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A highly efficient tris-cyclometalated iridium complex based on phenylphthalazine derivative for organic light-emitting diodes

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ABSTRACT

A novel ligand 9-(4-(4-chlorophenyl)phthalazin-1-yl)-9*H*-carbazole (HCPC) was designed and prepared, and the corresponding tris-cyclometalated iridium(III) complex $Ir(CPC)_3$ was readily synthesized by the reaction of the ligand with $IrCl_3 \cdot 3H_2O$ at 80 °C for 20 h. A highly efficient organic light-emitting device using this complex as a dopant was obtained. The device fabricated by solution process showed a maximum luminance of 2948 cd/m^2 at a current density of 115.6 mA/cm^2 and a maximum external quantum efficiency of 20.2% at 0.18 mA/cm^2.

1. Introduction

Organic light-emitting diodes (OLEDs) have attracted great attention in the past decades because of their potential advantages in full-color displays [1]. As phosphorescent dopants in the emitting layers, heavy metal complexes, such as iridium(III), platinum(II), osmium(II), and ruthenium(II) complexes, can harvest both singlet and triplet excitons for emission and hence lead to efficient OLEDs with the internal quantum efficiency potentially as high as 100% [2–5]. By reason of the theoretical limit from simple classical optics, the external quantum efficiency is confined to ~20% [6]. An external quantum efficiency of 19.2% ph/el at a current density of 0.55 mA/cm^2 was achieved by using tris(2-phenylpyridine) iridium(III) ($Ir(ppy)_3$) as a dopant [7]. Kang et al. reported the efficient OLEDs based on $Ir(2\text{-}(1\text{-cyclohexenyl})pyridine)_3$ with a maximum external quantum efficiency of 18.7% [8]. Most of reported highly efficient OLEDs were achieved by vacuum deposition technique, and yet solution process owning low cost and convenience advantages is expected to be important to OLEDs for large-area displays [9]. A solution processed OLEDs with an external quantum efficiency of 13.3% at 6.3 mA/cm^2 has been prepared by adopting a neutral osmium(II) complex other than iridium complexes [10].

Recently, we have reported the red device using tris-(1-(2,6-dimethylphenoxy)-4-(4-chlorophenyl)phthalazine) iridium(III) as a dopant by solution process with internal quantum efficiency of nearly 100% [11]. Mi et al. reported

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efficient and thermally stable OLEDs using tris(1,4-bis(phenyl) phthalazine) iridium(III) [12]. Based on these works, it is found that the ligands which have the structure of an sp^2-hybrid N-atom adjacent to the chelating N-atom, such as phenylpyridazine and phenylphthalazine derivatives [13], are beneficial for iridium(III) complexes due to the shorter bonding length and the stronger bonding strength between the chelating N-atom and Ir-atom, compared with these analogues which have a C-atom instead of the non-chelating N-atom. It is well known that carbazole-based compounds have been widely used as the host materials for OLEDs due to their high triplet energy and good hole-transporting ability. In addition, there are many reports about the integrations of carbazole modules and iridium(III) complexes which possess the dual functions of light emission and hole transportation [14–20].

In this article, we introduce carbazole unit into a phenylphthalazine derivative to get a novel ligand 9-(4-(4-chlorophenyl)phthalazin-1-yl)-9*H*-carbazole (HCPC). The ligand can bond to the Ir-atom more strongly, which leads to efficient mixing of the singlet and triplet excited states [12], and the incorporation of carbazole unit is beneficial to improve the charge balance in the electroluminescent (EL) process and enhance thermal stability [21–25]. The corresponding tris-cyclometalated iridium complex Ir(CPC)$_3$ is synthesized by the reaction of the ligand with $IrCl_3 \cdot 3H_2O$ at 80 °C for 20 h, directly. The thermal, photophysical and electrochemical properties of the complex are studied, and the performance of OLEDs fabricated by solution process is investigated.

