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Sensitized near-infrared luminescence from erbium ion-associated complex with IR140 dye

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ABSTRACT

In this study, the near-infrared luminescence emission of erbium ions is enhanced by 6-folds in intensity with an ion-associated erbium complex consisting of an IR140 dye cation. The energy-transfer process from the IR140 dye cation to the erbium ion has been modeled. Near-infrared luminescence of the erbium ion is sensitized intra-molecularly by the IR140 dye cation effectively, although the residual IR140 dye emission is still observed, indicating an incomplete energy transfer between the IR140 dye cation and the erbium ion. The main path of energy-transfer process is through the $T_1 \rightarrow {}^4I_{11/2}$ transition with the operative mechanism to be Förster type.

1. Introduction

Near-infrared luminescence from organic lanthanide complex drives considerable interest in fields such as organic light emitting diodes and integrated circuits for optical communications. Among the contributions to develop high-efficiency near-infrared emitting lanthanide-related materials, many methods have been explored including one of the most common way by which the organic molecule with much higher absorption coefficient is adopted for sensitization of lanthanide ion's near-infrared luminescence [1–14]. Under this idea, organic dyes with much higher absorption coefficient in specific spectral ranges have been applied for sensitizing the near-infrared emission of lanthanide ions, such as Er^{3+}, Nd^{3+} and Yb^{3+} [6–9,15–17]. Sensitizing processes have been grafted adequately [7,18,19], indicating these systems as potential luminescent active substances for optical materials. It has been realized to enhance luminescence properties of erbium ions by employing a low-cost semiconductor laser diode at 980 nm as the excitation source for the IR5 dye [1,6]. However, IR5 is somewhat fragile that even thermal-degradation may play an important role on the photostability and excitation in other spectral regions should also be investigated. As a further aim to use excitation sources at other wavelengths, a novel ion-association complex with enhanced near-infrared luminescent properties will be introduced in this work. Here, a near-infrared dye, IR140 dye salt (5,5′-dichloro-11-diphenylamine-3,3′-diethyl-10,12-ethylene-thiatricarbocyanine-perchlorate) is adopted as a sensitizer for erbium ion's near-infrared luminescence. It's already well-known that IR140 dye has the peak absorption cross-section at around 800 nm [20–22]. Thus, it is possible to enhance luminescence properties of erbium ions at around 1500 nm by utilizing an 800 nm laser diode. Hence, an ion-associated erbium complex with IR140 dye, $Er(TTA)_4(IR140)$ (TTA, 2-theonyltrifluoroacetone), is proposed to be synthesized together with its gadolinium analogue $Gd(TTA)_4(IR140)$ for comparison.

2. Experimental section

The chemical structures for ion-associated complexes are depicted in Fig. 1. The synthesis and characterization of these complexes are similar as that presented in the literature [6]. Crystal Structure data of IR140 dye salt can be available online (CCDC 661219 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif). Measurements of absorption spectra were performed on a Hitachi U-4100 spectrometer with a spectral resolution of 1 nm. Near-infrared photoluminescence spectra were determined at room temperature by using a spectrometer equipped with a monochromatic (Spex 1269, SPEX Industries Inc. USA) and a liquid-nitrogen-cooled Ge detector (EO-817L, Yellow River Systems Inc. USA). The integral time was aptly selected as 0.8 s and the spectral resolution is 2 nm. Phosphorescence measurement was

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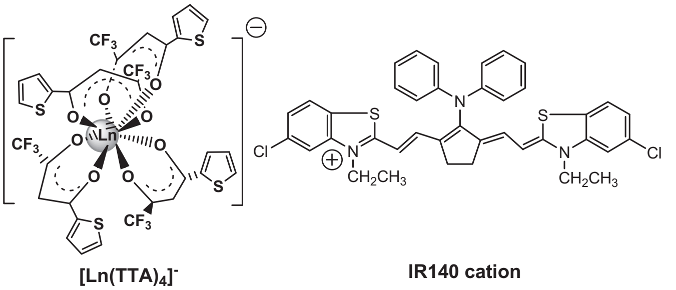

Fig. 1. Chemical structures of Ln(TTA)4(IR140) (Ln = Er, Gd).

performed at 77 K cooled by liquid nitrogen. The excitation source was a domestic semiconductor laser diode at the wavelength of 800 nm.

