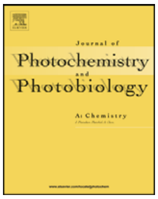
Journal of Photochemistry and Photobiology A: Chemistry 200 (2008) 371–376

Contents lists available at ScienceDirect

Journal of Photochemistry and Photobiology A: Chemistry

journal homepage: www.elsevier.com/locate/jphotochem



Triplet level-dependent photoluminescence and photoconduction properties of π-conjugated polymer thin films doped by iridium complexes

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ARTICLE INFO

Article history:
Received 3 June 2008
Received in revised form 23 July 2008
Accepted 22 August 2008
Available online 11 September 2008

Keywords:
π-Conjugated polymer
OLED
Photoluminescence
Phosphorescence
Energy transfer

ABSTRACT

Triplet energy level-dependent decay pathways of excitons populated on iridium (Ir) complexes within π-conjugated polymeric matrices were studied by means of photoluminescence (PL) and photoconduction action spectroscopy. We chose a set of matrices, poly(9-vinylcarbazole) (PVK), poly[9,9-bis(2-ethylhexyl)fluorene-2,7-diyl] (PF2/6), poly [2-(5′-cyano-5′-methyl-hexyloxy)-1,4-phenylene] (CNPPP), and poly [2-(5′-cyano-5′-methyl-hexyloxy)-1,4-phenylene-*co*-pridine] (CNPPP-py10 and CNPPP-Py20), having triplet energy levels ranging from 2.2 up to 3.0 eV. As Ir-complex dopants, we selected three phosphorescent emitters, iridium(III)bis(2-(2′-benzothienyl) pyridinato-*N*-acetylacetonate) ($Ir(btp)_2acac$), iridium(III)fac-tris(2-phenylpyridine) ($Ir(ppy)_3$), and iridium(III)bis[(4,6-fluorophenyl)-pyridinato-*N*,$C^{2'}$]picolinate (FIrpic), having triplet energy levels of 2.1, 2.5, and 2.7 eV, respectively. It was found that the triplet emission from the dopants, being populated via energy transfer from the matrices, was strongly dependent on the matching of triplet energy levels between matrix polymers and Ir-complexes. Photocurrent action spectra confirm effective exciton confinement at the dopants sites in the case of PVK matrix systems.

1. Introduction

Since Burroughes et al. [1] reported on an electroluminescent light-emitting diode (LED) using a soluble π-conjugated polymer, related solution processible polymers having optical and electric functionalities have attracted much attention due to their applicability to flexible optoelectronics [2,3]. In spite of offering significant advantages in fabricating flexible devices compared to conventional silicone-based device manufacturing, π-conjugated polymers have some drawbacks due to their intrinsic material properties, which can limit their maximum device efficiency. One of these problems is the 25% barrier of the singlet exciton-forming ratio due to the spin statistics of common organic materials. This problem was dramatically improved by Forrest and co-workers by doping triplet dopants as the main emitting species [4]. This concept was also applied to polymeric devices in order to achieve higher device efficiency [5].

Many groups have investigated-doped systems in efforts to clarify the underlying mechanisms and thereupon improve the device efficiency using this technique [6,7]. Soon after the breakthrough of the OLED efficiency via triplet doping and subsequent research activities, it was determined that the triplet level matching between the matrix and dopant is also crucial as efficient migration of singlet excitons on the matrix to the triplet dopant is important to tailoring an optimum host and guest combination. The importance of endothermic and exothermic transfer depending on the relative triplet energy level of the matrix to that of the dopant was reported by Forrest and co-workers [8,9] For polymer systems, similar behavior was studied with a doping system consisting of a phenyl-substituted poly(*para*-phenylenevinylene) derivative doped by Pt(II)octaethylporphine (PtOEP) in terms of back-transfer [10]. In addition, it was suggested that the triplet population mechanism onto dopant molecules from the matrix involves Förster-type resonant energy transfer (FRET), using a ladder type methyl-substituted poly(*para*-phenylene) (MeLPPP) [11].

