussed.

Experimental

Apparatus

All fluorescence intensities were made on a F-2500 spectrofluorimeter (Hitachi, Japan). All pH measurements were made with a pHS-2F meter (Leici, Shanghai). All absorbance spectra are recorded with UV-2401spectrophotometer (Shimadzu, Japan).

Reagents

Stock standard solutions of rare earth ions (Eu^{3+} and Gd^{3+}) (1.00×10^{-2} mol$\cdot$L^{-1}) was prepared by dissolving the corresponding oxides (99.9%, China National Medicines Corporation, Ltd. Shanghai) in hydrochloric acid and heating until nearly dry, then diluting with doubly distilled water.

Stock solution of Ofloxacin (OF) (1.00×10^{-3} mol$\cdot$L^{-1}) was prepared by dissolving the appropriate amount of OF with water.

A 0.1 mol$\cdot$L^{-1} Tris-HCl buffer was prepared by dissolving of 6.06 g Tris in 500 mL volumetric flask with water and adjusting the pH to 7.8 with HCl.

Stock solution of SDBS (1.00×10^{-2} mol$\cdot$L^{-1}) was prepared by dissolving the appropriate amount of SDBS with doubly distilled water.

All the chemicals used were of analytical grade and doubly distilled water was used throughout.

Procedure

To a 10 mL test tube, buffer solution, Eu^{3+}, Gd^{3+}, OF and SDBS were added in that order. The mixture was diluted to 10 mL with distilled water and allowed to stand for 20 min. The fluorescence intensity of Eu^{3+} was measured in a 1 cm quartz cell with excitation and emission wavelengths of 280 nm and 612 nm, respectively. The excitation and emission slits were both 10 nm.

Results and discussion

Fluorescence spectral

The excitation and emission spectra of systems are shown in Fig. 1. From this figure, it can be seen that the fluorescence intensity of Eu^{3+} is weakly enhanced after adding OF, SDBS and Gd^{3+}. But the fluorescence intensity is greatly
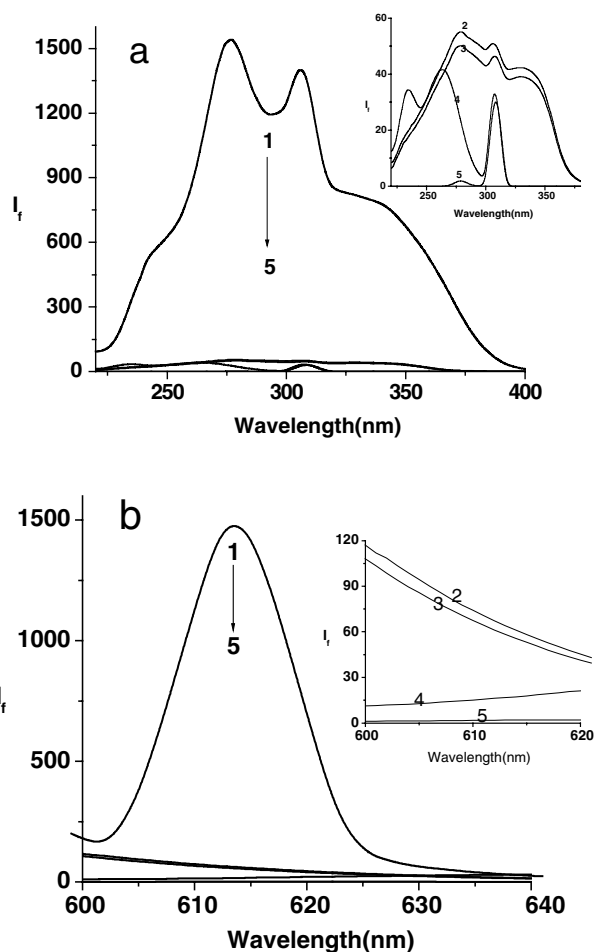
Fig. 1 Excitation spectra ($\lambda_{em} = 612$ nm) **a** and emission spectra ($\lambda_{ex} = 280$ nm) **b**. (1) Eu^{3+}–Gd^{3+}–OF–SDBS; (2) Eu^{3+}–Gd^{3+}–OF; (3) Eu^{3+}–OF;(4) Eu^{3+}–SDBS; (5) Eu^{3+}. Conditions: pH = 7.8; OF: 7×10^{-5} mol$\cdot$L^{-1}; SDBS: 3.6×10^{-4} mol$\cdot$L^{-1}; Gd^{3+}: 1.8×10^{-6} mol$\cdot$L^{-1}; Eu^{3+}: 1.0×10^{-7} mol$\cdot$L^{-1}