2. Experimental

2.1. General information

The solvents were purified by routine procedures and distilled under an atmosphere of dry nitrogen before use. All reagents, unless otherwise specified, were purchased from Aldrich and were used as received. The original compound 1-chloro-4-(4-chlorophenyl)phthalazine was prepared according to the procedure previously reported [26]. ^{1}H NMR spectra were recorded on a Varian Inova500NB spectrometer using $CDCl_3$ as solvent. Elemental analyses of carbon, hydrogen, and nitrogen were performed on a Vario EL microanalyzer. Thermal gravimetric analysis (TGA) was performed under a flow of nitrogen at a heating rate of 10 °C/min with a Perkin–Elmer Pyris Diamond TG/DTA thermal analyzer. Differential scanning calorimetry (DSC) was performed on a Netzsch DSC-200PC instrument at a heating rate of 10 °C/min. Photoluminescence (PL) spectra were measured with a Shimadzu RF-5301PC fluorescence spectrophotometer. Luminescence lifetime was determined on an Edinburgh FL920 time-correlated pulsed single-photon-counting instrument. UV–vis absorption spectra were recorded on a Shimadzu UV-2501 PC spectrophotometer. PL quantum yield was measured at room temperature in an argon prepurged tetrahydrofuran (THF) solution and compared with quinine sulfate in 1.0 N H_2SO_4 ($\Phi_{PL} = 0.546$) as a standard [27]. Electron ionization (EI) mass spectrum were recorded on a Shimadzu GC–MS-QP2010 PLUS mass spectrometer, and electron spray ionization (ESI) mass spectra on a Thermo LCQ DECA XP mass spectrometer. Cyclic voltammetry (CV) was performed with a Solartron SI 1287 voltammetric analyzer. It was conducted at room temperature in a typical three-electrode cell with a working electrode (Pt disk), a reference electrode (saturated calomel electrode), and a counter electrode (Pt wire) under a nitrogen atmosphere at a sweeping rate of 100 mV/s in a solution of 0.1 M tetra-*n*-butylammounium hexafluorophosphate (Bu_4NPF_6) in anhydrous CH_2Cl_2. Each oxidation potential was calibrated with that of ferrocene.

2.2. Synthesis of 9-(4-(4-chlorophenyl)phthalazin-1-yl)-9H-carbazole (HCPC)

A 60.5% suspension of sodium hydride (1.0 g, 25 mmol) in paraffin oil was added slowly to a solution of carbazole (4.18 g, 25 mmol) in anhydrous dimethylformamide (70 mL) under nitrogen as a protective gas. After stirred for 1 h at room temperature, this mixture was added dropwise to a solution of 1-chloro-4-(4-chlorophenyl)phthalazine (6.88 g, 25 mmol) in anhydrous dimethylformamide (70 mL) with an ice bath. Then the mixture was stirred at room temperature for 12 h. After reaction, the resulting mixture was poured into water and extracted with chloroform. The extracts were combined, washed with brine, dried over anhydrous magnesium sulfate, and filtered. Then the solvent was evaporated. The product thus obtained was purified by silica gel column chromatography using chloroform as the eluent to give a yellow solid. Yield: 78%. mp: 191–193 °C. ^{1}H NMR ($CDCl_3$): δ 8.22–8.18 (t, 3H), 7.96–7.92 (t, 1H), 7.88–7.84 (d, J = 8.6 Hz, 2H), 7.82–7.78 (t, 1H), 7.76–7.72 (d, 1H), 7.65–7.61 (d, J = 8.6 Hz, 2H), 7.4–7.33 (m, 4H), 7.29–7.26 (d, 2H). EI-MS (m/z): 404 (100%), 405 (68%), 406 (43%), 407 (23%).