Those triplet energy level-related exciton dynamics was also investigated by van Dijken et al. with oxadiazole containing polycarbazole copolymeric systems with electroluminescence (EL) efficiency analysis [12]. And Rothe et al. have shown systematic triplet kinetics using time-resolved measurements to depict

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doi:10.1016/j.jphotochem.2008.08.019

triplet exciton transfer between Ir emitters and analogous polycarbazole copolymers including polyspirobifluorene homopolymer [13]. Recently, Baluschev et al. [14] and Castellano and co-workers [15] have shown efficient up-conversion results with photoluminescence quantum yields as high as 0.04 using various types of heavy metal containing triplet sensitizers in solutions whose underlying mechanism might be explained by means of sensitized triplet–triplet annihilation as reported by Lquai et al. previously [16].

In a previous study on this topic, we revealed that the samples bearing a 1 wt% FIrpic and $Ir(ppy)_3$-doped PF2/6 system showed no detectable dopant emission at room temperature, although the spectral overlap between PF2/6's emission band and $Ir(ppy)_3$'s absorption band was not negligible [17]. The PF2/6 system displayed significant quenching upon doping of Ir-complexes. This behavior is analogous to that of the PVK system except for a subsequent intense emission from the dopant. It was thus suggested that the underlying mechanism for the effective PL quenching of PF2/6 via triplet doping is either efficient back-transfer from the triplet population on the dopant which was originally photogenerated on the matrix as singlet excited state to the matrix's triplet due to the lower lying triplet level of the matrix or dopant-induced electron dissociation due to a molecular heterojunction.

We also characterized a series of polymer having a triplet energy level between PVK and PF2/6 by means of low-temperature time-resolved photoluminescence (PL) spectroscopy [18]. Thus, in the present study, we investigate the triplet energy level dependence of dopant emission intensity in the matrix in order to verify the back-transfer phenomena. In addition, we prepared doped films with a red emitting Ir-complex having the lowest triplet energy among the used materials to confirm the triplet energy level dependency in a reverse manner by controlling the dopant's triplet energy level. Finally, photocurrent action spectroscopy was performed to probe whether the exciton dissociation and subsequent charge carrier generation takes place competitively with back-transfer.

2. Experiments

Poly(9-vinylcarbazole) (PVK, Mw = 100,000) was purchased from Aldrich Co., and poly[9,9-bis(2-ethylhexyl)fluorene-2,7-diyl] (PF2/6), poly [2-(5′-cyano-5′-methyl-hexyloxy)-1,4-phenylene] (CNPPP), and poly [2-(5′-cyano-5′-methyl-hexyloxy)-1,4-phenylene-*co*-pridine] (For CNPPP-py10 and CNPPP-Py20, the numbers denote the feeding mol ratio of pyridine comonomer) were synthesized [18] and used for this study. These π-conjugated polymers were used as matrix materials, where triplet emitters were doped. FIrpic, $Ir(ppy)_3$ and $Ir(btp)_2acac$ were used as phosphorescent dopants for the polymers. The chemical structure of $Ir(btp)_2acac$ is shown in the inset of Fig. 1.

Thin films were prepared on quartz substrates by spin coater at 2000 rpm under an ambient condition. For the spin coating, a 1 wt% chloroform solution of a mixture contaning triplet dopant's were used. The doping ratio of the phosphorescent materials was either 0.1 wt% or 1.0 wt% relative to the corresponding matrix polymer. The obtained films had a thickness of about 80 nm and showed a transparent and homogenous film quality. UV–visible absorption (UV–vis) was measured using a conventional UV-vis absorption spectrometer (Cary 100, Varian Co.) for the spin-coated films. The PL and PLE spectra of the thin films were recorded with the aid of a commercial luminescence spectrometer (LS-50B, PerkinElmer Co.) at room temperature using quartz plates.

