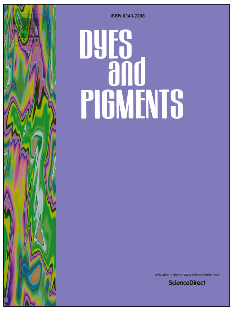

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Fluorine substituted triazine acceptor based thermally activated delayed fluorescent emitter as an assistant dopant of fluorescent emitter

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Abstract

A fluorine substituted triazine acceptor was developed for blue thermally activated delayed fluorescent (TADF) emitters. 9,9'-((6-(4-Fluorophenyl)-1,3,5-triazine-2,4-diyl)bis(2,1-phenylene))bis(3,6-di-tert-butyl-9H-carbazole) (FTrzTCz) was synthesized by connecting 4-fluorophenyltriazine with 3,6-di-tert-butylcarbazole at ortho position of a phenyl linker. The FTrzTCz emitter showed high efficiency of 18.5% and improved efficiency roll-off. The FTrzTCz also showed high quantum efficiency of 17.8% as an assistant dopant in the green fluorescent devices. The FTrzTCz worked well as a TADF emitter and an assistant dopant of hyperfluorescent devices by suppressed Dexter energy transfer.

Key words : high efficiency, Dexter energy transfer, delayed fluorescence, sensitizer

Introduction

In recent years, thermally activated delayed fluorescence (TADF) devices have emerged as third generation organic light-emitting diodes (OLEDs) because TADF is one of the methods to resolve the low efficiency of fluorescent OLEDs. The TADF emitters can harvest the wasted triplet excitons of conventional fluorescent emitters through reverse intersystem crossing (RISC) and theoretically reach 100% internal quantum efficiency[1-6].

Generally, TADF materials are designed based on a molecular platform with donor and acceptor structure to induce the RISC transition[1,7-17] and the emission characteristics of the TADF emitters are determined by the donor or acceptor moieties[18-22]. Therefore, molecular engineering of the donor and acceptor moieties is of great importance to the TADF emitters. There have been many studies for the donor and acceptor units. In the case of the acceptors, diphenyltriazine, diphenylsulfone and benzonitrile have been mostly used as acceptor moieties for TADF emitters[1,7,8,10-17]. In particular, the diphenyltriazine has been one of the most widely used acceptors of TADF emitters due to moderate acceptor strength and good stability[23-28]. A lot of diphenyltriazine derived TADF materials have been documented in the literatures and showed good TADF features. However, the acceptor strength of the diphenyltriazine acceptor itself is rather limited, and the development of even stronger acceptor moiety than the diphenyltriazine is necessary while keeping the merit of the diphenyltriazine.

In this work, 9,9'-((6-(4-fluorophenyl)-1,3,5-triazine-2,4-diyl)bis(2,1-phenylene))bis(3,6-di-tert-butyl-9H-carbazole) (FTrzTCz) was synthesized as a TADF emitter and an assistant dopant for green fluorescent OLEDs. A fluorine substituted triazine was developed as an acceptor moiety to control the acceptor strength and 3,6-di-tert-butylcarbazole was introduced as a donor moiety for blue emission energy and suppressing the Dexter energy transfer. The FTrzTCz showed a high EQE of 18.5% as the TADF emitter without color shift according to doping concentration. As an assistant dopant, FTrzTCz worked efficiently by providing high EQE of 17.9% in the hyperfluorescent devices.

