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White-Light-Emitting Diodes Based on Iridium Complexes via Efficient Energy Transfer from a Conjugated Polymer**

By Tae-Ho Kim, Hang Ken Lee, O Ok Park,* Byung Doo Chin, Soo-Hyeong Lee, and Jai Kyeong Kim*

Efficient white-light-emitting diodes (WLEDs) have been developed using a polyfluorene-type blue-emitting conjugated polymer doped with green and red phosphorescent dyes. The emission spectrum of the conjugated polymer, which has a very high luminescent efficiency, shows a large spectral overlap with the absorbance of green and red iridium complexes. Also, efficient energy transfer from the conjugated polymer to the iridium complexes is observed. Poly(*N*-vinyl carbazole) is used to improve the miscibility between conjugated polymer and iridium complexes because of their poor chemical compatibility and phase separation. A white emission spectrum is easily obtained by varying the contents of the three materials and controlling the phase morphology. Moreover, these WLEDs show a voltage-independent electroluminescence owing to the threshold and driving voltage of the three materials being similar as a result of energy transfer.

1. Introduction

White-light-emitting diodes (WLEDs) have many advantages that can be applicable to illumination light sources, backlights for liquid crystal displays, and full-color flat-panel displays with color filters.^[1,2] Many attempts to achieve WLEDs have been reported, including the use of small organic molecules,^[3] specially synthesized polymers,^[4] polymer blends,^[5-7] and polymer bilayers,^[8] mixing the above with phosphorescent iridium complexes,^[9] and also using luminescent semiconducting polymers blended with organometallic emitters.^[10]

However, the vacuum deposition of small molecules in organic light-emitting diodes (LEDs) is not effective, especially for large-area displays. Moreover, for vacuum-deposited WLEDs very precise control of ratios of red-, green-, and blue-emitting dopants is needed in order to obtain pure white emission. In contrast, conjugated polymers have the advantages of easy processing in solution and relative insensitivity to dopant ratio compared to evaporated WLEDs. This means that they can be suitable for the large-area fabrication of polymer films via spin-coating. [5-7] These WLEDs that are made from poly-

efficiency and brightness, but also color instability, depending on the applied voltage, resulting from the phase separation of different polymer components. Recently, efficient WLEDs based on a non-conjugated polymer, poly(N-vinyl carbazole) (PVK), doped with phosphorescent dyes, where the singlet and triplet excitons of the molecules contribute to light generation, have been demonstrated. [9] However, a high driving voltage is required owing to the charge trapping of the iridium complexes.[10,11] Also, low brightness is a problem that needs to be solved. Research on white-light emission from devices fabricated using luminescent semiconducting polymers blended with organometallic emitters by spin-casting have been reported.[12,13] In these studies, energy transfer from the organic emissive material to the phosphor was facilitated by synthesizing an iridium complex with fluorene-modified phenylpyridine ligands, [12,14] because energy transfer from conjugated polymer to ordinary phosphorescent dopants does not occur. In spite of this effort, it is difficult to expect high efficiency and brightness from these devices, owing to the restrictions on the choice of the host emissive material that can be synthesized. In addition, the use of two conjugated polymers for blue and green emission made this WLED less efficient compared with phosphorescent WLEDs. Recently, a multilayer structure system to improve the white electroluminescence (EL) performance has been suggested.[15]

mer blends usually exhibit not only relatively low luminance

Here, we would like to report an alternative approach to the preparation of white-light-emitting devices that uses a highly efficient conjugated polymer doped with iridium complexes and solution processing. In this system, a polyfluorene-type blue-light-emitting polymer (Lumation BlueJ Light-Emitting Polymers, Trademark of Dow Chemical Company, BlueJ) is used as an energy-donating conjugated polymer, and *fac*-tris [2-(2-pyridinyl- \tilde{v} N)(5-(3,4-di-*sec*-butoxyphenyl)phenyl)- \tilde{v} C]iridium(III) [Ir(PBPP)₃; for green emission] and tris(1-phenyliso-quinoline)iridium(III) [Ir(PIQ)₃; for red emission] are used as

