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Journal of Nanoscience and Nanotechnology Vol. 12, 5613–5618, 2012

Phosphorescence Color Tuning of Oxadiazole-Based Iridium(III) Complexes for Organic Light Emitting Diode

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The new heteroleptic iridium complexes bearing 2-(5-phenyl-1,3,4-oxadiazol-2-yl)phenolate (ODZ), were synthesized and characterized for application to organic light-emitting diodes (OLEDs). As main ligands (C^N), the anions of 2-phenylpyridine (ppy), 2-phenylquinoline (pq) and 2-(2,4-difluorophenyl)pyridine (F₂-ppy) were chelated to the iridium center and 2-(5-phenyl-1,3,4-oxadiazol-2-yl)phenolate (ODZ) was introduced as an ancillary ligand for luminescence modulation of their iridium complexes. We expected that the relative energy levels of the main and ancillary ligands in the complexes could lead to emission color tuning and luminous efficiency improvement by possible inter-ligand energy transfer (ILET). The photoabsorption, photoluminescence and electroluminescence of the complexes were studied. $Ir(F_2-ppy)_2(ODZ)$, $Ir(ppy)_2(ODZ)$ and $Ir(pq)_2(ODZ)$ exhibited the photoluminescence maxima between 505–610 nm at room temperature in CH_2Cl_2 , depending on both main and ancillary ligands. The longer π conjugation in the cyclometallating pq ligands leads to the bathochromic shift in luminescence of their iridium complexes. The electroluminescence properties of the complexes were influenced by ILET

Keywords: Organic Light-Emitting Diode, Iridium Complex, Oxadiazolylphenolate, Inter-Ligand Energy Transfer.

1. INTRODUCTION

In the last few years, organic light emitting materials have been extensively studied owing to their excellent phosphorescent quantum yields and easy tuning of emission color by their molecular structures.^{1–2} The iridium complexes have attracted increasing interests as highly emissive dopants in organic light emitting diodes (OLEDs).^{3–4} Because the heavy iridium center allows both singlet and triplet excitons to be used in an electroluminescence process, internal quantum efficiency of 100% is theoretically possible.

In general, OLEDs exhibit several advantages such as a wider viewing angle, faster response time, and selfemission over liquid crystal display (LCD). However, fullcolor OLEDs still require improvement in terms of device efficiency, color purity, manufacturing process, and low cost. These factors in turn depend on the development of efficient OLED materials, including red, green, and blue (RGB) emitters.^{5–7} Therefore, the design and synthesis of phosphorescent emitting materials containing heavy-metal complexes are particularly important in achieving high-efficiency of the OLEDs.⁸

For the development of full-color, and efficient emitting materials of OLEDs, we have introduced various main ligands (C^N) and a new ancillary ligand to the iridium complexes. The C^N ligands have been known for their major contribution to emission color determination of their complexes while the ancillary ligand has shown a minor effect on the color tuning. On the other hand, the recent reports on inter-ligand energy transfer (ILET) between the main and ancillary ligands emphasize the contribution of the ancillary ligand to the emission color and efficiency.^{9–10} Thus, the modulation of the relative triplet states of the C^N and ancillary ligands could be considered an important factor in determining the emission color of their iridium complexes. We applied these concepts to the design of the iridium complexes and prepared them containing the anions of F₂-ppy, ppy and pq as main ligands and 2-(5-phenyl-1,3,4-oxadiazol-2-yl)phenolate (ODZ) as an ancillary ligand.

The main ligands, F_2 -ppy, ppy and pq, which have drastically different energy gaps between the HOMO and LUMO energy levels were chosen. As an ancillary ligand,

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an oxadiazole derivative, ODZ, was introduced; it is known to have promising electron transport and emission characteristics when used in organic EL devices.¹¹⁻¹³ ODZ can form a stable six-membered metallacycle when chelated. This class of compounds can offer numerous attractive properties such as a double role of electron transport and phosphor, high thermal stability, and ease of sublimation.

We investigated the photoabsorption properties of the iridium complexes with their UV-Vis spectra and studied the electrochemical characteristics with their cyclic voltammetric diagrams. The photoluminescence (PL) and electroluminescence (EL) of the complexes were also investigated. The emitting colors and the device performances of the complexes, $Ir(F_2-ppy)_2(ODZ)$, $Ir(ppy)_2(ODZ)$ and $Ir(pq)_2(ODZ)$, were compared to find the effects of the main ligands and the new ancillary ligand, ODZ, on the luminescence properties of their complexes.

2. EXPERIMENTAL DETAILS

2.1. Synthesis and Characterization

All reagents, purchased from Aldrich Co. and Strem Co., were used without further purification. All reactions were carried out under an argon atmosphere. Solvents were dried by standard procedures. Mass spectra were determined on JEOL, JMS-AX505WA, HP 5890 Series II Hewlett-Packard 5890A at Seoul National University. ¹H NMR was taken with a 400 MHz NMR spectrometer at Sogang University in Korea.

2.1.1. Synthesis of Ligands and Iridium Complexes

Synthesis of F_2 -*ppy.* 2,4-difluoro-2-phenylpyridine was obtained from the reaction of 2-chloropyridine and 2,4-difluorophenylboronic acid by Suzuki coupling.¹⁴ Yield: 69%.

Synthesis of ODZ.¹³ In 25-ml anhydrous pyridine, acetylsalicyloyl chloride (10.0 g, 50.3 mmol) and 5-phenyl-1,2,3,4-tetrazole (7.5 g, 51.3 mmol) were added and stirred at reflux for 2 h under an argon atmosphere. After removal of the solvent under reduced pressure, the residue was treated with aqueous sodium hydroxide solution (3.5 g) in ethanol at reflux for 1 h. The precipitates were collected by glass filter and acidified in methanol. Yield: 71%.

Synthesis of $Ir(C^N)_2(ODZ)$ ($C^N = F_2$ -ppy, ppy, pq). The cyclometallated Ir(III) μ -chloro-bridged dimer, $(C^N)_2Ir(\mu$ -Cl)_2Ir(C^N)₂ (0.5 mmol), was first prepared according to the Nonoyama method.¹⁵ The resulting dimer (1.73 g, 1.9 mmol) and an excess of the ancillary ligands were next mixed with Na₂CO₃ (500 mg) in 2-ethoxyethanol (30 mL). The mixture was refluxed for 2 h. The solution was cooled to room temperature and the solid was filtered. The crude product was chromatographed on silica gel column with dichloromethane and purified by recrystallization.

Data for $[Ir(F_2-ppy)_2(ODZ)]$: Yield: 68%. FAB-MS: calculated 809; found 810. ¹H NMR(CDCl₃, 400 MHz): $\delta 8.21 (d, 3 \text{ H}) 8.12 (d, 2 \text{ H}) 7.98 (d, 3 \text{ H}) 7.82 (d, 3 \text{ H}) 7.66 (d, 1 \text{ H}) 7.56 (s, 4 \text{ H}) 7.43 (t, 2 \text{ H}) 7.20 (d, 2 \text{ H}) 6.87 (d, 1 \text{ H}).$

Data for $[Ir(ppy)_2(ODZ)]$: Yield: 65%. FAB-MS: calculated 737; found 738. ¹H NMR(CDCl₃, 400 MHz): δ 8.63 (*d*, 1 H) 8.38 (*d*, 1 H) 8.13 (*d*, 2 H) 7.87 (*d*, 2 H) 7.79 (*m*, 3 H) 7.72 (*d*, 2 H) 7.55 (*d*, 3 H) 7.30 (*t*, 1 H) 7.17 (*t*, 2 H) 6.81 (*m*, 2 H) 6.66 (*d*, 2 H) 6.55 (*d*, 1 H) 6.43 (*t*, 1 H) 6.18 (*d*, 1 H) 6.05 (*d*, 1 H).

Data for $[Ir(pq)_2(ODZ)]$: Yield: 71%. FAB-MS: calculated 837; found 838. ¹H NMR(CDCl₃, 400 MHz): δ 8.48 (*d*, 2 H) 8.37 (*d*, 2 H) 8.13 (*d*, 1 H) 8.01 (*d*, 1 H) 7.91 (*d*, 1 H) 7.85 (*d*, 1 H) 7.74 (*d*, 2 H) 7.64 (*d*, 1 H) 7.55 (*m*, 3 H) 7.42 (*t*, 1 H) 7.33 (*d*, 2 H) 7.16 (*d*, 2 H) 6.94 (*d*, 2 H) 6.87 (*t*, 1 H) 6.59 (*m*, 3 H) 6.25 (*d*, 1 H) 6.19 (*d*, 1 H) 6.05 (*t*, 1 H) 5.76 (*s*, 1 H).

2.2. Optical Measurements

UV-visible absorption spectra were obtained from Hewlett Packard 8425A spectrometer. PL spectra were measured on a Perkin Elmer LS 50B spectrometer. The UV-Vis and PL spectra of iridium complexes were measured in 10^{-5} M dilute CH₂Cl₂ solution. Cyclic voltammograms were obtained at a scan rate of 100 mV/s with electrochemical Analyzer of CH Instruments. ¹H NMR spectra were obtained from a 400 MHz NMR at Sogang University, and mass spectra were determined on JEOL, JMS-AX505WA, HP 5890 Series II Hewlett-Packard 5890A (capillary column) at Seoul National University in Korea.

2.3. Device Fabrication and Measurements

The OLEDs containing the heteroleptic iridium complexes as a dopant in emitting layers were fabricated. Glass substrate coated with a 180-nm-thick indium tin oxide (ITO) layer had a sheet resistance of 12 Ω /sq. All organic layers were sequentially deposited onto the substrate without breaking vacuum at a pressure of about 5×10^{-7} torr, using the thermal evaporation equipment. The organic materials were deposited as the following structure: 4,4'-bis[*N*-(naphthyl)-*N*-phenylamino]biphenyl (NPB, 50 nm) as hole transporting layer (HTL)/4,4',4"tris(N-carbazolyl)triphenylamine (TCTA, 10 nm) as buffer layer/iridium complex phosphors (10%) doped in 4,4'bis(9-carbazolyl)-1,1'-biphenyl (CBP, 30 nm) as emitting layer/4,7-Diphenyl-1,10-phenanthroline (Bphen, 30 nm) as electron transporting layer (ETL)/lithium quinolate (Liq, 2 nm) as electron injection layer (EIL). The deposition rates were 1.0-1.1 and 0.1 Å/sec for organic materials and lithium quinolate (Liq), respectively. Finally, the aluminum Park et al.

cathode was deposited at a rate of 10 Å/sec. The devices were encapsulated in a glove box with O_2 and H_2O at concentrations below 1 ppm. The devices had emission areas of 3×3 mm². The electrical characteristics of devices and the EL spectra were measured and immediately recorded with a Chroma meter CS-1000A (Minolta). The current and voltage were controlled with a measurement unit (model 236, Keithely).

3. RESULTS AND DISCUSSION

As previously reported,^{16–19} the main and/or ancillary ligands could have a significant effect on the emission properties of their complexes. Synthesis of the main ligand, F_2 -ppy, was straightforward, according to the modified Suzuki coupling method.¹⁴ The ancillary ligand, ODZ, was prepared from the reaction of acetylsalicyloyl chloride with 5-phenyl-1,2,3,4-tetrazole, according to the reported procedure.¹³ The heteroleptic iridium complexes of ODZ were synthesized as reported by Nonoyama.¹⁵ The overall synthetic schemes are illustrated in Figure 1.

Figure 2 presents the UV-Vis absorption spectra of the complexes in CH₂Cl₂. The strong absorption bands between 240 and 300 nm in the ultraviolet region are attributed to the spin-allowed ${}^{1}\pi - \pi^{*}$ transition of the cyclometallated ligands, F2-ppy, ppy and pq, in the complexes. The weak band of Ir(F2-ppy)2(ODZ) and Ir(ppy)₂(ODZ) at 350-450 nm in the visible region can be assigned to the spin-allowed metal-to-ligand charge transfer band (¹MLCT), and the weaker absorption bands at the longer wavelengths can be attributed to the spin-forbidden ³MLCT and spin-orbit coupling enhanced ${}^{3}\pi - \pi^{*}$ transition. The absorption patterns of $Ir(F_2-ppy)_2(ODZ)$ and $Ir(ppy)_2(ODZ)$ are similar with the absorption maxima between 260 and 400 nm. The similar absorption patterns indicate that change of the main ppy-based ligands in the complexes does not make significant contribution to the absorption process. In contrast, $Ir(pq)_2(ODZ)$ had different absorption patterns, showing the maxima at 275, 330 and 427 nm, which could be



Fig. 1. The synthesis of main ligands, ancillary ligands and their iridium complexes.

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Fig. 2. UV-Vis absorption spectra of $Ir(C^{\wedge}N)_2(ODZ)$ in a 10^{-5} M CH_2Cl_2 solution ($C^{\wedge}N = F_2$ -ppy, ppy, pq).

ascribed to the red emitting ligand, pq. The UV-Vis spectra of $Ir(pq)_2(ODZ)$ has also a distinct feature of strong absorption peak around 310–370 nm.

Figure 3 shows the PL spectra of the Ir complexes in 10^{-5} M CH₂Cl₂ solution. The emission maxima of Ir(F₂-ppy)₂(ODZ), Ir(ppy)₂(ODZ) and Ir(pq)₂(ODZ) appear at 505, 515 and 608 nm, respectively. Ir(F₂ppy)₂(ODZ) and Ir(ppy)₂(ODZ) have similar luminescence aspects with the emission peaks around 510 nm. Such similarities in PL spectra of these two complexes are in fact expected, considering the similar photoabsorption patterns in their UV-Vis spectra. The ppy-based main ligands did not seem to affect luminescence as well as absorption of their complexes coordinated with ODZ. In contrast, Ir(pq)₂(ODZ) exhibited the distinct emission peak at 608 nm.

We attribute such PL difference between the ppy-based iridium complexes and the pq-based complex to the different emission mechanisms. The emission maxima of the iridium complexes were substantially shifted to the



Fig. 3. PL spectra of $Ir(C^N)_2(ODZ)$ in a 10^{-5} M CH_2Cl_2 solution $(C^N = F_2$ -ppy, ppy, pq).

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Table I. Physical parameters for the iridium complexes.									
Ir complex	$\lambda_{\rm em}/{\rm nm}^a$	$E_{\rm ox}/{\rm V}^b$	HOMO/ eV ^c	LUMO/ eV ^d	$\Delta E/\mathrm{eV}^e$				
$Ir(F_2-ppy)_2(ODZ)$	505	0.56	-5.36	-2.27	3.09				
$Ir(ppy)_2(ODZ)$	515	0.54	-5.34	-2.36	2.98				
Ir(pq) ₂ (ODZ)	608	0.39	-5.19	-2.44	2.75				

Notes: ^{*a*}Measured in CH₂Cl₂ solution. ^{*b*}Scan rate: 100 mV/s, Electrolyte: tetrabutylammonium hexafluorophosphate. The potentials are quoted against the internal ferrocene standard. ^{*c*}Deduced from the equation HOMO = $-4.8 - E_{ox}$, LUMO = $-4.8 - E_{red}$. ^{*d*}. ^{*e*}Calculated from the optical edge and the relation ΔE = HOMO – LUMO.

longer wavelength upon changing the main ligand from F_2 -ppy to pq. The energy gap of pq main ligand in the complex is smaller than those of ppy and F_2 -ppy analogs, preventing the possibility of ILET. Therefore, we assign that the resulting emission is originated mostly from pq ligand in the complex. Previously, the emission of the iridium complexes chelated with pq was reported to appear at the similar red region of 597 nm.²⁰ On the other hand, $Ir(F_2$ -ppy)₂(ODZ) and $Ir(ppy)_2(ODZ)$ exhibited the similar photophysical characteristics, regardless of the main ligands, *vide supra*. Their similar photophysical results imply that these two complexes undergo interligand energy transfer (ILET), resulting in the emission mainly originated from electronic transition of the ancillary ligand, ODZ.

We investigated the electrochemical properties of the Ir complexes with the cyclic voltammetry (CV) which could reveal the positions of their HOMOs and LUMOs.^{21–22} Table I summarizes the CV data. The oxidation potentials which indicates the HOMOs of Ir(F₂-ppy)₂(ODZ) and Ir(ppy)₂(ODZ) were irreversible at 0.56 and 0.54 V relative to an internal ferrocenium/ferrocene reference (Fc⁺/Fc), respectively. By comparison, the oxidation potential of Ir(pq)₂(ODZ) was semi-reversible at 0.39 V. The reduction curves of all complexes were not clearly observed up to -2.2 V. Therefore, the LUMOs of the complexes were estimated from their respective absorption spectra, using the optical edge and band gap equation ($\Delta E = E_{ox} - E_{red}$). Their calculated reduction potentials were -2.27, -2.36 and -2.44 eV for Ir(F₂-ppy)₂(ODZ),



Fig. 4. The device structure of Ir complexes.

Ir(ppy)₂(ODZ) and Ir(pq)₂(ODZ), respectively. The energy gap of Ir(pq)₂(ODZ) was the smallest among the complexes in this study and this could be explained with the extended π conjugation length of the pq ligand relative to the other ligands in the respective complexes. As a result, the pq ligand caused the bathochromic luminescence shift and the luminescence mechanism change of its iridium complex.

The EL properties of the complexes were also investigated for their OLED application. The configuration of the EL device with $Ir(C^N)_2(ODZ)$ used as a dopant was ITO/NPB/TCTA/CBP:DOPANT 10%/Bphen/Liq/Al. In the EL spectra, Figure 5(a) shows that the emission peaks of $Ir(F_2-ppy)_2(ODZ)$, $Ir(ppy)_2(ODZ)$ and $Ir(pq)_2(ODZ)$ were



0 20 40 60 80 100 120 140 160 180 200 220 Current density [mA/cm²]

Fig. 5. (a) The EL spectra and (b) the power and luminous efficiencies versus current density of the devices with the iridium complexes.

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Table II.	OLED device characteristics of the iridium complexes.						
Ir complex	EL λ_{max} (nm)	Power efficiency (lm/W)	Quantum efficiency (%)	Luminance efficiency (cd/A)	CIE		
Ir(F ₂ -ppy) ₂ (ODZ)	500	5.52	2.62	7.91	(0.30, 0.56)		
Ir(ppy) ₂ (ODZ)	510	20.63	10.64	35.02	(0.33, 0.57)		
Ir(pq) ₂ (ODZ)	606	7.57	6.63	9.64	(0.61, 0.38)		

observed at 500, 510 and 606 nm, respectively. These results were consistent with the PL results and indicated that EL originated from the iridium complex dopant in the emitting layer. Figure 5(b) shows the luminance efficiency versus current density characteristics of the devices with iridium complexes. The maximum luminous efficiencies of the devices with $Ir(F_2-ppy)_2(ODZ)$, $Ir(ppy)_2(ODZ)$ and Ir(pq)₂(ODZ) were 7.91, 35.02 and 9.64 cd/A, respectively. The device of Ir(ppy)₂(ODZ) showed the best luminous efficiency of 35.02 cd/A. The ILET between the ppy main ligand and the ancillary ligand, ODZ, seemed to occur strongly, leading their iridium complex to have substantial improvement of the device efficiencies. The Ir complexes bearing a benzoquinoline ancillary ligand was recently reported to show similar improvement of the device performances by ILET.23 However, the device of $Ir(F_2-ppy)_2(ODZ)$ did not show high efficiency in spite of possible ILET. Delicate criteria seem to operate in relative triplet energy levels of the main and ancillary ligands for the ILET mechanism to be operated. Further studies are in progress regarding theoretical calculation of energy levels of the complexes studied herein. The Commission Internationale de L'Eclairage (CIE) coordinates of Ir(F₂-ppy)₂(ODZ), Ir(ppy)₂(ODZ) and Ir(pq)₂(ODZ) were (0.30, 0.56), (0.33, 0.57) and (0.61, 0.38), respectively. Table II and Figure 4 spresent the detailed data and device structure.

We also tried to synthesize the homoleptic iridium complex of ODZ, $Ir(ODZ)_3$, for luminescence comparison. The synthesis of homoleptic iridium complex was however failed. The failure in formation of $Ir(ODZ)_3$ is attributed to the difficulty in tri-cyclometallated six-membered ring formation with the iridium center and steric bulkiness of ODZ ligand.

4. CONCLUSIONS

We have focused our research on the development of iridium complexes, especially with respect to color tuning and efficiency improvement of luminescence for OLED. Herein, we synthesized $Ir(C^N)_2(ODZ)$ and studied their emission patterns and characteristics where F_2 -ppy, ppy and pq were introduced as main ligands (C^N). We expected that the complexes synthesized in this study

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might exhibit various emission colors due to the main ligand effect among the complexes. Generally, the energy gap of the main ligand has a major effect in determining the emitting color of its complex. The electroluminescence spectra of $Ir(F_2-ppy)_2(ODZ)$ and $Ir(ppy)_2(ODZ)$ exhibited the similar emission patterns with the maxima at 500 and 510 nm, while $Ir(pq)_2(ODZ)$ revealed the red luminescence around 606 nm with the CIE coordinates of (0.61, 0.38). Considering the consistent UV and PL patterns of the iridium complexes with ppy-based main ligands discussed in this study, we could infer that the ILET might operate in the phosphorescence process of $Ir(F_2-ppy)_2(ODZ)$ and $Ir(ppy)_2(ODZ)$. The EL device of $Ir(ppy)_2(ODZ)$ showed the best luminous efficiency of 35 cd/A. On the other hand, the emission of Ir(pq)₂(ODZ) was assigned to originate from electronic transition in the main ligand, pq. This study also showed the possibility of the application of iridium complexes containing oxadizole-based ligand which has a good electron transporting property to the phosphorescent material for OLED.

Acknowledgments: This study was supported by the Korea Research Foundation (No. 2011-0003765).

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Received: 28 June 2011. Accepted: 16 January 2012.

Delivered by Publishing Technology to: University of Waterloo IP: 23.24.70.177 On: Wed, 14 Oct 2015 07:09:55 Copyright: American Scientific Publishers