Results and discussion

Diphenyltriazine is a common acceptor mostly used for green and blue TADF materials. It was already proven that it can assist achieving high efficiency and long lifetime in the TADF OLEDs as a building block of the TADF materials[23-28]. However, the diphenyltriazine is a moderate acceptor, which restricts the application of the diphenyltriazine acceptor in the TADF material design. Therefore, fluorophenyltriazine was developed as a triazine derived acceptor unit to increase the acceptor strength while maintaining the advantages of triazine. The F functional group in the fluorophenyltriazine has an electron withdrawing character and can strengthen the acceptor character of the triazine unit. This would be advantageous in the development of TADF emitters because the singlet-triplet energy gap (ΔE_{ST}) can be reduced and the RISC process can be facilitated. However, the planar structure of fluorophenyltriazine can induce strong intermolecular interaction and bathochromic shift of emission energy in film state due to hydrogen bonding. Moreover, it negatively influences emission performances of the hyperfluorescence OLEDs because of the Dexter energy transfer from the TADF emitter to fluorescent emitters. Therefore, the FTrzTCz was designed to have 3,6-di-tert-butylcarbazole donor at ortho position to prevent intermolecular interaction and suppress the Dexter energy transfer through the protective function of the bulky t-butylcarbazole group.

The synthetic method of FTrzTCz was based on multi-step reaction procedure. Fluorophenyltriazine acceptor was synthesized by Grignard reaction of 1-bromo-4-fluorobenzene with 1,3,5-trichlorotriazine. Brominated phenylcarbazole with t-butyl groups at 3 and 6 positions of phenylcarbazole borylated through lithiation reaction followed by Suzuki coupling with the fluorophenyltriazine produced the FTrzTCz compound. The FTrzTCz was synthesized in good yield and purified by column chromatography to obtain high purity above 99%. The detail synthesis was described in **Scheme 1**.

Optical properties and molecular electronic distribution of the FTrzTCz were simulated using Gaussian software with B3LYP 6-31G basis set. The highest occupied molecular orbital (HOMO) was localized in t-butylcarbazole because of its electron donating character. The lowest unoccupied molecular orbital (LUMO) was mainly localized on triazine and phenyl linker. The LUMO was spread to a fluorine atom due to its strong electronegativity. In the geometrical configuration, the triazine was out of plane by large steric hindrance between triazine and ortho positioned t-butylcarbazoles. The dihedral angles between triazine and phenyl linker were nearly 40°. The t-butylcarbazole donor was distorted from the phenyl linker by more than 60 ^{o}. The large dihedral angle may increase charge transfer (CT) character of the TADF materials and promote RISC rate through small ΔE_{ST}. The calculated results are depicted in **Figure 1**.

The HOMO and LUMO of FTrzTCz were determined by oxidation and reduction potentials of cyclic voltammetry (CV). The measured value was corrected with ferrocene (-4.8 eV) standard and 0.1M tetrabutylammonium perchlorate solution in acetonitrile was used as an electrolyte. The measured CV curves are presented in **Figure 2**. The HOMO and LUMO energy levels were -6.13 and -3.42 eV from the onset potentials of the CV measurements. The measured potentials were calibrated with ferrocene to obtain the HOMO and LUMO energy levels. In comparison with 9,9'-((6-phenyl-1,3,5-triazine-2,4-diyl)bis(2,1-phenylene))bis(9H-carbazole) (BisCz-Trz) TADF emitter reported in other works[29], the HOMO level was similar, but the LUMO level of FTrzTCz was deeper than that of BisCz-Trz by employing a fluorine atom in the triazine acceptor.

Photophysical properties were measured by ultraviolet-visible (UV-Vis) absorption and photoluminescence (PL) analysis. The broad absorption from 350 to 400 nm in the UV-vis spectrum was due to CT absorption of FTrzTCz based on the donor and acceptor structure. Main absorption below 300 nm was assigned to π-π* transition of the backbone structure. The singlet energy (E_S) and triplet energy (E_T) were estimated from onset values of fluorescence at room temperature and phosphorescence spectra at 77K in toluene solution. Broad fluorescence spectrum indicates that the fluorescence is from CT based singlet excited state. The vibronic peaks in phosphorescence spectrum suggest that the triplet emission is from local excited (LE) state. The E_S and E_T of FTrzTCz were 2.97 and 2.75 eV, respectively, and ΔE_{ST} was 0.22 eV. The analysed data are presented in **Figure 3**.