3. Results and discussion

Absorption spectra of IR140 dye salt and $Ln(TTA)_4(IR140)$ (Ln = Er, Gd) in various solvents are given in Fig. 2. The absorption peak of IR140 dye salt blue-shifts from 818 nm in benzene (lower polarity), to 816 nm in H_2CCl_2 and 808 nm in ethanol (higher polarity). Absorption peaks for ion-associated complexes $Gd(TTA)_4(IR140)$ and $Er(TTA)_4(IR140)$ exhibit similar changes. The absorption band around 600–900 nm with the 818 nm peak of the IR140 cation in benzene can be deconvoluted into two components: one at ~780 nm assigned to the mono-cis isomer and the other ~820 nm corresponding to all-trans isomer [23,24]. Moreover, in solvents with lower polarity, IR140 cations tend to couple with its negatively charged counter-ion, which would force the equilibrium toward all-trans isomer due to steric hindering, whilst in higher polarity solvents, unpaired cations adopt mono-cis conformation more easily [25,26].

In nonpolar benzene, anions associate with anions by nucleophilic attack of cations on their positive charge centers, and positive charges are localized onto heteroatoms (here, nitrogen atoms). Due to higher electronegative and nucleophilicity of Fluorine atoms in $[Gd(TTA)_4]^-$ or $[Er(TTA)_4]^-$ than Oxygen atoms in ClO_4^-, $[Gd(TTA)_4]^-$ or $[Er(TTA)_4]^-$ are more likely to form ion-pairs, which reinforce their nucleophilic attacking of cations. Subsequently, this results in more delocalization of positive charge along polymethine chain and then more reduction on the first singlet level's energy of dye cations (bathochromic shift) [27–29]. As shown in Fig. 2, the absorption peaks of $Er(TTA)_4(IR140)$ or $Gd(TTA)_4(IR140)$ red-shifts ~2 nm in comparison with that of IR140 dye salt (see Fig. 2a), which demonstrates the intramolecular anion's effect on dye cation exactly. In Fig. 2b, the ion-pair stability of $Er(TTA)_4(IR140)$ in benzene is verified with little changes on the feature of absorption spectra at various concentrations.

In polar solvents such as CH_2Cl_2 or ethanol, $[Ln(TTA)_4]^-[IR140]^+$ tends to dissociate with increasing polarity, consequently the polymethine chain of cation retorts relatively while the interaction between anions and cations weakens. And $[Ln(TTA)_4]^-$ can be seen as a kind of solvent with higher polarity than that of CH_2Cl_2 or ethanol. Thus, the blue-shift of absorption peaks are observed, as that blue-shifts from 816 nm for IR140 dye in CH_2Cl_2 to 809 nm for $Gd(TTA)_4(IR140)$ and 809.5 nm for $Er(TTA)_4(IR140)$ respectively, and from 808 nm for IR140 dye salt in ethanol to 807 nm for $Gd(TTA)_4(IR140)$ and 807 nm for $Er(TTA)_4(IR140)$ respectively (see Fig. 2c and d). These results demonstrate the formation of ion-associated complexes.

Fig. 3 has illustrated a reduction on the fluorescence intensity in the sequence of IR140 dye salt, $Gd(TTA)_4(IR140)$ and $Er(TTA)_4(IR140)$ in *d*-chloroform. This reduction originates from

