ABSTRACT: Holmium(III) ions incorporated with an organic ligand generate \( \sim 2 \mu m \) optical emission which is characterized by steady and time-resolved photoluminescence. A potential efficient sensitization scheme is demonstrated by empirically calculating the Förster energy transfer rate and modeling the excited state dynamics of the ion. This is demonstrated by taking into account an ideal organic chromophore. The presented work proposes a promising material candidate for the 2 \( \mu m \) emission, which can be fabricated in thin films.

The 2 \( \mu m \) wavelength is favored for low propagation loss, attenuated photon energy, and large optical depth in water, which makes the light sources ideal in optical telecommunication, industrial laser fabrication, and ultrafast optical medical treatment.\(^1\) A typical 2 \( \mu m \) emitting material system is GaSb-based quantum dots, but their fabrication conditions are complicated because of the sensitivity of water.\(^1\) Another way of generating a 2 \( \mu m \) photon is via the 4f–4f transitions of lanthanide ions, Ho\(^{3+}\) and Tm\(^{3+}\) ions, which are narrow in line width and have relatively long lifetimes.\(^2\)–\(^11\) Ho\(^{3+}\) and Tm\(^{3+}\) ions can be easily doped to glass hosts for the fabrication of 2 \( \mu m \) lasers and optical amplifiers, whereas these devices require high pump power to induce forbidden 4f transitions due to the low absorption coefficients and lack of integrability for advanced integrated technologies. Despite some sensitization schemes using other lanthanide ions to excite Ho\(^{3+}\) indirectly or Tm\(^{3+}\) ions, the enhancement of the emission intensity is still trivial with a small factor of \(<10.\)\(^11\)–\(^16\) In this context, organic sensitized lanthanide materials provide a possible solution. In these systems, a light-harvesting organic chromophore efficiently sensitizes nearby lanthanide ions due to the coupling between organic excitons and lanthanide energy levels.\(^17\)–\(^20\) However, the organics commonly have high-energy vibration bonds, including C–H, O–H, and N–H bonds, and they quench the lanthanide emission severely.\(^21\)–\(^22\) The quenching effect becomes more challenging to eliminate when the lanthanide emission extends to a low energy region (e.g., long NIR wavelengths) because of the higher coupling grade of the vibration energy and the lanthanide transition energy. For example, measuring the 1.5 \( \mu m \) emission brightness of most organic erbium materials is challenging. Within numbers of those sensitization materials, a composite system of a chromophore, Zn(F-BTZ)\(_2\) and a perfluorinated organic complex, Ln(F-TPIP)\(_3\) (Ln = Nd, Er, and Yb) has a tremendous sensitization effect that provides the ion NIR emission with up to an enhancement factor of 10\(^5\) times.\(^23\)–\(^25\) In this system that has Zn(F-BTZ)\(_2\) and Ln(F-TPIP)\(_3\) molecules mixed at the molecular level, the average separation of the two molecules is believed to be within a range of \( \sim 1 \) to 2 nm, which is approximately the size of the molecule.\(^24\)–\(^27\) Meanwhile, the perfluorinated organic environment and bulky ligand cage significantly protect the ions from the quenching effect. Despite this success, the emitting region has only extended to an \( \sim 1.5 \mu m \) band with an internal quantum efficiency of \( \sim 30\%.\)\(^21\) How longer wavelengths of a measurable lanthanide emission can be extended using this approach is an open question. In this work, we empirically investigate and discuss the performance of the 2 \( \mu m \) emission of Ho\(^{3+}\), which presumes that similar fabrication techniques can fabricate a Ho(F-TPIP)\(_3\) and Zn(F-BTZ)\(_2\) composite film and that the Ho\(^{3+}\) ions share comparable energy transfer mechanics with those other lanthanide ions.

A cage of F-TPIP\(^-\) ligands is used to coordinate a Ho\(^{3+}\) ion to eliminate vibrational quenching from external environments. The photoluminescence (PL), photoluminescence excitation
(PLE), and time-resolved PL spectra of Ho(F-TPIP)3 powder are characterized to demonstrate the features of Ho3+ emission at the 2 μm band. A rate equation model is built to simulate the energy transfer process from Zn(F-BTZ)2 to Ho(F-TPIP)3. Zn(F-BTZ)2 refers to the conjugate base of 2-(tetrafluoro-2-hydroxyphenyl) tetrafluorobenzothiazole. The spectroscopy of Zn(F-BTZ)2 has been well studied in previous research. Fitting the emission spectra of Zn(F-BTZ)2 to Förster calculations gives a singlet energy transfer rate of 9 ± 1.1(109) s–1 and a triplet energy transfer rate of 900 ± 80 s–1. By feeding the energy transfer rate to the rate equation model, we obtain a potential PL enhancement of ~8000 times, which exceeds any reported sensitization system in Ho3+ material systems.