To further figure out the PL emission behaviour, transient PL of FTrzTCz was analysed (**Figure 4)**. In the transient PL data, the decay curve of FTrzTCz clearly showed delayed emission with long decay time. The excited state lifetime of the prompt component was 53.7 ns and that of delayed component was 5.91 μs. The temperature dependent transient PL data in **Figure S1** clearly confirms the TADF character by displaying intensified delayed fluorescence according to temperature. The absolute PL quantum yield (PLQY) was 85% under a N_2 atmosphere at doping concentration of 10%. The PLQY of FTrzTCz was higher than that of BisCz-Trz (76%). The PLQYs of prompt and delayed fluorescence were 28% and 57%, respectively. The measured photophysical properties and calculated rate constant are summed up in **Table 1**. Additionally, the thermal analysis data in **Figure S2** suggest good thermal stability by showing thermal decomposition temperature over 400 ^{o}C.

The FTrzTCz was used as an emitter in the TADF devices to identify the device performances. The device structure was depicted in **Figure 5**. The TADF emitter was doped at concentrations of 10~50%. Current density (J) and luminance (L) according to applied voltage (V) are provided in **Figure 6(a)**. The J and L were dramatically increased in accordance with the doping concentration because the FTrzTCz acted as trap sites due to the large energy barrier between DPEPO and FTrzTCz. As the doping concentration increased, the carriers were dominantly transferred by hopping process through emitters, increasing the J and L.

The EQEs of the FTrzTCz devices are presented in **Figure 6(b)**. Maximum EQE was 18.5% at doping concentration of 20%, which was higher than 12.2% of the BisCz-Trz device. The high PLQY of FTrzTCz enhanced the EQE of the TADF device. Furthermore, the efficiency drop at high luminance was improved because triplet exciton quenching process was suppressed successfully by short delayed fluorescence lifetime. The F functional group in the acceptor moiety mainly contributed to the weakened EQE roll-off at high luminance by shortening the delayed fluorescence lifetime through small ΔE_{ST} without sacrificing the PLQY. Although the PLQY was high at 30% doping concentration (Table S1), the large contribution of triplet excitons in the electric field driven emission process induces exciton quenching and decreases the EQE at high doping concentration.

The electroluminescence (EL) spectra of the FTrzTCz devices depending on doping concentration are shown in **Figure 6(c)**. The EL spectra showed no significant change although the doping concentration was increased from 10% to 50%. The constant EL spectra indicates that the bulky t-butyl groups and largely distorted structure of FTrzTCz disturbed intermolecular interaction between emitter molecules. The peak wavelength was in the range of 495~497 nm. All device data are summarized in **Table 2**.

Motivated by the intermolecular interaction suppressing effect of the FTrzTCz emitter as reflected in the EL spectra, the FTrzTCz was applied as an assistant dopant of green fluorescent emitter. Hyperfluorescent OLEDs were fabricated using 10-2-benzothiazolyl-1,1,7,7-tetramethyl-2,3,6,7-tetrahydro-1H,5H,11H-benzo [l]pyrano [6 7 8-ij]quinolizin-11-one (C545T) and 2,6-di-tert-butyl-N9,N9,N10,N10-tetrakis (4-(tert-butyl)phenyl)anthracene-9,10-diamine (6tBPA)[30] as green fluorescent emitters at 1% doping concentration. The EQEs of the green hyperfluorescent OLEDs were 11.5% and 17.9% for C545T and 6tBPA doped devices, respectively. The EQE of the 6tBPA device was higher than that of the C545T device because of effective Dexter energy management through the t-butyl blocking groups in the 6tBPA emitter. The high EQE of the 6tBPA demonstrated that the triplet excitons of FTrzTCz harvested through RISC transition were effectively used for fluorescent radiative decay of the 6tBPA. The highly twisted structure and t-butyl blocking groups in the donor structure were responsible for the effective control of the energy transfer from the FTrzTCz to 6tBPA. The EQE plots are presented in **Figure 7(a)**. The EQE of the pure C545T and 6tBPA devices was only about 6% as presented in **Figure S3** of supporting information.

