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Synthesis, characterization, photophysical and oxygen-sensing properties of a novel europium(III) complex

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1. Introduction

It is an important analytical problem to determine the molecular oxygen concentration in gas phase, liquid phase, or both in different branches of chemical and food industries, medicine, analytical chemistry, and environmental monitoring [1,2]. Traditionally, oxygen concentration measurements are based on the Clark electrode and the Winkler titration approach which suffer from drawbacks such as oxygen consumption during sensing process, long response times and the tendency of electrodes to be poisoned by sample constituents (e.g., H₂S, proteins, and certain anesthetics) [3,4]. In addition, both techniques necessitate the use of sophisticated instrumentations and require complicated pretreatment procedures, not suitable for on-line or in-field monitoring. Given these limitations, the search for new oxygen-sensing systems and their fabrication methods has attracted considerable interest in the past few decades.

Sensor technology is much simpler in instrumental implementation and sample preparation. Owing to the advantages of simple, rapid, and non-destructive characteristics, many oxygen-sensing systems have been reported. Among them, the development of optical oxygen sensors has attracted considerable attention in recent years, as such sensors can offer advantages in terms of size, electrical safety, costs, not requiring a reference element, and the fact that analytical signals is free of influence from electromag-

ABSTRACT

In this paper, we report the synthesis, characterization, crystal structure, and photophysical properties of a novel Eu³⁺ complex of Eu(DBM)₃IPD, where DBM=1,3-diphenyl-propane-1,3-dione and IPD=4-(1H-imidazo[4,5-f][1,10]phenanthrolin-2-yl)-N,N-diphenylaniline. Its elementary application for oxygen-sensing application is also investigated by doping it into a silica matrix of MCM-41. Experimental data suggest that the 20 mg/g doped Eu(DBM)₃IPD/MCM-41 system exhibits a high sensitivity of 3.6 towards molecular oxygen with a good linear relationship of R^2 = 0.9987. In addition, the 20 mg/g doped Eu(DBM)₃IPD/MCM-41 system owns a quick response of 8 s towards oxygen, along with its excellent atmosphere insensitivity and photobleaching resistance. All these results suggest that both Eu(DBM)₃IPD/MCM-41 systems are promising candidates for oxygen-sensing optical sensors.

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netic filed as well as easy to transmit over a long distance [5,6]. For practical applications in optical oxygen-sensing devices, it is necessary to embed sensors into a solid matrix acting as a supporting medium, allowing oxygen transportation from surroundings. The commonly used matrixes are silica-based materials, polymers, and Langmuir–Blodgett films. Indeed, the support may have quite stringent criteria for suitable performances [7]. For example, a high diffusion coefficient is necessary for a rapid response, while, a highly locally effective quenching around the sensor molecule is necessary for good sensitivity, and long distance on-line monitoring necessitates a high degree of photostability. Thus, the exploration for sensors with intense luminescence, excellent antijam ability, high photostability, and good compatibility with supporting matrix is still a challenge for analytical chemistry.

It seems that rare-earth (RE)-based emitters which are usually excellent emitters can well satisfy the above requirements. Due to the unique excitation mechanism of antenna effect and f-f radiative transition, RE³⁺ based emitters can generate characteristically sharp and narrow emissions without being affected by surrounding environment or reagents, offering an excellent antijam ability. In addition, RE³⁺ based emitters' good solubility in common solvents allow themselves to be easily loaded into supporting matrixes. All these excellent characters make RE³⁺ based emitters promising candidates for oxygen-sensing materials. Guided by above results, in this paper, we report the synthesis, characterization, crystal structure, and photophysical properties of a novel Eu³⁺ complex of Eu(DBM)₃IPD, where DBM = 1,3-diphenyl-propane-1,3-dione and IPD = 4-(1Himidazo[4,5-f][1,10]phenanthrolin-2-yl)-N,N-diphenylaniline. Its

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Scheme 1. A synthetic procedure for IPD diimine ligand, Eu(DBM)₃IPD, and Eu(DBM)₃IPD/MCM-41 oxygen-sensing systems.

elementary application for oxygen-sensing application is also investigated by doping it into a silica matrix of MCM-41. Experimental data suggest that the 20 mg/g doped Eu(DBM)₃IPD/MCM-41 system exhibits a high sensitivity of 3.6 towards oxygen with a good linear relationship of R^2 = 0.9987. In addition, the 20 mg/g doped Eu(DBM)₃IPD/MCM-41 system owns a quick response of 8 s towards oxygen, along with its excellent atmosphere insensitivity and photobleaching resistance. All these results suggest that both Eu(DBM)₃IPD and Eu(DBM)₃IPD/MCM-41 systems are promising candidates for oxygen-sensing optical sensors.