The blue curve illustrates the emission spectrum of Ho3+ in Figure 1. A 440 nm pulsed Nd:YAG OPO laser pumps the Ho(F-TPIP)3 powder to excite the Ho3+ ions from 3F7 to 5G6 states. The excited states relax to the lowest excited state of 3I8 and decay back to the ground state 1I8 to give the emission band from 1850 to 2100 nm. Herein we exclude the contribution of the F-TPIP ligand to the emission peak as the energy gap does not match the emission energy at 2 μm. The excitation spectra shown by the red curve are recorded by monitoring the PL intensity at the wavelength of 2050 nm while the powder is pumped with wavelengths from 430 to 550 nm. The excitation peaks at 420 nm, 450 nm, 470 nm, 480 nm, and 490 nm are in good agreement with the Ho3+ absorption spectrum, representing the transition from ground state 1I8 to excited states of 5G6, 5F7, 5F4, 5F3, 5F2, 5G6, and 5G4, respectively. The differences of the PLE intensities reflect the relative strength of the dipole oscillation for each transition.

The time-resolved PL spectrum of the Ho(F-TPIP)3 powder at the wavelength of 2050 nm is shown in Figure 2. The spectrum is fitted with the combination exponential rising and exponential decay process, giving a fitted decay lifetime of 11 ± 0.7 μs and a rise of 7 ± 0.3 μs, respectively. The 7 μs rise time is an overall relaxation time for electrons at the high energy excited states to decay to the lowest excited state. This fast relaxation process indicates that most electrons accumulate at the lowest excited state before they decay back to the ground state. If we use the literature reported 3 ms lifetime as the intrinsic lifetime of Ho3+, an internal quantum yield of 0.3% is obtained from the powder sample. We assume that the lifetime of Ho3+ could be prolonged when it is mixed into a composite with Zn(F-BTZ)2 along with proper encapsulation techniques and dilutes of some nonemissive organic complexes such as Y(F-TPIP)3.28 Also, further purification and waterproof encapsulation that suppress the nonradiative quenching effect from X–H (X = O, C, N, ...) bonds is a practical approach to improve quantum efficiency.

As the absorption spectra illustrate in Figure 3, Ho3+ ions have multiple absorption peaks in the visible range, providing energy transfer routes to sensitized Ho3+ ions with some light harvesting materials. The reason the PLE spectrum has more fine structures than the absorption spectrum is that the PLE spectrum has more absorption peaks in the visible range, providing energy transfer routes to sensitized Ho3+ ions with some light harvesting materials. The reason the PLE spectrum has more fine structures than the absorption spectrum is that the PLE process involves the internal relaxation processes coupled with possible multiphonon interactions within the excited states of Ho3+ ions. In this work, we take an organic chromophore Zn(F-BTZ)2 as a sensitizer to theoretically demonstrate the PL intensity enhancement of Ho3+ ions in an organic codoped system. The singlet and triplet emission spectrum of Zn(F-BTZ)2 is shown by the red and blue curves in Figure 3 in which the curves are normalized to the area of 1. As mentioned, Zn(F-BTZ)2 molecules should allocate nearby Ln(F-TPIP)3 molecules. The energy transfer from the singlet and triplet to the excited states of the Ho3+ ions can be predicted to be primarily driven by the Förster energy transfer mechanism. The Förster energy transfer rate can be quantified with the equations below:

\[
R_{\text{FRET}} = \frac{1}{\tau_D} \quad (1)
\]

\[
R_0^6 = 8.79 \times 10^{-5} \times \left( \frac{\kappa^2 \times Q}{n^4} \right) \times J(\lambda) \quad (2)
\]

\[
J(\lambda) = \int F_0(\lambda) \times \epsilon_0(\lambda) \times \lambda^4 \, d\lambda \quad (3)
\]
In the equations above, $J(\lambda)$ is the spectral overlap between the absorption spectra of Ho$^{3+}$ and the emission spectra of Zn(F-BTZ)$_2$. $Q_d$ is the quantum efficiency of the donor, where we use 0.3% for the triplet and 100% for the singlet. $k^2$ is a constant with a value of 2/3. Furthermore, $r$ is the distance from the donor to the acceptor, and we use 7–10 Å in this paper, which is defined by the radii of Ln(F-TPIP)$_3$ and Zn(F-BTZ)$_2$. The calculated singlet and triplet energy transfer rates are $\sim 9 \pm 1.1 (10^5)$ s$^{-1}$ and $\sim 900 \pm 80$ s$^{-1}$, respectively.

