

High triplet energy n-type dopants for high efficiency in phosphorescent organic light-emitting diodes

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ABSTRACT

High triplet energy n-type dopants, lithium 2-(oxazol-2-yl)phenolate (LiOx) and lithium 2-(1-methyl-imidazol-2-yl)phenolate (Lilm), were synthesized as n-type doping materials for phosphorescent organic light-emitting diodes and the effect of the n-type doping materials on the electron mobility and device performances of the phosphorescent organic light-emitting diodes was investigated. The LiOx and Lilm n-type dopants were effective to increase the electron mobility of electron transport materials and improve the quantum efficiency of green and blue phosphorescent organic light-emitting diodes.

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1. Introduction

Driving voltage and quantum efficiency are two important parameters to manage the power consumption of organic light-emitting diodes (OLEDs). Low driving voltage and high quantum efficiency can improve the power efficiency of OLEDs and reduce the power consumption of OLEDs. In particular, the driving voltage is critical to enhance the power efficiency of white OLEDs which are applied in displays and lighting devices [1].

There have been several approaches to lower the driving voltage of OLEDs and one of the most well-known methods was to use p-type or n-type doped charge transport layers [2–18]. The p-type or n-type doping increased the conductivity of the charge transport materials and reduced the driving voltage of the OLEDs [2]. In particular, the n-type doping was effective to reduce the driving voltage of OLEDs due to the low mobility of common electron transport materials [5–7,3,8–15]. Several n-type doping materials have been used to increase the conductivity of

the electron transport materials. Alkali metals such as Cs and Li were typical n-type doping materials [5–7,3,8,9] and several derivatives of Cs or Li were also developed as the n-type doping materials, which include ionic compound [10–12] or organometallic complexes [13–17]. Cs₂CO₃ [10], CsN₃ [11], and LiF [12] were reported as n-type doping materials and increased the conductivity of an electron transport layer, decreasing the driving voltage of OLEDs. However, the ionic compounds quenched the excitons in the emitting layer and had negative effect on the quantum efficiency of OLEDs. Therefore, organometallic complexes such as 8-hydroxyquinolinatolithium (Liq) were utilized as a molecular dopant to reduce the driving voltage [13–16]. The organometallic Liq dopant increased the mobility and conductivity of electron transport materials, resulting in low driving voltage in OLEDs. However, the Liq dopant can act as a luminance quencher in phosphorescent organic light-emitting diodes (PHOLEDs) because of low triplet energy of Liq (2.0 eV) although there has been no report about the triplet exciton quenching effect by Liq. In those works listed above, the Liq dopant was applied as the dopant for electron transport layer of fluorescent OLEDs. Alternatively, exciton blocking layer was inserted

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between the n-doped layer and emitting layer to block triplet exciton quenching of emitting materials. However, this approach increases the driving voltage of the device due to low electron mobility of the non-doped exciton blocking layer. Therefore, it is strongly required to develop high triplet energy n-type doping materials to suppress the triplet exciton quenching by the n-type doping material.

In this work, high triplet energy n-type doping materials, lithium 2-(oxazol-2-yl)phenolate (LiOx) and lithium 2-(1-methyl-imidazol-2-yl)phenolate (LiIm), were developed to reduce the driving voltage of PHOLEDs and suppress triplet exciton quenching by Liq dopant. It was demonstrated that the high triplet energy n-type dopant increased the mobility of the electron transport layer and improved the quantum efficiency of PHOLEDs by reducing exciton quenching by the n-doped electron transport layer.

2. Experimental

2.1. General information

All chemicals and reagents were purchased from Aldrich and TCI Chem., and were used without further purification. 2-(Oxazol-2-yl)phenol was prepared according to the method described in the literature [19]. The ^1H and ^{13}C nuclear magnetic resonance (NMR) were recorded on Jeol 400 (400 MHz) NMR spectrometer. The mass spectra were recorded using a JEOL, JMS-600W spectrometer in fast atom bombardment mode. Elemental analysis of the materials was carried out using Flash2000 (ThermoFisher). The photoluminescence (PL) spectra were recorded on a fluorescence spectrophotometer (HITACHI, F-7000) and the ultraviolet–visible (UV–Vis) spectra were obtained by means of a UV–Vis spectrophotometer (Shimadzu, UV-2501PC). Triplet energy analysis was carried out using low temperature PL measurement in liquid nitrogen. UV–Vis and solution PL measurement was carried out using tetrahydrofuran solution and the excitation wavelength for PL measurement was 300 nm. Low temperature PL measurement was performed using tetrahydrofuran solution after applying delay time of 100 μs .