enhanced in the system of Eu^{3+}–Gd^{3+}–OF–SDBS. The excitation wavelength is located at 280 nm, the Eu^{3+}–Gd^{3+}–OF–SDBS system can emit intrinsic emission peaks of Eu^{3+} appeared at 612 nm, corresponding to the $^5D_0 \rightarrow {}^7F_2$ transitions of Eu^{3+}. So, the emission wavelength of 612 nm was selected for the further experiment.

Optimization of the general procedure

Effects of pH

Figure 2 shows the effect of pH on the fluorescence intensity of the system. The maximum fluorescence intensity obtained in the range of pH 7.0–8.5. In this paper, the Tris–HCl buffer for pH = 7.8 was chosen for assay and the optimum volume of buffer is 1.0 mL.
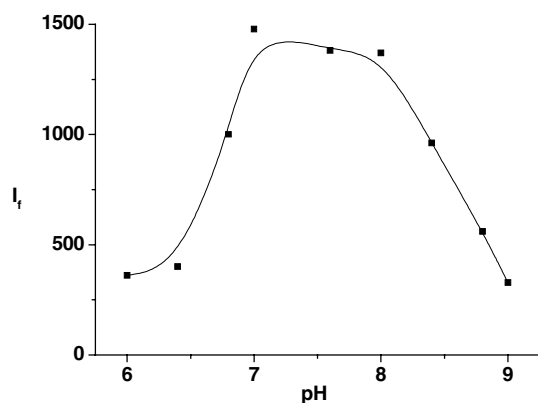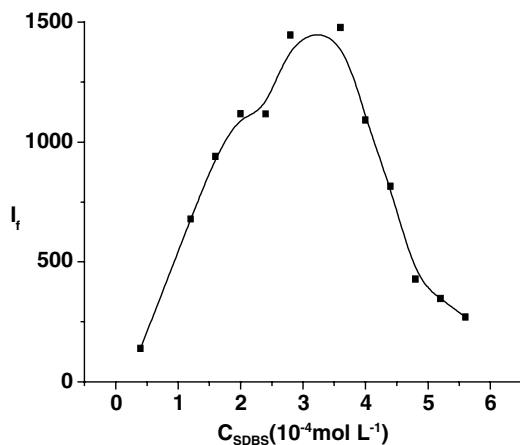
Fig. 2 The effect of pH on the fluorescence intensity. Conditions: OF: 7×10^{-5} mol $\cdot$ L^{-1}; SDBS: 3.6×10^{-4} mol $\cdot$ L^{-1}; Gd^{3+}: 1.8×10^{-6} mol $\cdot$ L^{-1}; Eu^{3+}: 1.0×10^{-7} mol $\cdot$ L^{-1}

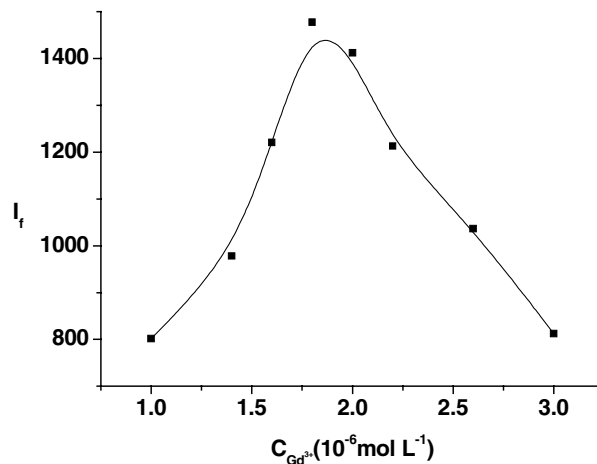*Effect of surfactants and its concentrations*

Different kinds of surfactants have different effects on the fluorescence intensity of the system. Some cationic surfactants CTAB, CPB and nonionic surfactant TX-100 have little affect on the system but some anionic surfactants SDS and SDBS could obviously increase the fluorescence intensity of the system. The test indicates that SDBS is the best in the surfactants studied. So, SDBS was selected for further experiments. The effect of its concentration on the fluorescence intensity is shown in Fig. 3. The maximum fluorescence intensity is obtained at 3.6×10^{-4} mol $\cdot$ L^{-1} of SDBS.