For steady-state photocurrent measurement, single layer devices with an ITO/polymer/Al sandwich structure were prepared. For this, 0.8 wt% of polymer mixture (polymer 99 wt%: dopant 1 wt%) in a chloroform solution was spin-coated onto an ITO substrate at a speed of 2000 rpm for 30 s. Aluminum (Al) top electrodes of 9 mm^2 active area were evaporated onto the spin-coated polymer films. Photocurrent measurements were performed in a temperature-controlled vacuum chamber with a combination of a monochromator and a xenon arc lamp as a light source. Photocurrent signals were detected with a lock-in amplifier (Model 7260, EG&G Instrument), which was connected to a chopper operated at a frequency of 20–40 Hz. The benefit of employing the lock-in technique is that it automatically subtracts the dark current from the measured signals. To calculate the electrical field in ITO/polymer/Al diodes, a built-in potential of 0.6 V between the Al and ITO electrodes has been taken into account. The charge carrier photogeneration quantum yield (QY) was calculated from the measured photocurrents, taking into account corrections for the light source intensity and absorption by glass substrates, the ITO electrodes, the active area of the film, and reflection from the Al electrode.

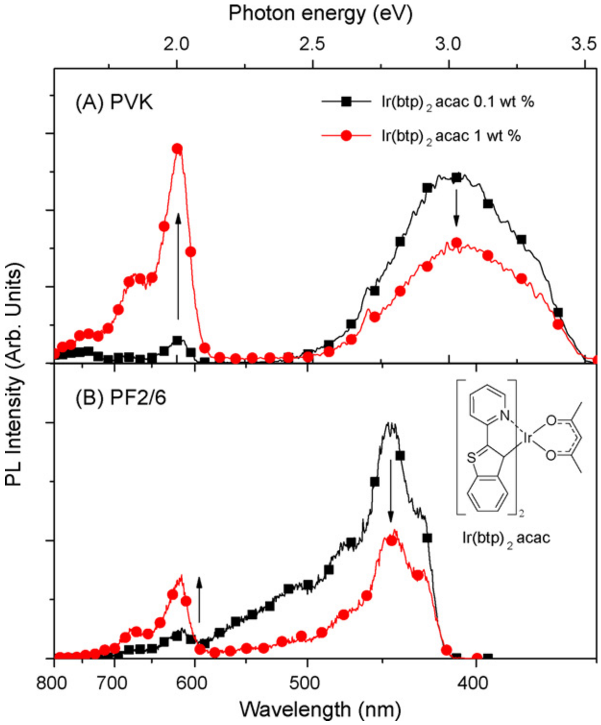

Fig. 1. PL spectra of PVK (A) and PF2/6 (B) film samples doped by 1 wt% of $Ir(btp)_2acac$.

3. Results

3.1. Doping of $Ir(btp)_2acac$

Both PVK and PF2/6 thin films doped with 1 wt% of $Ir(btp)_2acac$ showed clear emission behaviors from the triplet dopant with a reduction of the mutual matrix emission upon increasing the doping ratio from 0.1 to 1 wt%, as shown in Fig. 1. This reduction of matrix PL was also observed with FIrpic and $Ir(ppy)_3$ doping as reported in the previous study regardless whether there was subsequent emission from dopant molecules or not [17].

The three-dimensionally mapped PL and PLE spectra of the PVK and PF2/6 films doped by 1 wt% of $Ir(btp)_2acac$ are shown in Fig. 2, where the contour lines have a logarithmic scale of the PL intensity. Darker grey color between the contour lines was used to indicate higher PL intensity. The horizontal cross-section of the spectra,

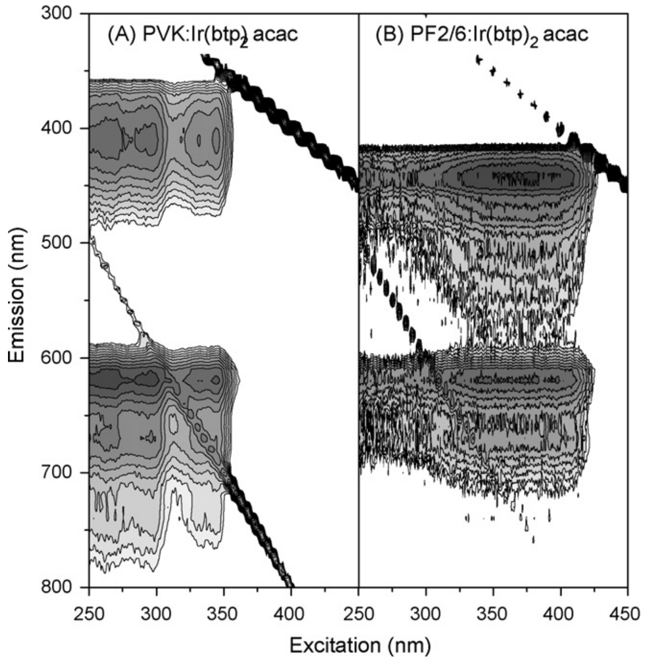

Fig. 2. Three-dimensional mapping of PLE and PL spectra of PVK (A) and PF2/6 (B) film samples doped by 1 wt% of Ir(btp)$_2$acac.