2. Experimental

2.1. Synthesis

A synthetic procedure for the diimine ligand of 4-(1Himidazo[4,5-f][1,10]phenanthrolin-2-yl)-N,N-diphenylaniline (IPD) and its corresponding Eu³⁺ complex with 1,3-diphenylpropane-1,3-dione (DBM) as the first ligand is shown in Scheme 1.

Triphenylamine (TPA), 1,10-phenanthroline (Phen), and 1,3diphenyl-propane-1,3-dione (DBM) were purchased from Aldrich Chemical Co. and used without further purification.

4-(Diphenylamino)benzaldehyde (TPA-CHO), 1,10phenanthroline-5,6-dione (Phen-O), and MCM-41 were synthesized according to the literature procedures [7,8].

IPD was synthesized as follows. A mixture of 2.1 g of Phen-O, 2.86 g of TPA-CHO, 15.4 g of NH₄Ac, and 30 mL of HAc was stirred at 90 °C for 4 h. Then the mixture was poured into cold water and extracted with CH₂Cl₂. The crude product was further purified by recrystallization from EtOH. ¹H NMR (300 Hz, CDCl₃): δ 9.17 (t, 2H), 7.72 (m, 2H), 7.62 (m, 2H), 7.33–7.14 (m, 14H).

Eu(DBM)₃IPD was synthesized according to the classic literature procedure [9]. Anal. Calcd. for $C_{76}H_{54}N_5O_6Eu$: C, 71.02; H, 4.23; N, 5.45. Found: C, 71.16; H, 4.34; N, 5.33. The identity of Eu(DBM)₃IPD was further confirmed by its single-crystal structure (CCDC 734798).

A typical synthetic procedure for $Eu(DBM)_3IPD/MCM-41$ systems is described as follows. $Eu(DBM)_3IPD$ was dissolved in 5 mL of CH₂Cl₂ to form a light yellow transparent solution, then 1 g of MCM-41 was added. The solution was stirred at room temperature for 5 h and filtered to give a yellowish powder. The crude powder was washed with CH₂Cl₂ and dried under N₂ atmosphere at room temperature.

2.2. Methods and measurements

Single-crystal data were collected on a Siemens P4 single-crystal X-ray diffractometer with a Smart CCD-1000 detector and graphitemonochromated Mo K α radiation, operating at 50 kV and 30 A at 298 K. Elemental analysis was performed on a Carlo Erba 1106 elemental analyzer. Luminescence lifetimes were obtained with a 355 nm light generated from the third-harmonic-generator pump, using a pulsed Nd:yttrium aluminium garnet (YAG) laser as the excitation source. The Nd:YAG laser possesses a line width of 1.0 cm⁻¹, a pulse duration of 10 ns, and a repetition frequency of 10 Hz. All photoluminescence (PL) spectra were measured with a F-4500 fluorescence spectrophotometer. In the measurement of Stern-Volmer plots, oxygen and nitrogen were mixed at different concentrations via gas flow controls and passed directly into sealed gas chamber. UV-vis absorption spectra were recorded using a HP 8453 UV-Vis-NIR diode array spectrophotometer. ¹H NMR spectra were obtained with the use of a Varian INOVA 300 spectrometer. All measurements were carried out in the air at room temperature without being specified.

3. Results and discussion

3.1. Crystal structure of Eu(DBM)₃IPD

Fig. 1 shows the molecular structure obtained from $Eu(DBM)_3IPD$ single crystal. Clearly, the Eu^{3+} adopts a square antiprism coordination sphere. Selected geometric parameters listed in Table 1 suggest that the central Eu^{3+} is coordinated by



Fig. 1. ORTEP drawing of Eu(DBM)₃IPD. All hydrogen atoms are not shown for clarity.

six oxygen atoms from three DBM molecules with Eu-O bond lengths ranging from 2.347 Å to 2.368 Å and two nitrogen atoms from one IPD molecule with Eu-N bond lengths ranging from 2.572 Å to 2.583 Å. It is found that the Eu-N coordination bond lengths are obviously longer than the corresponding bond lengths in a tetrahedral coordination environment (~ 2.1 Å), such as in Cu(I) complexes [10,11]. In addition, the N–Eu–N bite angle in Eu(DBM)₃IPD is also much smaller than the corresponding bite angle in a tetrahedral coordination environment ($\sim 80^{\circ}$) [10,11]. The crowded coordination environment around Eu³⁺ center may further distort the coordination sphere, leading to the abovementioned longer Eu-N bond lengths and smaller N-Eu-N bite angle. In DBM moiety, the average bond lengths of C-C and C-O are 1.348 Å and 1.243 Å, respectively, lying between typical single- and double-bond distances, reflecting the delocalization of π -electrons in the chelate ring system. As for IPD moiety, it is observed that there is a distortion between Phen and TPA planes, with a dihedral angle of 31.06° between Phen and TPA planes, making the conjugation between Phen and TPA moieties somewhat inefficient.