2.3. Synthesis of Ir(9-(4-(4-chlorophenyl)phthalazin-1-yl)-9H-carbazole)$_3$ [Ir(CPC)$_3$]

9-(4-(4-Chlorophenyl)phthalazin-1-yl)-9*H*-carbazole (0.408 g, 1.00 mmol) was dissolved in 2-ethoxyethanol (12 mL) in a 25 mL round-bottom flask. Iridium trichloride hydrate (0.1 g, 0.284 mmol) and water (4 ml) were then added to the flask. The mixture was stirred under nitrogen at 80 °C for 20 h and then cooled to room temperature. The precipitate formed in the mixture was collected and washed with methanol, ether and dried. The product thus obtained was purified by silica gel column chromatography using dichloromethane as the eluent to give a red solid. Yield: 34%. ^{1}H NMR ($CDCl_3$): δ 8.98–8.95 (d, J = 8.7 Hz, 1H), 8.30–8.27 (d, J = 8.7 Hz, 1H), 7.90–7.86 (t, 1H), 7.82–7.78 (d, J = 7.5 Hz, 1H), 7.70–7.67 (d, J = 7.7 Hz, 1H), 7.53–7.48 (t, 1H), 7.23–7.20 (d, J = 8.5 Hz, 1H), 7.20–7.19 (d, J = 2.3 Hz, 1H), 7.13–7.09 (dd, J = 8.5, 2.3 Hz, 1H), 6.92–6.88 (t, 1H), 6.65–6.60 (t, 1H), 6.55–6.52 (d, J = 8.2 Hz, 1H), 6.47–6.43 (t, 1H), 6.00–5.92 (m, 2H). ESI-MS (m/z): [M$^+$+H] Calcd. for $C_{78}H_{46}N_9Cl_3Ir$ 1404.26 (29%), 1405.23 (30%), 1406.25 (97%), 1407.25 (82%), 1408.21 (100%), 1409.21 (74%), 1410.20 (44%),1411.23 (22%). Found 1404.25 (33%), 1405.25 (30%), 1406.25 (100%), 1407.25 (82%), 1408.24(99%), 1409.25 (68%), 1410.25 (46%), 1411.24 (24%). Anal. Calcd. For $C_{78}H_{45}N_9Cl_3Ir$: C, 66.59; H, 3.22; N, 8.96. Found: C, 66.21; H, 3.45; N, 8.81%.

2.4. OLED fabrication and measurements

The indium tin oxide (ITO) glass substrate was washed in turn with a substrate-cleaning detergent, deionized water, acetone, and ethanol for 15 min, under ultrasonic condition, and finally treated with ozone for about 20 min. Poly(3,4-ethylenedioxythiophene) doped with poly (styrene sulfonic acid) (PEDOT) (Baytron P4083, Bayer AG) in water was spin-coated at a rate of 3000 rpm on the ITO substrate and dried by baking in air at 120 °C for 10 h. Then the solution of poly(vinylcarbazole) (PVK, $M_w = 81{,}800$) (68 wt%) blended with 2-tert-butylphenyl-5-biphenyl-1,3,4-oxadiazole (PBD) (30 wt%) and $Ir(CPC)_3$ (2 wt%) in chloroform was spin-coated at a rate of 1300 rpm on top of the PEDOT layer. The assembly was transferred into a deposition chamber with a base pressure of 3×10^{-6} Torr, a film of 1,3,5-tris(N-phenylbenzimidazol-2-yl)-benzene (TPBI) was evaporated onto the polymer layer. Finally, the Ba and Al layers were evaporated. Current density–voltage–luminance data were collected using a Keithley 236 source measurement unit and a calibrated silicon photodiode. External quantum efficiency was obtained by measuring the total light output in all directions in an integrating sphere (IS080, Labsphere). Luminance and luminous efficiency were measured by a silicon photodiode and calibrated using a PR-705 photometer (Photo Research).

3. Results and discussion

3.1. Synthesis and characterization

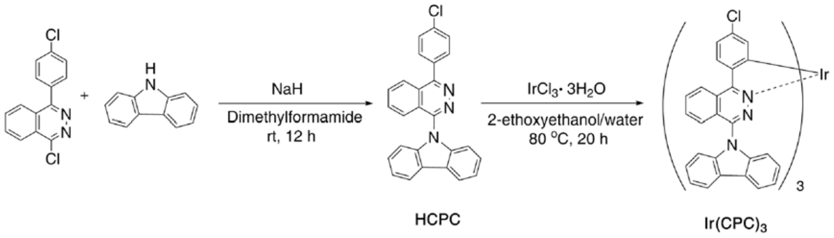
The synthetic procedures of the ligand and the complex are shown in Scheme 1. The cyclometalating ligand HCPC was prepared from 1-chloro-4-(4-chlorophenyl)phthalazine reacted with carbazole by an alkali of NaH in a polar solvent. Cl-atom on phthalazine has been replaced by carbazole unit, but Cl-atom on benzene ring did not take part in reaction due to low activity.

