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Efficient rare earth cerium(III) complex with nanosecond d-f emission for blue organic light-emitting diodes

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Abstract
Blue organic light-emitting diode (OLED) is still a challenge among the RGB ones due to the lack of emitter simultaneously has short excited state lifetime and high theoretical external quantum efficiency (EQE). We demonstrate herein a blue emissive rare earth cerium(III) complex Ce-2 showing a high photoluminescence quantum yield of 95% and a short excited state lifetime of 52.0 ns in doped film, which is considerably faster than that achieved in typical efficient phosphorescence or thermally activated delayed fluorescence emitters (typical lifetimes > 1 μs). The corresponding OLED shows a maximum EQE up to 20.8% and a still high EQE of 18.2% at 1000 cd m⁻², as well as a 70 times longer operation lifetime than a classic phosphorescence OLED. The excellent performance indicates that cerium(III) complex could be a candidate for efficient and stable blue OLEDs because of its spin- and parity-allowed d-f transition from Ce³⁺ ion.

Keywords: cerium complex, doublet emission, d-f transition, organic light-emitting diodes
INTRODUCTION

During decades of efforts, organic light-emitting diodes (OLEDs) achieved theoretical 100% internal quantum efficiency (IQE) by using phosphorescence,\(^{[1-4]}\) thermally activated delayed fluorescence (TADF)\(^{[5-7]}\) and organic radical\(^{[8-9]}\) materials as emitters. At the same time, tremendous progress in device operation lifetime has been made, which allows the commercialization of high efficiency red and green OLEDs in display and lighting applications.\(^{[10]}\) Nevertheless, blue OLED, which combines high efficiency and long device operation lifetime, is still a difficult challenge. For efficient blue phosphorescence and TADF emitters in OLEDs, the high-energy (above 2.6 eV) and long excited state lifetime (around microseconds) triplet excitons are easily occurring annihilation and/or chemical reactions at high current density, leading to efficiency roll-off and device degradation.\(^{[11]}\) Therefore, lots of efforts have been made on molecule design to shorten the excited state lifetime for better device stability.\(^{[11-12]}\) Theoretically, rare earth cerium(III) complex owns a short excited state lifetime\(^{[13-16]}\), and a high theoretical IQE up to 100% ascribed to the spin- and parity- allowed doublet \(d-f\) transition of \(\text{Ce}^{3+}\) ions, though this concept has not been proposed and demonstrated. Besides, the emission wavelength of cerium(III) complex could be adjusted by varying coordinate environment,\(^{[17]}\) and the cost of cerium is much lower than iridium and platinum because of the rich abundance of cerium in earth (even higher than copper) and the simple isolation process from other lanthanide elements.\(^{[18]}\) All these advantages reveal the huge potential of cerium(III) complex in OLEDs. However, electroluminescence investigations on cerium(III) complexes are rare and the reported maximum external efficiency (EQE) is below 1%,\(^{[19-21]}\) because most reported cerium(III) complexes are non-emissive.\(^{[22]}\) As a breakthrough, we demonstrate herein that cerium(III) complex Ce-2 shows a maximum EQE up to 20.8%, corresponding to an IQE close to 100%, and an operation lifetime (LT\(_{70}\)) about 70 times longer than bis(4,6-difluorophenylpyridine)(picolinate)iridium.
(Flrpic) in OLEDs, arising from its doublet $d$-$f$ transition mechanism and short excited state lifetime.

**RESULTS AND DISCUSSION**

The complex Ce-2 is synthesized by stirring potassium hydrotris(3,5-dimethylpyrazolyl)borate\textsuperscript{[23]} ($\text{KTp}^{\text{Me}_2}$) with Ce(CF$_3$SO$_3$)$_3$ in tetrahydrofuran, accompanied with hydrolysis due to the presence of water (Figure 1a). Though the reaction is found accidentally, it is reproducible with the synthetic method showed in methods section. The analogous hydrolysis reaction and its mechanism was reported in literature.\textsuperscript{[24]} The complex is precipitated from the mixture and then purified by thermal gradient sublimation under 290 $^\circ$C, much lower than its decomposition temperature of 356 $^\circ$C (Figure S1). The single crystals are obtained during sublimation, and the structure is shown with ORTEP and space-filling views in Figure 1b-c. The complex Ce-2 is a dinuclear compound with two Ce$^{3+}$ ions possessing the same coordination environment (Figure S2). The center Ce$^{3+}$ ions are shielded very well by surrounding ligands (Figure 1c), which could prevent luminescence quenching. Besides, the air stability of Ce-2 powder is quite good. Even when exposed to air for 750 hours, the photoluminescence quantum yield (PLQY) of Ce-2 powder does not decrease (Figure S3).

Since Ce-2 is insoluble in common solvents, the UV-Vis absorption and photoluminescence spectra are recorded in the thermal evaporated neat film state on a quartz substrate. As Figure 2a shows, two absorption bands locating at 330 nm and 399 nm with absorbance around 0.01 could be assigned to $4f\rightarrow5d$ transition of Ce$^{3+}$ ions, while the strong absorption under 260 nm arises from $\pi-\pi^*$ transition of the ligand. The Ce-2 neat film exhibits strong emission under UV excitation (Figure 2a, inset), with a maximum emission peak at 477 nm and a high PLQY of 74%. The crystalline powder of Ce-2 exhibits a similar emission spectrum at room temperature. However, it shows better resolved emission spectrum with two peaks at 476 nm and 524 nm at 77 K (Figure 2b). The energy difference between the two peaks is close to 2000 cm$^{-1}$, in agreement with the energy splitting between $^2\text{F}_{5/2}$ and $^2\text{F}_{7/2}$, two
ground levels of Ce$^{3+}$ ion.$^{[20]}$ Besides, the excited state lifetime of Ce-2 neat film is measured as 43.3 ns at room temperature. As for the crystalline powder, 56.9 ns at room temperature and 52.3 ns at 77 K are recorded (Figure 2c), respectively. All these properties are demonstrating that the emission of Ce-2 can be attributed to Ce$^{3+}$ ion, more specifically to the two electric-dipole $5d \rightarrow 4f$ transitions of Ce$^{3+}$ ion from the lowest excited state ($^2D_{3/2}$) to the ground states $^2F_{5/2}$ and $^2F_{7/2}$.

The complex Ce-2 shows high PLQY and short excited state lifetime, it is worthy of being investigated as the emitter in OLEDs. Thus electroluminescence properties of Ce-2, including efficiency and operation stability, are studied by fabricating its OLEDs with a vacuum deposition method. The frontier molecular orbital (FMO) energy levels of Ce-2 were estimated by ultraviolet photoelectron spectroscopy (UPS) (Figure S4) and the absorption edges of the UV-Vis spectrum. Based on the energy levels of Ce-2, 3,3'-bis(carbazol-9-yl)biphenyl (mCBP), [9-[3-(9H-carbazol-9-yl)phenyl]-9H-carbazol-3-yl]diphenylphosphine oxide (mCPPO1), $N,N'$-dicarbazolyl-3,5-benzene (mCP) and bis[2-(diphenylphosphino)phenyl]ether oxide (DPEPO) were estimated as host materials during photoluminescence study. The PLQY of mCBP, mCPPO1, and DPEPO with 10% Ce-2 doped films are estimated as 59%, 82%, and 75%, respectively. Especially, the mCP doped film exhibits the highest PLQY of 95% and an excited state lifetime of 52.0 ns. Thus it was introduced as the emission layer in OLEDs. Meanwhile, 1-bis[4-[N,N'-di(4-tolyl)amino]phenyl]cyclohexane (TAPC) and 1,3,5-tri(m-pyrid-3-yl-phenyl) benzene (TmPyPB) are used to fabricate hole and electron transport layers, respectively. After optimizing film thickness and doping concentration, the best performance is achieved in device D1 with a structure of ITO/MoO$_3$ (2 nm)/TAPC (40 nm)/mCP:Ce-2 (10 wt%, 30 nm)/TmPyPB (40 nm)/LiF (0.7 nm)/Al (100 nm) (Figure 3a). The device shows no electroluminescence from mCP, which is different from the photoluminescence spectrum of the mCP:Ce-2 (10 wt%) emission layer (Figure 3b). It is reasonable because the bandgap of
mCP is much wider than that of Ce-2, hence carriers may dominantly recombine on dopant rather than host molecule. The device D1 shows a turn-on voltage of 3.9 V, a maximum luminance of 31,160 cd m$^{-2}$, a current efficiency of 45.6 cd A$^{-1}$, and a power efficiency of 30.8 lm W$^{-1}$. The maximum EQE reaches 20.8% and remains 18.2% and 11.6% at 1,000 cd m$^{-2}$ and 10,000 cd m$^{-2}$, respectively. This performance is comparable and even better than those achieved in OLEDs with phosphorescence or TADF materials as emitters.$^{[25-28]}$ For further comparison, a reference device R1 using the classic phosphorescent material FIrpic as the emitter is fabricated with an identical device configuration. As showing in Table 1, complex Ce-2 shows similar emission color to FIrpic but much higher efficiencies in OLEDs. Notably, the transient electroluminescence lifetimes of device D1 and R1 are 64 ns and 1022 ns (Figure 3e), which are consistent with the excited state lifetime of the corresponding emitter, Ce-2 and FIrpic, respectively.

Table 1. Summarized parameters of key OLEDs in this work.

| Device | Emitter | $V_{on}$ a)$[V]$ | EQE$_{max}$ b)$[\%]$ | EQE$_{1000}$ c)$[\%]$ | $L_{max}$ d)$[cd m^{-2}]$ | CIE e)$[(0.17, 0.33)]$ |
|--------|---------|-----------------|-------------------|-----------------|------------------|---------------------|
| D1     | Ce-2    | 3.9             | 20.8             | 18.2            | 31,160           | (0.17, 0.33)        |
| R1     | FIrpic  | 3.8             | 17.4             | 14.7            | 31,680           | (0.15, 0.31)        |
| D2     | Ce-2    | 3.8             | 15.3             | 12.1            | 102,900          | (0.18, 0.35)        |
| R2     | FIrpic  | 3.4             | 16.2             | 13.6            | 18,060           | (0.15, 0.33)        |

a$)^{Turn on voltage, is taken as a reference point at which the luminance is 1 cd m$^{-2}$; b$)^{Maximum EQE; c$)^{EQE at 1000 cd m$^{-2}$; d$)^{Maximum luminance; e$)^{Coordinates at 1000 cd m$^{-2}$.}

The electroluminescence stability of Ce-2 is assessed in device D2 with a structure of ITO/MoO$_3$ (2 nm)/mCP:MoO$_3$ (20 wt%, 30 nm)/mCP (10 nm)/mCP:Ce-2 (10 wt%, 30 nm)/TmPyPB (40 nm)/LiF (0.7 nm)/Al (100 nm) under constant current density at an initial luminance of 1000 cd m$^{-2}$. We chose mCP as the hole transport material (HTL) rather than TAPC in D2 for two reasons. First, TAPC is easily degraded during device operation due to
the low bond dissociation energy (BDE) of the C (sp\(^2\))-N (sp\(^3\)) bond. The higher BDE of the C (sp\(^2\))-N (sp\(^2\)) bond of mCP leads to the better stability.\(^{29}\) Second, the charge accumulation at interfaces is considered as an important factor in OLED degradation.\(^{10}\) In D2, the charge barrier between HTL and emitting layer was eliminated by replacing the TAPC to mCP. Considering the device operation lifetime is greatly affected by materials, device configuration, fabrication environment, and encapsulation technique,\(^{10}\) a reference device R2 using FIrpic as the emitter is also fabricated. The performance of these devices is exhibited in Table 1 and Figure S5. Compared to device R2 with an operation lifetime (LT\(_{70}\)) of 158 s, the device D2 shows a dramatically increased LT\(_{70}\) to 10,940 s (Figure 3f). The emission color of device D2 remains stable over a much longer time range, while that of device R2 shows substantial change during the aging test (Figure S5c). Such results indicate that the electroluminescence stability of Ce-2 is significantly better than that of FIrpic. Furthermore, device D2 exhibits much lower efficiency roll-off at high luminance; thus, an ultrahigh maximum luminance over 100,000 cd m\(^{-2}\) is achieved. The EQE remains 11.1% and 8.9% at 10,000 cd m\(^{-2}\) and 80,000 cd m\(^{-2}\), respectively. The long operation lifetime and small efficiency roll-off could be attributed to the short excited state lifetime of Ce-2.

CONCLUSION

In summary, we demonstrated a blue emission rare earth cerium(III) complex for high performance OLEDs with a maximum EQE exceeding 20%, and a 70 times improved operation stability than a typical phosphorescence emitter FIrpic under the same condition. The excellent performance could be assigned to nearly 100% IQE of the investigated cerium(III) complex and its nanosecond excited state lifetime originating from spin- and parity-allowed 5d→4f transition of Ce\(^{3+}\) ion. Along with adjustable emission color and low cost, the cerium(III) complex would be a new type emitter in OLEDs.
METHODS

Synthesis of Ce-2: KTp^{Me2} (2.68 g, 8 mmol), Ce(CF_3SO_3)_3 (2.34 g, 4 mmol), H_2O (0.074 g, 4 mmol) and dry tetrahydrofuran (100 mL) were added to a 250 mL round-bottom flask. The mixture was stirred in a glovebox at room temperature for two days. The yellow-green powder was obtained by filtering the suspension and then loaded into a thermal sublimator. With gradient temperature of 290 °C – 230 °C – 130 °C and pressure around 2×10^{-4} Pa, 0.533 g Ce-2 was obtained as crystalline powder in 12 hours. Yield: ~ 20%. Anal. calcd. for Ce-2: N 21.37%; C 45.82%; H 5.69%; found: N 21.46%; C 45.76%; H 5.65%.

General characterization: Elemental analyses were performed on a VARIO EL analyzer (GmbH, Hanau, Germany). The crystal structure was obtained from Rigaku XtaLAB PRO 007HF(Mo) single crystal X-ray diffractometer. UV-vis absorption spectra were recorded on a Shimadzu UV3600Plus UV-VIS-NIR spectrophotometer. Fluorescence and transient PL decay spectra were measured on an Edinburgh Analytical Instruments FLS980 spectrophotometer. PLQYs were measured on C9920-02 absolute quantum yield measurement system from Hamamatsu Company. Thermogravimetric analysis was undertaken with a Q600SDT instrument. Ultraviolet photoelectron spectroscopy was measured on AXIS Supra X-ray photoelectron spectrometer.

OLEDs fabrication and measurement: Indium tin oxide (ITO) patterned anode was commercially available with a sheet resistance of 14 Ω square^{-1} and 80 nm thickness. ITO substrates were cleaned with deionized water, acetone, and ethanol. The organic and metal layers were deposited in different vacuum chambers with a base pressure better than 1×10^{-4} Pa. The active area for each device is 4 mm^2. All electrical testing and optical measurements were performed under ambient conditions with encapsulation of devices in a glovebox. The EL spectra, current density-voltage-luminance (J-V-L) and EQE characteristics were measured by computer-controlled Keithley 2400 source meter and absolute EQE.
measurement system (C9920-12) with photonic multichannel analyzer (PMA-12, Hamamatsu Photonics).

Transient electroluminescence measurement: Short-pulse excitation with a pulse width of 15 μs was generated using an Agilent 8114A. The amplitude of the pulse was 9 V, and the baseline was −3 V. The period was 50 μs, delayed time 25 μs, and the duty cycle 30%. The decay curves of devices were detected using an Edinburgh FL920P transient spectrometer. [CCDC 1943674 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.]

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SUPPLEMENTARY DATA

The supplementary data is available at NSR online.

Author contributions

Z. L. and L. W. synthesized and characterized the compounds; L. W., Z. Z. and G. Z. collected and analyzed the spectroscopic data; L. W. and Z. Z. fabricated and tested the
OLEDs; Z. Z., and Z. L. wrote the manuscript. All authors discussed the results and commented on the manuscript. Z. L., Z. B. and C.H. directed the project.

CONFLICT OF INTEREST

The authors declare the following competing interest: Z. Zhao, L. Wang, Z. Liu, Z. Bian and C. Huang are inventors on a patent application based partly on the intellectual property in this report.

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Figure 1. Synthesis and molecule structure of Ce-2. 

a. Synthesis route of Ce-2. 

b. Oak ridge thermal ellipsoid plot (ORTEP) drawing of Ce-2 at a 50% probability level, where purple for Ce, green for B, blue for N, red for O, grey for C. All hydrogen atoms have been omitted for clarity. 

c. The space-filling view of Ce-2, where white for H.
Figure 2. Photophysical properties of Ce-2. 

a. Absorption and photoluminescence spectra of Ce-2 neat film. The excitation wavelength is 380 nm. Inset: the photograph of Ce-2 neat film on quartz under 365 nm irradiation. 

b. Photoluminescence spectra of Ce-2 crystalline powder at 300 K and 77 K, the excitation wavelength is 360 nm. Inset: the photograph of Ce-2 crystal under 365 nm irradiation. 

c. The transient photoluminescence decays of Ce-2 as neat film and crystalline powder at 300 K and 77 K. The excitation wavelength is 380 nm.
Figure 3. Electroluminescence performance of Ce-2. a. The schematic device configuration of device D1. The labels give the energy levels in electronvolts and the thickness of layers in nanometers. b. The electroluminescence spectrum of device D1 and photoluminescence spectrum of the mCP:Ce-2 emission layer. The excitation wavelength is 280 nm. c. The plot of current density-voltage-luminance characteristics of device D1. d. The power efficiency-luminance-EQE traces of device D1. e. The transient electroluminescence decays of device D1 (Ce-2 as the emitter) and R1 (FIrpic as the emitter). f. The operation lifetime decay of devices D2 (Ce-2 as the emitter) and R2 (FIrpic as the emitter) at an initial luminance of 1000 cd m$^{-2}$. 