3.2. UV-vis and excitation spectra of Eu(DBM)₃IPD

Fig. 2 shows the UV–vis absorption and excitation spectra of Eu(DBM)₃IPD in CH₂Cl₂ solution with a concentration of 1×10^{-4} mol/L. Eu(DBM)₃IPD renders a broad absorption band centering at 350 nm which is attributed to $\pi \rightarrow \pi^*$ transition of DBM moiety [9]. Eu(DBM)₃IPD excitation spectrum ranging from 300 nm to 420 nm fits well with its absorption spectrum as shown in Fig. 2, with a maximum of 352 nm. It is observed that the optimal excitation window of Eu(DBM)₃IPD overlaps the emission region of commercially available ultraviolet InGaN/GaN light-emitting diodes (LEDs), suggesting that Eu(DBM)₃IPD based optical sensors

Table	21

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selecteu	geometric	parameters	OI EU	DDIVI 31PD

Bond length	(Å)	Bond angle	(°C)
Eu-O1 Eu-O2 Eu-O3 Eu-O4 Eu-O5	2.373 2.360 2.362 2.347 2.355	01–Eu–02 03–Eu–04 05–Eu–06 N1–Eu–N2	70.86 71.10 71.61 63.63
Eu–O6 Eu–N1 Eu–N2	2.347 2.583 2.572		

can use ultraviolet InGaN/GaN LEDs as excitation sources, which is much helpful to further simplify instrumental implementation. Considering that the excitation spectrum extends to visible region of 420 nm, we may choose lower-energy LEDs to depress photobleaching phenomenon which is defined as the photochemical destruction of a fluorophore.

3.3. PL properties of Eu(DBM)₃IPD

Fig. 2 shows the PL spectra of Eu(DBM)₃IPD in solid state under pure N₂ and O₂ atmospheres upon excitation wavelength of 350 nm. No surprise, characteristic lines from Eu³⁺ center peaking at 578 nm for ${}^5D_0 \rightarrow {}^7F_0$, 590 nm for ${}^5D_0 \rightarrow {}^7F_1$, and 610 nm for ${}^5D_0 \rightarrow {}^7F_2$ are observed. Even though the emission intensity under N₂ atmosphere (intensity = 1.00 at 610 nm) is nearly two times stronger than that under O₂ atmosphere (intensity = 0.51 at 610 nm), their emission band shapes are exactly the same, along with the identical emission peak, which can be explained as follows. Eu³⁺ emissions originate from *f*-*f* radiative transitions, and the partially filled 4f orbitals are perfectly shielded from environmental influence by the filled 5d and 5p orbitals, leading to the immunity of emissions to various atmospheres. Since sensor tech-



Fig. 2. Absorption and excitation spectra recorded at room temperature of Eu(DBM)₃IPD in CH₂Cl₂ solution with a concentration of 1×10^{-4} mol/L, along with PL spectra of Eu(DBM)₃IPD in solid state under pure N₂ and O₂ atmospheres upon excitation wavelength of 350 nm. Inset: luminescence decay curves of ${}^{5}D_{0} \rightarrow {}^{7}F_{2}$ transition under pure N₂ and O₂ atmospheres.



Fig. 3. SAXRD curves of pure MCM-41 and $Eu(DBM)_3IPD/MCM-41$ systems with various doping concentrations. Inset: absorption spectra of $Eu(DBM)_3IPD/MCM-41$ systems.

nology works on the basis of emission intensity measurements at a certain wavelength, the immunity to various atmospheres of emission peak makes Eu(DBM)₃IPD a promising candidate for oxygen sensors.

