Novel rare-earth(III)-based water soluble emitters for Fe(III) detection

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ABSTRACT

In this paper, we report the synthesis, characterization, and Fe(III)-sensing properties of two easily synthesized rare-earth(III)-based emitters, $Na_3Eu(PDA)_3$ and $Na_3Tb(PDA)_3$. Results suggest that the two emitters exhibit high sensitivity and linear spectral response towards Fe(III) (Stern–Volmer constant = 2.21×10^4 L/mol, linearity = 0.988). In addition, the two emitters are oxygen- and temperature-insensitive, making themselves promising candidates to be utilized in actual applications.

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1. Introduction

The detection of metal ions has always been an important consideration in the fields of environmental protection, biochemistry, and analytical chemistry. Particularly, the detection of ferrous ions [Fe^{3+}] and ferric ions [Fe^{3+}] is one of the most important measurements due to their biological participation in many animal species, including human being. Iron is an essential element for the formation of hemoglobin of red cells and plays an important role in the storage and transportation of oxygen to tissues. Iron is indispensable for most organisms, and both its deficiency and overload can induce various disorders. Iron metabolism disorders have been reported to cause anemia as well as the liver and kidney damage which might ultimately cause liver cancer, liver cirrhosis, arthritis, diabetes or heart failure [1]. Recent studies have linked neurodegenerative disorders such as Parkinson's disease to elevated iron levels [2]. Iron also plays a key role in some important infectious diseases such as malaria [3]. The development of reliable sensing methods for iron ions is, therefore, of considerable importance for environment protection and human health. Several techniques such as spectrophotometry, atomic absorption spectroscopy, chemiluminescence and voltammetry have been used for iron assay in various samples [4–9]. Some techniques necessitate the use of sophisticated instrumentations and require complicated pretreatment procedures, not suitable for on-line or in-field monitoring. The sensor technology is much simpler in instrumental implementation and sample preparation. Owing to the advantages of simple, rapid and non-destructive characteristics, many sensors for iron-selective assay have been reported in the past decades [10–13]. The development of optical sensors for physiologically relevant alkali, alkaline earth metal ions, as well as heavy and transition metal ions 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 the analytical signal is free of the influence of an electromagnetic field and easy to transmit over a long distance [14–17]. Besides intense luminescence, searching for new emitters for iron ions with excellent antijam ability and large Stokes shifts is still a challenge for analytical chemistry research.

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, RE^{3+} based emitters can generate characteristically sharp and narrow emission bands without being affected by surrounding environment or reagents [18]. In addition, RE^{3+} based emitters' long excited state lifetimes make the elimination of fluorescence jamming generated by samples possible. All these excellent characters make RE^{3+} based emitters promising candidates for iron sensors. Unfortunately, most of the reported RE^{3+} based emitters suffer badly from a poor water solubility, preventing them from actual applications for iron sensors [19–21].

In this paper, we report two easily synthesized RE^{3+} based emitters and their elementary applications for Fe^{3+} detection. Results

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Na$_3$RE(PDA)$_3$, RE = Eu, Tb
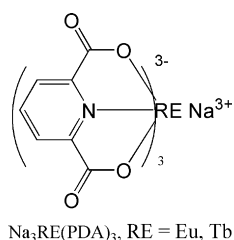
Scheme 1. Molecular structures of Na$_3$RE(PDA)$_3$.

suggest that RE^{3+} based emitters exhibit good spectral responses towards Fe^{3+}, and may be utilized in actual applications.

2. Experimental details

Molecular structures of the two emitters are shown in Scheme 1. Pyridine-2,6-dicarboxylic acid (referred as PDA, ≥98.0%) was purchased from Aldrich Chemical Company. Eu$_2$O$_3$ (99.99%), Tb$_4$O$_7$ (99.99%), NaCl (AR), K$_2$CO$_3$ (AR), MgCl$_2$ (AR), CaCl$_2$ (AR), Bi(NO$_3$)$_3$·5H$_2$O (AR), and NaOH (AR) were purchased from Sinopharm Chemical Reagent Company. All reagents and chemicals were used without further purification. Solvent water was redistilled and bubbled by N$_2$ for 1 min to eliminate O$_2$ without being specified.