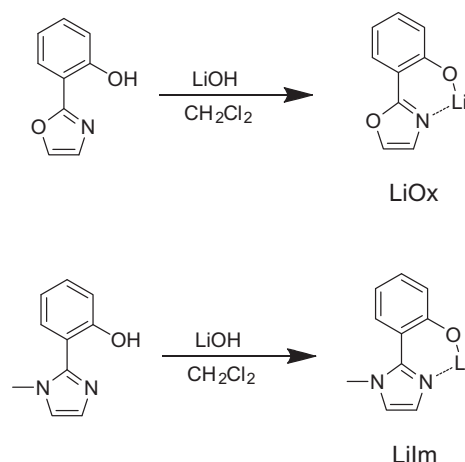
2.2. Synthesis

Synthetic scheme of LiOx and LiIm is shown in Scheme 1.

2.2.1. Synthesis of lithium 2-(oxazol-2-yl)phenolate (LiOx)

2-(Oxazol-2-yl)phenol (1.00 g, 6.21 mmol) and lithium hydroxide (0.16 g, 6.83 mmol) were dissolved in methylene chloride (50 ml) and the solution was stirred at room temperature for 12 h under nitrogen. The mixture was filtered and washed with methylene chloride. After drying in vacuum oven, the product was purified by vacuum train sublimation. The product was obtained as a white powder (0.61 g).

Yield 59%. Tm 311 $^{\circ}\text{C}$. ^1H NMR (400 MHz, DMSO): δ 8.09 (s, 1H), 7.67 (d, J = 6.00 Hz, 1H), 7.27 (s, 1H), 7.14 (t, J = 8.00 Hz, 1H), 6.66 (d, J = 6.00 Hz, 1H), 6.48 (s, 1H), ^{13}C NMR (100 MHz, DMSO): δ 140.28, 138.54, 129.64, 128.14,



Scheme 1. Synthetic scheme of LiOx and LiIm.

126.44, 124.58, 124.10, 123.65, 107.34, MS (FAB) m/z 167 $[(M + H)^+]$. Elemental analysis, Anal. Calcd for $\text{C}_9\text{H}_6\text{LiNO}_2$: C, 64.69; H, 3.62; Li, 4.15; N, 8.38; O, 19.15 Found C, 64.60; H, 3.58; N, 8.35.

2.2.2. Synthesis of lithium 2-(1-methyl-imidazol-2-yl)phenolate (LiIm)

2-(1-Methyl-1H-imidazol-2-yl)phenol (1.10 g, 6.31 mmol) and lithium hydroxide (0.17 g, 6.95 mmol) were dissolved in methylene chloride (50 ml). The solution was stirred for 12 h at room temperature in nitrogen atmosphere and was filtered off using methylene chloride. The filtered product was purified by vacuum train sublimation and white powder was obtained (0.32 g).

Yield 28%. Tm 297 $^{\circ}\text{C}$. ^1H NMR (400 MHz, DMSO): δ 7.13–7.11 (m, 2H), 6.94 (t, J = 6.40 Hz, 1H), 6.85–6.84 (m, 1H), 6.52 (d, J = 4.00 Hz, 1H), 6.25 (t, J = 6.80 Hz, 1H), 3.65 (s, 3H) ^{13}C NMR (100 MHz, DMSO): δ 148.62, 129.69, 129.63, 126.05, 125.85, 121.22, 120.50, 117.01, 109.90, 34.48 MS (FAB) m/z 180 $[(M + H)^+]$. Anal. Calcd for $\text{C}_{10}\text{H}_9\text{LiN}_2\text{O}$: C, 66.68; H, 5.04; Li, 3.85; N, 15.55; O, 8.88 Found C, 66.59; H, 5.00; N, 15.51.