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energy acceptors to radiate red, green, and blue light simultaneously. The structures of these components are shown in Scheme 1. In the case of fluorescent–phosphorescent complex devices for WLEDs, the light-emitting properties and efficiency of the energy-donating polymer are very important, because the performance of the devices is dependent mainly on an energy donor. The BlueJ polymer used in this work has a very high luminance efficiency with high brightness, [16,17] and the driving voltage of the device fabricated using this polymer is relatively low compared with devices that use other blue-emitting conjugated polymers. However, this BlueJ polymer is not chemically compatible with iridium complexes, so that energy transfer from this polymer to the iridium complexes does not occur when blended, as energy transfer from most conjugated polymers to the phosphorescent materials does not occur owing to their poor miscibility. [18] Therefore in order to induce the energy transfer we use a third component, PVK (see Scheme 1), which has good chemical compatibility with both iridium complexes and BlueJ polymer, to improve the overall miscibility. [17] This is a very effective method to accelerate energy transfer in the luminescent conjugated polymer-phosphorescent dye system.

2. Results and Discussion

Figure 1a shows the absorption and photoluminescence (PL) spectra of BlueJ polymer, Ir(PBPP)₃, and Ir(PIQ)₃, and the PL spectrum of neat PVK film. The emission spectrum of PVK peaking at $\lambda_{\text{max}} = 400$ nm shows the large spectral overlap with the absorption band of BlueJ (λ_{max} =390) (Fig. 1b), and the emission spectrum of BlueJ overlaps the metal-to-ligand charge transfer (MLCT) absorption band of Ir(PBPP)₃. The peak around 290 nm is the absorption of the ligand center,

Scheme 1. Molecular structures of BlueJ, PVK, Ir(PBPP)₃, and Ir(PIQ)₃.

while the peaks at 390 nm and 420 nm are the absorption of the transition from the ground state to the singlet MLCT and triplet MLCT excited states.[11] Therefore, efficient cascade Förster energy transfer from PVK and BlueJ polymer to Ir(PBPP)₃ is expected. The emission spectrum of BlueJ also broadly overlaps the MLCT absorption band of Ir(PIQ)₃. The peak around 288 nm is the absorption of the ligand center of Ir(PIQ)3, while the peaks at 430 nm and 475 nm are the absorption of the transition from the ground state to the singlet MLCT and triplet MLCT excited states (Fig. 1c). [11] Recently, it has been reported that efficient electrophosphorescence is obtained from a class of heavy-metal complexes that are characterized by a MLCT excited state with a short lifetime of phosphorescence.^[19,20] Therefore, an efficient cascade Förster energy transfer from PVK and BlueJ polymer to Ir(PIQ)3 is also expected. Because Ir(PIQ)₃ is not completely chemically compatible with Ir(PBPP)₃, the partial energy transfer from Ir(PBPP)₃ to Ir(PIQ)₃ occurs through both Förster and Dexter energy transfer.

Figure 2 shows the normalized PL spectra of the blended films made from all four materials. We varied the blend ratios of PVK and BlueJ, fixing the ratio of Ir(PBPP)₃ at 9.7 wt.-% and Ir(PIQ)₃ at 0.3 wt.-%, and used excitation at the wavelength of 345 nm, where the absorption of the host PVK is dominant. After blending the iridium complexes in various ratios, we found the ratio of 9.7 wt.-% Ir(PBPP)₃ and 0.3 wt.-% Ir(PIQ)₃ produced the same luminescent intensity of green and red light. The much higher concentration of Ir(PBPP)₃ relative to Ir(PIQ)3 indicates the existence of energy transfer from Ir(PBPP)₃ to Ir(PIQ)₃. [9] As shown in Figure 1, the emission spectrum of BlueJ overlaps with the absorption band of Ir(PIQ)₃ more than that of Ir(PBPP)₃. However, energy from BlueJ which can not contribute to the red emission is transferred to Ir(PBPP)₃ for green emission because of the low con-

centration of Ir(PIQ)₃ and phosphor saturation^[21] owing to the relatively long triplet decay time. [22]