2.1. Synthesis of emitters

Na$_3$Eu(PDA)$_3$·14H$_2$O: 1 mmol of EuCl$_3$·6H$_2$O and 3 mmol of PDA was dissolved in hot water, NaOH a.q. with a concentration of 1 mol/L was added until pH = 5–5.3. The crude product was obtained by solvent vaporization and then purified by recrystallization from hot water [22]. Anal. calcd. for C$_{21}$H$_{37}$N$_3$O$_{26}$Na$_3$Eu: C, 26.04; H, 3.85; N, 4.34. Found: C, 26.01; H, 3.79; N, 4.15.

Na$_3$Tb(PDA)$_3$·14H$_2$O: Na$_3$Tb(PDA)$_3$·14H$_2$O was synthesized similar with Na$_3$Tb(PDA)$_3$·14H$_2$O except that EuCl$_3$·6H$_2$O was replaced by TbCl$_3$·6H$_2$O. Anal. calcd. for C$_{21}$H$_{37}$N$_3$O$_{26}$Na$_3$Tb: C, 25.86; H, 3.82; N, 4.31. Found: C, 25.75; H, 3.77; N, 4.19.

2.2. Methods and measurements

All the photoluminescence spectra were measured with a Hitachi F-4500 fluorescence spectrophotometer. Elemental analyses were performed on a Carlo Erba 1106 elemental analyzer. All measurements were carried out in the air at room temperature (20 °C) without being specified. The semiempirical calculation on [Tb(PDA)$_3$]$^{3-}$ was finished by MOPAC2007 using PM6 Hamilton. The initial geometry was obtained from its single crystal structure.

3. Results and discussion

3.1. Absorption and photoluminescence

The obtained two complexes are hardly soluble in organic solvents but water diffluent, making themselves convenient to be utilized in the field of metal ion detection in water. Fig. 1 shows the absorption and excitation spectra of Na$_3$Eu(PDA)$_3$ and Na$_3$Tb(PDA)$_3$ in water with a concentration of 1 × 10^{-5} mol/L, as well as their photoluminescence (PL) spectra, which definitely confirms the large scale Stoke's shifts as expected. Not surprisingly, both emitters exhibit characteristic emission bands of their corresponding central RE^{3+} ions, peaking at 592 and 613 nm for Na$_3$Eu(PDA)$_3$, while 489, 543, 582 and 620 nm for Na$_3$Tb(PDA)$_3$. Due to the same ligand molecular structure, absorption spectra of Na$_3$Eu(PDA)$_3$ and Na$_3$Tb(PDA)$_3$ are quit similar with each other,
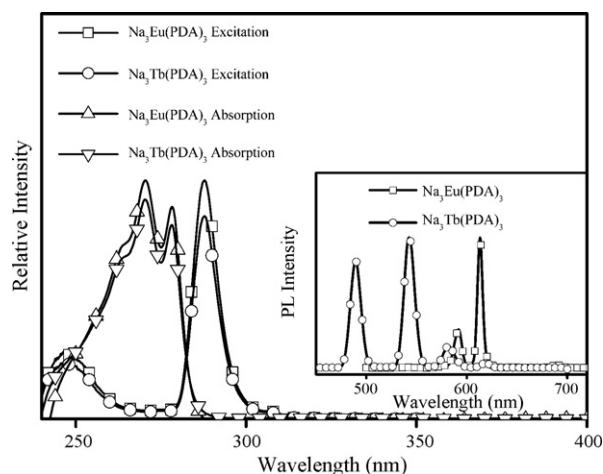