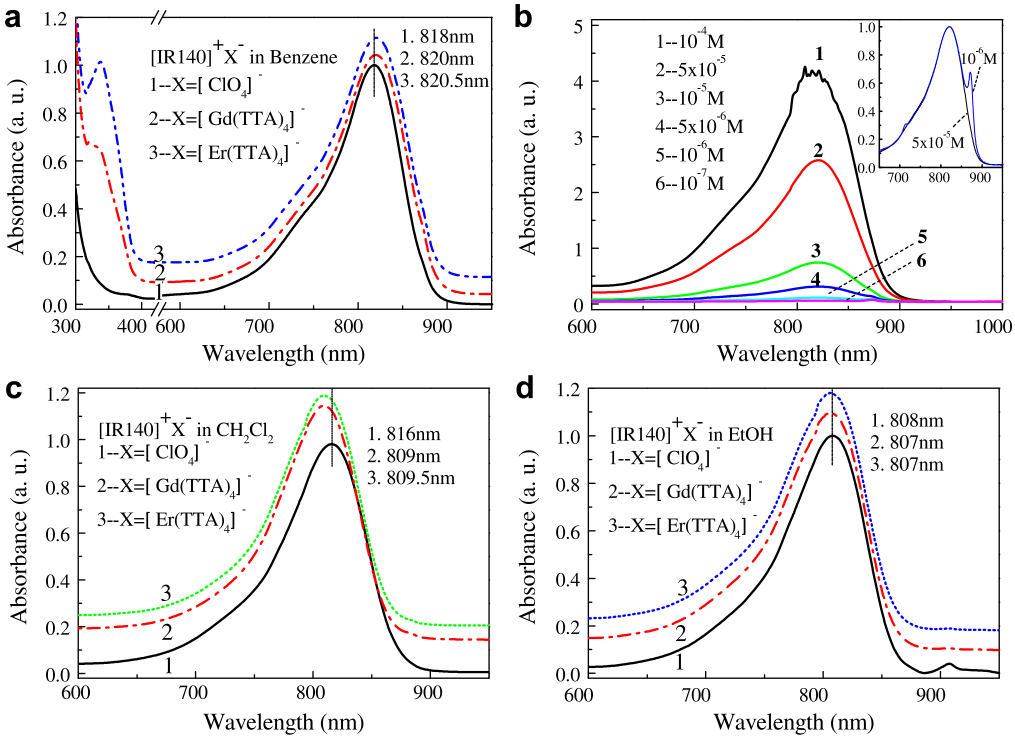

Fig. 2. (a) Absorption spectra of IR140 dye salt, $Gd(TTA)_4(IR140)$ and $Er(TTA)_4(IR140)$ dissolved in benzene (1×10^{-5} M). (b) Dependence of absorption on the concentration for $Er(TTA)_4(IR140)$ dissolved in benzene (1×10^{-7}–1×10^{-4} M). The inset is the normalization of absorption spectra for 5×10^{-5} M and 1×10^{-6} M benzene solution. Absorption spectra of IR140 dye salt, $Gd(TTA)_4(IR140)$ and $Er(TTA)_4(IR140)$ dissolved in CH_2Cl_2 (1×10^{-5} M) (c) And ethanol (1×10^{-5} M) (d).

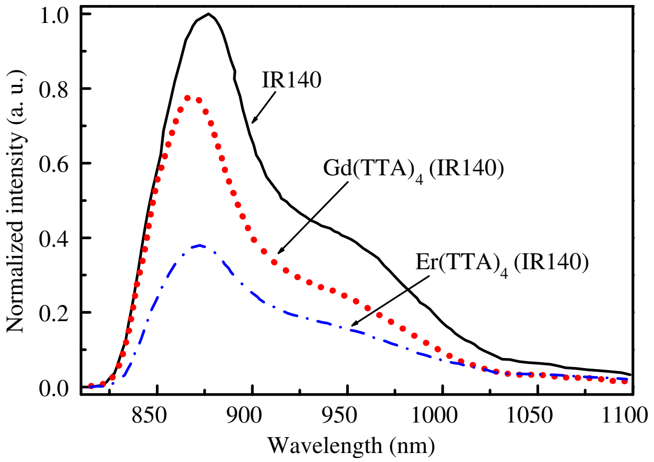

Fig. 3. Normalized near-infrared fluorescence spectra of IR140 dye salt, Gd(TTA)$_4$(IR140) and Er(TTA)$_4$(IR140) dissolved in *d*-chloroform (1 × 10^{-4} M) Excited at 800 nm respectively.