According to the calculated energy transfer rate, the dynamics of the excited state population is analyzed by rate equation modeling. With the assumption that the system is pumped by a continuous wave to a steady state, the dynamics of the excited state population could be described by the equations below:

$$N_{50} \times R_p - N_{51} \times (R_{ETS} + R_S + R_{ISC}) = 0 \quad (4)$$

$$-N_{50} \times R_p + N_{51} \times (R_{ETS} + R_S + R_{ISC}) + N_{T1} \times (R_T + R_{ETT}) = 0 \quad (5)$$

$$N_{51} \times R_{ISC} - N_{T1} \times (R_T + R_{ETT}) = 0 \quad (6)$$

$$N_{50} + N_{51} + N_{T1} = N_{Zn} \quad (7)$$

$$R_{ETS} \times N_{51} + R_{ETT} \times N_{T1} \times N_{Ho_{-g}} \times \frac{1}{C_{Ho}} C_{Ho} = 0 \quad (8)$$

$$-R_{ETS} \times N_{51} + R_{ETT} \times N_{T1} \times N_{Ho_{-g}} \times \frac{1}{C_{Ho}} C_{Ho} + N_{Ho_{e_sensitize}} \times R_{Ho} = 0 \quad (9)$$

$$N_{Ho_{-g}} + N_{Ho_{-s}} = N_{Ho} \quad (10)$$

$$R_p = \sigma_{Ho} \times \Phi_p \times P \quad (11)$$

In eqs 4 to 7, $N_{50}$,$N_{51}$, and $N_{T1}$ describe the dynamics of the ground state, the lowest singlet state, and the lowest triplet state of the Zn(F-BTZ)$_2$ chromophore. Their sum equals the total number of Zn(F-BTZ)$_2$ chromophores in the film. $R_p$, $R_{ETT}$, $R_{SC}$, $R_{ETS}$, and $R_{ETT}$ indicate the singlet decay rate, triplet decay rate, intersystem crossing rate, singlet energy transfer rate, and triplet energy transfer rate. Indicated by eqs 8 to 10, the population of the singlet and triplet states, $N_{51}$ and $N_{T1}$, determines the number of Ho$^{3+}$ ions that can be sensitized. The terms $N_{Ho_{-g}}$ and $N_{Ho_{-s_sensitize}}$ give the population in the Ho$^{3+}$ ground state and the lowest excited state. $N_{Ho_{g}}$ and $R_{Ho}$ represent the total number of Ho$^{3+}$ ions and its decay rate. $C_{Ho}$ indicates the concentration of Ho(F-TPIP)$_3$.

Since the relaxation rate from the Ho$^{3+}$ ions' excited state is swift, we approximate the energy state of Ho$^{3+}$ to a two-level system. Hence, the rate equations used to simulate the direct excitation process are shown below.

$$R_{p, Ho} \times N_{Ho_{-g}} - N_{Ho_{-s_sensitize}} \times N_{Ho_{sensitize}} \times R_{Ho} = 0 \quad (12)$$

$$-R_{p, Ho} \times N_{Ho_{-g}} + N_{Ho_{-s_sensitize}} \times N_{Ho_{sensitize}} \times R_{Ho} = 0 \quad (13)$$

$$N_{Ho_{-g}} + N_{Ho_{-s}} = N_{Ho_{g}} \quad (14)$$

$$R_{p, Ho} = \sigma_{Ho} \times \Phi_p \times P \quad (15)$$

In the equations above, $R_{p, Ho}$ indicates the direct excitation rate, which could be calculated with the absorption cross-section of Ho$^{3+}$. It is noteworthy that the lifetime of Ho$^{3+}$ only determines the quantum efficiency at the lowest excited states. Also, we consider that there is no back energy transfer from Ho$^{3+}$ to Zn(F-BTZ)$_2$, which allows us to assume that the radiative rate of Ho$^{3+}$ remains the same when the film is excited via either sensitization or direct excitation. Hence, the PL intensity enhancement can be given by Equation 16:

$$PL \text{ intensity enhancement} = \frac{N_{Ho_{-s_sensitize}}}{N_{Ho_{-s_sensitize}}} \quad (16)$$