Fig. 1. Absorption, excitation and photoluminescence spectra of Na$_3$RE(PDA)$_3$ in water with a concentration of 1 × 10^{-5} mol/L.

as well as their nearly identical excitation spectra. However, there is an obvious inconsistency between the absorption and excitation spectra. Besides the largely different spectral band shapes, the maximum excitation peak of Na$_3$Eu(PDA)$_3$ (285 nm) red shifts by ~10 nm compared with the 0–0 onset absorption peak (275 nm). The largely different spectral band shapes and spectral shift suggest that the effective excitation of emitters corresponds to a charge-transfer n → π* transition [23]. Due to the relatively small molar absorption coefficient of n → π* transition, it is covered up by the following higher energy π → π* transition whose molar absorption is much bigger, leading to the mentioned band shape difference and spectral shift. In order to get a further understanding on the electronic transition, we perform a semiempirical calculation on [Tb(PDA)$_3$]$^{3-}$. As shown in Fig. 2, the highest occupied molecular orbital (HOMO) localizes mainly on carboxylic O atoms, admixed with some contributions from π orbital between carboxylic C atom and pyridine ring, while the lowest unoccupied molecular orbital (LUMO) is essentially π* orbital of pyridine ring. It is thus confirmed that the effective excitation of emitters is a charge-transfer n → π* transition. Furthermore, the effective excitation wavelength of Na$_3$Eu(PDA)$_3$ and Na$_3$Tb(PDA)$_3$ localizes in the UV light emitting diode (LED) emitting region of ~280 nm, making themselves convenient to utilize the UV-LED as an excitation light source and further simplify instrumental implementation.

3.2. PL spectral responses towards Fe^{3+}

Figs. 3 and 4 demonstrate the PL intensity variations of Na$_3$Eu(PDA)$_3$ and Na$_3$Tb(PDA)$_3$ in water with a concentration of 1 × 10^{-3} mol/L upon Fe^{3+} concentration increase from 0 to 30 × 10^{-5} mol/L. Clearly, both emitters are sensitive towards Fe^{3+}: a trace mount of Fe^{3+} (1 × 10^{-5} mol/L) leads to obvious PL intensity decreases in both Na$_3$Eu(PDA)$_3$:Fe^{3+} and Na$_3$Tb(PDA)$_3$:Fe^{3+} systems. With a high Fe^{3+} concentration of 20 × 10^{-5} mol/L, the absorption spectra of Na$_3$Eu(PDA)$_3$:Fe^{3+} and Na$_3$Tb(PDA)$_3$:Fe^{3+} systems are still nearly the same with the corresponding ones of pure Na$_3$Eu(PDA)$_3$ and Na$_3$Tb(PDA)$_3$, as shown in Fig. 5. This result suggests that the sensing mechanism is a dynamic one, and can be described as:

$$[RE(PDA)_3]^{3-*} + Fe^{3+} \rightarrow [RE(PDA)_3]^{3-} + Fe^{3+*} \qquad (1)$$
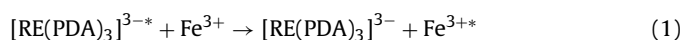
where * stands for an excited state. The Stern–Volmer plots of the two systems can be seen in Figs. 3 and 4, where I is the steady-state luminescence intensity, and 0 denotes the absence of Fe^{3+}. As shown in Fig. 3, PL intensity variation curve of
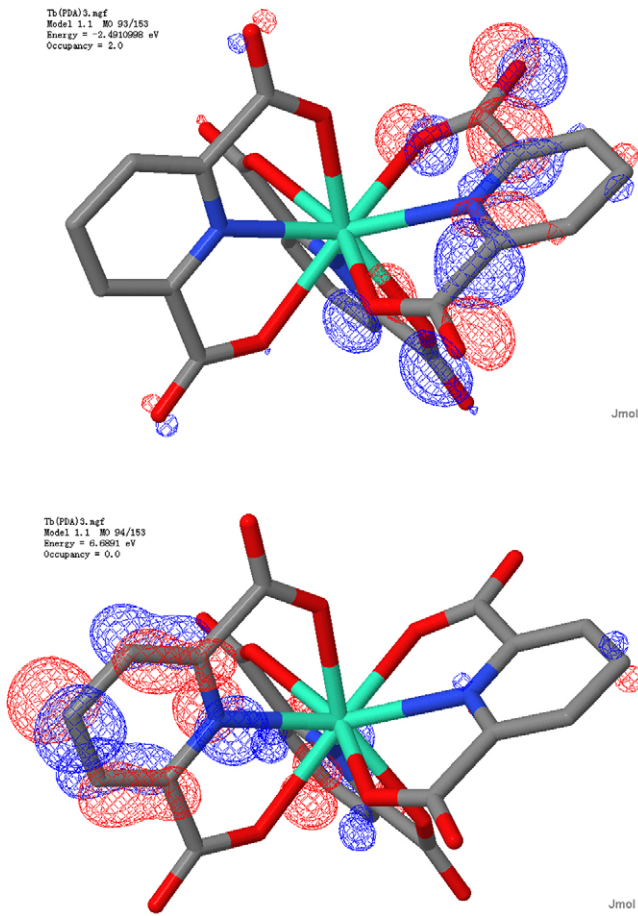
Fig. 2. HOMO (up) and LUMO (down) of $[Tb(PDA)_3]^{3-}$.

Fig. 4. Stern–Volmer plot of $Na_3Tb(PDA)_3$ in water with a concentration of 1×10^{-3} mol/L upon concentration increase of Fe^{3+} from 0 to 30×10^{-5} mol/L. Inset: PL spectral variation (up), magnified view from 0 to 10×10^{-5} mol/L (down).

$Na_3Eu(PDA)_3$ offers a good linear response ($R^2 = 0.988$) with a Stern–Volmer constant of 1.62×10^4 L/mol when Fe^{3+} concentration increases from 0 to 10×10^{-5} mol/L. Upon a higher Fe^{3+} concentration of 30×10^{-5} mol/L, $Na_3Eu(PDA)_3$'s PL intensity loses nearly 95% of its initial value, suggesting that Fe^{3+} is an efficient killer for $Na_3Eu(PDA)_3$'s emission. Similar case is also observed in $Na_3Tb(PDA)_3$:Fe^{3+} system as shown in Fig. 4. It seems that Fe^{3+} is even more efficient on quenching $Na_3Tb(PDA)_3$'s emission. PL intensity variation cur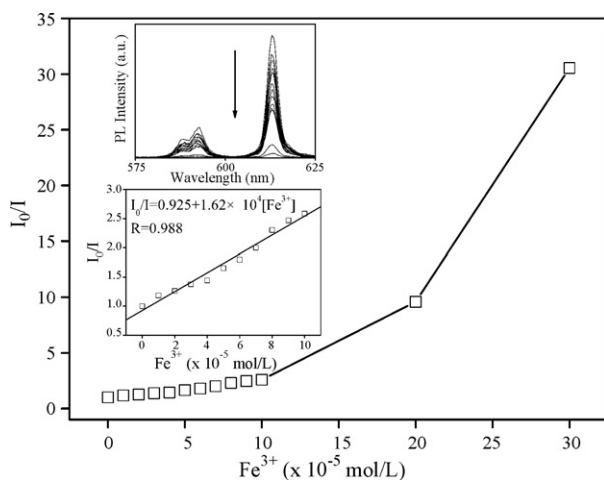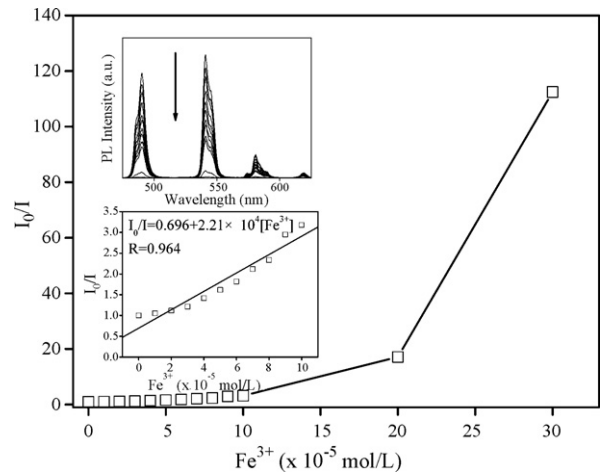ve of $Na_3Tb(PDA)_3$ presents a sharp decrease when Fe^{3+} concentration increases from 0 to 10×10^{-5} mol/L, with a Stern–Volmer constant of 2.21×10^4 L/mol, suggesting that $Na_3Tb(PDA)_3$'s optimal sensitive window towards Fe^{3+} localizes in a low concentration region from 0 to 10×10^{-5} mol/L. It is also notable that the characteristic emission bands of Eu^{3+} and Tb^{3+} ions are insensitive to Fe^{3+} existance as expected, making the instrumental detection more easier to perform.

