

Three novel Iridium(III) complexes containing double chiral atoms for OLEDs

Gan Cheng¹, Junjian Lu¹, Li-ya Zhu¹, and Gaozhao Lu²

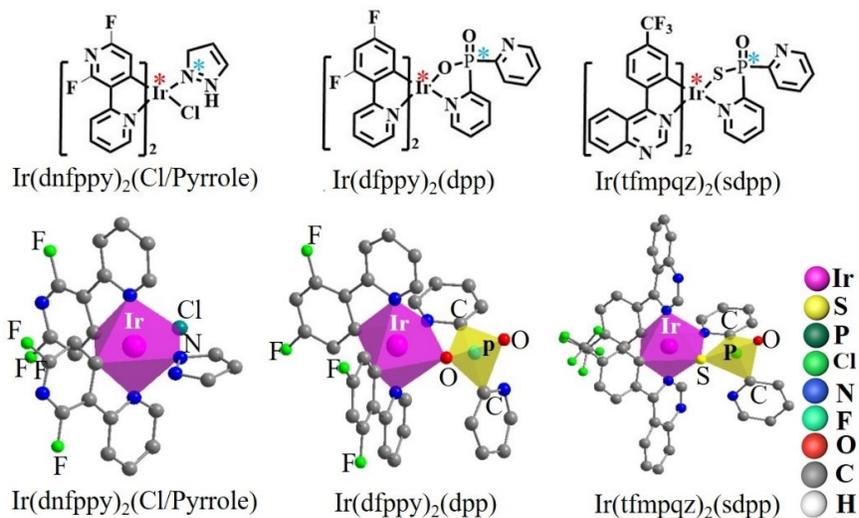
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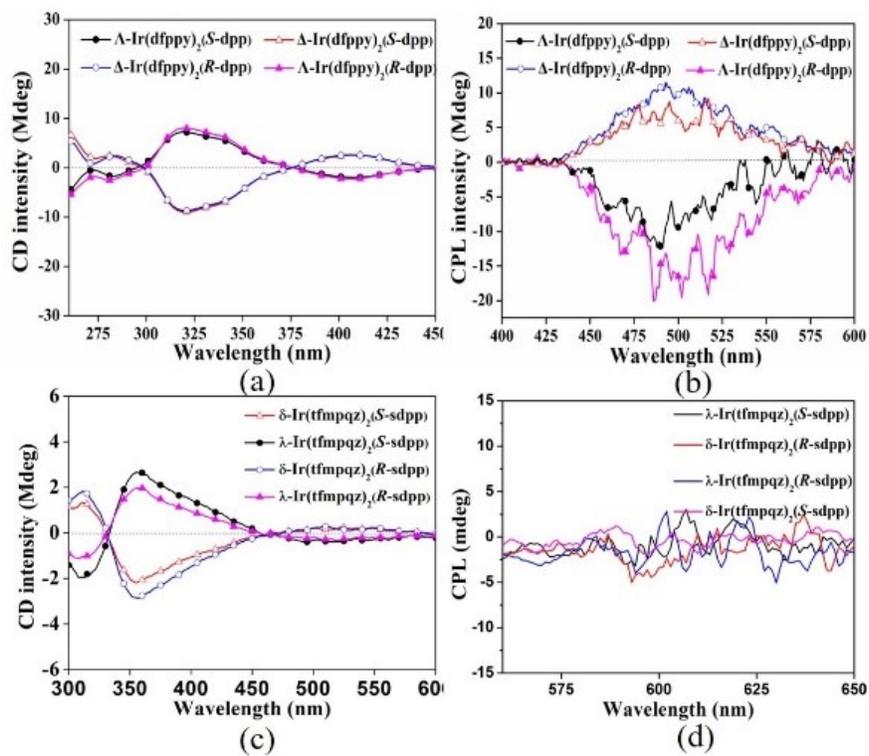
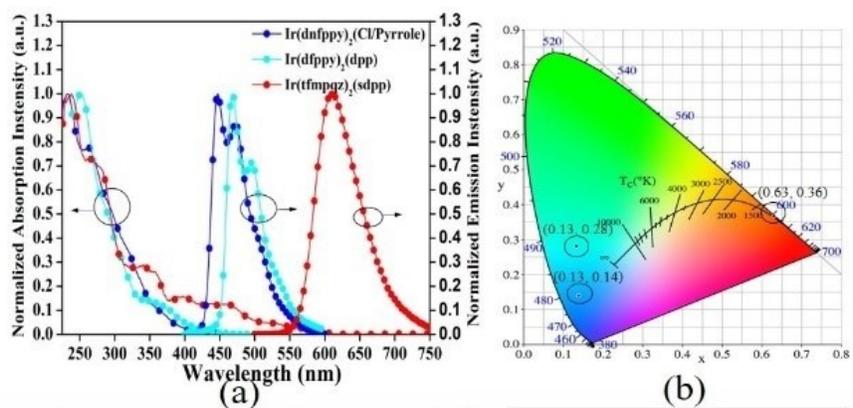
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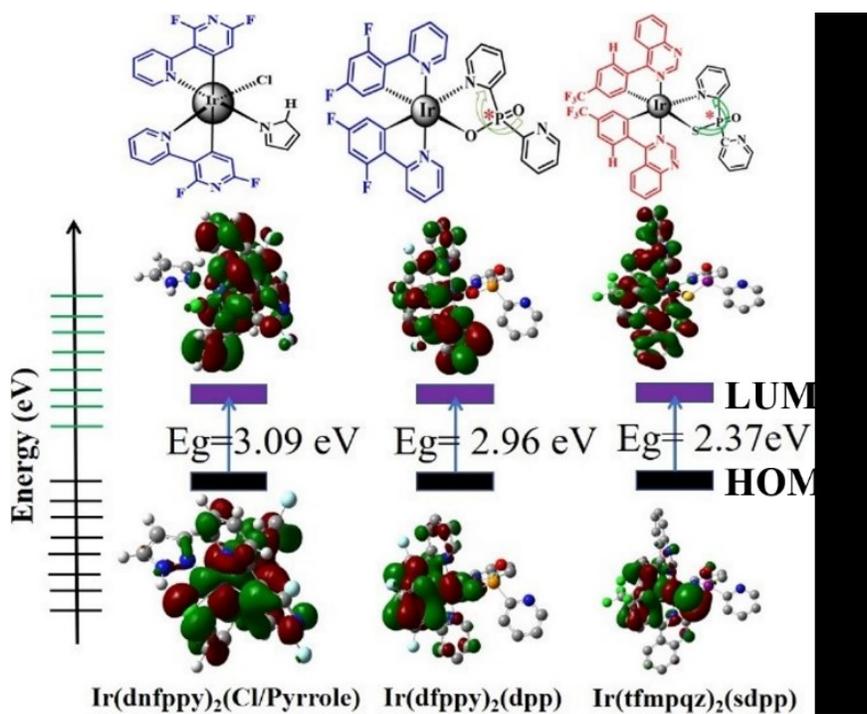
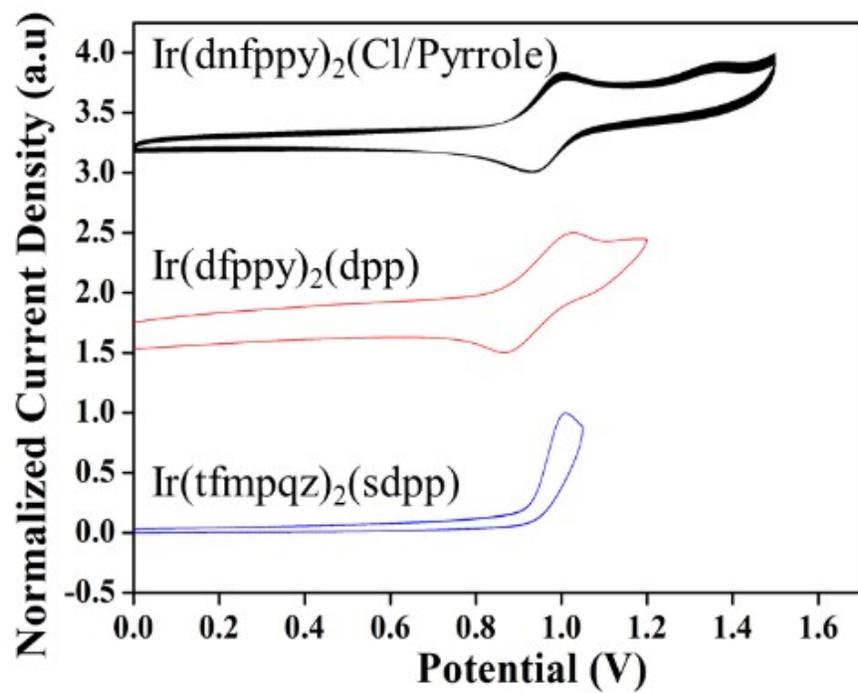
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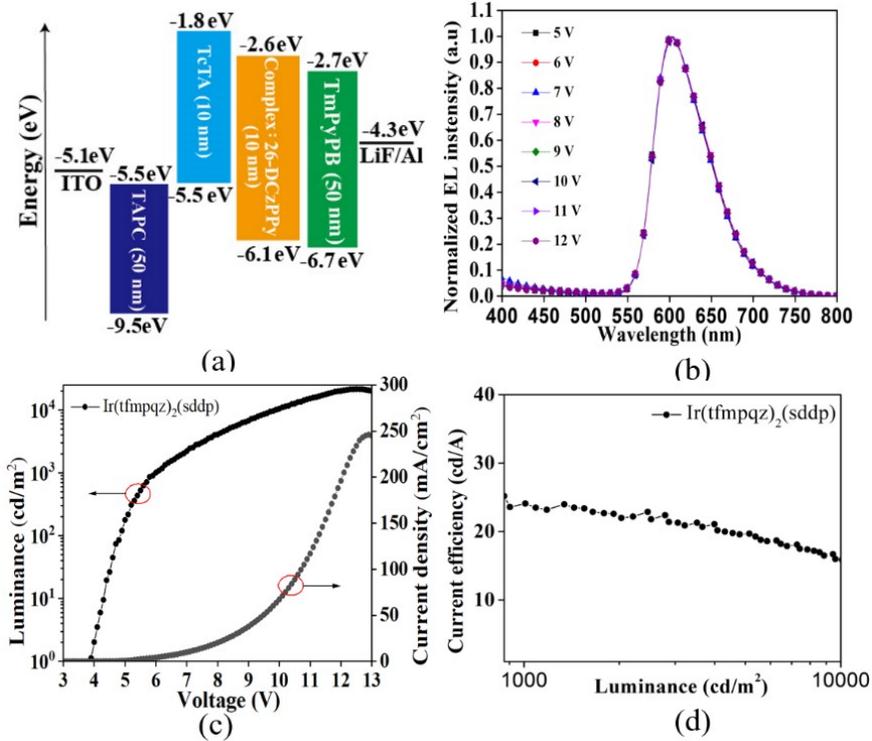
Abstract

Chiral Iridium complexes are very important for the preparation of circularly polarized light emitting diodes (CP-OLEDs). Three novel Iridium(III) complexes, Ir(dnfppy)₂(Cl/pyrrole), Ir(dfppy)₂(dpp) and Ir(tfmqz)₂(sdpp) have been synthesized and characterized, respectively. These Iridium(III) complexes emitted deep-blue, blue and red photoluminescence with high quantum yields, for ((Ir(dnfppy)₂(Cl/Pyrrrole): λ_{max}=447 nm, F=62.4%; Ir(dfppy)₂(dpp): λ_{max}=467 nm, F= 25%; Ir(tfmqz)₂(sdpp): λ_{max}=609 nm, F=73.7%). Compared with Ir(dnfppy)₂(Cl/pyrrole), HOMO energy level of Ir(dfppy)₂(dpp) and Ir(tfmqz)₂(sdpp) was calculated to be -5.71/-5.73 eV, and LUMO energy level increased to be -2.75/-3.36 eV, respectively. CD spectra of two pairs of the enantiomers for Ir(dfppy)₂(dpp) or Ir(tfmqz)₂(sdpp) displayed symmetry with opposite polarization between 300 and 600 nm. The maximum external quantum efficiency (EQEmax) of OLEDs based on Ir(tfmpqz)₂(sdpp) was 13.8%, showing a relatively low efficiency roll-off with the EQE of 10.7% at 5000 cd/m².









Three novel Iridium(III) complexes containing double chiral atoms for OLEDs

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ABSTRACT

Chiral Iridium complexes are very important for the preparation of circularly polarized light emitting diodes (CP-OLEDs). Three novel Iridium(III) complexes, Ir(dnfppy)₂(Cl/pyrrole), Ir(dfppy)₂(dpp) and Ir(tfmqz)₂(sdpp) have been synthesized and characterized, respectively. These Iridium(III) complexes emitted deep-blue, blue and red photoluminescence with high quantum yields, for ((Ir(dnfppy)₂(Cl/Pyrrrole): λ_{\max} =447 nm, F=62.4%; Ir(dfppy)₂(dpp): λ_{\max} =467 nm, F=25%; Ir(tfmqz)₂(sdpp): λ_{\max} =609 nm, F=73.7%). Compared with Ir(dnfppy)₂(Cl/pyrrole), HOMO energy level of Ir(dfppy)₂(dpp) and Ir(tfmqz)₂(sdpp) was calculated to be -5.71/-5.73 eV, and LUMO energy level increased to be -2.75/-3.36 eV, respectively. CD spectra of two pairs of the enantiomers for Ir(dfppy)₂(dpp) or Ir(tfmqz)₂(sdpp) displayed symmetry with opposite polarization between 300 and 600 nm. The maximum external quantum efficiency (EQE_{max}) of OLEDs based on Ir(tfmqz)₂(sdpp) was 13.8%, showing a relatively low efficiency roll-off with the EQE of 10.7% at 5000 cd/m².

Keywords: iridium compounds, chirality, electroluminescence

1. Introduction

Circularly polarized luminescence (CPL) has attracted considerable attention because of its wide applications in optical data storage, backlights in 3D displays or liquid-crystal display, spin sources in optical spintronics and information carriers in quantum computation, and so on [1]. Although chiral luminophores with circularly polarized photoluminescence (CPPL) have made considerable progress so far [2], the development of circularly polarized electro-luminescence (CPEL) emitters, which would be more practical for future display and photonic technologies, still lag behind [3]. For CPEL, the luminescence dissymmetry factor (g factor) is an important parameter to evaluate the degree of polarization, which is defined as $g_{EL} = \Delta I / I = 2(I_L - I_R) / (I_L + I_R)$, where I_L and I_R represent the left and right-handed luminescence polarized intensity values, respectively [4]. At present, the $|g_{EL}|$ values were mostly in the order of 10^{-3} for the emitting devices based on CPL-active organic molecules. Therefore, it is necessary to study the relationship of the structure and photo-physical properties of chiral luminescence materials to further improve the $|g_{EL}|$ factors.

Phosphorescent materials based on complexes of heavy metals such as Pt(II) [5], Os(II) [6] and Ru(II) [7] are able to achieve theoretically 100% internal quantum efficiency through harvesting singlet and triplet excitons, because they can be synchronously utilized by spin-orbit coupling interactions due to the metal effect. Among the heavy-metal complexes used in phosphorescent OLEDs, Iridium complexes, which are used as emitters in organic light-emitting diodes (OLEDs) due to the high phosphorescent quantum efficiency and broad emission colors range [8]. To the best of our knowledge, there are only a few reports about the utilization of chiral phosphorescent Iridium(III) complexes to construct circularly polarized light emitting diodes (CP-OLEDs) [9]. For example, Zheng et al. reported a series of Iridium(III) complex isomers by introducing chiral phosphine atoms in the ancillary ligand. The two pairs of enantiomers for $\text{Ir}(\text{dfppy})_2(\text{L})$, (where dfppy is 2-(2,4-difluorophenyl) pyridine, and L is sulfur-dipyridinylphosphinate oxide), based on Λ (λ) or Δ (δ) isomer together with chiral phosphine center (R or S), have been designed and studied [10]. Meanwhile, the CP-OLEDs based on enantiomeric isomers of Iridium(III) complexes as emitters showed the obvious CPEL signals with the maximum external quantum efficiency (EQE) of 14.6%. As well as known, λ and δ isomers of Iridium(III) complexes can be obtained by chiral column separation. However, the process to separate or purify λ and δ isomers was complicated. In addition, most of Iridium(III) complexes also exhibited extremely low $|g_{PL}|$ factor [11, 12]. Meanwhile, blue and red iridium complexes containing two chiral atoms are also more important for the preparation of white light circularly polarized light-emitting devices. Therefore, the preparation of blue or red emitting chiral iridium complexes with high $|g_{EL}|$ factors is more practical in the research fields of CP-OLEDs.

To further investigate the effect of double chiral atoms in chiral Iridium(III) complexes on the chiral emitting properties, we have designed and synthesized three Iridium(III) complexes, $\text{Ir}(\text{dnfppy})_2(\text{Cl}/\text{Pyrrole})$, $\text{Ir}(\text{dfppy})_2(\text{dpp})$ and $\text{Ir}(\text{tfmqz})_2(\text{sdpp})$. The chemical structures were shown in Fig. 1. The chiral performance of isomers of $\text{Ir}(\text{dfppy})_2(\text{dpp})$ and $\text{Ir}(\text{tfmqz})_2(\text{sdpp})$ were mainly

by circular dichroism (CD) and circularly polarized luminescence (CPL) spectra. These research results would provide a new, adaptable method for designing chiral emitters in the field of CP-OLEDs.

2. Experiment section

The chloro-bridged dimers (0.20 mmol) and 2.2 times of the corresponding ligands (0.44 mmol) were dissolved in 10 mL of dichloromethane. After degassed, the reaction was maintained at room temperature for about 10 min under argon. The solvent was removed and the crude compound purified by column chromatography with CH₂Cl₂: MeOH = 20:1 as eluent. Further purification was taken by gradient sublimation. And then four chiral Iridium(III) isomers were obtained by column chromatography.

Ir(dnfppy)₂(Cl/Pyrrrole). EI-TOF-MS (M/Z): Calculated for C₂₃H₁₄F₄IrN₆, [M+H]⁺ 678.053, found 679.780.

Ir(dfppy)₂(dpp). ¹H NMR (400 MHz, CDCl₃) δ 8.79-8.68 (m, 2H), 8.53-8.38 (m, 2H), 8.37-8.27 (m, 2H), 8.24-8.10 (m, 2H), 8.00-7.85 (m, 4H), 7.81-7.71 (m, 4H), 7.69-7.61 (m, 1H), 7.35-7.24 (m, 1H), 7.16-7.04 (m, 3H), 7.03-6.96 (m, 1H), 6.59 (d, *J* = 1.8 Hz, 1H), 6.38 (dd, *J* = 14.9, 1.8 Hz, 1H). ¹⁹F NMR (376 MHz, CDCl₃) δ -106.59 (1F), -108.49 (1F), -109.02 (1F), -110.64 (1F). ³¹P NMR (162 MHz, CDCl₃) δ 33.64. EI-TOF-MS (M/Z): Calcd for C₄₀H₂₄F₆IrN₆OPS, [M+H]⁺ 997.089, found 997.089.

Ir(tfmpqz)₂(sdpp). ¹H NMR (400 MHz, CDCl₃) δ 8.79-8.68 (m, 2H), 8.53-8.38 (m, 2H), 8.37-8.27 (m, 2H), 8.24-8.10 (m, 2H), 8.00-7.85 (m, 4H), 7.81-7.71 (m, 4H), 7.69-7.61 (m, 1H), 7.35-7.24 (m, 1H), 7.16-7.04 (m, 3H), 7.03-6.96 (m, 1H), 6.59 (d, *J* = 1.8 Hz, 1H), 6.38 (dd, *J* = 14.9, 1.8 Hz, 1H). ¹⁹F NMR (376 MHz, CDCl₃) δ -106.59 (1F), -108.49 (1F), -109.02 (1F), -110.64 (1F). ³¹P NMR (162 MHz, CDCl₃) δ 33.64. EI-TOF-MS (M/Z): Calcd for C₄₀H₂₄F₆IrN₆OPS [M+H]⁺ 997.089, found 997.089.

3. Results and discussion

3.1 Synthesis and characterization of Iridium(III) complexes

The synthetical routes of Ir(dnfppy)₂(Cl/Pyrrrole), Ir(dfppy)₂(dpp) and Ir(tfmqz)₂(sdpp) were showed in Supplementary information (Fig. 1*). Three Iridium complexes were synthesized by chloro-bridged dimers and corresponding ligands. From Fig. 1, the double chiral atoms were Ir/N, Ir/P, Ir/P for Ir(dnfppy)₂(Cl/Pyrrrole), Ir(dfppy)₂(dpp) and Ir(tfmqz)₂(sdpp), respectively. Single crystals of these Iridium(III) complexes were obtained by solvent evaporation method. Ir(dnfppy)₂(Cl/Pyrrrole) adopted distorted octahedral coordination geometry with the distance of 2.45 Å between Ir and Cl atom. For Ir(dfppy)₂(dpp), the angle of C-P-C is 106.4°, indicating different positions of two pyridine groups in space. Meanwhile, because of the coordination between iridium and nitrogen atoms in the pyridine group, P-O-Ir-N-C forms stable five-membered ring. Ir(tfmpqz)₂(sdpp) also adopted distorted octahedral coordination geometry,

and P-O-C-C-S in the ancillary ligand exhibited triangular conformation. The distance between chiral P atom and plane centroid of S-C-C in the ancillary ligand for λ -Ir(tfmpqz)₂(S-sdpp) was 0.70 Å. Ir-N-C-P-S constituted a stable five-membered ring with the Ir-P distance of 3.43 Å. Ir(tfmpqz)₂(sdpp) exhibited excellent thermal stability with a high decomposition temperature (T_d) of 323 °C (5% weight loss). The excellent thermal stabilities are necessary for improving the performance of OLEDs.

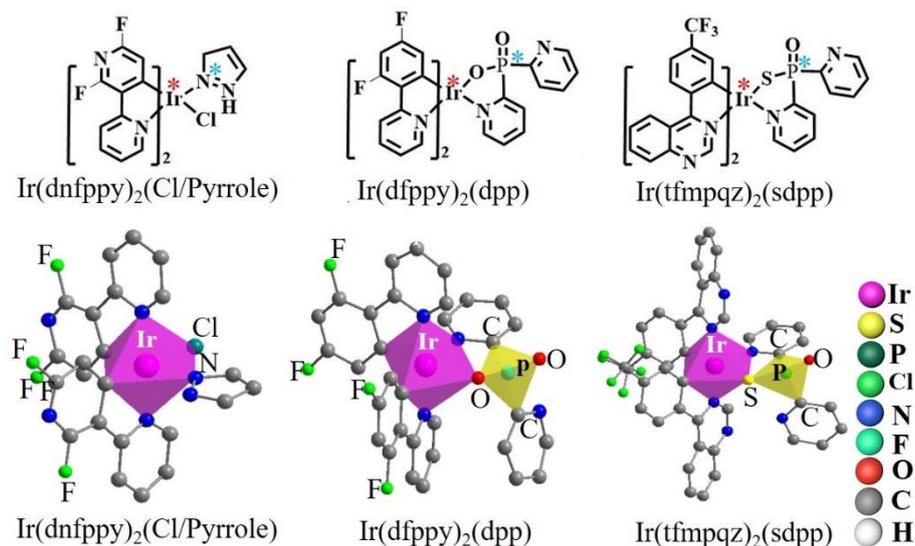


Fig. 1. The chemical and single crystal diffraction structures of Ir(dnfppy)₂(Cl/Pyrrrole) (CCDC no. 1956569), (b) Ir(dfppy)₂(dpp) (CCDC no. 2025773), (c) Ir(tfmqz)₂(sdpp) (CCDC no. 2053656).

3.2 Photophysical properties

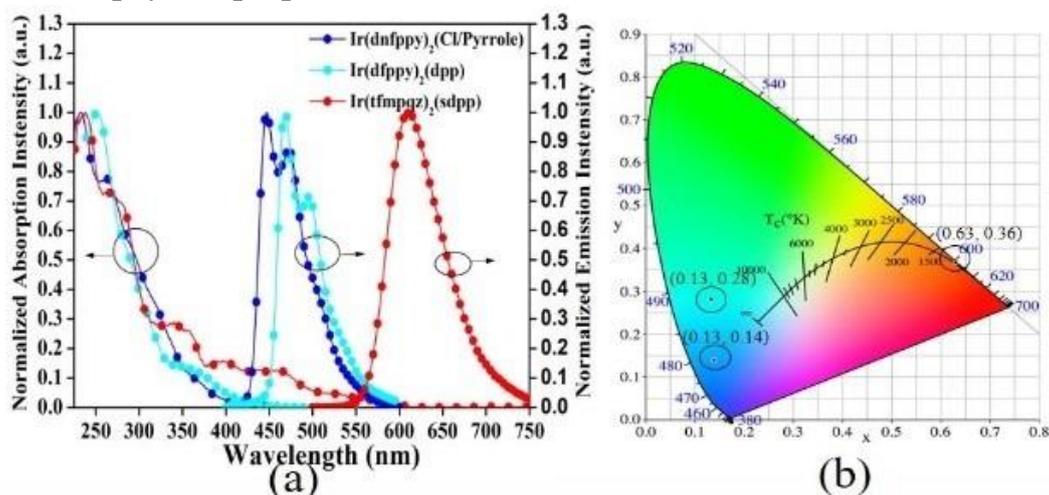


Fig. 2. (a) UV-vis absorption and emission spectra and (b) the Commission Internationale de L'Eclairage (CIE (x, y)) of Ir(dnfppy)₂(Cl/Pyrrrole), Ir(dfppy)₂(dpp) and Ir(tfmqz)₂(sdpp).

The photophysical properties of Iridium(III) complexes were investigated in CH₂Cl₂ solution. As shown in Fig. 2a, the maximum absorption peaks were 232, 238 and 254 nm for Ir(dnfppy)₂(Cl/Pyrrrole), Ir(dfppy)₂(dpp) and Ir(tfmqz)₂(sdpp), respectively. Broad and intense high-energy absorption bands were assigned to the π - π^* transition, which corresponded to the absorption bands of the main ligands. The weaker and broad low-energy transitions in the range

of 450–600 nm were attributed to the mixed $^1\text{MLCT}/^3\text{MLCT}$ transitions [13]. Meanwhile, these Iridium(III) complexes emitted deep-blue, blue or red photoluminescence with high quantum yields, for $(\text{Ir}(\text{dnfppy})_2(\text{Cl}/\text{Pyrrole}))$: $\lambda_{\text{max}}=447$ nm, $F=62.4\%$; $\text{Ir}(\text{dfppy})_2(\text{dpp})$: $\lambda_{\text{max}}=467$ nm, $F=25\%$; $\text{Ir}(\text{tfmqz})_2(\text{sdpp})$: $\lambda_{\text{max}}=609$ nm, $F=73.7\%$), respectively. From Fig. 2b, the Commission Internationale de L’Eclairage (CIE (x, y)) color coordinate was (0.13, 0.14), (0.13, 0.28) and (0.63, 0.36) for $\text{Ir}(\text{dnfppy})_2(\text{Cl}/\text{Pyrrole})$, $\text{Ir}(\text{dfppy})_2(\text{dpp})$ and $\text{Ir}(\text{tfmqz})_2(\text{sdpp})$, respectively. The triplet lifetime of $\text{Ir}(\text{tfmpqz})_2(\text{sddp})$ was 0.8 μs , suggesting the efficient intersystem crossing from the singlet to triplet state. $\text{Ir}(\text{dfppy})_2(\text{dpp})$ and $\text{Ir}(\text{tfmqz})_2(\text{sdpp})$ contain double chiral centers of iridium and phosphorus atoms, which lays a foundation for the preparation of circularly polarized light emitters. Two pairs of enantiomers for $\text{Ir}(\text{dfppy})_2(\text{dpp})$ and $\text{Ir}(\text{tfmqz})_2(\text{sdpp})$ have been separated by column chromatography. λ - $\text{Ir}(\text{dfppy})_2(\text{S-dpp})$ and δ - $\text{Ir}(\text{dfppy})_2(\text{R-dpp})$, λ - $\text{Ir}(\text{dfppy})_2(\text{R-dpp})$ and δ - $\text{Ir}(\text{dfppy})_2(\text{S-dpp})$ showed the emission spectra like $\text{Ir}(\text{dfppy})_2(\text{S-dpp})$ with the maximum emitting peak at 467 nm. Meanwhile, λ - $\text{Ir}(\text{tfmqz})_2(\text{S-sddp})$ and δ - $\text{Ir}(\text{tfmqz})_2(\text{R-sddp})$, λ - $\text{Ir}(\text{tfmqz})_2(\text{R-sddp})$ and δ - $\text{Ir}(\text{tfmqz})_2(\text{S-sddp})$ exhibited identical emission spectra to $\text{Ir}(\text{tfmqz})_2(\text{sddp})$ with the maximum emitting peak at 609 nm.

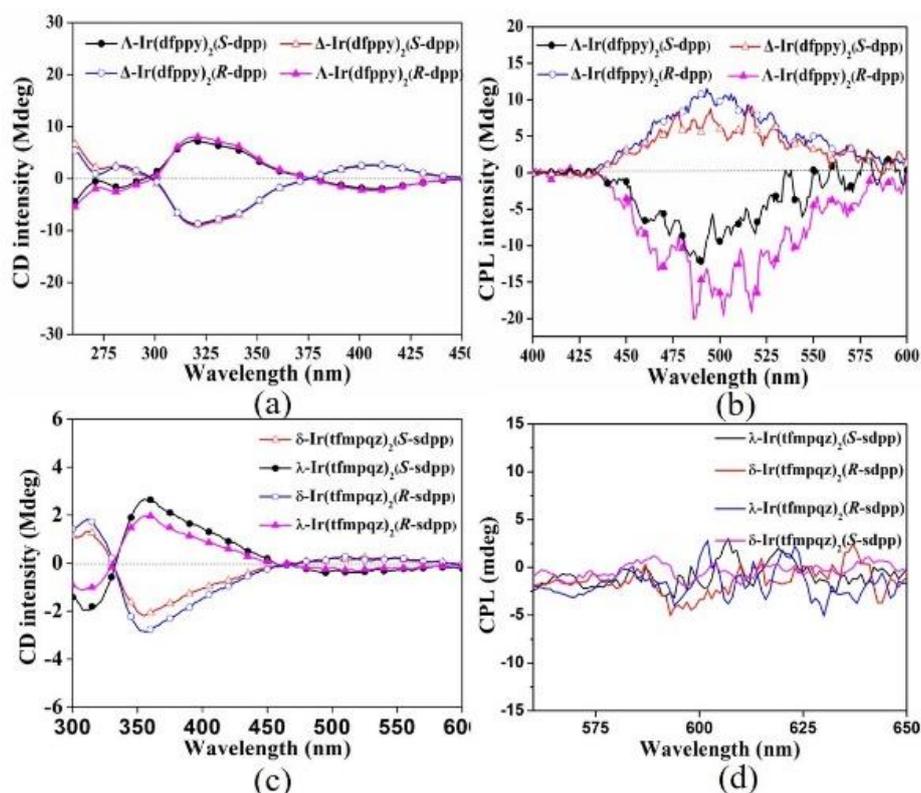


Fig. 3. (a) CD and (b) CPL spectra of the enantiomer for $\text{Ir}(\text{dfppy})_2(\text{dpp})$ in CH_2Cl_2 solution; (c) CD and (d) CPL spectra of the enantiomer for $\text{Ir}(\text{tfmqz})_2(\text{sdpp})$ and $\text{Ir}(\text{tfmpqz})_2(\text{sddp})$ in CH_2Cl_2 solution.

To investigate the chiral optical performance, CD and CPL spectra of the enantiomers of $\text{Ir}(\text{dfppy})_2(\text{dpp})$ and $\text{Ir}(\text{tfmqz})_2(\text{sdpp})$ were investigated in CH_2Cl_2 solution. From Fig. 3a, λ - $\text{Ir}(\text{dfppy})_2(\text{R-dpp})$ and δ - $\text{Ir}(\text{dfppy})_2(\text{S-dpp})$ exhibited mirror-image CD spectra with alternating negative and positive Cotton effects. By comparison, δ - $\text{Ir}(\text{dfppy})_2(\text{S-dpp})$ and λ - $\text{Ir}(\text{dfppy})_2(\text{R-dpp})$

ddp) showed symmetrical and opposite absorption trend. The intense Cotton effects in short wavelength regions were assigned to the absorption caused by the intramolecular π -system transition. Meanwhile, the weak cotton effect at the longest-wavelength absorption band was caused by the MLCT [14]. As show in Fig. 3b, the g_{lum} values of λ -Ir(dfppy)₂(R-ddp), δ -Ir(dfppy)₂(S-ddp) δ -Ir(dfppy)₂(S-ddp) and λ -Ir(dfppy)₂(R-ddp) was calculated to be -8.4×10^{-4} , 1.1×10^{-3} , -1.3×10^{-3} 和 8.4×10^{-4} at 470 nm, respectively. From Fig. 3c, CD spectra of the two pairs of the enantiomer of Ir(tfmqz)₂(sdpp) displayed symmetry with opposite polarization between 300 and 600 nm. From Fig. 4d, we can know that the CPL spectra of the isomers for Ir(tfmpqz)₂(sdpp) displayed weak symmetry in 500-650 nm. These research results indicated that blue emitting Iridium complex containing double chiral centers showed higher $|g_{EL}|$ values that that of red emitting Iridium emitters.

3.3 Electrochemical properties and DFT calculation

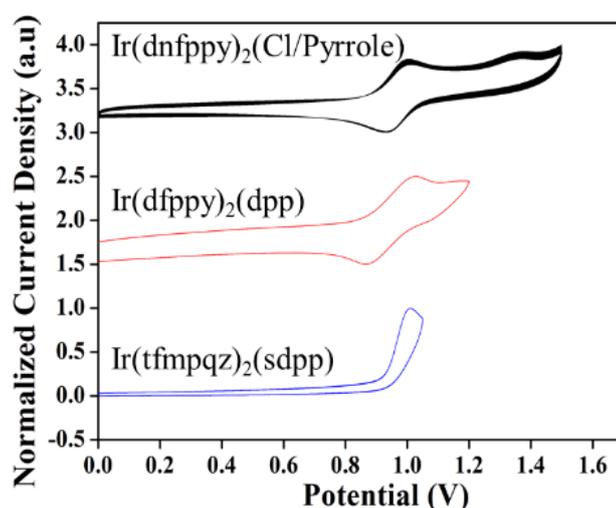


Fig. 4. Cyclic voltammograms of Ir(dnfppy)₂(Cl/Pyrrrole), Ir(dfppy)₂(dpp) and Ir(tfmqz)₂(sdpp) on platinum electrode in 0.1 mol/L Bu₄NClO₄, acetonitrile solution.

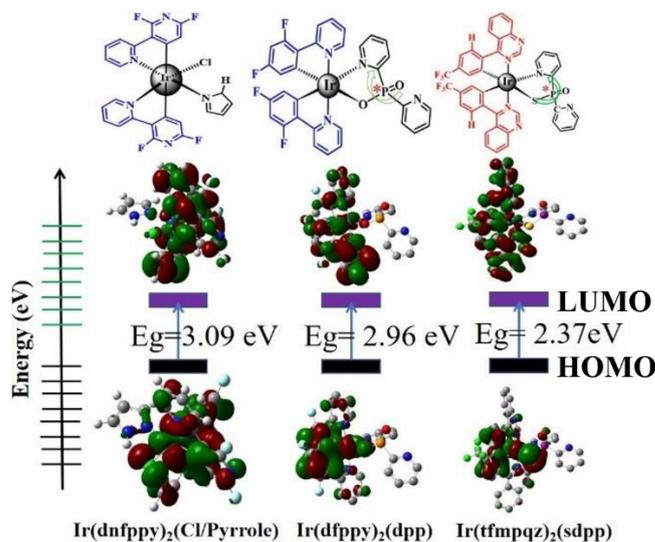


Fig 5. HOMO and LUMO electron distributions for Ir(dnfppy)₂(Cl/Pyrrrole), Ir(dfppy)₂(dpp) and Ir(tfmqz)₂(sdpp).

The redox properties have been measured by cyclic voltammetry (CV) experiments. The oxidation processes were observed for Ir(dnfppy)₂(Cl/Pyrrole), Ir(dfppy)₂(dpp) and Ir(tfmqz)₂(sdpp) with oxidation potential of 0.93, 1.01 and 1.0 V, respectively. HOMO energy level of Ir(dnfppy)₂(Cl/Pyrrole), Ir(dfppy)₂(dpp) and Ir(tfmqz)₂(sdpp) were calculated to be -5.43/-5.71/-5.73 eV, respectively. Based on the first anodic peak potentials and the onset of the UV-vis absorption spectra^[20], the energy level (E_g) between HOMO and LUMO for these iridium complexes was calculated as 3.09/2.96/2.37 eV, as shown in Fig 4. LUMO energy level of Ir(dnfppy)₂(Cl/Pyrrole), Ir(dfppy)₂(dpp) and Ir(tfmqz)₂(sdpp) was -2.34/-2.75/-3.36 eV, respectively. From Fig. 5, based on the B3LYP function, the 6-31G for C, H, O, S, P, lan12dz base set for Ir atoms, the electron distributions of four configuration isomers of chiral Iridium complex are studied by density functional theory with time variation. Theoretical results show that the enantiomers of Ir(dnfppy)₂(Cl/Pyrrole), Ir(dfppy)₂(dpp) and Ir(tfmqz)₂(sdpp) have bimanual emission characteristics.

3.4 Electroluminescent properties

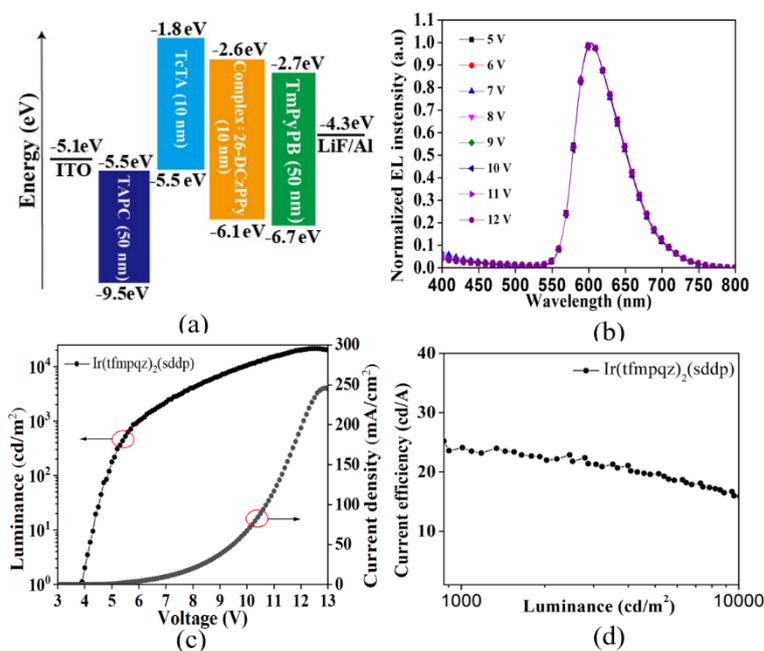


Fig. 6. (a) The structure and energy diagram of OLEDs, (b) EL spectra, (c) luminance-voltage-current density curves, and (d) current efficiency luminance curve of Ir(tfmpqz)₂(sddp).

CPL spectra of two pairs of enantiomers for Ir(tfmpqz)₂(sddp) were not remarkable, the performance of its circularly polarized light emitting devices was not investigated. The organic light-emitting diodes of Ir(tfmpqz)₂(sddp) was fabricated. Ir(tfmpqz)₂(sddp) was used as emissive dopant with the architecture: ITO/HATCN (5 nm)/TAPC (50 nm)/10 wt% Ir(tfmpqz)₂(sddp):26DCzPPY (20 nm)/TmPyPB (50 nm)/LiF (1 nm)/Al (100 nm) (Fig. 6a). The electroluminescence spectra of the devices displayed a prominent red EL centered at 602 nm (Fig. 6b). The light emitting device achieved a maximum luminance of 36020 cd/m² at 12V. Notably, the device featured a turn-on voltage (V_T) of 3.6 V, a maximum current efficiency, a

maximum power efficiency and the maximum external quantum efficiency (EQE_{\max}) of 25.2 cd/A, 17.9 lm/W and 13.8%, respectively. Moreover, the device also showed a relatively low efficiency roll-off, and the EQE was 10.7% at a luminance of 5000 cd/m².

4. Conclusions

In summary, three novel Iridium(III) complexes, Ir(dnfppy)₂(Cl/Pyrrole), Ir(dfppy)₂(dpp) and Ir(tfmqz)₂(sdpp), have been synthesized and characterized. These complexes emitted deep-blue, blue, and red photoluminescence with high quantum yields, respectively. The CD spectra of two pairs of enantiomers of Ir(dfppy)₂(dpp) and Ir(tfmpqz)₂(sddp) displayed symmetry with opposite polarization between 300 and 600 nm. The light emitting device based on Ir(tfmpqz)₂(sddp) achieved a maximum luminance of 36020 cd/m² at 12 V, a maximum current efficiency of 25.2 cd/A and EQE_{\max} of 13.8%, respectively. These research results indicate that the Iridium(III) complex double chiral atoms can provide a new pathway to design circularly polarized luminescent materials for CPOLEDs.

Acknowledgements

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Conflict of Interest

The authors declare no conflict of interest.

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