2.3. Device fabrication and measurements

The device structure of green PHOLEDs was indium tin oxide (ITO, 50 nm)/poly(3,4-ethylenedioxythiophene);poly(styrenesulfonate) (PEDOT:PSS, 60 nm)/4,4'-cyclohexyldienebis[N,N-bis(4-methylphenyl)aniline] (TAPC, 20 nm)/4,4'-N,N'-dicarbazolylbiphenyl (CBP):iridium (III) tris(2-phenylpyridine) ($\text{Ir}(\text{ppy})_3$) (25 nm, 5% doping)/2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline (BCP) or BCP: Liq or BCP:LiOx (40 nm)/Al (200 nm) and the device structure of blue PHOLEDs was ITO (50 nm)/PEDOT:PSS (60 nm)/TAPC (20 nm)/3,3-di(9H-carbazol-9-yl)biphenyl (mCBP):bis[2-(4,6-difluorophenyl)pyridinato- $\text{C}^2\text{,N}$] picolinate iridium(III) (Irpic) (25 nm, 5%)/1,3,5-tri[(3-pyridyl)-phen-3-yl]benzene (BmPyPb) or BmPyPb:LiOx or BmPyPb:LiIm (40 nm)/Al (200 nm). Doping concentration of Liq and LiOx was 30 mol%. Device structure of electron only

device was ITO (50 nm)/Ca (5 nm)/BCP:LiOx or BmPyPb:LiIm (100 nm)/LiF (1 nm)/Al (200 nm). The doping concentrations of LiOx and LiIm were 0, 10, and 30 mol%. All devices were fabricated by vacuum thermal evaporation and were encapsulated with a glass lid and CaO getter. Device performances were measured using Keithley 2400 source measurement unit and CS 1000 spectroradiometer.

3. Results and discussion

LiOx and LiIm were designed to have high triplet energy by coordinating high triplet energy 2-(oxazol-2-yl)phenol and 2-(1-methyl-1H-imidazol-2-yl)phenol ligands to Li metal core. The coordination of the high triplet ligands may yield high triplet energy Li compound which can be applicable in green and blue PHOLEDs. The LiOx and LiIm dopants are expected to suppress triplet exciton quenching by n-doped electron transport layer and can improve the quantum efficiency and power efficiency of PHOLEDs compared to common Liq n-type dopant.

The LiOx and LiIm dopants were simply synthesized by coordinating 2-(oxazol-2-yl)phenol and 2-(1-methyl-1H-imidazol-2-yl)phenol ligands to Li using LiOH as shown in Scheme 1. Synthetic yields of the LiOx and LiIm dopants were 59% and 28% after purification by vacuum train sublimation. Highly pure compound with a high purity level over 99% was confirmed by high performance liquid chromatography. Chemical structure of the LiOx and LiIm dopants was analyzed using ^1H and ^{13}C nuclear magnetic resonance spectrometer, mass spectrometer and elemental analysis.

Photophysical properties of LiOx and LiIm were analyzed using UV–Vis and PL spectrometers. Fig. 1 shows UV–Vis absorption, solution PL and low temperature PL emission spectra of LiOx and LiIm. LiOx exhibited strong UV–Vis absorption peaks at 263, 275, 321 and 355 nm assigned to π – π^* transition of the Li coordinated 2-(oxazol-2-yl)phenol ligand, while LiIm showed UV–Vis absorption peaks at 262, 320, and 341 nm by π – π^* transition of the Li coordinated 2-(1-methyl-imidazol-2-yl)phenol ligand. Peak positions of solution PL emission of LiOx and LiIm were 405 nm and 387 nm, respectively. The PL emission was shifted to short wavelength due to less aromaticity of imidazole group of LiIm. Phosphorescent emission of LiOx and LiIm appeared between 420 nm and 600 nm. First phosphorescent emission peaks of LiOx and LiIm were observed at 462 nm and 441 nm, which corresponded to triplet energies of 2.65 eV and 2.82 eV. The triplet energy of LiOx and LiIm was high because high triplet energy ligands were coordinated to Li metal. Therefore, LiOx can suppress triplet exciton quenching of green triplet emitter with a triplet energy of 2.40 eV and LiIm can block triplet exciton quenching of blue triplet emitter with a triplet energy of 2.65 eV.