The EL spectra of hyperfluorescent devices are provided in **Figure 7(b)**. The EL spectra showed C545T or 6tBPA emission, verifying that the energy transfer from the assistant dopant to fluorescent emitters occurred efficiently. Color coordinates of the C545T and 6tBPA hyperfluorescent devices were (0.24, 0.64) and (0.24, 0.58), respectively. The hyperfluorescent device performances were tabulated in **Table 3**.

Conclusions

The FTrzTCz was designed as an efficient sky-blue TADF emitter and assistant dopant of green fluorescent OLEDs. A fluorine atom in triazine acceptor induced strong CT character by intensified acceptor strength, short delayed fluorescent lifetime and fast RISC. The EQE of the FTrzTCz device was 18.5% and the efficiency roll-off was improved. The FTrzTCz performed well as an assistant dopant of green fluorescent OLEDs and enabled high maximum EQEs of 17.9% in the green hyperfluorescent device. Therefore, the F functionalized triazine acceptor can be a potential candidate of the acceptor to design TADF materials and assistant dopants of hyperfluorescent OLEDs.

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Table 2. Device characteristics of FTrzTCz

Table 3. Device characteristics of hyperfluorescence OLEDs

Table 1. Photophysical properties and rate constant of FTrzTCz

	E_S/E_T (eV)[a]	Φ_F/Φ_{TADF} (%)[b]	τ_p (ns)[c]	τ_d (μs)[d]	k_{ISC} ($10^7 s^{-1}$)[e]	k_{RISC} ($10^5 s^{-1}$)[f]	k_{nr}^T ($10^4 s^{-1}$)[g]
FTrzTCz	2.97/2.75	28/57	53.7	5.91	1.35	4.83	3.53

[a] Singlet and triplet energies were onset values of fluorescence and phosphorescence emission spectrum

[b] Φ_F and Φ_{TADF} represent the PLQYs of prompt and delayed fluorescence component

[c] τ_p is decay time of prompt component

[d] τ_d is decay time of delayed component

[e] k_{ISC} is rate constant of intersystem crossing

[f] k_{RISC} is rate constant of reverse intersystem crossing

[g] k_{nr}^T is rate constant of non-radiative decay from T_1 to S_0

Table 2. Device characteristics of FTrzTCz

	Doping concentration (%)	η_{max} (%)[a]	η_{1000} (%)[b]	Color coordinate	λ_{EL} (nm)[c]
FTrzTCz	10	16.9	5.1	(0.20, 0.44)	497
	20	18.5	14.5	(0.20, 0.45)	496
	30	17.4	15.9	(0.20, 0.46)	495
	50	15.3	14.4	(0.20, 0.47)	497

[a] η_{max} is maximum external quantum efficiency

[b] η_{1000} is external quantum efficiency at 1000cdm^{-2}

[c] λ_{EL} is peak wavelength of EL spectrum

Table 3. Device characteristics of hyperfluorescence OLEDs

	V_{on} (V)[a]	η_{max} (%)[b]	η_{1000} (%)[c]	Color coordinate	λ_{EL} (nm)[d]
C545T	7.06	11.5	7.1	(0.24, 0.64)	515
6tBPA	7.11	17.9	14.0	(0.24, 0.58)	517