3.3. Selectivity of the two emitters

Considering that other metal ions usually accompany Fe^{3+} in actual detections, ion selectivity is thus an important consideration for sensors. Herein, we select four commonly existed metal ions in water, Na^+, K^+, Mg^{2+}, and Ca^{2+}, to explore the two emitters' selectivity. Fig. 6 shows the PL intensities of $Na_3Eu(PDA)_3$ and $Na_3Tb(PDA)_3$ in water with a concentration of 1×10^{-4} mol/L and those of solutions with the existence of 1×10^{-4} mol/L metal ions. It seems that Na^+ and Mg^{2+} exert no obvious effect on the two emitters. On the other hand, K^+ and Ca^{2+} can quench the emission, and the quenching effect increases with the increasing metal ion charge. But the quenching effect is relatively unobvious in $Na_3Tb(PDA)_3$:metal ion systems, indicating that $Na_3Tb(PDA)_3$ owns a better antijam ability than $Na_3Eu(PDA)_3$.

Fig. 3. Stern–Volmer plot of $Na_3Eu(PDA)_3$ in water with a concentration of 1×10^{-3} mol/L upon concentration increase of Fe^{3+} from 0 to 30×10^{-5} mol/L. Inset: PL spectral variation (up), magnified view from 0 to 10×10^{-5} mol/L (down).

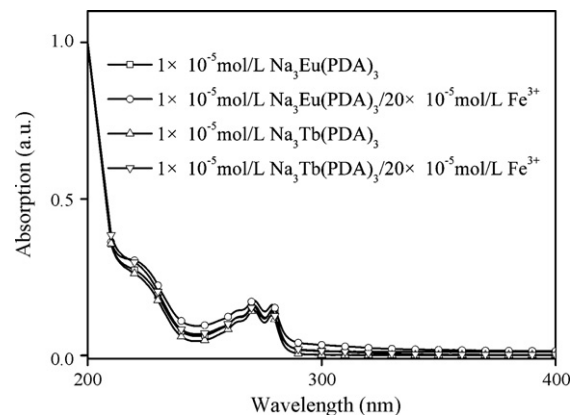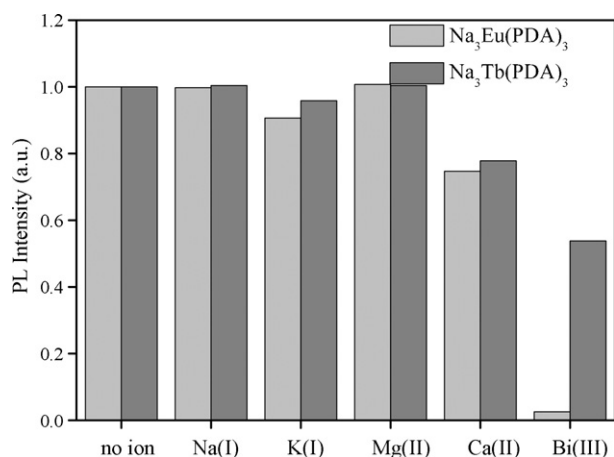**Fig. 5.** Absorption spectra of pure RE emitters and $Na_3RE(PDA)_3$/Fe^{3+} systems.