In a sensitization process, both triplet and singlet states sensitize the excited state of Ho$^{3+}$ with a triplet energy transfer rate $R_{ETT}$ and a singlet energy transfer rate $R_{ETS}$. Whereas, at room temperature, the quantum efficiency of the triplet state is extremely low due to thermal quenching, which only builds a small triplet population for transfer energy. Thus, in this...
modeling, we only investigate the influence of $R_{ETS}$ on the sensitized PL intensity enhancement. It is worth noting that the triplet energy transfer process could be enhanced by reducing the temperature to suppress the thermally activated processes such as triplet–triplet annihilation and reversed intersystem crossing.

Considering the excited state density of Ho$^{3+}$ under sensitization and direct excitation for the PL intensity, we use eqs 1–16 to simulate the sensitized PL intensity and PL intensity enhancement, which are shown in Figure 4a,b. The details of the simulation method, including Mathematica programming, codes, and numerical calculations, are presented in the SI. Given a concentration of 10% and a $R_{ETS}$ equal to 9 \( (10^8) \) s$^{-1}$, with the increase in pump power density, the population at the excited state of Ho(F-TPIP)$_3$ is saturated at a high pump power density. The reason the lowest doping concentration has the brightest sensitized PL intensity is because the low quantum efficiency of the Ho$^{3+}$ quenches most of the excited state population. That suggests even when the sensitization process is designed to be efficient by creating a large spectral overlap between the lanthanide ions and the chromophore, the lanthanide ions still need to have a significant quantum efficiency to give bright light emission.

Illustrated by the red curve in Figure 4b, with the calculated $R_{ETS}$ of 9 \( (10^8) \) s$^{-1}$, the PL enhancement could reach 12 000 times compared to direct excitation with a Ho(F-TPIP)$_3$ concentration of 10%. Increasing the doping concentration would reduce the PL intensity enhancement due to the decreased excitation probability for individual Ho$^{3+}$ ions. Herein, we validate our simulation by considering the uncertainties of the parameters we use in this model. The most significant simulation error comes from the Förster energy transfer rate calculation that is introduced by the uncertainty of the donor–acceptor distance. If we give a range of separation distances from 7 to 10 Å, the calculated singlet energy transfer rate would vary form $2.9 \ (10^8)$ s$^{-1}$ to $2.5 \ (10^8)$ s$^{-1}$. In such a case, the maximum PL intensity enhancement at a low Ho(F-TPIP)$_3$ concentration increases from 8000 to 16 000 times. That strongly indicates that a potential efficient energy transfer route exists between Ho$^{3+}$ and Zn(F-BTZ)$_2$. Even with the lowest $R_{ETS}$ from the Förster calculation, the simulation still gives 8000 times PL intensity enhancement, which is three magnitudes larger than the current reported sensitization schemes in the inorganic material system.

This work demonstrates the 2 μm emission from a perfluorinated Ho$^{3+}$ complex, Ho(F-TPIP)$_3$. The PLE and TRPL spectra of the Ho(F-TPIP)$_3$ powder are presented to give a perspective of the luminescence property of Ho$^{3+}$. A theoretical model predicts the efficiency energy coupling between a light-harvesting organic chromophore Zn(F-BTZ)$_2$ and Ho$^{3+}$ ions, which may yield ~8000 to 16 000 times PL intensity enhancement via the sensitization process. Incorporating Zn(F-BTZ)$_2$ and Ho(F-TPIP)$_3$ in thin films opens a new opportunity to fabricate integrated light emitting devices at 2 μm.

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**EXPERIMENTAL METHOD**

The synthesis of the organic complex Ho(F-TPIP)$_3$ can be found in the Supporting Information. The crude Ho(F-TPIP)$_3$ is purified by a train vacuum purification system before performing any optical characterization. The PL signals are collected by a Triax 550 spectrometer and detected by a Hamamatsu R5509-72 nitrogen cooled photomultiplier.

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**ASSOCIATED CONTENT**

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.jpclett.2c02516.

Chemical properties and synthesis procedure of Ho(F-TPIP)$_3$ and the parameters for the Förster energy transfer calculation (PDF)

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Author Contributions
H.Y. designed the experiment and drafted the paper. C.L. prepared samples, performed experiments, and analyzed data.

Notes
The authors declare no competing financial interest.

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