i.e. the PLE spectra of both films at a detection wavelength of 615 nm, followed the coincident spectral dependences as the intrinsic UV–vis spectra of their corresponding matrices. This means that the triplet excitons on Ir(btp)$_2$acac were mainly populated from the photogenerated primary excitons on the matrix molecules. This trend was also observed for CNPPP and its pyridine containing statistical copolymers (data not shown).

3.2. CNPPP and CNPPP-pyxs as matrices for Ir(ppy)$_3$

PL spectra of CNPPP, CNPPP-py10, and CNPPP-py20 doped by various triplet emitters with a 1 wt% doping ratio are shown in Fig. 3. There were no emissions from FIrpic, while Ir(btp)$_2$acac-doped films exhibited a typical emission from Ir(btp)$_2$acac, having a band maxima centered at 615 nm. Interesting behaviors of dopant emission were observed with the samples doped by 1 wt% of Ir(ppy)$_3$ having a 2.4 eV triplet energy level precisely located between FIrpic and Ir(btp)$_2$acac's triplet energy levels. The PL emission from the green triplet dopant could be observed for all of the CNPPP derivatives and increased with increasing triplet energy level of the matrix. This transition of Ir(ppy)$_3$ emission upon increasing the matrix triplet level is shown in Fig. 4 in greater detail as normalized PL spectra with (Fig. 4B) and without (Fig. 4A) doping for a clear comparison. The triplet energy levels of CNPPP derivatives characterized by low-temperature time-resolved PL spectroscopy and conventional cyclic voltammetry (CV) were reported in a previous work [18] and the values are illustrated in Fig. 5 with other relevant triplet energy levels. It should be noted that there is a slight difference between the currently used triplet energy levels and previously reported energy values, because we have used the values taken at the front edge of the PL spectra instead of the maximum peak value for all emitting species except PVK. This was performed in order to remove uncertainty by comparing triplet energy levels, such as that caused by intrinsic conformational disorder induced bandwidth variation. Since PVK has a much wider S_0–T_1 gap compared to the triplet dopants used in this study, we used the triplet energy value of PVK as reported by Burrows and co-workers [19] without any change. The value for PF2/6 was taken from the spectra reported by Hertel et al. [20] in the same manner as for CNPPP. In Fig. 6, relative PL intensities from the Ir(ppy)$_3$ dopant as a function of the matrix's triplet level with a fitting curve using a Boltzmann function are shown.