Fig. 6. PL intensity responses of $Na_3RE(PDA)_3$ in water with a concentration of 1×10^{-4} mol/L towards metal ions (1×10^{-4} mol/L).

Fig. 7. PL spectral responses of $Na_3RE(PDA)_3$ in water with a concentration of 1×10^{-5} mol/L towards oxygen.

3.4. Sensing mechanism of the two emitters

According to the above find, it seems that metal ion charge plays an important role when explaining metal ions' different activities on emitters' PL intensity: interaction between metal ions and emitters increases with the increase of metal ion charge, which is consistent with the commonly believed quenching mechanism of charge-transfer (CT) [24]. As we mentioned, the emitters effective excitation is a charge-transfer $n \rightarrow \pi^*$ transition of PDA moiety. Upon the existence of Fe^{3+}, the excited PDA moiety may transfer its energy to Fe^{3+} instead of RE^{3+}, leading to RE^{3+} emission decrease. And this hypothesis is supported by the following experimental data. Na^+ and K^+ own relative poor interactions with $[RE(PDA)_3]^{3-}$ and thus are ineffective on quenching $[RE(PDA)_3]^{3-}$'s emission. While, as for Ca^{2+}, the stronger interaction leads to more obvious quenching effects on $[RE(PDA)_3]^{3-}$'s emissions. Fe^{3+} is a reasonable highly efficient killer due to the fact that Fe^{3+} owns the largest metal ion charge, thus, there may be a strong coulombic attraction between $[RE(PDA)_3]^{3-}$ and Fe^{3+}, leading to the emission quenching. In order to further confirm this coulombic interaction between $[RE(PDA)_3]^{3-}$ and analyte, we explore the PL response of $[RE(PDA)_3]^{3-}$ towards Bi^{3+}. Not surprisingly, both emitters demonstrate obvious intensity decrease upon Bi^{3+} as shown in Fig. 6, which further confirms the coulombic interaction mechanism.

3.5. Oxygen and thermal sensitivity of the two emitters

Our previous result indicates that molecular oxygen is an efficient killer for emitters with long excited state lifetimes, leading to shortened excited state lifetimes and decreased emission intensities [25]. In addition, it has been reported that RE^{3+} complexes' emission intensities depend some extend on temperature [26]. Considering that sensor technology bases on the detection of emitter's emission intensity, it is thus an important consideration to get an understanding on oxygen and thermal effects on the $Na_3Eu(PDA)_3$ and $Na_3Tb(PDA)_3$.