The complex $Ir(CPC)_3$ was synthesized by a simple procedure. HCPC reacted with $IrCl_3 \cdot 3H_2O$ directly formed the tris-cyclometalated iridium complex $Ir(CPC)_3$ at 80 °C for 20 h. Generally, the synthesis of tris-cyclometalated iridium(III) complexes is in need of Ir(III) organic complexes, such as $(C\hat{}N)_2Ir(O\hat{}O)$, $(C\hat{}N)_2Ir(\mu\text{-}Cl)_2Ir(C\hat{}N)_2$ or $Ir(O\hat{}O)_3$ (C^N = cyclometalating ligand, O^O = β-diketonate anion), reacted with excessive free cyclometalating ligand precursors at high temperature [28]. We believe this special reaction is relative to the strong bonding strength and the small steric hindrance between the ligand and the centric Ir-atom. Research on the mechanism of this reaction is currently underway. The complex was also characterized by ^1H NMR spectroscopy. The spectra indicate the facial isomer is formed, because three ligands surrounding the iridium atom are magnetically equivalent [29].

3.2. Thermal, absorption, photophysical and electrochemical properties

The thermal properties of the complex were characterized with TGA and DSC under a nitrogen stream. The TGA data reveal that the 5% weight-reduction temperature of $Ir(CPC)_3$ is 444 °C. In comparison, the analogous complex $Ir(MPCPPZ)_3$ which contains 2,6-dimethylphenoxy group instead of carbazole commences decomposition at 388 °C [11]. So, the introduction of carbazole unit can observably improve the thermal stability of the complex. The DSC curve shows no crystallization and melting peaks, and it is revealed that the complex is amorphous. The absorption and photoluminescence spectra of $Ir(CPC)_3$ in dichloromethane at room temperature are shown in Fig. 1. The absorption spectra show broad bands from 270 to 600 nm, and the most intense bands between 270 and 330 nm are attributed to the spin allowed π–π^* transitions from the cyclometalating ligand. The absorption bands around 350–470 nm can be assigned to the spin-allowed metal-ligand charge transfer band (^{1}MLCT). The absorption features at the lowest energy ($\lambda > 470$ nm) are attributed to the formally spin-forbidden ^{3}MLCT transition. Upon irradiation with 400 nm light, the complex gives a strong photoluminescence in dichloromethane at 615 nm. The PL quantum yield of $Ir(CPC)_3$ in THF at room temperature is 0.46. The emission decay curve of $Ir(CPC)_3$ in solid state is shown in Fig. 2. The lifetime of $Ir(CPC)_3$ shows double exponentials as 0.182 and 0.837 μs, respectively. Because long lifetime will give rise to the emission saturation and elevate the triplet–triplet annihilation, the suitable lifetime of Ir(III) complex (~0.1–3 μs) is beneficial to improve the efficiency of OLEDs [30].

The electrochemical properties of $Ir(CPC)_3$ were examined by cyclic voltammetry. The complex showed reversible oxidation processes during the anodic scan in dichloromethane solution, but no clear reduction peak was observed. The highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital



Scheme 1. Synthesis of $Ir(CPC)_3$.

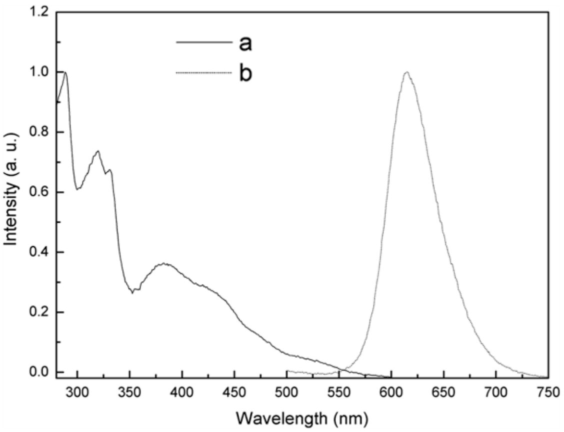


Fig. 1. UV–vis absorption (a) and photoluminescence (b) spectra of Ir(CPC)$_3$.