The blend of BlueJ and iridium complexes without PVK shows no energy transfer from BlueJ to iridium complexes as a result of the low chemical compatibility and phase separation. Only emission from the BlueJ polymer is evident (Fig. 2a), but when blended with PVK, the films begin to show a partial energy transfer. The more the PVK content of the blend film increases, the more conspicuous the energy transfer from BlueJ to the iridium complexes becomes. However, the optimal blend ratio for white emission is BlueJ 65:PVK 25:Ir(PBPP)₃ 9.7:Ir(PIQ)₃ 0.3 [wt.-%], which produces a similar luminescent intensity form each of the three emissive materials. When the PVK content of the blend film reaches 70 wt.-% (BlueJ 20:PVK 70:Ir(PBPP)₃ 9.7:Ir(PIQ)₃ 0.3 [wt.-%]), the domain size of the polymers is minimized to give a perfect energy transfer from BlueJ.[17]

The atomic force microscopy (AFM) images of the blend film with the optimal ratio (BlueJ 65:PVK 25:Ir(PBPP)₃ $9.7:Ir(PIQ)_3 0.3$ [wt.-%]) shows phase-separated domains in the blend, as

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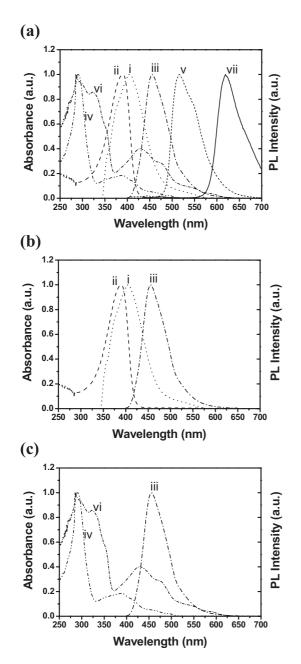


Figure 1. a) Normalized absorption (Abs) and photoluminescence (PL) spectra of PVK, BlueJ, Ir(PBPP)3, and Ir(PIQ)3. i) PL of PVK, ii) Abs of BlueJ, iii) PL of BlueJ, iv) Abs of Ir(PBPP)3, v) PL of Ir(PBPP)3, vi) Abs of Ir(PIQ)₃, vii) PL of Ir(PIQ)₃). b) Spectra of (i,ii,iii). c) Spectra of (iii,iv,vi).

shown in Figure 3. In our previous work, it was confirmed from morphology changes that the bright and dark regions of similar AFM images were occupied by a PVK-Ir complex and BlueJ polymer, respectively.^[17] Because PVK is highly compatible with iridium complexes, the bright side of this AFM image is the domain in which miscible PVK and iridium complexes coexist, and the dark side is the region occupied by the BlueJ polymer. Although BlueJ and the iridium complexes are immiscible, a partial energy transfer occurs at the interface of

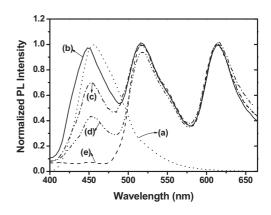
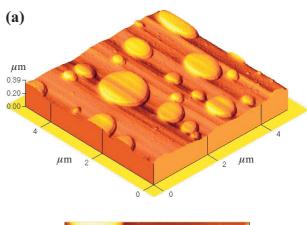


Figure 2. Normalized photoluminescence spectra of thin blend films: a) BlueJ 90:Ir(PBPP)3 9.7:Ir(PIQ)3 0.3 [wt.-%]; b) BlueJ 65:PVK 25:Ir(PBP-P)₃ 9.7:Ir(PIQ)₃ 0.3 [wt.-%]; c) BlueJ 45:PVK 45:Ir(PBPP)₃ 9.7:Ir(PIQ)₃ 0.3 d) BlueJ 30:PVK 60:Ir(PBPP)₃ 9.7:Ir(PIQ)₃ 0.3 [wt.-%]; e) BlueJ 20:PVK 70:Ir(PBPP)₃ 9.7:Ir(PIQ)₃ 0.3 [wt.-%].



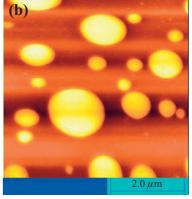


Figure 3. AFM images of the surfaces of BlueJ 65:PVK 25:Ir(PBPP)₃ 9.7:Ir (PIQ)₃ 0.3 [wt.-%] film. The size of each image is 5 μ m × 5 μ m.

the phase in spite of microphase separation, since BlueJ and the iridium complexes are both highly compatible with PVK. The light intensity from the iridium complexes excited by the energy of BlueJ is quite large compared with the light intensity from the iridium complexes excited by the energy of



The EL spectra of BlueJ:PVK:Ir(PBPP)3:Ir(PIQ)3 blends for different blend ratios of polymers doped with 9.7 wt.-% Ir(PBPP)₃ and 0.3 wt.-% Ir(PIO)₃ at an applied voltage of 7 V are shown in Figure 4. As for Figure 2, it is observed that the energy transfer from BlueJ to the iridium complexes occurs primarily according to an increase in PVK content. The BlueJ polymer transfers the energy which is absorbed from PVK and its own energy to iridium complexes. Simultaneous red-, green-, and blue-light emission, which have similar intensity, are obtained in a device with a 65:25:9.7:0.3 ratio of blend components, which is the optimal ratio for WLEDs.

Figure 5 shows the normalized EL spectra of a ITO/PED-OT:PSS/BlueJ 65:PVK 25:Ir(PBPP)₃ 9.7:Ir(PIQ)₃ 0.3 [wt.-%]/ BCP/Li:Al (ITO: indium tin oxide; PEDOT: poly(3,4-ethylenedioxythiophene); PSS: poly(styrene sulfonate); BCP: 2,9dimethyl-4,7-diphenyl-1,10-phenanthroline) device measured for various applied voltages. The spectra contain three main peaks located at 460 nm, 518 nm, and 618 nm, which are ascribed to light emission from the BlueJ host, from Ir(PBPP)₃, and from Ir(PIQ)3, respectively. For the previously reported WLEDs based on immiscible polymer blends, the composition

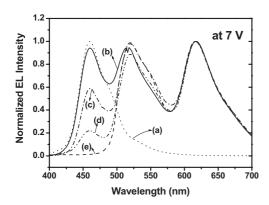


Figure 4. Normalized EL spectra of the devices: a) BlueJ 90:Ir(PBPP)₃ 9.7:Ir(PIQ)₃ 0.3 [wt.-%]; b) BlueJ 65:PVK 25:Ir(PBPP)₃ 9.7:Ir(PIQ)₃ 0.3 [wt.-%]; c) BlueJ 45:PVK 45:Ir(PBPP)₃ 9.7:Ir(PIQ)₃ 0.3 [wt.-%]; d) BlueJ 30:PVK 60:Ir(PBPP)₃ 9.7:Ir(PIQ)₃ 0.3 [wt.-%]; and e) BlueJ 20:PVK 70:Ir(PBPP)₃ 9.7:Ir(PIQ)₃ 0.3 [wt.-%].

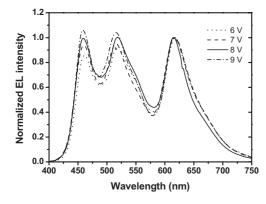


Figure 5. Normalized EL spectra of the ITO/PEDOT:PSS/BlueJ: PVK:Ir(PBPP)₃:Ir(PIQ)₃/BCP/Li:Al device measured at various voltages.

is not so homogeneous normal to the surface owing to the different wettability of each component on the surface that the emissive zone changes with the bias voltage, which results in non-pure white emission.^[23,24] The performance of the polymer-based electrophosphorescent device depends considerably on the properties of the host polymer, and the electrical and optical properties (e.g., driving voltage, light intensity, etc.) of the phosphorescent materials are also similar to those of host polymer if the energy of the host is transferred to them completely. In our system, the iridium complexes have a similar maximum brightness and driving voltage as the BlueJ polymer because these guest materials completely radiate the energy transferred from BlueJ polymer as light. As a result, the Commission Internationale de l'Eclairage (CIE) coordinates of the observed white light are insensitive to the applied voltage. PVK plays the important roles of acting as the hole-transporting material as well as improving the miscibility between the conjugated polymer and the iridium complexes. So, as a holeblocking layer, BCP makes the driving voltage of the iridium complexes effectively lower in the PVK region (Fig. 3) than in the BlueJ region. At lower voltages, the red emission from Ir(PIQ)₃ is somewhat dominant, owing to the efficient excitonenergy transfer and the relatively low energy bandgap. As the voltage is increased, the BlueJ band rises slightly.

We realized nearly "pure" white light, close to CIE (Commission Internationale d'Eclairage) coordinates of (0.333, 0.333), by controlling the concentration of dyes and the phase morphology of the blended film. Aided by a microphase-separated morphology, blue light is emitted from the conjugated polymer, and green and red light are emitted from Ir(PBPP)₃ and Ir(PIQ)3, respectively. Unlike other WLEDs in which white-light emission is obtained from a mixture of two colors, our system is suitable for application in full-color displays with color filters, owing to its simultaneous red, green, and blue emission. The performance of the white-light-emitting device is summarized in Table 1. The CIE coordinates under driving voltages of 6, 7, 8, and 9 V are (0.34,0.34), (0.34,0.33), (0.33,0.33), and (0.32,0.32), respectively, corresponding to brightness values of 708, 1996, 4169, and 6945 cd m⁻². The EL spectrum of fluorescence/phosphorescence complex WLED investigated in this work shows color stability over different operating voltages. The EL spectra of this device cover a wide range of the visible region, and its color rendering index (CRI) value is in the range 83-86, which is appropriate for generalpurpose illumination applications.

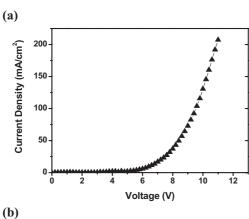
Table 1. The performance of the white-light-emitting device is summarized as luminance, luminance efficiency, and CIE coordinates corresponding to each voltage.

Voltage	Efficiency	Luminance	CIE
[V]	$[cd A^{-1}]$	$[cd m^{-2}]$	(x,y)
6	12.11	708	(0.34,0.34)
7	11.95	1996	(0.34, 0.33)
8	11.23	4169	(0.33, 0.33)
9	9.55	6945	(0.32, 0.32)



The current-density versus voltage and luminance versus voltage characteristics of the ITO/PEDOT:PSS/ BlueJ 65:PVK 25:Ir(PBPP)₃ 9.7:Ir(PIQ)₃ 0.3 [wt.-%]/BCP/Li:Al device are shown in Figures 6a,b, respectively. The turn-on voltage (defined as the voltage required to give a luminance of 1 cd m⁻²) of the 65:25:9.7:0.3 blend device was 3.4 V. This result is the lowest driving voltage among the polymer-based whiteemitting electrophosphorescence devices reported to date. While energy transfer from the PVK to the iridium complex leads to the relatively high working voltage of the devices as a result of charge-trapping effects, [10,11] the energy transfer from BlueJ to the iridium complexes lowers the driving voltage of the device, because the dominant mechanism in EL in this latter system is Förster energy transfer rather than charge trapping.^[17] The device achieves a maximum brightness of 11730 cd m⁻² at 11 V and a current density of 207.22 mA cm⁻².

Figures 7a,b show luminance efficiency and external quantum efficiency as a function of current density, respectively. The peak luminance efficiency of the BlueJ 65: PVK 25:Ir(PBPP)₃ 9.7:Ir(PIQ)₃ 0.3 [wt.-%] device is 12.52 cd A⁻¹ at a current density of 7.23 mA cm⁻², a voltage of 6.2 V, and a brightness of 905 cd m⁻², corresponding to an external quantum efficiency of 3.2 %. The external quantum efficiency of the device was increased by using two phosphorescent materials in



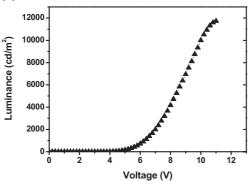
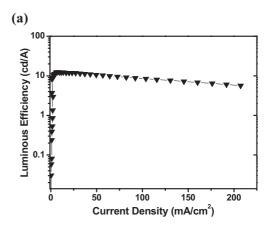


Figure 6. a) Current-density versus voltage characteristics of the ITO/ PEDOT:PSS/BlueJ:PVK:Ir(PBPP)3:Ir(PIQ)3/BCP/Li:Al device. b) Luminance versus voltage characteristics of the ITO/PEDOT:PSS/BlueJ:PVK:Ir (PBPP)3:Ir(PIQ)3/BCP/Li:Al device.



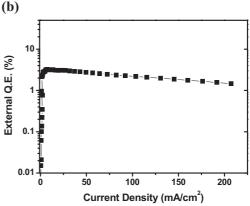


Figure 7. a) Luminance-efficiency versus current-density characteristics of the ITO/PEDOT:PSS/BlueJ:PVK:Ir(PBPP)3:Ir(PIQ)3/BCP/Li:Al device. b) External-quantum-efficiency versus current-density characteristics of the ITO/PEDOT:PSS/BlueJ:PVK:Ir(PBPP)3:Ir(PIQ)3/BCP/Li:Al device.

the WLED. Also, we obtained polymer-based electrophosphorescent WLEDs with high brightness and efficiency by using a highly efficient luminescent conjugated polymer as both an energy host and blue emitter.

Figure 8 shows a photo of such a device during operation at 7 V. Uniform, pure white emission is observed, even though simultaneous red-, green-, and blue-light emission is occurring in the microphase-separated system.

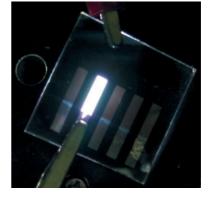


Figure 8. Photo of a white-light-emitting device during operation at 7 V. The size of the emitting device is 2 mm × 8 mm.



So far, we have considered efficient energy transfer from the conjugated polymer to the phosphorescent dopants from the point of view of miscibility, but it can be also explained by a triplet energy level. Recently, it was reported that energy transfer from conjugated polymers to phosphorescent dyes is difficult because of phosphorescence quenching owing to the relatively low triplet energy levels of the conjugated polymers.^[25,26] On the other hand, PVK has a higher triplet energy than that of commonly used heavy-metal phosphorescent dopants, which means that it cannot quench the triplet state of the dopant. [27] In our system, besides energy transfer resulting from the improved chemical compatibility, it is expected that the triplet energy level of the polymer blend at the PVK/BlueJ interface will be higher when BlueJ is mixed with PVK that has the high triplet excited state. Both factors, i.e., improved chemical miscibility and the raised triplet excited state of host polymer blend, lead to efficient energy transfer from BlueJ to the iridium complexes. A more detailed evaluation of the physical mechanism, including the triplet energy level of the host-polymer blend, is necessary to understand the energy-transfer mechanism in a phosphorescent dye-doped polymer-blend system. Related further studies are ongoing in our laboratory.

3. Conclusions

We have fabricated efficient polymer-based electrophosphorescent WLEDs using iridium complexes with the BlueJ polymer as a new efficient host material. The color of the devices can be tuned by varying the blend ratios of the emitting materials. PVK was used to lead energy transfer from conjugated polymer to phosphorescent dyes by improving the chemical compatibility between the conjugated polymer and phosphorescent dyes. Luminescence efficiency was improved owing to efficient energy transfer from the conjugated polymer to the iridium complexes, and the turn-on and driving voltages of the WLED were greatly decreased. We obtained excellent whitelight emission, and color stability resulted from simultaneous red-, green-, and blue-light emission through controlled microphase-separated morphology. This approach for white-light emission is promising compared to phosphorescent-dye-doped polymer-blend systems in terms of both efficiency and color stability. We expect that this work will be useful in the development of inexpensive large-area WLEDs, full-color flat-panel displays with color filters, and new illumination light sources.

4. Experimental

Materials: Poly(3,4-ethylenedioxythiophene):poly(styrene sulfonate) (PEDOT:PSS) was purchased from Baytron PH, H. C. Starck (Germany). PVK was purchased from Sigma–Aldrich and refined in our laboratory using the following procedure. PVK (4 wt.-%) was dissolved in tetrahydrofuran and then precipitated in stirred ethanol and filtered using a 0.2 μ m injector filter. The product was dried in a vacuum oven for 12 h at 50 °C. Ir(PIQ)₃, purchased from American Dye Sources, Inc., was used for red emission. Other materials were used as received.

Ir(PBPP)₃ was used as a green emitter, and BlueJ polymer was used as not only the host but also a blue emitter. PVK, BlueJ, Ir(PBPP)₃, and Ir(PIQ)₃ were dissolved in chlorobenzene. The highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) energy levels of PVK, BlueJ, Ir(PBPP)₃, Ir(PIQ)₃, and BCP are depicted in Scheme 2. The electrochemical behavior of the polymers and the iridium complexes was investigated by cyclic voltammetry. Cyclic voltammetry studies were performed with an eDAQ EA160 potentiostat. All measurements were carried out in an electrolyte of 0.1 M tetra-n-butylammunium tetrafluoro borate solution in acetonitrile using a three-electrode configuration with sample thin films as

Scheme 2. Energy-level diagram of PVK, BlueJ, Ir(PBPP)3, Ir(PIQ)3, and RCP

the working electrode, platinum as the counter electrode, and Ag/AgCl serving as the reference electrode. Thin films were deposited by casting from a dilute chlorobenzene solution on an ITO electrode of ca. 1 cm² surface area. The HOMO levels of the materials were measured using cyclic voltammetry and the LUMO levels were found by measuring the bandgap of the materials via UV-vis spectra investigation.

Device Fabrication: ITO-coated glass substrates were cleaned by sonication in chloroform for 30 min and then in acetone/isopropyl alcohol (1:1 ν/ν) solution for 20 min and treated with oxygen plasma before use. A layer of PSS:PEDOT with a thickness of 20 nm was first spin-coated on the treated substrate, and annealed at 120 °C for 1 h under vacuum. Then, blends of BlueJ, PVK, Ir(PBPP)₃, and Ir(PIQ)₃ in chlorobenzene (10 mg mL⁻¹) were spin-coated on top of the substrate (precoated with a PSS:PEDOT layer. For device fabrication, we employed a single-active-layer architecture (ITO/PEDOT:PSS/BlueJ: PVK:Ir(PBPP)₃:Ir(PIQ)₃/BCP/Li:Al. The BCP layer (20 nm) acted as a hole-blocking layer and was thermally evaporated onto the blend layer at below 1×10^{-6} torr (1 torr = 133 Pa) before electrode deposition, and then a cathode layer of 150 nm thick Li:Al alloy was vacuum deposited at a pressure of 1×10^{-6} torr.

Measurement: All measurements of the EL properties were performed on the devices encapsulated by a cover glass by using an epoxy adhesive. The emission light of the devices was measured by a luminance meter (Minolta, LS-100) and a luminous spectrometer (Minolta, CS-1000). The electrical and luminescence characteristics of the devices were analyzed with a current and voltage-source measurement unit (Keithley 236) and a luminance meter (Minolta, LS-100). Photoluminescence and EL spectra were measured using a fluorospectroscope (ISS PC1 Photon Counter Meter). The CRI was quantitatively evaluated from the EL spectra using reported numerical formulas [28,29].

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