Correspondingly, the luminescence decay data shown by the inset of Fig. 2 confirm that Eu(DBM)₃IPD emission is sensitive towards O₂: the $^5D_0 \rightarrow {}^7F_2$ lifetime decreases largely from 590 μs under pure N_2 atmosphere to 400 µs under pure O_2 atmosphere. According to the previous report, there are three potential intermolecular energy transfer mechanisms in RE(III) complexes [12]. The one that seems to agree with most of the experimental data is described as follows. After an efficient intersystem crossing between the lowest singlet and triplet excited states of the ligand, energy transfer from triplet excited state of the ligand to a lowerenergy state of the RE(III) ion, leading to the *f*-*f* radiative decay of central RE(III) ion. Even though the intermolecular energy transfer experiences a ligand's triplet state which is generally believed to be vulnerable to O_2 , the energy transfer process is so fast that the triplet state is hardly to be quenched by O₂ [13]. Thus, combined with the largely decreased ${}^5D_0 \rightarrow {}^7F_2$ lifetime of Eu³⁺ center, we come to a conclusion that O₂ molecules directly quench the excited state Eu³⁺ center instead of excited state ligands. The quenching mechanism is then assigned as a dynamic one, and can be described as follows:

 $\operatorname{Eu}(\operatorname{III})^* + \operatorname{O}_2 \to \operatorname{Eu}(\operatorname{III}) + \operatorname{O}_2^* \tag{1}$

where "*" denotes an excited state.

3.4. Oxygen-sensing performances of Eu(DBM)₃IPD/MCM-41 systems

Oxygen-sensing performances of Eu(DBM)₃IPD are initially tested by physically incorporating it into mesoporous silica MCM-41 (referred as Eu(DBM)₃IPD/MCM-41) with various loading concentrations of 10 mg/g, 20 mg/g, 30 mg/g, respectively. Their oxygen-sensing performances are discussed on the basis of luminescence intensity quenching.

3.4.1. Confirmation of sensor systems

As shown in Fig. 3, powder small angle X-ray diffraction (SAXRD) measurements reveal that blank MCM-41 owns three well-resolved broad Bragg reflections that can be indexed as d_{100} , d_{110} , and d_{200} , which is the characteristic of a well-ordered hexag-

onal mesostructure [14]. SAXRD curves of the composite materials shown in Fig. 3 display an identical pattern with that of MCM-41, indicating that the hexagonal arrangement of channels in MCM-41 remains after the incorporation procedure. The absorption spectra of Eu(DBM)₃IPD/MCM-41 systems in CH₂Cl₂ exhibit characteristic absorption peaks of Eu(DBM)₃IPD as shown by the inset of Fig. 3. Combined with the characteristic emission lines of Eu(DBM)₃IPD/MCM-41 systems which will be discussed later, it is safe to say that Eu(DBM)₃IPD is successfully incorporated into MCM-41 matrix.

3.4.2. Sensitivity

The sensitivity (I_0/I_{100}) , where I_0 is the luminescence intensity under pure N_2 atmosphere and I_{100} is that under pure O2 atmosphere) values of Eu(DBM)3IPD/MCM-41 systems with various loading concentrations are 2.7 for 10 mg/g doped Eu(DBM)₃IPD/MCM-41, 3.6 for 20 mg/g doped Eu(DBM)₃IPD/MCM-41, and 3.4 for 30 mg/g doped Eu(DBM)₃IPD/MCM-41, respectively. Sensitivity values of Eu(DBM)₃IPD/MCM-41 systems are found to be larger than that of pure Eu(DBM)₃IPD in solid state (1.00/0.51 as mentioned), suggesting that MCM-41 provides an excellent supporting matrix for Eu(DBM)₃IPD molecules. The 20 mg/g doped Eu(DBM)₃IPD/MCM-41 system exhibits a higher sensitivity than the others, which means that the sensitivity is not simply proportional to the loading concentration. We are giving an explanation as follows. There may be at least two opposite factors affecting sample sensitivity: emission intensity from probe molecules and adverse interaction between probe molecules (aggregation, for example) [7,14]. When loading concentration is low, emission from the probe is weak, leading to a low sensitivity. On the other hand, a much high loading concentration may accelerate the intermolecular aggregation which also decreases the sensitivity. The two opposite factors may achieve a balance in the 20 mg/g doped Eu(DBM)₃IPD/MCM-41 system, showing the highest sensitivity of 3.6.

Despite the long ${}^{5}D_{0} \rightarrow {}^{7}F_{2}$ lifetime of Eu³⁺ center (590 µs), the sample's sensitivity is lower than those of sensors based on Ru(II) and Cu(I) complexes whose excited lifetimes are shorter than 100 µs [15,7]. We believe the luminescence mechanism difference between Eu(III)-based and Cu(I)-based emitters should be responsible for this phenomenon. As for a Eu(III)-based emitter, the emission originates from metal-centered (MC) *f*-*f* transitions. The emissive center is thus covered by surrounding ligands and the outer orbitals of Eu(III) ion, which prevents molecular oxygen from closing in, leading to the low sensitivity. On the other hand, as for a typical Cu-based emitter, the highest occupied molecular orbital has a predominant Cu d character, while the lowest unoccupied molecular orbital is essentially π^* orbital localized on diimine ligand. The photoluminescence corresponds to the lowest triplet T₁ and is thus assigned as a character of metal-to-ligand-chargetransfer ³MLCT [d(Cu) $\rightarrow \pi^*$ (diimine ligand)] [10]. The excited state electron localizes on diimine ligand and thus is open for molecular oxygen attack, leading to a high sensitivity, even though the excited state lifetime is shorter than that of the Eu(III)-based emitter.