Effect of Gd^{3+} concentration

With the change in the concentration of Gd^{3+}, the corresponding change of fluorescence intensity of this system is shown in Fig. 4. It can be seen that the fluorescence inten-

Fig. 4 The effect of the concentration of Gd^{3+} on the fluorescence intensity. Conditions: pH = 7.8; OF: 7×10^{-5} mol $\cdot$ L^{-1}; SDBS: 3.6×10^{-4} mol $\cdot$ L^{-1}; Eu^{3+}: 1.0×10^{-7} mol $\cdot$ L^{-1}
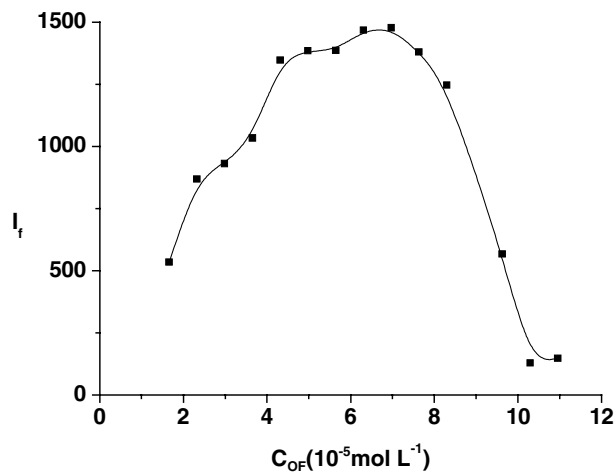
sity enhanced to a maximum at the concentration of Gd^{3+} is 1.8×10^{-6} mol $\cdot$ L^{-1}. So, in this paper, the concentration of Gd^{3+} for study is chosen as 1.8×10^{-6} mol $\cdot$ L^{-1}.

Effect of OF concentration

Effect of the OF concentration on the fluorescence intensity is shown in Fig. 5. The maximum fluorescence intensity is obtained at the concentration of 7.0–8.0×10^{-5} mol $\cdot$ L^{-1} OF. So the concentration of OF is fixed at 7.0×10^{-5} mol $\cdot$ L^{-1} in further experiments.

The addition order and stability of this system

Under the optimum condition, the effect of time on the fluorescence intensity was studied. The result showed that

Fig. 3 The effect of the concentration of SDBS on the fluorescence intensity. Conditions: pH = 7.8; OF: 7×10^{-5} mol $\cdot$ L^{-1}; Gd^{3+}: 1.8×10^{-6} mol $\cdot$ L^{-1}; Eu^{3+}: 1.0×10^{-7} mol $\cdot$ L^{-1}

Fig. 5 The effect of the concentration of OF on the fluorescence intensity. Conditions: pH = 7.8; SDBS: 3.6×10^{-4} mol $\cdot$ L^{-1}; Gd^{3+}: 1.8×10^{-6} mol $\cdot$ L^{-1}; Eu^{3+}: 1.0×10^{-7} mol $\cdot$ L^{-1}

Table 1 Interferences from foreign substances

Foreign substances	Concentration $(10^{-9} \text{ mol} \cdot \text{L}^{-1})$	ΔI_f (%)	Foreign substances	Concentration $(10^{-9} \text{ mol} \cdot \text{L}^{-1})$	ΔI_f (%)
K^+, Cl^-	20	0.91	Sm^{3+}	30	4.8
NH_4^+, Cl^-	20	−0.1	Tb^{3+}	8	3.20
Zn^{2+}, Cl^-	40	1.4	Dy^{3+}	3	4.56
Mn^{2+}, SO_4^{2-}	160	2.9	Tm^{3+}	2	3.46
Na^+, SO_4^{2-}	20	−0.07	Er^{3+}	5	4.76
K^+, NO_3^-	20	−1.98	Sc^{3+}	4	4.60
Ca^{2+}, Cl^-	20	−4.1	Nd^{3+}	2	5.20
Mg^{2+}, SO_4^{2-}	20	−2.17	Lu^{3+}	5	3.98
Fe^{3+}, SO_4^{2-}	20	5.7	Gd^{3+}	25	4.24
Fe^{2+}, SO_4^{2-}	60	2.7	La^{3+}	20	2.86
Y^{3+}	20	5.4	Dy^{3+}	100	3.81