As LiOx and LiIm were developed as n-type dopants to improve the mobility of electron transport materials, electron only device of LiOx and LiIm doped electron transport material was prepared. Device structure of electron only devices were ITO (50 nm)/Ca (5 nm)/BCP:LiOx (100:0, 90:10, 70:30)/Al (200 nm) and ITO (50 nm)/Ca (5 nm)/

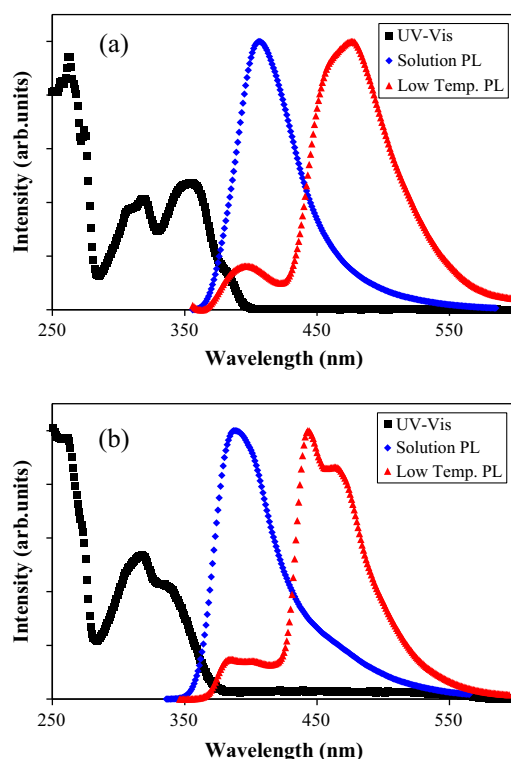


Fig. 1. UV–Vis absorption, solution PL and low temperature PL spectra of LiOx (a) and LiIm (b). Solution PL measurement was carried out using tetrahydrofuran as a solvent at a concentration of 1.0×10^{-5} M. Low temperature PL spectra were obtained in liquid nitrogen.

BmPyPb:LiIm (100:0, 90:10, 70:30)/Al (200 nm). BCP was used as the matrix material for LiOx for application in green PHOLEDs, while BmPyPb was utilized as the matrix material for LiIm for application in blue PHOLEDs. The thickness of the BCP:LiOx and BmPyPb:LiIm was 100 nm. Current density–voltage curves of the electron only devices are shown in Fig. 2. The current density of the LiOx doped BCP and LiIm doped BmPyPb devices was increased compared with that of the non-doped BCP and BmPyPb devices due to n-type doping effect caused by anion formation [12] or reduction of energetic disorder [16] as reported in other works.

Mobility of the LiOx doped BCP and LiIm doped BmPyPb was calculated from space charge limited current curve of the single carrier devices. Electron mobility–(electric field) $^{1/2}$ curves of LiOx doped BCP electron only devices and LiIm doped BmPyPb electron only devices are shown in Fig. 3(a) and (b). The electron mobility was calculated from the space charge limited current region of the current density–voltage curves of the electron only devices. The electron mobility of BCP was 5×10^{-7} cm ^{2} /V s at an (electric field) $^{1/2}$ of 650 (V/cm) $^{1/2}$, but it was increased to 4×10^{-5} and 1×10^{-4} cm ^{2} /V s at the same electric field by doping 10 mol% and 30 mol% of LiOx. Two order increase of the electron mobility was observed by doping LiOx in the BCP electron transport layer. This result confirms that the LiOx dopant plays a role of n-type dopant in the BCP electron transport layer. Similar results were

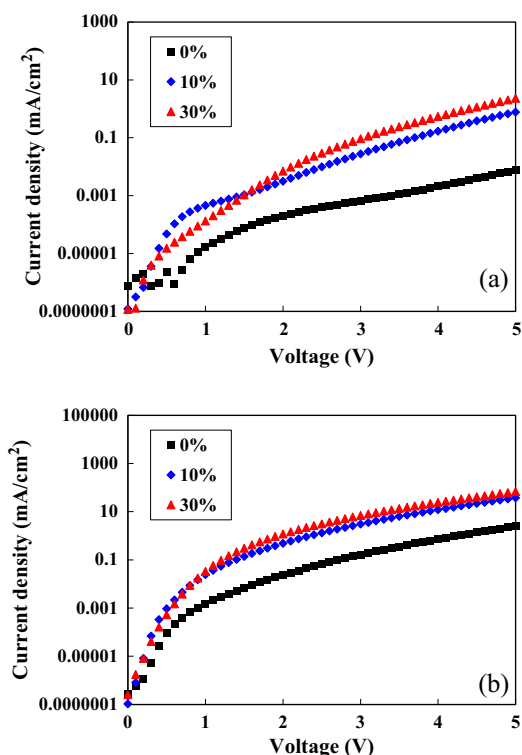


Fig. 2. Current density–voltage curves of electron only devices of LiOx doped BCP electron transport material (a) and LiIm doped BmPyPb electron transport material (b) according to doping concentration of LiOx and LiIm.