the external heavy atom effect which can be induced by a heavy and paramagnetic lanthanide ion in close proximity of IR140 cation. The nearby lanthanide ion can increase the intersystem crossing yield of the dye cation. This effect enhances not only spin-orbit coupling of the system which relaxes the selection rules for electronic transitions, but also exchange interaction of lanthanide ion-unpaired electrons with the π-electrons of the dye cation according to Tobita et al [30–32]. The main fluorescence peaks with shoulders locate at 878, 869 and 873 nm respectively, which is decided by relative fluorescence intensity from both mono-cis and all-trans conformation of IR140 cations [25]. In the case of Gd(TTA)$_4$(IR140) and Er(TTA)$_4$(IR140), fluorescence intensity from mono-cis conformation of IR140 cation is weakened relatively, then their main peaks blue-shift because of the limitation on torsion of polymethine chains with the presence of anions. This result is also consistent with that obtained from IR140 dye salt doped into poly(methyl methacrylate) (PMMA) [33].

Singlet state energy of 11,750 cm^{-1} for IR140 cation moiety in Gd(TTA)$_4$(IR140) is estimated from the spectra presented in Fig. 4 by the crossing of the absorption and fluorescence spectra [8]. Gd(TTA)$_4$(IR140) in a *d*-chloroform glass at 77 K shows strong phosphorescence band next to a very weak fluorescence band. Since Gd^{3+} ion has no energy level below 32,000 cm^{-1}, Gd^{3+} ion cannot accept any energy from IR140 cation's triplet state. The triple state energy of 11,150 cm^{-1} for the IR140 cation is determined from the 0-0 transition in the phosphorescence spectrum (see Fig. 4) [21,22].

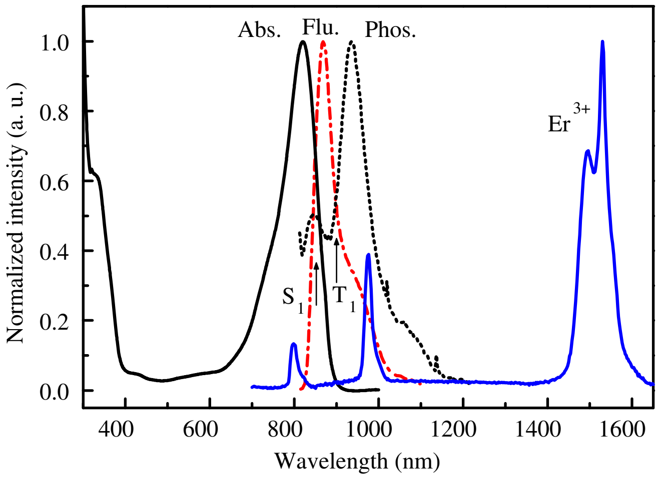

Fig. 4. Normalized absorption spectrum of Gd(TTA)^4(IR140) dissolved in *d*-chloroform (1 × 10^{-5} M). Normalized near-infrared fluorescence spectrum of Gd(TTA)$_4$(IR140) in *d*-chloroform solution (1 × 10^{-4} M) and phosphorescence spectrum of Gd(TTA)$_4$(IR140) in solid *d*-chloroform solution at 77 K excited at 800 nm respectively, together with the absorption spectrum of Er^{3+}. Arrows indicate the positions for S$_1$ and T$_1$.