3.4.3. Stern–Volmer plots

The emission spectra of 20 mg/g doped Eu(DBM)₃IPD/MCM-41 system under various oxygen concentrations from 0% to 100% are presented in Fig. 4. With increasing oxygen concentrations, the luminescence intensity at 610 nm decreases smoothly with no affection on emission band or peak. It has been reported that the quenching behavior of a probe is affected by the microstructure of matrix and micro-environment in which the probe is located. In a homogeneous media with a single-exponential decay, the intensity form of Stern–Volmer equations with dynamic quenching is



Fig. 4. Emission spectra recorded at room temperature of 20 mg/g doped Eu(DBM)₃IPD/MCM-41 system under various oxygen concentrations from 0% to 100% with an interval of 10%.



Fig. 5. Stern–Volmer plots of 20 mg/g doped Eu(DBM)₃IPD/MCM-41 system at various oxygen concentrations. Inset: PL intensity response of 20 mg/g doped Eu(DBM)₃IPD/MCM-41 system when exposed to periodically varied 100% N₂ and 100% O₂ atmospheres.

described as follows [16]:

$$\frac{I_0}{I_{100}} = 1 + K_{SV}[O_2] \tag{2}$$

where *I* is luminescent intensity. The subscript 0 denotes a value in the absence of quencher, K_{SV} is the Stern–Volmer constant, and $[O_2]$ is O_2 concentration. A plot of I_0/I_{100} versus $[O_2]$ should be linear with identical slopes of K_{SV} .

Fig. 5 shows the Stern–Volmer plots of 20 mg/g doped Eu(DBM)₃IPD/MCM-41 system at various oxygen concentrations. It is observed that all plots fit well with Eq. (1) and exhibit good linear relationship ($R^2 = 0.9987$) with increasing oxygen concentration, indicating that the micro-environment around Eu(DBM)₃IPD molecules is homogenous and Eu(DBM)₃IPD molecules are uniformly distributed within MCM-41 matrix.

3.4.4. Response time and photostability

The inset of Fig. 5 demonstrates a PL intensity response of 20 mg/g doped Eu(DBM)₃IPD/MCM-41 system when exposed to periodically varied 100% N₂ and 100% O₂ atmospheres. Here, we define 95% response time as the time taken for 20 mg/g doped Eu(DBM)₃IPD/MCM-41 system to lose 95% of its initial

emission intensity when changed from 100% N₂ atmosphere to 100% O₂ atmosphere, and 95% recovery time as the time taken when changed from 100% O₂ atmosphere to 100% N₂ atmosphere. Correspondingly, 20 mg/g doped Eu(DBM)₃IPD/MCM-41 system renders a response time of only 8s and a recovery time of 15s. The quick response towards O_2 and N_2 suggests that 20 mg/gdoped Eu(DBM)₃IPD/MCM-41 system is highly sensitive towards O₂. In addition, it is found that the recovery time is obviously longer than the response time, which can be explained by the diffusion-controlled dynamic response and recovery behavior of a hyperbolic-type sensor reported by Mills and Lepre [17]. 20 mg/g doped Eu(DBM)₃IPD/MCM-41 system exhibits reversible time response plots, with slight intensity decrease which is called photobleaching. For future studies, we are hoping to further improve the photostability and eliminate photobleaching by covalently grafting the probe into silica matrix.

4. Conclusion

In this paper, we report the synthesis, characterization, crystal structure, and photophysical properties of $Eu(DBM)_3IPD$. Its elementary application for oxygen-sensing application is also investigated by doping it into a silica matrix of MCM-41. Experimental data suggest that the 20 mg/g doped $Eu(DBM)_3IPD/MCM-41$ system exhibits a high sensitivity of 3.6 towards oxygen with a good linear relationship of $R^2 = 0.9987$. In addition, the 20 mg/g doped $Eu(DBM)_3IPD/MCM-41$ system owns a quick response of 8 s towards oxygen, along with its excellent atmosphere insensitivity and photobleaching resistance. All these results suggest that both $Eu(DBM)_3IPD$ and $Eu(DBM)_3IPD/MCM-41$ systems are promising candidates for oxygen-sensing optical sensors.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.saa.2010.05.025.

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