obtained by doping LiIm in the BmPyPb electron transport layer. The electron mobility of BmPyPb was $1.2 \times 10^{-5} \text{ cm}^2/\text{V s}$ at an (electric field) $^{1/2}$ of $400 (\text{V}/\text{cm})^{1/2}$ and the electron mobilities of 10 mol% and 30 mol% LiIm doped BmPyPb were $1.7 \times 10^{-4} \text{ cm}^2/\text{V s}$ and $6.6 \times 10^{-4} \text{ cm}^2/\text{V s}$, respectively. Therefore, the LiIm compound was effective to enhance the electron mobility of the BmPyPb electron transport material.

As LiOx and LiIm were effective to increase the electron mobility of the BCP and BmPyPb electron transport materials, they were tested as the n-type dopants in PHOLEDs. Green PHOLEDs were fabricated using LiOx as the triplet energy of the LiOx dopant was 2.65 eV compared with the triplet energy of 2.40 eV of green triplet emitter. Blue PHOLEDs were grown using LiIm because the triplet energy of LiIm (2.82 eV) was suitable for application in blue PHOLEDs. Liq was used as the standard n-type dopant for the BCP and BmPyPy electron transport material. Device structure of green PHOLEDs was ITO (50 nm)/PEDOT:PSS (60 nm)/TAPC (20 nm)/CBP:Ir(ppy) $_3$ (25 nm, 5% doping)/BCP or BCP:Liq or BCP:LiOx (40 nm)/Al (200 nm), while device structure of blue PHOLEDs was ITO (50 nm)/PEDOT:PSS (60 nm)/TAPC (20 nm)/mCBP: Irpic (25 nm, 10% doping)/BmPyPb or BmPyPb:Liq or BmPyPb:LiIm (40 nm)/Al. Electron injection layer was not used in the PHOLEDs. Fig. 4 shows current density–voltage–luminance curves of the green PHOLEDs with BCP electron transport layer doped with Liq and LiOx. Device data of the green

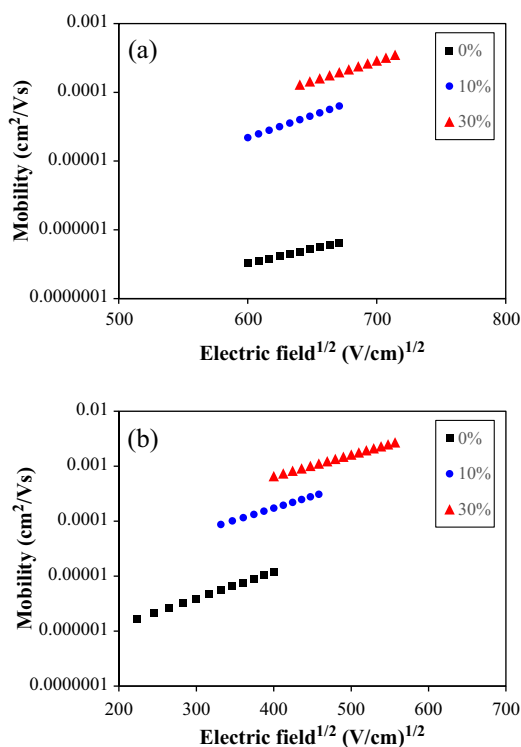


Fig. 3. Electron mobility–(electric field) $^{1/2}$ curves of LiOx doped BCP electron transport material (a) and LiIm doped BmPyPb electron transport material (b) according to doping concentration of LiOx and LiIm.

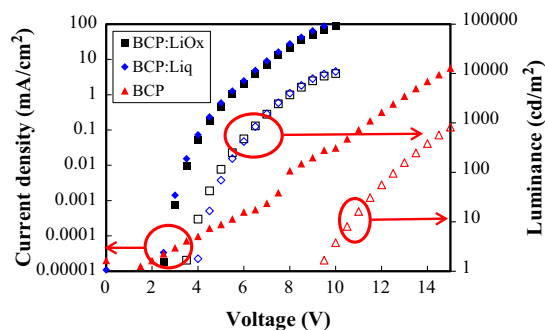


Fig. 4. Current density–voltage–luminance curves of green PHOLEDs with BCP, BCP:Liq or BCP:LiOx electron transport layers.