To make sure whether it is possible to realize energy transfer from the dye cation to the erbium-centered anion, it is necessary to estimate the distance between centers of $[Er(TTA)_4]^-$ and $[IR140]^+$. Since it is difficult to obtain X-ray crystallography parameters of Er(TTA)$_4$(IR140) for its single crystal is not so easy to realize, the distance is indirectly estimated from the disassociation constant K_D of Er(TTA)$_4$(IR140) in different solvents, according to the method introduced by Schuster et al. [33]. Accordingly, the conductivity of $[Er(TTA)_4]^-[IR140]^+$ solutions in a series of solvents with different dielectric constants have been measured (not given here), and the derived K_D values were obtained. Then according to the relation equation (Fuoss equation) between K_D and the solvent dielectric constant ε [33], the natural log of K_D plots against the reciprocal of ε. The slope of the line provides the center-to-center distance between $[Er(TTA)_4]^-$ and $[IR140]^+$ in $[Er(TTA)_4]^-[IR140]^+$ ion-pair, with the value estimated as 9 ± 2 Å. According to crystallology data from IR140 dye salt's single crystal, the distances between central chlorine atom of ClO_4^- and 3 nitrogen atoms, 2 carbon atoms and 2 sulfur atoms of IR140 cation are 16.90, 14.40, 12.42, 13.74, 12.98, 16.53 and 13.00 Å respectively, and the average value is estimated as 13.7 Å. The difference of two values is due to the presence of CF_3 groups in TTA ligands and different diameters between $[Er(TTA)_4]^-$ and ClO_4^-.

As shown in Fig. 5, near-infrared photoluminescence spectrum of bulky-powdered Er(TTA)$_4$(IR140) with that of Er(TTA)$_4$K for comparison has been obtained at room temperature by using a 800 nm semiconductor laser diode as the excitation source. The emission peak around 1500 nm is corresponding to the transition $^4I_{13/2} \rightarrow {}^4I_{15/2}$ of erbium ion. It should be noted that difference between the formulas for two complexes is that Er(TTA)$_4$(IR140) has ion-associated with IR140 dye cation, which leads to the 6-fold enhancement on the 1500 nm emission in comparison with that of Er(TTA)$_4$K (see Fig. 5). When excited at 800 nm, the direct excitation through $^4I_{9/2}$ for Er(TTA)$_4$K will take place because the absorption of the TTA ligands is far beyond 800 nm. While for Er(TTA)$_4$(IR140), IR140 cation has 4 orders of magnitude larger absorption coefficient than that of erbium ion at around 800 nm. Therefore, most of excitation photons are absorbed by IR140 cation segment in Er(TTA)$_4$(IR140) and indirect excitation of erbium ion via IR140 cation is expected. The fluorescence intensity descending from Gd(TTA)$_4$(IR140) to Er(TTA)$_4$(IR140) as shown in Fig. 3 can also be regarded as another proof.

For further detailed descriptions on the indirect excitation mechanism responsible for the sensitization of the erbium ion by IR140, the schematic diagram of the energy transfer processes are illustrated in Fig. 5, in which the energy levels of each chemical are included. As shown in Fig. 5, upon excitation at 800 nm, IR140 cation is excited to its first singlet state S$_1$, then undergoes intersystem crossing to its first triplet state T$_1$ at an enhanced rate due to the external heavy atom effect of paramagnetic erbium ion. Subsequently main pathway from IR140 cation to erbium ion is from the former's first triplet state other than its first singlet state. The possible levels of erbium ion for accepting transferring energy are $^4I_{11/2}$ (10,200 cm^{-1}) and $^4I_{13/2}$ (6500 cm^{-1}). However, it is evident that the energy transfer to the latter may be more unlikely due to the large energy difference (up to 4650 cm^{-1}) between T$_1$