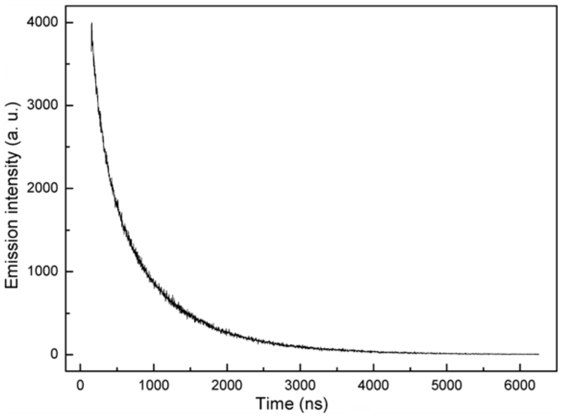


Fig. 2. Emission decay curve of Ir(CPC)$_3$ in solid state.

(LUMO) energy levels of Ir(CPC)$_3$ are −5.35 eV and −3.15 eV, estimated by using the oxidation potential and UV–vis absorption spectrum edge. Because its HOMO level is above that of the hosts (−5.8 eV for PVK, −6.2 eV for PBD) and its LUMO level is below that of the hosts (−2.2 eV for PVK, −2.6 eV for PBD), the complex can function as trap for both electrons and holes [31].

3.3. Electroluminescent properties

For studying EL properties of the complex, the OLEDs using Ir(CPC)$_3$ as a dopant was fabricated by solution process with the following structure: ITO/PEDOT(45 nm)/ 2 wt% Ir(CPC)$_3$+ 68 wt% PVK+ 30 wt% PBD (80 nm)/ TPBI(30 nm)/Ba(4 nm)/Al(100 nm). PEDOT was used as a hole-injecting material. PVK and PBD were selected as the host material and the electron transport material, respectively. TPBI acted as both a hole-blocker and an electron-transporter. Fig. 3 shows the general structure of the device and the molecular structures of the compounds used in the device.

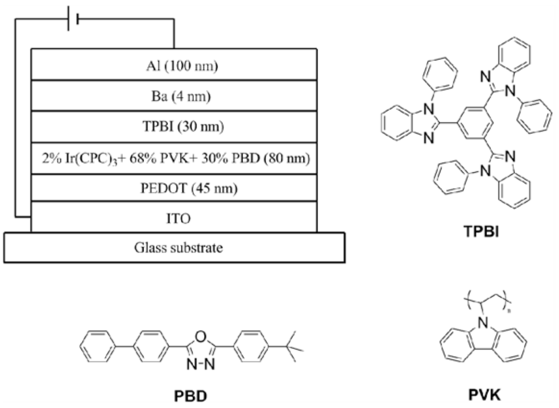


Fig. 3. General structure for the device and molecular structures of the relevant compounds.

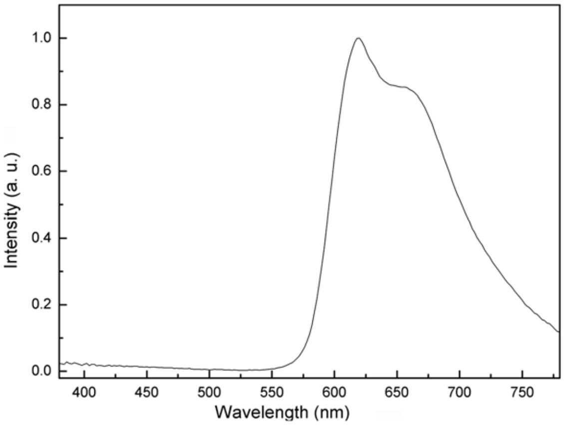


Fig. 4. Electroluminescence spectra of the device.

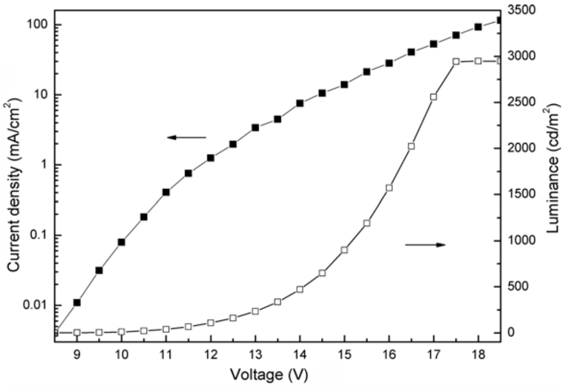


Fig. 5. Current density–voltage–luminance curves of the device.

The device has a maximum emission at 620 nm (Fig. 4), and the fine vibronic structure of the EL spectra leads us to conclude that EL emission originates from a mixed ^{3}MLCT/$^3\pi$–π^* state [30]. The Commission Internationale de L'Eclairage (CIE) coordinate is (0.65, 0.33) at a driving voltage of 12 V. No emission from the PVK–PBD blend is detected, which implies that the energy transfer from the blend to Ir(CPC)$_3$ is efficient under electrical excitation.

As shown in Fig. 5, the device gives a maximum luminance of 2948 cd/m^2 at a current density of

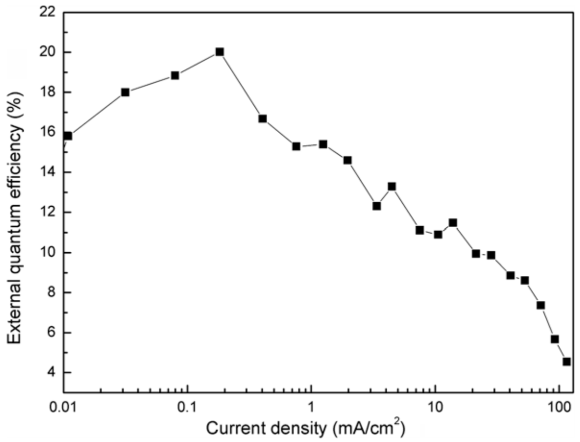


Fig. 6. External quantum efficiency vs. current density curve of the device.

115.6 mA/cm^2. A maximum external quantum efficiency of 20.2% corresponding to a luminous efficiency of 11.3 cd/A is obtained at a current density of 0.18 mA/cm^2. To the best of our knowledge, it is one of the highest reported efficiencies for red-emission OLEDs. Such an outstanding performance can be explained by the high PL quantum yield of $Ir(CPC)_3$ ($\Phi_{PL} = 0.46$), which is higher than those of other known high-performance iridium(III) complexes, such as $Ir(ppy)_3$ ($\Phi_{PL} = 0.40$) and $Ir(piq)_3$ (piq = 1-phenylisoquinolinato) ($\Phi_{PL} = 0.26$) [7,30]. The rigid ligand can significantly reduce the non-radiative transition. Moreover, the hole-transporting carbazole unit in the complex can improve the charge-balance properties in the EL process [21–23]. Compared to other OLEDs fabricated by vacuum deposition, this solution processed device has more advantages for large-area displays, such as low cost, simple preparation.

However, the efficiency roll-off, mostly due to triplet–triplet annihilation of phosphor-bound excitons, is severe at high current density as shown in Fig. 6. An external quantum efficiency of 10.9% is obtained at a current density of 10.6 mA/cm^2. When the current density increases to 92.7 mA/cm^2, the external quantum efficiency is 5.7% with a decrease of 47.7%. We believe this poor result arises from unstable thin-film morphology probably induced by the planar structure of the cyclometalating ligand, which tends to cause aggregation of molecules at high current density and then leads to excited-state intermolecular interactions.

4. Conclusions

In summary, we synthesized a new tris-cyclometalated iridium(III) complex by the reaction of 9-(4-(4-chlorophenyl)phthalazin-1-yl)-9*H*-carbazole with $IrCl_3 \cdot 3H_2O$ at 80 °C for 20 h. The device using the complex at 2 wt% doping concentration exhibited excellent performance. A maximum external quantum efficiency of 20.2% corresponding to a luminous efficiency of 11.3 cd/A at a current density of 0.18 mA/cm^2 and a maximum luminance of 2948 cd/m^2 at 115.6 mA/cm^2 were obtained. The optimization to the device for suppressing efficiency roll-off at high current density is in progress.

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