PHOLED with only BCP layer without doping were also added for comparison. The current density of the Liq or LiOx doped devices was similar, indicating that LiOx shows similar n-type doping effect to Liq. As confirmed by the electron mobility data, the LiOx increased the electron mobility of the BCP electron transport layer and exhibited similar n-type doping effect to Liq. Both Liq and LiOx doped devices showed much higher current density than the pure BCP device due to high mobility and better electron injection. The luminance was relatively high in the LiOx doped device compared to Liq device.

Quantum efficiency–luminance curves of Liq and LiOx doped green PHOLEDs are shown in Fig. 5. The quantum

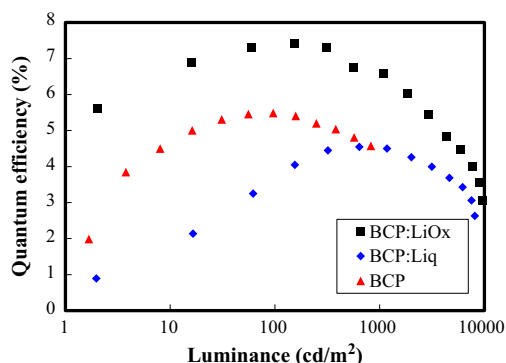


Fig. 5. Quantum efficiency–luminance curves of green PHOLEDs with BCP, BCP:Liq or BCP:LiOx electron transport layers.

efficiency of green PHOLEDs was high in the LiOx doped BCP device, while it was degraded in the Liq doped BCP device. This behavior is related with the triplet energy of Liq and LiOx. There are several device parameters affecting the quantum efficiency of PHOLEDs, which include charge balance, charge confinement and exciton confinement in the emitting layer [20]. Comparing the Liq and LiOx devices, the charge balance may have little effect on the quantum efficiency because the current density was similar in the two devices. The charge confinement would be also similar because the same BCP was used as the electron transport material. Although Liq and LiOx were doped in the BCP electron transport material, BCP dominates the charge confinement because of only 30% doping concentration of Liq or LiOx. Therefore, the different quantum efficiency is related with the exciton confinement. In particular, triplet exciton confinement is critical to the quantum efficiency of PHOLEDs because triplet emitters were used in the emitting layer. The triplet energy of Ir(ppy)_3 is 2.40 eV, while the triplet energies of Liq and LiOx are 2.00 eV and 2.65 eV from our measurement. The triplet energy of the Liq dopant is lower than that of Ir(ppy)_3 and quenches triplet excitons, while the high triplet energy of the LiOx dopant suppresses the triplet exciton quenching by n-type dopant, leading to high quantum efficiency compared to Liq. This can be proved by PL measurement of the Liq and LiOx doped BCP: Ir(ppy)_3 films in Fig. 6. PL intensity of the LiOx doped film was higher than that of

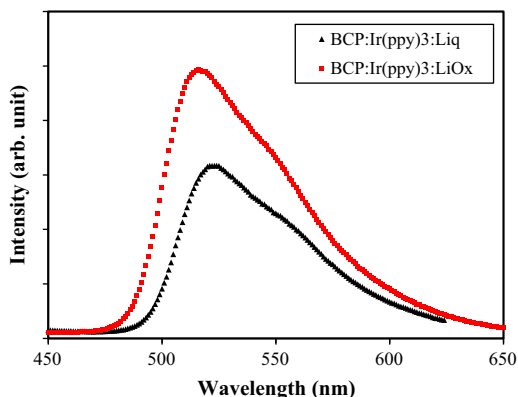


Fig. 6. PL spectra of BCP: Ir(ppy)_3 :Liq and BCP: Ir(ppy)_3 :LiOx films.

Liq doped film, proposing suppression of triplet exciton quenching of Ir(ppy)_3 by LiOx. Therefore, the use of LiOx dopant was effective to obtain high quantum efficiency in green PHOLED without any electron injection layer while increasing the electron mobility of electron transport materials. The quantum efficiency of the LiOx doped device was improved by more than 50% compared to that of the Liq doped device. The low quantum efficiency of the pure BCP device is due to carrier imbalance in the emitting layer.