[a] Von is turn-on voltage at 1cdm^{-2}

[b] η_{max} is maximum external quantum efficiency

[c] η_{1000} is external quantum efficiency at 1000cdm^{-2}

[d] λ_{EL} is peak wavelength of EL spectrum

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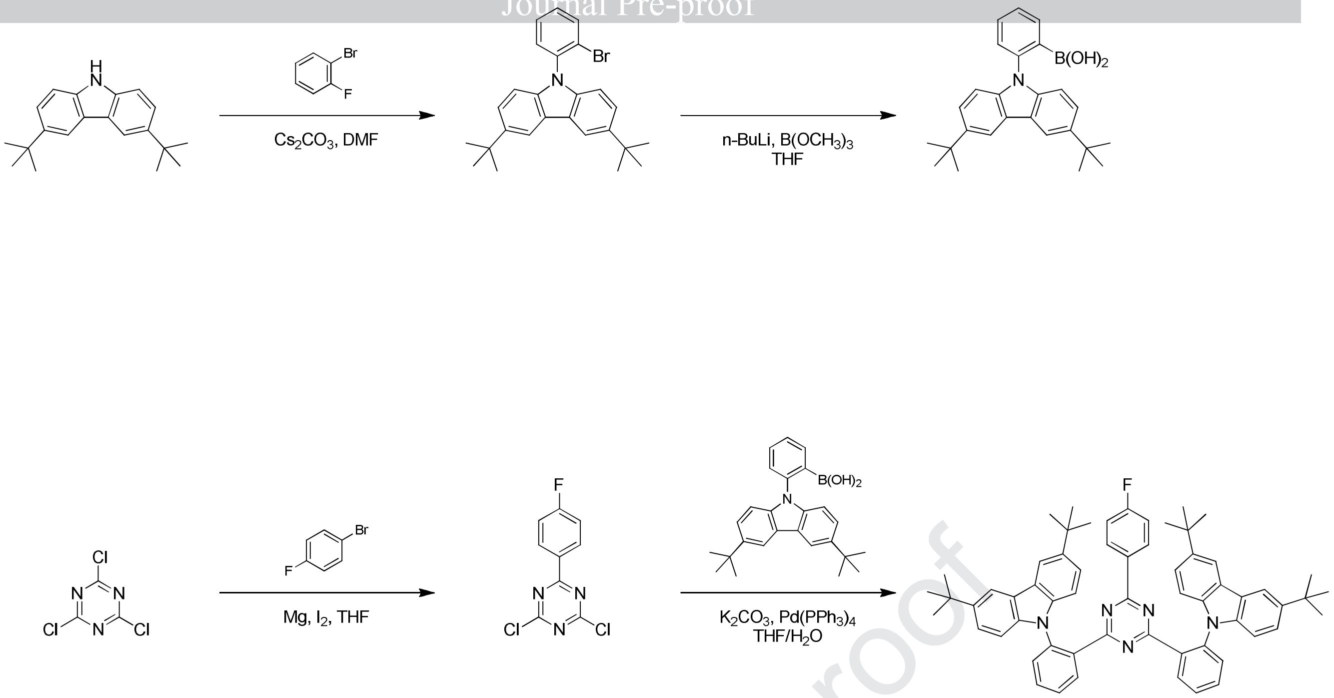
Figure 7. (a)EQE plots and (b)EL spectra of hyperfluorescence OLEDs at 1% doping concentration of C545T and 6tBPA



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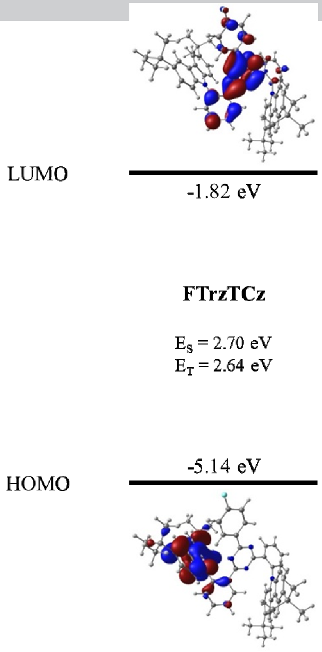