Note. Conditions: pH = 7.8; OF: $7 \times 10^{-5} \text{ mol} \cdot \text{L}^{-1}$; SDBS: $3.6 \times 10^{-4} \text{ mol} \cdot \text{L}^{-1}$; Gd^{3+}: $1.8 \times 10^{-6} \text{ mol} \cdot \text{L}^{-1}$; Eu^{3+}: $1.0 \times 10^{-7} \text{ mol} \cdot \text{L}^{-1}$
$^a 10^{-8} \text{ g} \cdot \text{mL}^{-1}$.

the fluorescence intensity reached a maximum after all the reagents had been added and remained stable for over 1 h. Therefore, this system exhibits good stability.

Effect of foreign substances

The interference of foreign substances was tested at the concentration of Eu^{3+} is $8.0 \times 10^{-10} \text{ mol} \cdot \text{L}^{-1}$ and the results are shown in Table 1. It was found that most of metal ions and other lanthanides had little effect on the determination of Eu, under the permission of ±5% relative errors.

Analytical application

The calibration graph and detection limit

Under the optimum condition defined, the calibration graphs for Eu^{3+} are obtained and showed in Table 2. It can be seen that there is a linear relationship between the fluorescence intensity of the system and the concentration of Eu^{3+} in the range of 5.0×10^{-10} to $2.0 \times 10^{-7} \text{ mol} \cdot \text{L}^{-1}$, and the detection limit is $1.46 \times 10^{-10} \text{ mol} \cdot \text{L}^{-1}$.

Determination of synthetic sample

Because of the effects of other lanthanide ions on the fluorescence intensity of the system, the standard addition method is used for the determination of Eu^{3+} in samples. A synthetic sample, prepared based on the interference of foreign substances (Table 3) was analyzed. As Table 3 shows, the results are reproducible and reliable. It can be seen that the proposed method is suitable for the determination of trace amount of Eu^{3+}.

Interaction mechanism of the system

Formation of Eu^{3+}-Gd^{3+}-OF-SDBS complex

From Figure 1, it can be seen that when Gd^{3+}, OF and SDBS are together added to Eu^{3+}, the fluorescence intensity of the system is greatly enhanced. This indicates that there is the interaction among Gd^{3+}, OF, SDBS and Eu^{3+}.

The turbidity measurement shows that OF–SDBS solution remained transparent at the studied concentration. Particles are too small to produce strong light scattering. When Eu^{3+} is added into the system, the solution remains transparent, too. Once Gd^{3+} is added sequentially, it is found that the solution appears cloudy like diluted milk. This means that the larger complex particles are formed. RLS technique is available to provide some insight into the process responsible for the formation of the complex, which is shown in Fig. 6. It can be seen that RLS intensity is obviously enhanced when Eu^{3+} or Gd^{3+} are added into OF–SDBS system, indicating that Eu^{3+}, Gd^{3+} is effective in binding cations to form the complex with OF–SDBS due to the similar chemical properties of all lanthanide elements. As the concentration of Gd^{3+} is higher than that of Eu^{3+} and the number of whole complexes is increased, which causes Gd^{3+}–OF–SDBS the higher enhanced RLS intensity. From Fig. 6, it also can be seen that the intensity of RLS is greatly enhanced when Gd^{3+} is added compared to that of Eu^{3+}–OF–SDBS. This indicates that there exists a large congeries in Eu^{3+}–Gd^{3+}-OF–SDBS system. In order to predigest the discussion to understand it easily, we hypothesize that there are two complexes (Eu^{3+}–OF–SDBS and Gd^{3+}–OF–SDBS). The two complexes are in close proximity in the system, because SDBS possesses the tendency to self-assemble as pre-micelle

Table 2 Analytical parameters of this method

Linear regression equation	Linear range $(\text{mol} \cdot \text{L}^{-1})$	Correlation coefficient r	Limit of detection $(\text{mol} \cdot \text{L}^{-1})$
$I = 144.3 + 1.332 \times 10^{10}C$	5.0×10^{-10}–2.0×10^{-7}	0.9974	1.46×10^{-10}

Table 3 Determination of Eu^{3+} in the sample

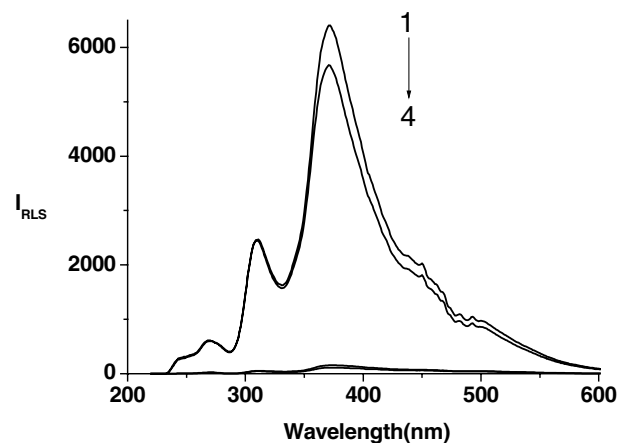| Added (%)	Environment (Oxides Presence, weigh %)	Found (%)	RSD (%) ($n = 5$)	Recovery (%)
Eu 0.25	La13.83 Ce0.455 Pr5.76 Nd14.43 Sm1.83 Gd0.42 Tb0.027 Dy0.257 Ho0.019 Yb0.015 Er0.022 Tm0.0076 Lu0.001 Y0.05 Fe < 0.01 Mg < 0.0005 Ca < 0.15 Si < 0.1 Th < 0.005	0.256	2.42	102.4

or micelles. Self-aggregation of anionic surfactants SDBS shortened the distance between these two ternary complexes and there are large numbers of Gd^{3+} complexes around Eu^{3+} complexes.

The energy transfer of the systems

Figure 7 shows that when excited at 280 nm, the emission peak of OF in the presence of SDBS is at 453 nm. When Eu^{3+} is added into OF–SDBS system, the latter's fluorescence decreased and the characteristic fluorescence peak of Eu^{3+} at 612 nm increased, indicating that the intra molecular energy transfer is happening from OF–SDBS to Eu^{3+}. Furthermore, the fluorescence intensity of Eu^{3+} can be greatly enhanced near 1000 times in the complex of Eu^{3+}–Gd^{3+}–OF–SDBS.

In order to know more details about this transfer, the energy transfer efficiency (E_a) and the interaction distance (r) between donor and acceptor can be evaluated using Förster theory [13, 14]:

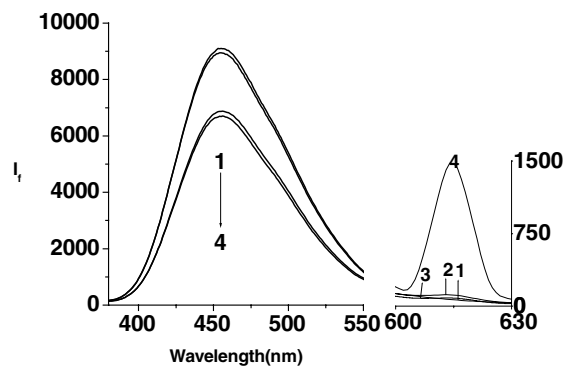$$E_a = 1 - \frac{I_{da}}{I_d} = \frac{R_0^6}{R_0^6 + r^6} \tag{1}$$

$$R_o^6 = 8.8 \times 10^{-25} \cdot k^2 \cdot n^{-4} \cdot \phi_d \cdot J \tag{2}$$

$$J = \int_0^\infty F(\lambda)\varepsilon(\lambda)\lambda^4 d\lambda / \int_0^\infty F(\lambda)d\lambda \tag{3}$$

where, R_0 is the critical transfer radius at which half of the energy is transferred; I_{da} and I_d are the fluorescence intensities of the donor with and without the acceptor, respectively; k^2 is a factor describing the relative orientation in space of the transition dipoles of donor and acceptor. In solutions of low viscosity, where rotation of the donor and the acceptor is sufficiently fast, an average value of $k^2 = 2/3$ may be assumed [15, 16]; φ_d is the fluorescence quantum yield of the donor (OF) in the absence of acceptor, which is determined based on the reference method with quinine sulfate as the criterion [17]; n is the refractive index of the medium and $n = 1.336$; J is the spectral overlap integral between the emission spectrum of donor and the absorption spectrum of acceptor. $F(\lambda)$ and $\varepsilon(\lambda)$ are the fluorescence intensity of the fluorescence donor (OF) and the molar absorption coefficient of the acceptor (Eu^{3+}), respectively. The values of E_a, R_0, and r are listed in Table 4.

From Table 4, it can be seen that in ternary complex of Eu^{3+}-OF-SDBS, the energy transfer efficiency is low. In the

Fig. 6 RLS spectra of the system. (1) Eu^{3+}–Gd^{3+}–OF–SDBS; (2) Gd^{3+}–OF–SDBS; (3) Eu^{3+}–OF–SDBS; (4) OF–SDBS. Conditions: pH = 7.8; OF: 7 × 10^{-5} mol·L^{-1}; SDBS: 3.6 × 10^{-4} mol·L^{-1}; Gd^{3+}: 1.8 × 10^{-6} mol·L^{-1}; Eu^{3+}: 1.0 × 10^{-7} mol·L^{-1}

Fig. 7 Emission spectra of the system. (1) OF–SDBS; (2) Eu^{3+}–OF–SDBS; (3) Gd^{3+}–OF–SDBS; (4) Eu^{3+}–Gd^{3+}–OF–SDBS. Conditions: pH = 7.8; OF: 7 × 10^{-5} mol·L^{-1}; SDBS: 3.6 × 10^{-4} mol·L^{-1}; Gd^{3+}: 1.8 × 10^{-6} mol·L^{-1}; Eu^{3+}: 1.0 × 10^{-7} mol·L^{-1}

Table 4 The efficiency of energy transfer (E_a) and the critical transfer radius (R_o)

| System	Donor	Acceptor	E_a	J (10^{-13} cm^6 M^{-1})	R_0 (nm)	r (nm)
Eu^{3+}–OF–SDBS	OF	Eu^{3+}	0.017	2.394	4.25	8.36
Eu^{3+}–Gd^{3+}–OF–SDBS	OF	Eu^{3+}	0.026	6.200	4.27	7.83

tetra complex of Eu^{3+}–Gd^{3+}–OF–SDBS, the energy transfer is more efficient than in ternary complex. It is considered that the distances between OF and Eu^{3+} in the tetra complex is decreased to 7.83 nm from 8.36 nm in ternary complex. Therefore, the energy transfer efficiency increases and the fluorescence intensity of Eu^{3+} is enhanced.

In the Gd^{3+}–OF–SDBS system, the characteristic emission of Gd^{3+} is not observed. This is probably due to the fact that Gd^{3+} possesses a relatively stable half-full 4f shell and the luminescence level of Gd^{3+} is higher than the triplet state of OF–SDBS in the Gd^{3+} complex, so the energy absorbed by OF–SDBS can not be transferred to Gd^{3+} by the intra molecular energy transfer. It can be seen from Fig. 7 that when Gd^{3+} is added into Eu^{3+}-OF-SDBS system the fluorescence of Eu^{3+} is greatly enhanced accordingly. Since the concentration of the Gd^{3+} complex is much greater than that of the Eu^{3+} complex and the distance between Gd^{3+}–OF–SDBS and Eu^{3+}–OF–SDBS complexes is short enough, Eu^{3+} in the Eu^{3+}–OF–SDBS can accept the energy from Gd^{3+}–OF–SDBS through intermolecular energy transfer in this system, resulting in the enhanced fluorescence of Eu^{3+} compared to the system in the absence of Gd^{3+}.

It is found that when the concentration of Eu^{3+} is 1.9×10^{-6} mol·L^{-1} in the Eu^{3+}–OF–SDBS system the fluorescence intensity of Eu^{3+} is still under that of Eu^{3+}–Gd^{3+}–OF–SDBS (the sum concentration of Eu^{3+} and Gd^{3+} is 1.9×10^{-6} mol·L^{-1}). This result shows that there are another factor improved the fluorescence intensity of Eu^{3+}–Gd^{3+}–OF–SDBS along with the intra and inter molecular energy transfer. It is asserted that in the Eu^{3+}–Gd^{3+}–OF–SDBS system, the concentration of the Gd^{3+} complex is much higher than that of the Eu^{3+} complex; each of Eu^{3+}–OF–SDBS complex molecule is surrounded by many Gd^{3+}–OF–SDBS complex molecules. This surrounding complex could act as an energy-insulating sheath, which could prevent collision with water molecules and decrease the energy loss of Eu^{3+}–OF–SDBS complex, thus improving the fluorescence quantum yield and the fluorescence intensity of Eu^{3+} enhanced.

On the basis of the results above mentioned, the conclusion for the fluorescence enhancement of the system comprises two effects: one is the intramolecular energy transfer from OF to Eu^{3+} in Eu^{3+}–OF–SDBS system and the intermolecular energy transfer from Gd^{3+}–OF–SDBS to Eu^{3+}–OF–SDBS; the other is that the increasing of the fluorescence quantum yield of Eu^{3+}.
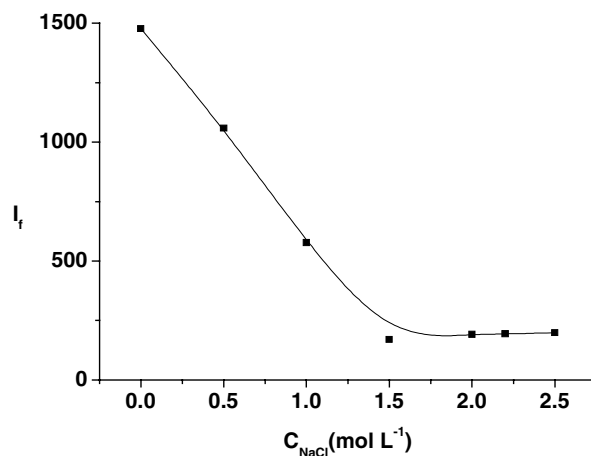
Fig. 8 The effect of the ionic strength on the fluorescence intensity. Conditions: pH = 7.8; OF: 7×10^{-5} mol·L^{-1}; SDBS: 3.6×10^{-4} mol·L^{-1}; Gd^{3+}: 1.8×10^{-6} mol·L^{-1}; Eu^{3+}: 1.0×10^{-7} mol·L^{-1}

The effect of the ionic strength

The influence of ionic strength on the fluorescence intensity of this system is tested as shown in Fig. 8. Experiments indicate that the fluorescence intensity of the system has obvious decreased when NaCl is added into the system. So, it is assumed that the interaction of Gd^{3+} or Eu^{3+} and OF–SDBS in this system forming the ternary complex is through electric attraction. At high concentration of salt, the fluorescence of this system decreased because charges could be screened and the interaction via electric attraction will be weakened.

Conclusion

In this paper, a new co-luminescence system Eu^{3+}–Gd^{3+}–OF–SDBS is found. Based on it, a method is established to determine Eu^{3+} sensitively and its detection limit reached as low as 1.46×10^{-10} mol·L^{-1}. The interaction mechanism of this system is studied. In Eu^{3+}–Gd^{3+}–OF–SDBS system, it is considered that there existed simultaneously two complexes: Eu^{3+}–OF–SDBS and Gd^{3+}–OF–SDBS. Because of the self-aggregation of SDBS, these two complexes are close to one another to form large complexes. Thus, intermolecular energy transfer is possible to enhance the fluorescence of Eu^{3+} through accepting energy from the Gd^{3+}–OF–SDBS besides of the intra molecule energy transfer from OF to

Eu^{3+} in the complex of Eu^{3+}–OF–SDBS. In addition, large numbers of complexes of Gd^{3+} surrounded the complex of Eu^{3+} to form an energy-insulating sheath, resulting in the prevention of collision with water molecules. Thus, the fluorescence quantum yield is improved and fluorescence of Eu^{3+} is significantly enhanced.

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