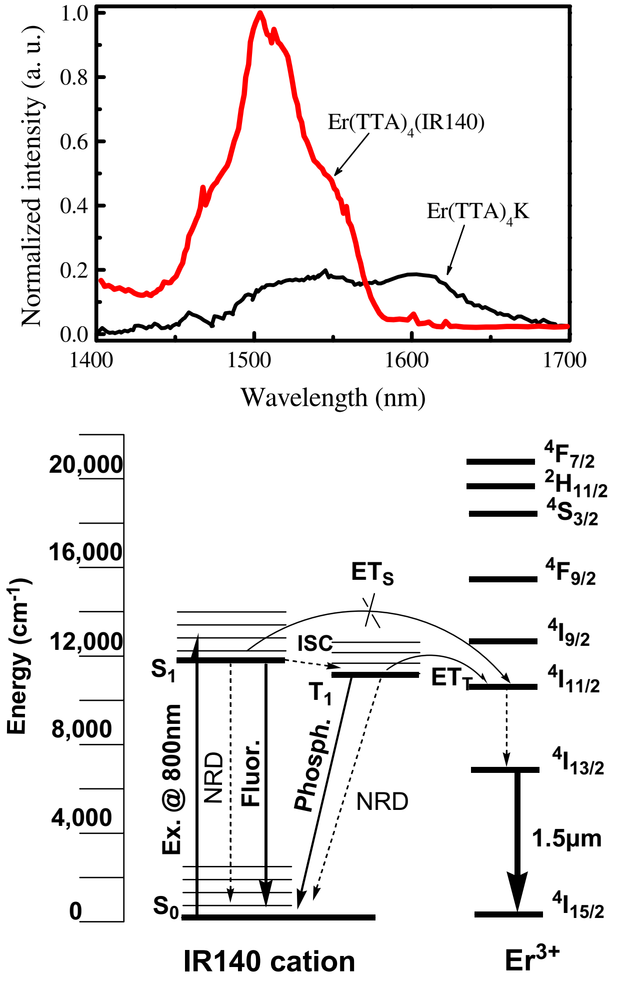

Fig. 5. Near-infrared photoluminescence spectra of the complexes Er(TTA)$_4$K and Er(TTA)$_4$(IR140) in bulky powder excited at 800 nm at room temperature (Top). Sensitization mechanism for erbium ion by IR140 dye cation (Bottom). Here the abbreviations IC, NRD, ISC and ET are the phases internal conversion, non-radiation deactivation, intersystem crossing and energy transfer respectively.

(11,150 cm^{-1}) and ^{4}I$_{13/2}$ (6500 cm^{-1}). And the energy difference between T$_1$ and ^{4}I$_{11/2}$ (10,200 cm^{-1}) is 950 cm^{-1}, an actually matching value for energy transferring for IR140 to erbium ion. Furthermore, the selection rule for energy transfer from the excited triplet state of sensitizer to $^{(2S+1)}\Gamma_J$ level of erbium ion is depicted by Malta et al. as follows [18,19]: $|\Delta J| = 2,4,6$ for the dipolar or multipolar energy transfer mechanism (Förster mechanism). For accepting level ^{4}I$_{11/2}$, $|\Delta J|$ is 2 to the ground state ^{4}I$_{15/2}$, as Förster mechanism will play a major role in sensitization of erbium ion via IR140 cation.

As mentioned above, the distance between the centers of IR140 cation and erbium ion is around 10 Å, which is beneficial for energy transfer with Förster mechanism. Meanwhile, according to Dexter [34], the degree of overlap between the phosphorescence spectrum of IR140 cation and the absorption spectrum of erbium ion, the spectral overlap integral (J), can be given by $J = \int f_D(\lambda)\varepsilon_A(\lambda)d\lambda$ with both of their spectra normalized ($\int f_D(\lambda)d\lambda = 1$ and $\int \varepsilon_A(\lambda)d\lambda = 1$ respectively, in Fig. 5). Then the calculated J value in this case of T$_1$ and ^{4}I$_{11/2}$ is 3.47×10^{-3}, which is a moderate value to ensure the energy transfer process. The possible energy transfer via singlet state of IR140 cation is precluded though the calculated J value for S$_1$ and ^{4}I$_{11/2}$ is 4.18×10^{-3}, since it is evaluated that the triplet state dominating the contribution of energy transfer validates in the similar case [18] and since the singlet state is short-lived, this process is often not efficient. Therefore, the energy transfer process via singlet state is negligible. Thus, main operative process goes essentially through S$_1$ → T$_1$ → ^{4}I$_{11/2}$, pointing to Förster mechanism.

4. Conclusions

In summary, this study provides results on a novel stable near-infrared dye based emitter. The complete emission spectra of IR140 ion-associated erbium complex are reported for the first time and characterized. This ion-associated complex is shown to be potentially useful as a candidate material for near-infrared emission doping.

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