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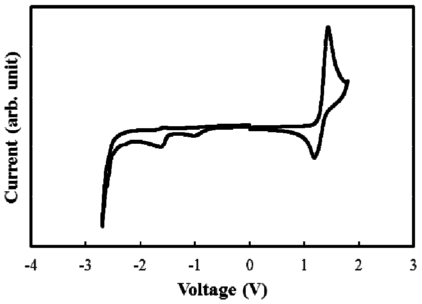

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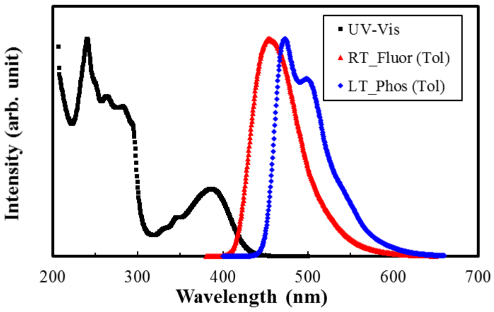

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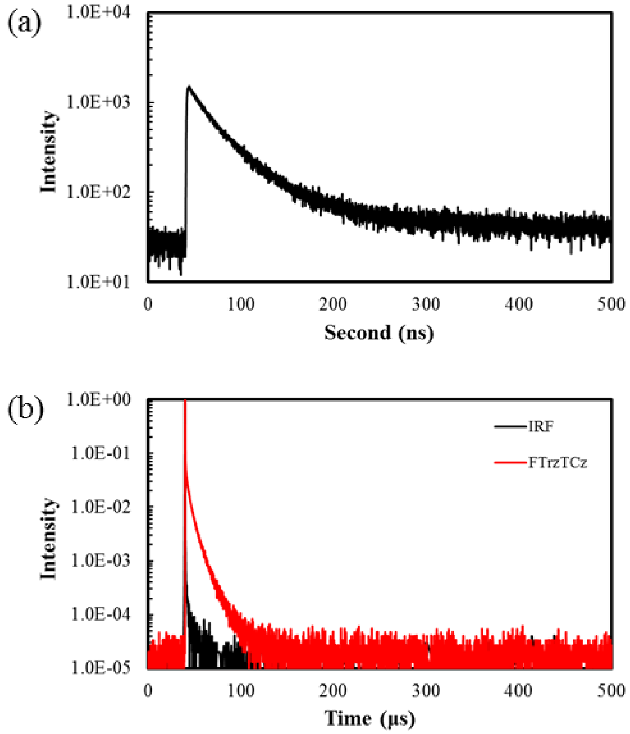

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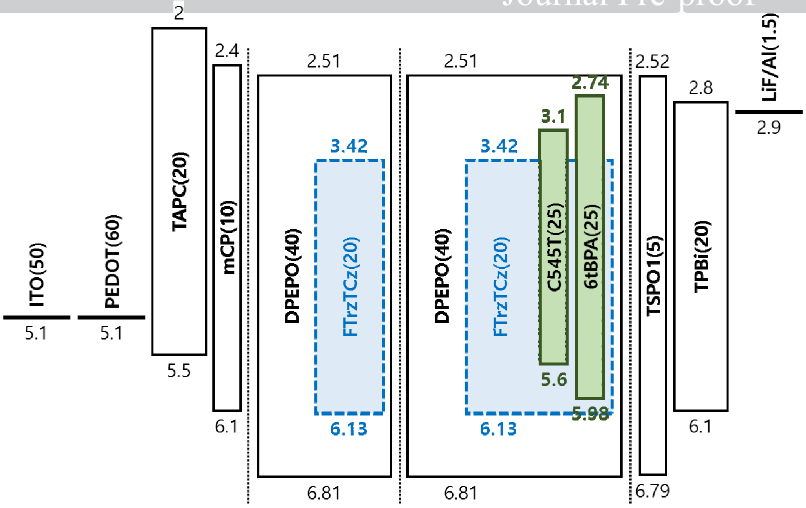