Fig. 7 shows the PL spectra of $Na_3Eu(PDA)_3$ and $Na_3Tb(PDA)_3$ in degassed water with a concentration of 1×10^{-5} mol/L and those of $Na_3Eu(PDA)_3$ and $Na_3Tb(PDA)_3$ solutions after being bubbled by O_2. Clearly, $Na_3Eu(PDA)_3$ exhibits nearly identical PL spectra, including band shape and emission intensity, in degassed solution and O_2 bubbled solution. Similarly, $Na_3Eu(PDA)_3$'s PL spectra in degassed solution and O_2 bubbled solution are also the same, indicating that both emitters are oxygen insensitive. According to the previous report, there are three potential intermolecular energy transfer mechanisms in RE^{3+} complexes. The one that seems to agree with
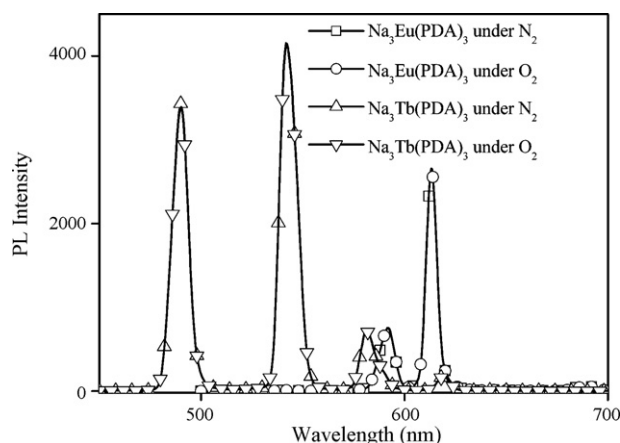
most of the experimental data is described as follows. After an efficient intersystem crossing between the lowest singlet and triplet excited states of the ligand, energy transfer from triplet excited state of the ligand to a lower energy state of the RE^{3+} ion, leading to the $f–f$ radiative decay of central RE^{3+} 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 O_2. As for the excited RE^{3+} ion, although its excited state lifetime is usually as long as microseconds, the partially filled 4f orbitals are shielded from environment by the filled 5d and 5p orbitals, and thus is O_2 insensitive. The O_2 insensitivity of $Na_3Eu(PDA)_3$ and $Na_3Tb(PDA)_3$ may largely simplify the pretreatment procedures and sample preparation in actual applications.

Fig. 8 shows the emission intensity variations of $Na_3Eu(PDA)_3$ and $Na_3Tb(PDA)_3$ solutions in water upon temperature increases from 0 to 30 °C. It is notable that both $Na_3Eu(PDA)_3$ and $Na_3Tb(PDA)_3$ are very sensitive towards temperature: emission intensity decrease with the temperature increase, and this trend is obviously clear in the region from 0 to 10 °C. This is because the excited RE^{3+} ion suffers from vibration decay process in water solutions due to RE^{3+} ion's long excited state lifetime, while low temperature can efficiently suppress this decay process, leading to the improved emission intensity. However, in the region of 10 to 30 °C, the decrease trends are largely suppressed and follow nearly linear decrease curves, suggesting that emission intensities can be easily corrected under different temperatures from 10 to 30 °C.
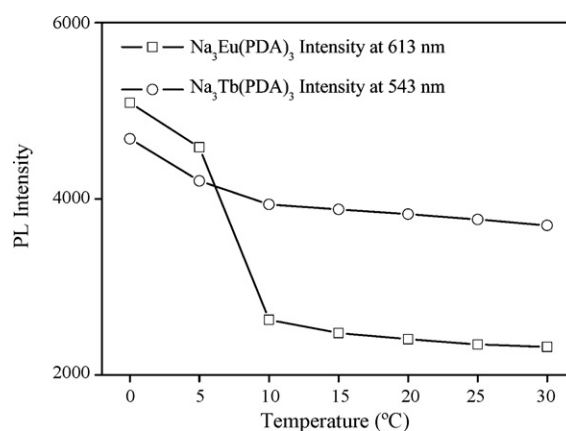

Fig. 8. PL intensity responses of $Na_3RE(PDA)_3$ in water with a concentration of 1×10^{-5} mol/L upon temperature increases from 0 to 30 °C.

Considering that most measurements are carried out at room temperature, the thermal sensitivity of $Na_3Eu(PDA)_3$ and $Na_3Tb(PDA)_3$ should not cause too much trouble in actual applications.

4. Conclusion

In this paper, we report two easily synthesized RE^{3+} based emitters and their elementary applications for Fe^{3+} detection. The results suggest that RE^{3+} based emitters exhibit good spectral responses towards Fe^{3+}, and may be utilized in the actual applications.

Acknowledgements

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