Current density–voltage–luminance curves of blue PHOLEDs with LiIm doped BmPyPb electron transport layer are shown in Fig. 7. The current density of the blue PHOLEDs with LiIm doped BmPyPb electron transport layer was higher than that of the blue PHOLED with Liq doped BmPyPb, indicating better n-type doping effect of LiIm than Liq in the BmPyPb electron transport layer. The luminance was also higher in the LiIm doped BmPyPb device than in the Liq doped BmPyPb device. The pure BmPyPb device showed very low current density and luminance owing to low electron mobility and poor electron injection.

Quantum efficiency–luminance curves of blue PHOLEDs are presented in Fig. 8. The quantum efficiency of the blue PHOLEDs was enhanced by doping LiIm instead of common Liq dopant. The difference of the quantum efficiency of the blue PHOLEDs is originated from suppressed triplet exciton quenching effect by LiIm as explained in the LiOx device.

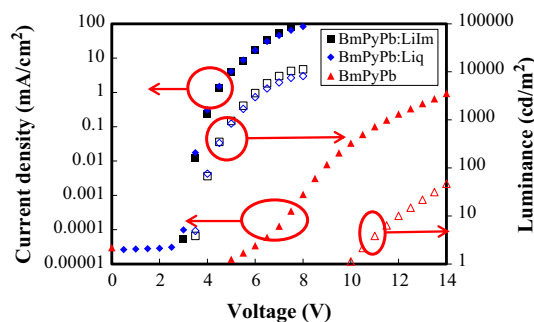


Fig. 7. Current density–voltage–luminance curves of blue PHOLEDs with BmPyPb, BmPyPb:Liq or BmPyPb:LiIm electron transport layers.

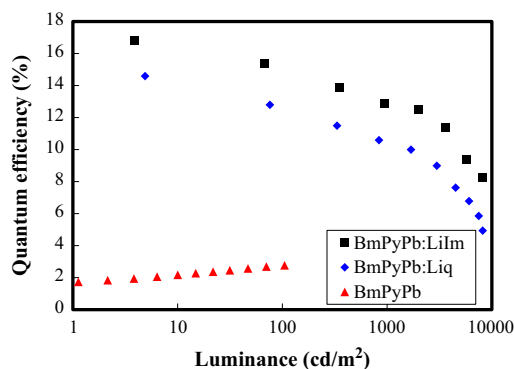


Fig. 8. Quantum efficiency–luminance curves of blue PHOLEDs with BmPyPb, BmPyPb:Liq or BmPyPb:LiOx electron transport layers.

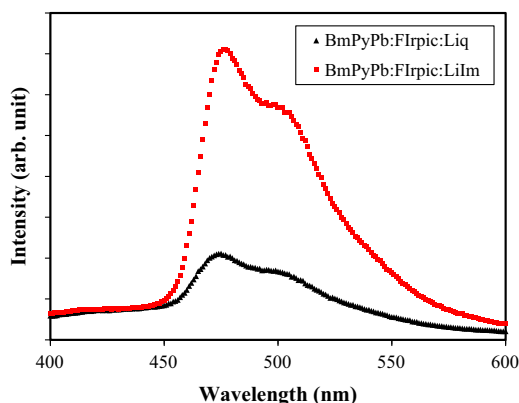


Fig. 9. PL spectra of BmPyPb:Flrpic:LiQ and BmPyPb:Flrpic:LiIm films.

As the triplet energy of LiIm (2.82 eV) was higher than that of Flrpic dopant (2.65 eV), the LiIm dopant can block triplet exciton quenching of Flrpic by n-type dopant. However, the LiQ dopant quenches the triplet excitons of Flrpic due to low triplet energy (2.0 eV) as can be confirmed by the PL spectra of LiQ and LiIm doped BmPyPb:Flrpic film in Fig. 9. Therefore, the LiIm dopant enhanced the quantum efficiency of blue PHOLEDs without any electron injection layer while improving the electron mobility of the BmPyPb electron transport layer. The quantum efficiency was improved by 22% by replacing LiQ with LiIm.

4. Conclusions

In conclusion, high triplet energy n-type dopants, LiOx and LiIm, were effectively synthesized to increase the mobility of electron transport materials and suppress triplet exciton quenching in green and blue PHOLEDs. Almost two order increase of electron mobility was observed by doping of LiOx and LiIm, and the quantum efficiency of electron injection layer free PHOLEDs was improved by

reduced triplet exciton quenching effect by LiOx and LiIm compared to LiQ. Therefore, the high triplet energy n-type dopant can replace current LiQ dopant and simplify the device structure by omitting an electron injection layer.

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