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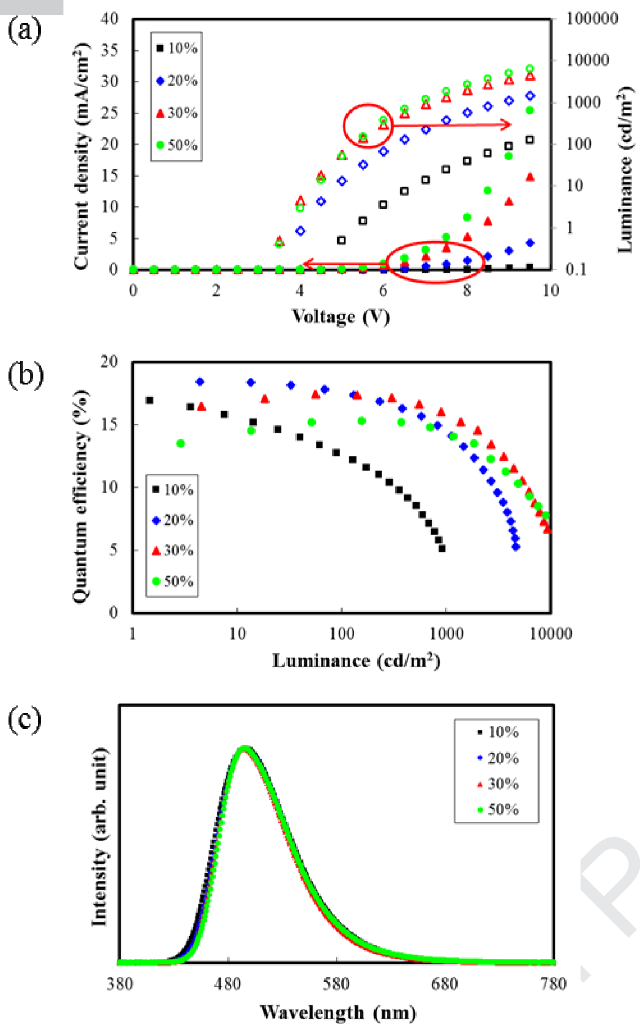

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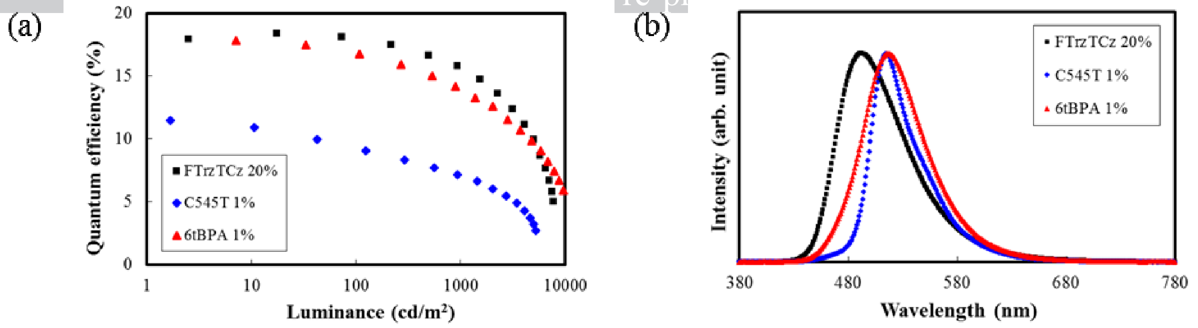

Figure 7. (a)EQE plots and (b)EL spectra of hyperfluorescence OLEDs at 1% doping concentration of C545T and 6tBPA

- **Fluorine substituted triazine acceptor for the design of thermally activated delayed fluorescent emitter**
- **High efficiency and small efficiency roll-off in the thermally activated delayed fluorescent devices**
- **High efficiency in fluorescent organic light-emitting diodes by energy transfer**

Declaration of interests

☒ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: