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抗菌医药左氧氟沙星在有机电致发光二极管中的应用

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摘要: 左氧氟沙星(LOFX)是一种知名的抗菌药物, 它的价格非常便宜, 且有成熟的合成和纯化技术. 本文中首次将 LOFX 作为一种蓝光发光材料和电子传输材料应用于有机电致发光器件(OLED)中. 通过热重分析、UV-Vis 吸收光谱、发射光谱以及循环伏安曲线详细地表征了 LOFX 的热学及光物理特性. LOFX 有高的分解温度, 为 327 °C; HOMO、LUMO 能级分别为 -6.2 和 -3.2 eV, 光学带隙为 3.0 eV. 以 LOFX 作为客体材料, 掺杂在主体材料 4,4'-二(9-咔唑)联苯(CBP)中制备了蓝光 OLED, 该器件的电致发光(EL)发射峰位于 452 nm, 最大亮度为 2315 cd·m⁻². 进一步, 选择 8-羟基喹啉铝(Alq₃)作为参考材料, 分别以 LOFX 和 Alq₃ 作为电子传输材料制备了结构相同的单载流子器件和绿色磷光 OLED. 在相同的电压下, 以 LOFX 作为电子传输材料的单载流子器件的电流密度比以 Alq₃ 作为电子传输材料的单载流子器件更高. 同时, 以 LOFX 作为电子传输材料的绿色磷光 OLED 获得更高的器件效率. 从这些 EL 性能可以看出, LOFX 同时也是一很好的电子传输材料.

关键词: 有机电致发光器件; 左氧氟沙星; 蓝光发光材料; 电子传输材料; 电致发光光谱; 电致发光性能

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Antimicrobial Drug Levofloxacin Applied to an Organic Light-Emitting Diode

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Abstract: Levofloxacin (LOFX) is a well-known and inexpensive antimicrobial drug that can be easily synthesized and purified. We report the first application of LOFX to an organic light emitting diode (OLED). Its thermal and photophysical properties were thoroughly investigated using thermogravimetric analysis (TGA), UV-Vis absorption spectra, emission spectra, and cyclic voltammetry. LOFX has HOMO and LUMO energies

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of -6.2 and -3.2 eV, respectively, and high molecule decomposition temperature (T_d) of 327 °C. An OLED with a LOFX emitter shows electroluminescence (EL) at 452 nm and maximum luminance of 2315 $\text{cd} \cdot \text{A}^{-1}$, which can be used in a white OLED. To investigate the electron transporting ability of LOFX, an electron-carrier only OLED was made. In addition, a green OLED based on $\text{Ir}(\text{ppy})_3$ (fac-tris(2-phenylpyridine)iridium) with electron transporting layer of LOFX was made, comparing with that with electron transporting layer of tris(8-hydroxyquinoline) aluminum (Alq_3). The former exhibited higher device efficiencies than that of the latter. The results show that LOFX has a higher electron transport ability than Alq_3 .

Key Words: Organic light-emitting diode; Levofloxacin; Blue-light emitting material; Electron transport material; Electroluminescence spectrum; Electroluminescence performance

1 Introduction

Organic light-emitting diodes (OLEDs) have attracted attention because of applications to full-color flat-panel displays, lighting, and sensor.¹⁻⁷ The fabrication cost is one of the important factors for the mass-production OLED companies. Organic materials, which are less-expensive and established in the synthesis and purification technologies, are preferable for the mass production of OLED devices. In our research to find such a material, beyond the field of chemical compounds for conventional light emitting semiconductors, we have extended our attention to a field of medicament and noticed a blue fluorescence emitting antimicrobial medicament, levofloxacin.

Levofloxacin $\text{C}_{18}\text{H}_{20}\text{FN}_3\text{O}_4$ ((S)-9-fluoro-2,3-dihydro-3-methyl-10-(4-methylpiperazin-1-yl)-oxo-7H-pyrido[1,2,3-de]-1,4-benzoxazine-6-carboxylic acid, called LOFX hereafter) is an oral broad spectrum antibiotic of the fluoroquinolone drug, which is widely used in the treatment of certain bacterial infections including pneumonia, urinary tract infections, and abdominal infections.⁸⁻¹² A blue emission has been reported for LOFX.¹³ The molecular structure is shown in Fig.1.

Gunasekaran *et al.*¹⁴ have reported that the intense absorption bands from LOFX were observed at 323 , 281 , and 255 nm. The absorption bands which appeared below 323 nm were confirmed from the photoluminescence excitation (PLE) spectra of LOFX in solution, which contain the 330 and 286 nm PLE bands corresponding to the 323 and 281 nm absorption bands, respectively.¹³ Here, the 286 nm band is attributed to $\pi-\pi^*$ transition, while the 330 nm band to the $n-\pi^*$ transition.¹⁴

Although the infrared vibrational, UV-Vis absorption, photoluminescence (PL), and PLE spectra have been reported for LOFX,^{13,14}

the optical properties have not fully established yet. Its application to OLED has never been examined. In the present paper, we report the detailed optical properties of LOFX and the possibility of application to OLED materials.

Here, we investigate the physical properties (spectroscopic properties, electronic energy levels, and thermal stability) of LOFX, and fabricate three types of OLEDs, which are named as blue-light Device series-B, electron-only Device series-E, and green-light Device series-G, to examine whether LOFX is useful as OLED materials such as blue emitter and electron transporter. Molecular structures of all materials involved in this paper are shown in Fig.1. Schematic device structures and the energy levels of functional materials used in this work are shown in Fig.2.

2 Experimental details

LOFX was purchased from J&K Chemical and other materials involved in devices were purchased from Luminescence Technology Corp. The purity of all materials is $>98\%$ and directly used without further purification.

The thermogravimetry analysis (TGA) of LOFX was performed in a NETZSCH STA409C TGA system at a ramping rate of 10 °C $\cdot$ min⁻¹ under an argon flow of 10 mL $\cdot$ min⁻¹ from room temperature to 600 °C. The differential scanning calorimetry (DSC) of LOFX was performed in a NETZSCH STA409C TGA system at a ramping rate of 10 °C $\cdot$ min⁻¹ under an argon flow rate of 10 mL $\cdot$ min⁻¹ from room temperature to 300 °C. The UV-Vis absorption spectrum of LOFX aqueous solution (10^{-5} mol $\cdot$ L⁻¹) was recorded by Hitachi U3900 UV-Vis spectrophotometer. The photoluminescence spectrum of LOFX solid powder was measured by Cary Eclipse fluorescence spectrophotometer. The photoluminescence quantum yield (PLQY) of LOFX was measured by a FluoroMax-4 fluorescence spectrophotometer equipped with an integrating sphere. To examine the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) energies, the cyclic voltammetry (CV) curve was measured with Autolab/PG STAT302 electrochemical workstation in a three-electrode cell containing tetrabutyl perchloric acid amine (TBAP) (0.1 mol $\cdot$ L⁻¹ in the mixed solution of acetonitrile and methylene chloride (2:1, molar ratio)) as an electrolyte at the scan speed of 50 mV $\cdot$ s⁻¹. A platinum wire, a platinum electrode, and a calomel electrode were used as a working electrode, a counter electrode, and a reference electrode, respectively.

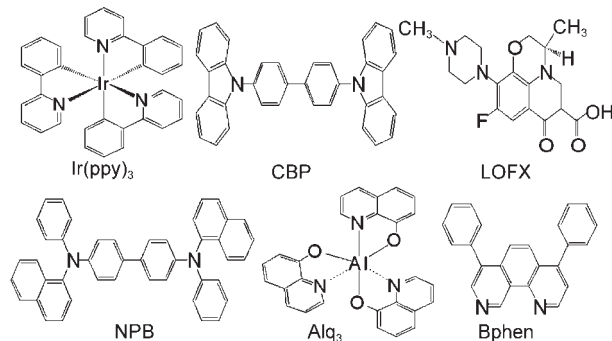


Fig.1 Molecular structures of materials involved in this paper

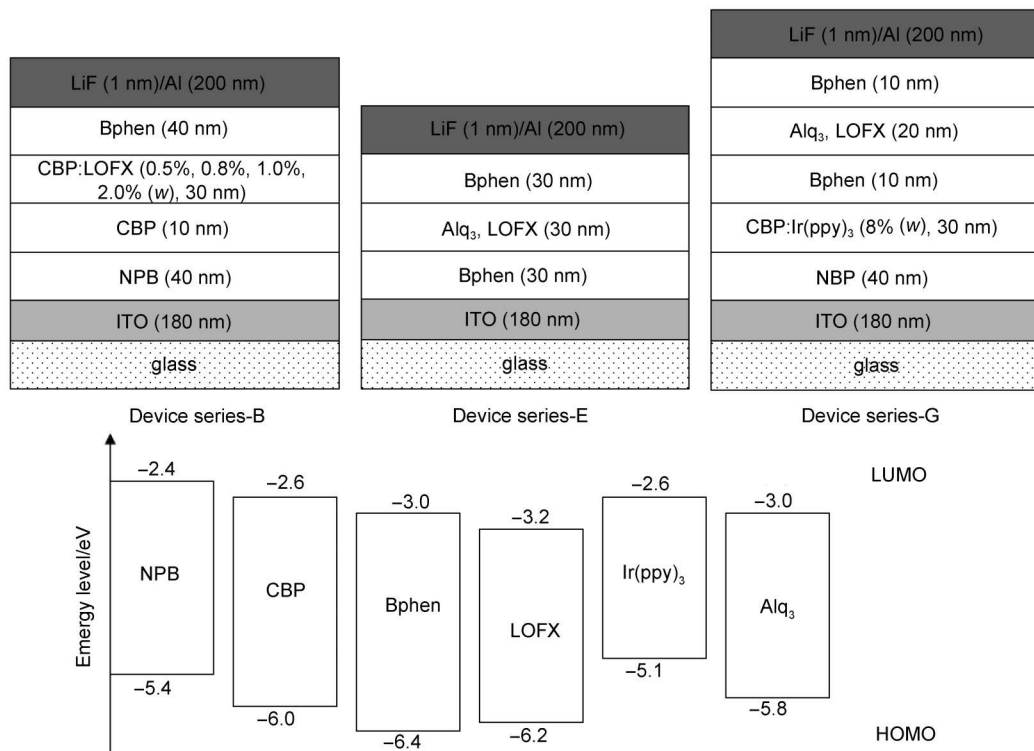


Fig.2 Schematic device structures of Device series-B, Device series-E, and Device series-G, and the energy levels of functional materials used in these devices

The doping concentration is in mass fraction (w).

OLEDs with the emission area of 3 mm×3 mm were fabricated on the pre-patterned indium tin oxide (ITO) glass substrate with sheet resistance of $15 \Omega \cdot \square^{-1}$. ITO substrates were cleaned by ultrasonication in baths of detergent water, deionized water, and acetone for 15 min successively, and then blown dry nitrogen and treated with UV ozone for 8 min, respectively. Then, the substrates were transferred into a vacuum chamber for sequential deposition of all organic functional layers by thermal evaporation below a vacuum of 5×10^{-4} Pa. The deposition rate for organic materials, LiF, and Al were about 0.1, 0.01, and 0.6 nm·s⁻¹, respectively. The device performances of OLEDs were characterized by Keithley 2400 source meter combined with Photo Research PR655 spectrometer simultaneously. All measurements were performed at room temperature in ambient atmosphere without device encapsulation.

3 Results and discussion

3.1 Physical properties of LOFX

Fig.3 shows the UV-Vis absorption and PL spectra of LOFX in deionized water. Absorption begins from about 400 nm, giving an absorption band with peak at 302 nm and a sideband at about 330 nm. Much intense band continues from about 230 nm. An intense PL band with peak at 445 nm is observed. Our absorption spectrum of LOFX in solution is consistent with the PLE spectrum of LOFX in aqueous solution by Polishchuk *et al.*¹³, although our PL spectrum is shifted from 485.3 nm¹³ to 445 nm. However, our absorption spectrum is not consistent with the absorption spec-

trum by Gunasekaran *et al.*¹⁴ who observed an absorption band at 400 nm. It seems that the 400 nm band is due to an aggregate.

The thermal stability is an important factor for organic electroluminescent materials. Fig.4(a) shows the TG and differential thermogravimetry (DTG) curves of LOFX, and Fig.4(b) shows the DSC curves of LOFX. In TG curve, a 5% weight loss was observed at 327 °C, indicating a high decomposition temperature (T_d) and good thermal stability of LOFX. Meanwhile, in DSC curve, LOFX exhibited a high glass transition temperature (T_g) of 161 °C. The high T_g and T_d values render LOFX an OLED material capable of forming stable amorphous films through vacuum thermal evaporation and upon heating.¹⁵

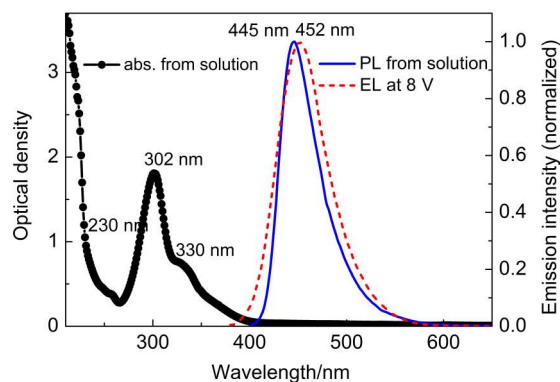


Fig.3 UV-Vis absorption and PL spectra of LOFX in deionized water solution excited at 360 nm, compared with the electroluminescence (EL) spectrum

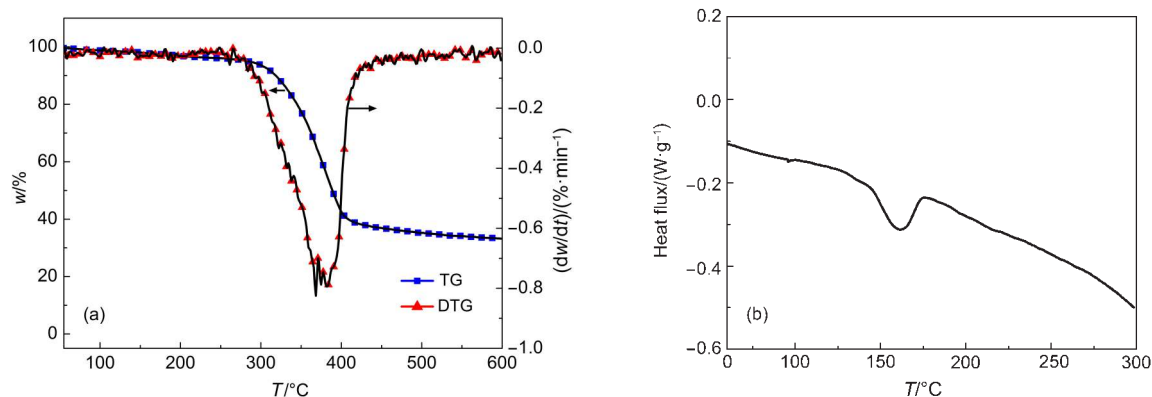


Fig.4 (a) TG and DTG curves of LOFX; (b) DSC curve of LOFX

Fig.5 shows the CV curve of LOFX. From the CV curve, two oxidation peaks at about 1.0 and 1.5 V can be observed. The oxidation peaks at about 1.0 V and the onset oxidation potential at about 0.8 V should be ascribed to the oxidation of acetonitrile. And the oxidation peak at about 1.5 V and onset oxidation potential at about 1.4 V should be assigned to the oxidation of LOFX. There is an empirical equation $E_{\text{HOMO}} = -(E_{\text{onset}}^{\text{ox}} + 4.8)$ (eV), where $E_{\text{onset}}^{\text{ox}}$ stands for the onset potential for oxidation, the potential of saturated calomel electrode (SCE) relative to the vacuum level is 4.8 eV.^{16,17} Therefore, the HOMO level is -6.2 eV for LOFX. From the absorption spectrum of LOFX (see Fig.3), the wavelength of absorption edge (λ_{edge}) is above 414 nm. The optical gap (E_g) was obtained according to the following equation: $E_g = 1240/\lambda_{\text{edge}}$.¹⁸ So, the E_g of LOFX is 3.0 eV, and $E_{\text{LUMO}} = E_g + E_{\text{HOMO}}$, is -3.2 eV. Here, the LUMO level of LOFX is lower than that of Alq₃ (-3.0 eV) but higher than the work function of Al (-4.1 eV), indicating that LOFX has an electron transport characteristic.

3.2 LOFX as blue-light emitting material in OLED

From above experimental results, it can be seen that LOFX expresses remarkable blue fluorescence effect, indicates that LOFX has potential application in OLED. Further, we measured the fluorescence quantum yield of LOFX solid powders, pure LOFX film, and CBP doped with 1% LOFX film (in mass fraction). The results indicate that CBP doped with 1% LOFX film shows a high PLQY of 12.53%, is higher than those of LOFX solid powders (5.54%) and pure LOFX film (6.25%). It is ex-

pected that LOFX is doped into host material to structure high performance device. For identifying it, a series of blue light OLED (Device series-B)(see Fig.2) are fabricated using LOFX as blue emitter with the device configuration of indium tin oxide (ITO)/NPB (40 nm)/CBP (10 nm)/CBP:wLOFX (30 nm)/Bphen (40 nm)/LiF (1 nm)/Al (200 nm), where the concentrations (in w , mass fraction) of LOFX are changed as 0.5%, 0.8%, 1.0%, and 2.0%. Here, NPB is *N,N'*-bis-(naphthyl)-*N,N'*-diphenyl-1,1'-biphenyl-4,4'-diamine, which is used as hole transport layer (HTL); CBP is 4,4'-bis(carbazol-9-yl)biphenyl, which is used as the exciton-block layer; Bphen is 4,7-diphenyl-1,10-phenanthroline, which is used as electron transport layer (ETL). The layer of CBP doped with 1.0% LOFX is a light emitting layer (EML), while LiF and Al are used as electron injection layer (EIL) and cathode, respectively.

Fig.6(a) shows the luminance-voltage ($L-V$) curves of Device series-B with different doping concentrations of LOFX in CBP. As doping concentrations increase from 0.5% to 1%, the maximum luminance of Device series-B increases from 1593 $\text{cd} \cdot \text{m}^{-2}$ at 0.5% to 2315 $\text{cd} \cdot \text{m}^{-2}$ at 1%. When doping concentration is 2%, the maximum luminance of Device series-B lowers to 736.8 $\text{cd} \cdot \text{m}^{-2}$. Lower doping concentrations limit radioactive recombination on dopant sites and induce in inadequate utilization of excited energy originated from CBP. On the contrary, higher doping concentrations result in serious concentration quenching of LOFX.¹⁹ So, the optimum doping concentration of LOFX in CBP is determined to be 1.0%. The Device series-B ($w=1.0\%$) exhibited a turn-on voltage, defined as the voltage measured at 1 $\text{cd} \cdot \text{m}^{-2}$, of around 4.5 V and a maximum luminance of 2315 $\text{cd} \cdot \text{m}^{-2}$ driven by voltage of 7.5 V.

Fig.6(b) shows the EL spectra of Device series-B ($w=1.0\%$) driven by various bias voltages, inset is the photograph of Device series-B ($w=1.0\%$) under voltage of 8 V. An EL band with emission peak at 452 nm due to LOFX is observed clearly when driven voltage is above 6 V. The EL band red-shifted by 7 nm from the PL band (Fig.3) observed in solution owing to a solid state effect.²⁰ From Fig.6(b), it can be seen that no obvious EL band shift was observed by changing the operating voltage. The Device series-B ($w=1.0\%$) emitted a pure blue light with com-

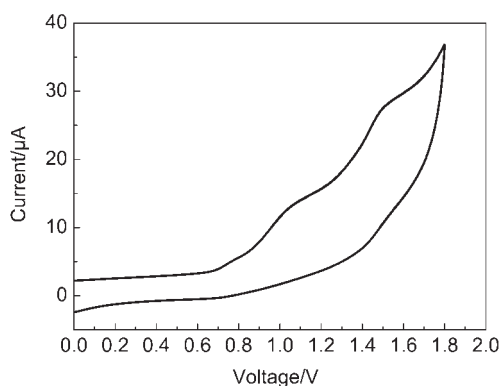


Fig.5 CV curve of LOFX

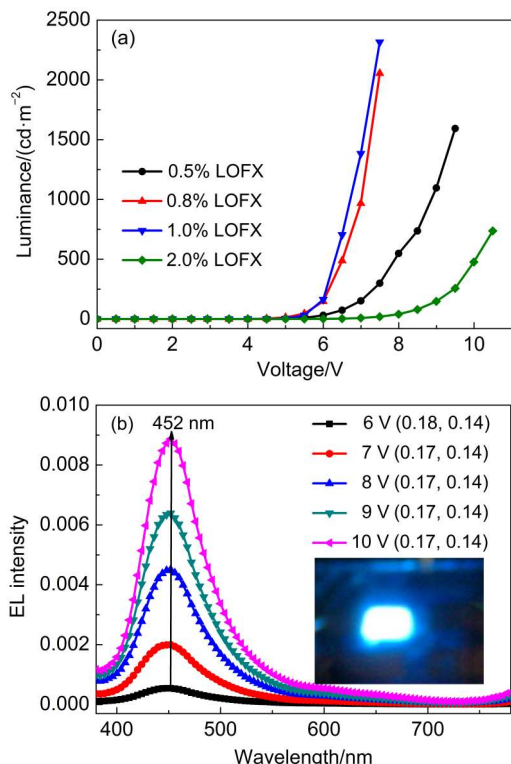


Fig.6 (a) Luminance–voltage (L – V) curves of Device series-B with different doping concentrations of LOFX in CBP; (b) EL spectra of Device series-B ($w=1.0\%$) at various voltages with CIE coordinates

inset in figure (b): the photograph of Device series-B ($w=1.0\%$) at 8 V

mission internationale de l'éclairage (CIE) 1931 coordinates coordinates of (0.17, 0.14) at 7–10 V, which is close to the National Television System Committee (NTSC) blue standard.²¹ These EL performances indicate that LOFX as blue emitter is useful for OLEDs.

3.3 LOFX as electron transport material in OLED

Further, we investigate if LOFX is useful as electron transporting material in OLEDs. To check this point, we investigated the current density–voltage characteristic by fabricating an electron-carrier-only OLED (called Device E-2) with structure of ITO/Bphen (30 nm)/LOFX (30 nm)/Bphen (30 nm)/LiF (1 nm)/Al (200 nm) (see Fig.2). We compare its characteristics with another electron-carrier-only OLED (called Device E-1) based on Alq₃, which is well-known as good electron transporting material. Device E-1 has the quite similar layer structure as Device E-2, i.e., ITO/Bphen (30 nm)/Alq₃ (30 nm)/Bphen (30 nm)/LiF (1 nm)/Al (200 nm) (see Fig.2). The current densities of these two devices are plotted against applied voltage in Fig.7. Device E-2 with LOFX has higher current density than Device E-1 with Alq₃ at the same driving voltage. Device E-2 has much lower turn-on voltage than Device E-1. From these results, LOFX is confirmed its superiority to Alq₃ in electron-transporting capability.

To further check the electron-transporting superiority of LOFX to Alq₃, we fabricate two green-emitting OLEDs with Ir(ppy)₃ ((fac-tris(2-phenylpyridine) iridium) emitter (Devices G-1 and G-

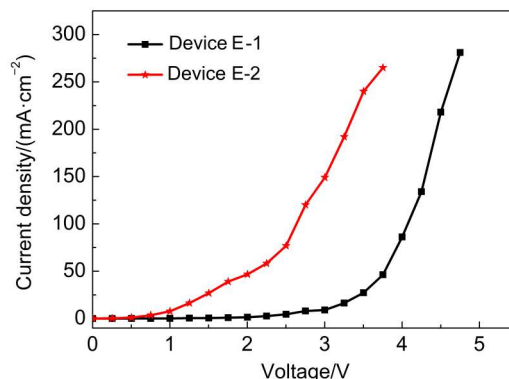


Fig.7 Current density–voltage curves of Device E-1 and Device E-2

2). Devices G-1 and G-2 have the same structure except ETL layer, i.e., ITO/NPB (40 nm)/BP:Ir(ppy)₃ (8%, 30 nm)/Bphen (10 nm)/Alq₃ or LOFX (20 nm)/Bphen (10 nm)/LiF (1 nm)/Al (200 nm) (see Fig.2), respectively, where NPB is used as HTL, CBP: Ir(ppy)₃ complex as EML, and Bphen adjacent to LiF as EIL. Fig.8(a, b, c, d) show the luminance–voltage, current density–voltage, current efficiency–current density, and power efficiency–voltage characteristics of Device G-1 with Alq₃ and G-2 with LOFX, respectively. For Devices G-1 and G-2, the turn-on voltages are 3.5 and 4.0 V, the high luminance of 36267 cd·m⁻² at 7.5 V and 36600 cd·m⁻² at 8.5 V, the maximum current efficiency of 15 cd·A⁻¹ at 47.5 mA·cm⁻² and 17.7 cd·A⁻¹ at 16.7 mA·cm⁻², the maximum power efficiency of 8.86 lm·W⁻¹ at 4.5 V and 9.96 lm·W⁻¹ at 5.5 V, respectively. The current efficiency is higher at low current densities of 8–200 mA·cm⁻² in Device G-2 than that in Device G-1 (Fig.8(a, c)), and the maximum power efficiency is also higher in Device G-2 than that in Device G-1 (Fig.8(d)). One of the reasons of higher efficiency of Device G-2 than Device G-1 is higher electron transport of LOFX than Alq₃. Another reason is that Device G-2 blocks holes more efficiently than Alq₃ because LOFX has lower HOMO level (–6.2 eV) than Alq₃ (–5.8 eV).

Regarding the low operational voltage, Alq₃ is superior to LOFX. This is understood by higher electron injection barrier (0.2 eV) at LOFX/Bphen interface relative to the barrier (0 eV) at Alq₃/Bphen interface, leading to more difficulty to enter electrons from cathode to EML in Device G-2 than that in Device G-1.

The maximum current efficiency of Device G-2 with LOFX is superior to Device G-1 with Alq₃. However the efficiency of 17.7 cd·A⁻¹ is smaller than the conventional efficiencies of OLEDs with phosphorescence Ir(ppy)₃. For example, Baldo *et al.*²² obtained 28 cd·A⁻¹ using an OLED with EML of CBP doped with 6% Ir(ppy)₃.

To find this reason, we examine the EL spectra of Device G-2 carefully by semi-log plotting. As seen in Fig.9, a small EL band appears at about 445 nm besides the 510 nm EL band due to Ir(ppy)₃. We compare the PL spectrum of NPB neat film with the EL spectra (Fig.9). NPB gives PL band with peak at about 445 nm, which coincides with the weak band. Therefore the 445 nm EL band is attributable to NPB. This indicates that electrons from

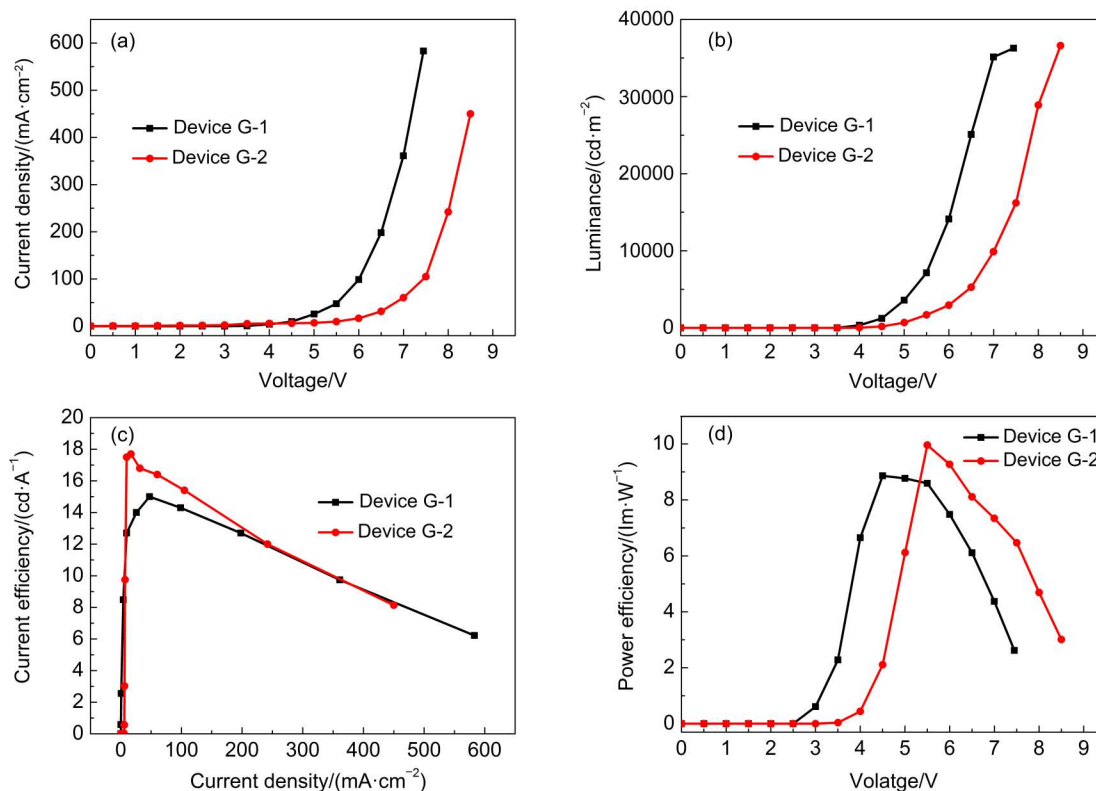


Fig.8 (a) Current density–voltage curves, (b) luminance–voltage characteristic, (c) current efficiency–current density curves, and (d) power efficiency–voltage curves of Devices G-1 and G-2

cathode are leaked to the NPB layer. This leakage is understood from the energy level diagram of Device G-2 which shows that the energy gap (0.2 eV) of LUMO energy between CBP of EML and NPB of HTL is small (Fig.2).

Device G-2 is more enhanced the roll-off effect than Device G-1. This is understood as follows. Better electron transportation leads to higher triplet-triplet annihilation and roll-off.^{23–25} It is suggested that the electron injection to EML is better to LOFX layer than to Alq₃ layer because of better electron transportation in LOFX layer than in Alq₃ layer as mentioned above. This leads to higher electron accumulation in EML with phosphorescent Ir(ppy)₃ emitter of Device G-2 at high current densities than in EML of Device G-1, resulting in stronger roll-off for Device G-2

than for Device G-1. In this way, the superior of LOFX electron transportation to Alq₃ is confirmed from the roll-off.

4 Conclusions

To investigate whether LOFX, well-known as an antimicrobial medicament, is useful as OLED materials such as blue emitter and electron transporter, we have studied the spectroscopic properties, electronic energy levels, thermal stability, electroluminescence, and OLED characteristics. LOFX shows a blue PL band at 446 nm, HOMO and LUMO energies of -6.2 and -3.2 eV, respectively, and high molecule decomposition temperature (T_d) at 327 °C. The blue OLED with LOFX emitter shows a pure blue emission with a peak at 452 nm and a maximum luminance of 2315 cd m⁻². Further, LOFX is found to be higher in electron-transporting ability than Alq₃, which was obtained using the electron-transport-only device. In the case that LOFX is used as ETL in green emitting OLED with Ir(ppy)₃, the maximum current density of 17.7 cd A⁻¹ and the maximum power efficiency of 9.96 lm W⁻¹ are obtained, which are higher than 15.0 cd A⁻¹ and 8.86 lm W⁻¹ in the OLED with ETL of Alq₃. The current efficiency is lower than the conventional efficiency of OLEDs with Ir(ppy)₃. This is attributed to leakage of electrons from EML to HTL of NPB in the present OLED device. From the EL performances, it is suggested that LOFX can act as a desired bifunctional material: not only a pure blue emitter, but also a excellent electron transport material in OLED devices, which is useful for OLEDs.

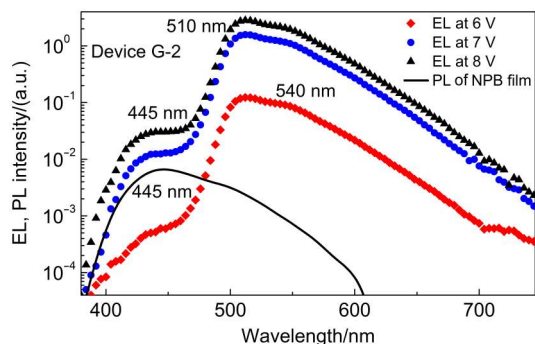


Fig.9 Semi-log plotted EL spectra of Device G-2 at various voltages, compared with the PL spectrum of NPB neat film excited at 350 nm

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