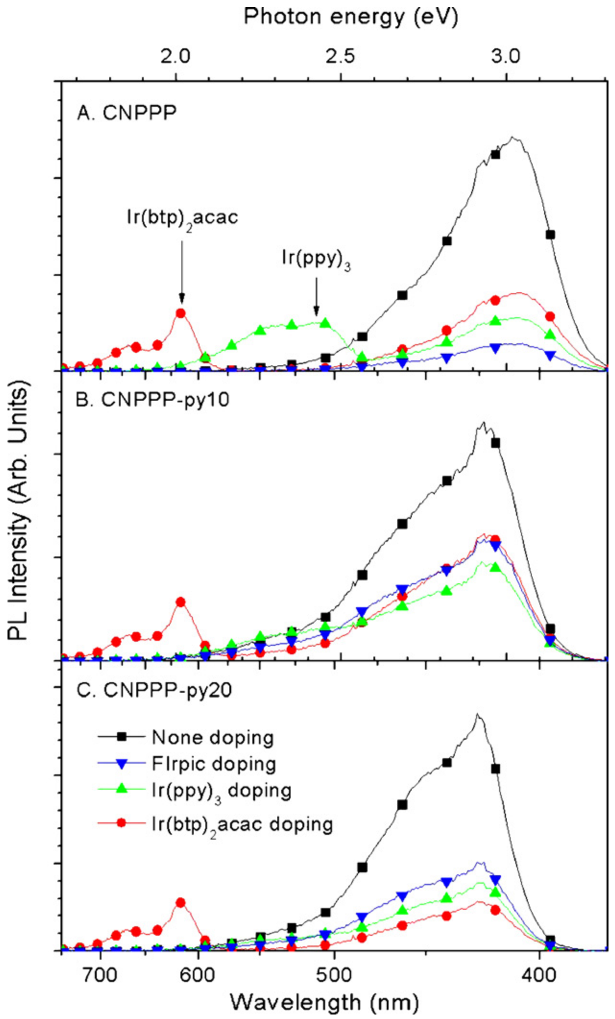

Fig. 3. PL spectra of triplet emitter-doped samples: (A) CNPPP, (B) CNPPP-py10 and (C) CNPPP-py20.

3.3. Photocurrent action spectra

We have performed a set of steady-state photocurrent measurements for verifying whether a doped system having no subsequent dopant emission could provide a higher charge carrier photogeneration quantum yield (QY) as discussed previously. Interestingly, the PF2/6 system showed a less pronounced increase of photocurrent upon 1 wt% doping of FIrpic, while the PVK system showed considerably pronounced photocurrent at the same electric field of about 5×10^5 V/cm under applying the forward bias, as shown in Fig. 7. We also observed a steep increase of QY above 0.6 eV from the absorption edge in the case of PVK and above 0.8 eV in the case of PF2/6.

4. Discussion

Upon doping of Ir(btp)$_2$acac into those conjugated polymers, corresponding red emission from the dopant, being mainly populated from the matrix polymers, as evidenced by PLE spectra,

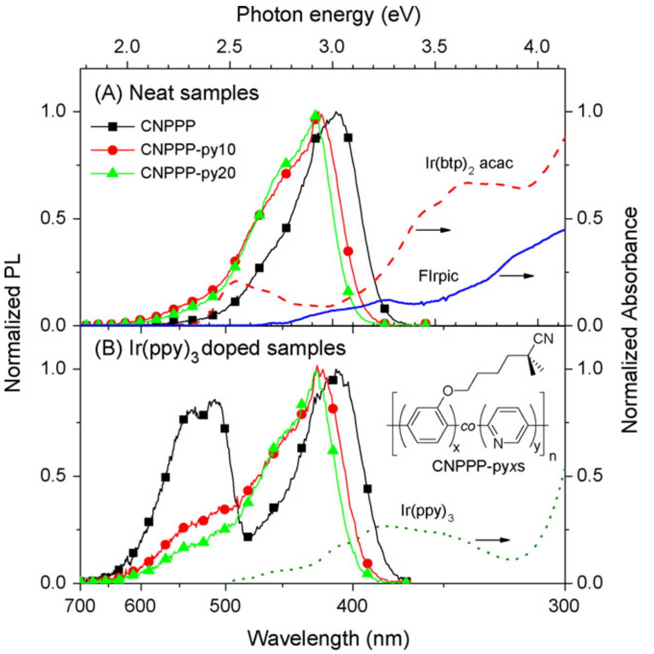

Fig. 4. Normalized PL spectra of un-doped (A) and Ir(ppy)$_3$-doped (B) films of CNPPP derivatives. Normalized absorbance spectra of dopant molecules are also shown for comparison. (Inset: the chemical structure of CNPPP-py*x*s).

was observed regardless of the triplet energy levels, since all of the triplet energy levels of the matrices were lower than that of Ir(btp)$_2$acac. A transition of Ir(ppy)$_3$ emission intensity from strong to weak was observed with CNPPP, CNPPP-py10 and CNPPP-py20 having triplet energy levels between those of PVK and PF2/6, as shown in Fig. 6. The transition trend of the Ir(ppy)$_3$ emission precisely follows the triplet levels of the matrices: PVK (3.0 eV) > CNPPP (2.6 eV) > CNPPP-py10 (2.5 eV) ≥ CNPPP-py20 (2.4 eV) ≫ PF2/6 (2.2 eV) ~ 0 (no dopant emission). Similarly, the transition trend for the CNPPP and CNPPP-py*x*s matrices was as follows: Ir(btp)$_2$acac ≥ Ir(ppy)$_3$ ≫ FIrpic ~ 0 (no dopant emission). This trend about the triplet energy level-dependent PL from the Ir-complexes is summarized as Fig. 6, where the relative Ir(ppy)$_3$ emission intensity is plotted as a function of the triplet level of the matrix. It should be pointed out that the triplet level of the matrix should be at least 0.2 eV higher than that of the Ir-complex in order to ensure effective dopant emission, as determined from a comparison of the CNPPP and CNPPP-py*x*s matrices. A difference of higher than 0.5 eV might be needed to achieve highly efficient triplet exci-

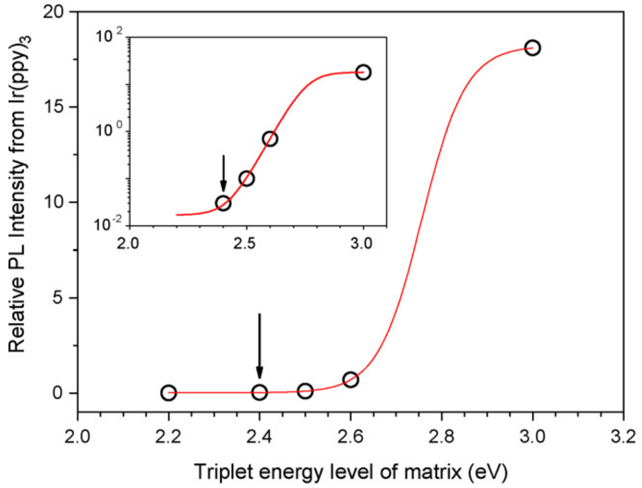

Fig. 6. Plot of relative Ir(ppy)$_3$ emission intensity as a function of matrix's triplet energy level, inset: same plot in half logarithmic scale. (Arrow indicates the triplet level of Ir(ppy)$_3$ and the solid line is an interpolated curve using a Boltzmann function.)

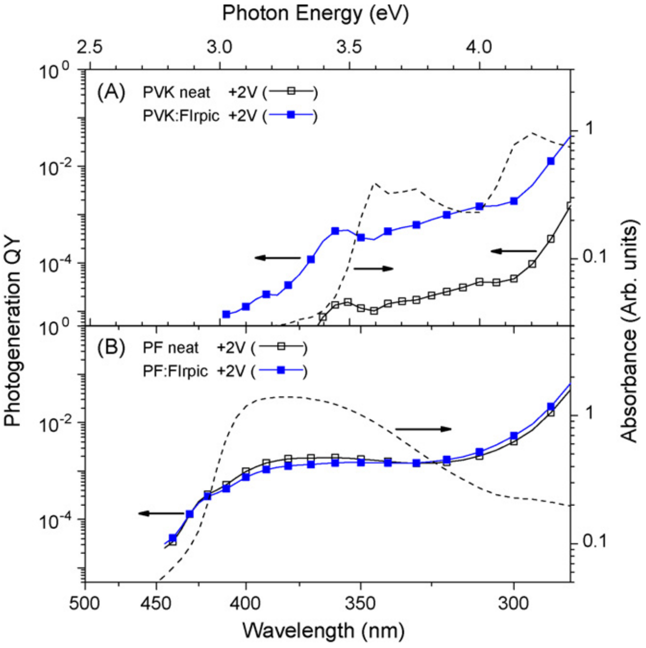

Fig. 7. Photocurrent action spectra of the triplet-doped PVK and PF2/6 devices.

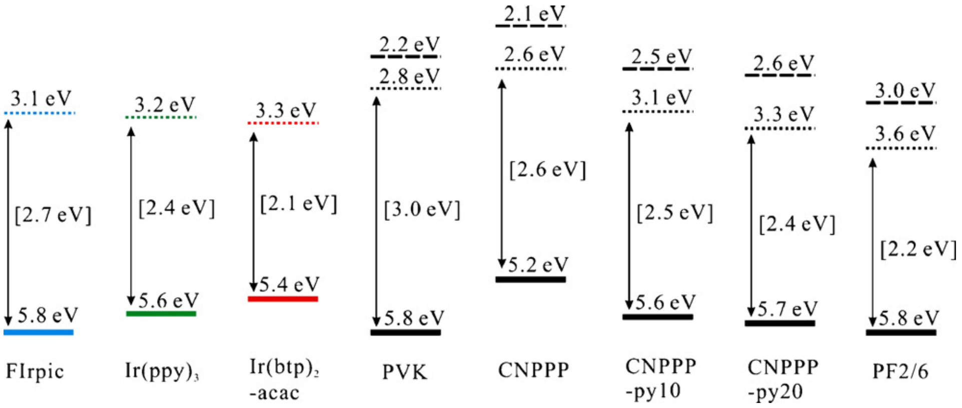

Fig. 5. Energy level diagram of studied materials (solid lines: S_0, dotted lines: T_1 and dashed lines: S_1). Values in brackets "[ ]" are the differences between the triplet energy level (T_1) and the HOMO energy level (S_0).

ton confinement at the dopant sites, and hence quasi loss-less dopant PL emission at room temperature having a relatively low doping ratio compared to the conventional doping ratio when fabricating phosphorescent LEDs [21]. The value of 0.5 eV might be considered as barrier amplitude to avoid band overlapping between the matrix and dopant's triplet bands at or beyond which back-transfer could occur in such π-conjugated polymeric systems. The barrier amplitude of about 0.5 eV can be deemed reasonable if one considers the full width at half maximum of about 0.3 eV for typical semiconducting π-conjugated polymers. The significant barrier amplitude value is a result of their inhomogeneously broadened feature due to strong conformational and environmental disorder effect, as verified by numerous spectroscopic studies [22,23].

In a previous study [17], higher photocurrent QY for a PF2/6-doped system was suggested as one of the possible reasons why PF2/6 has absolutely no emission from triplet dopant, although excitons from the PF2/6 matrix was effectively removed upon doping of triplet emitters as PVK system. The removal of excitons from mutual matrix sites might be caused either by electron transfer to the triplet level of the dopants or by energy transfer to the dopant. In the former case, migrated electrons could contribute to the generation of free charge carriers or undergo fast non-radiative recombination [24]. In the latter case, the migrated excitons could be dissipated via effective back-transfer to the lower-lying triplet level of the matrix from the dopant's triplet population and subsequent non-radiative recombination, as is common for π-conjugated polymers, especially at room temperature [25].

Interestingly, from the results of steady-state photocurrent measurements, the doped PVK systems show a significant increase of charge carrier photogeneration QY while PF systems have no significant photocurrent change upon doping of 1 wt% triplet emitters. This supports the notion that the triplet excitons at the Ir-complexes within PVK films have a pronounced triplet exciton confinement effect due to sufficiently lower triplet energy levels of Ir-complex dopants compared to their PVK matrix. Meanwhile, triplet excitons on dopants within PF2/6 films should rapidly non-radiatively recombine via fast back-transfer to the triplet level of the PF2/6 matrix.

The action spectra of the PF2/6 system revealed similar spectral dependences and amplitudes, as were reported with a poly(*para*-phenylenevinylene) derivative doped by electron-accepting trinitro-fluorenone (TNF) systems. Here, the photocurrent QY response was mainly contributed by acceleration of the extrinsic photocurrent at the interface between the ITO and polymer layers in conjunction with the bulk sensitized intrinsic photocurrent via TNF doping [26]. Furthermore, the PVK neat sample showed a significantly lower value than that of PF2/6. This might be explained by the lower intrinsic charge carrier mobility between PVK [27] and PF2/6 [28]. Thus, the photocurrent value of PVK-based sample was recovered by doping of the Ir-complex, which improved not only charge carrier photogeneration but also the mobility. It should also be emphasized that the exciton binding energy of the PVK system is about 0.2 eV smaller than that of PF2/6, extracted from the energy gap from the absorption edge to the point where the steep increase of charge carrier photogeneration QY started [29]. It is noteworthy that the PVK system is not directly comparable to the PF2/6 system due to the absence of extended π-conjugation along the polymer backbone. Therefore, this could account for the different charge carrier photogeneration behavior between PVK and PF2/6. However, detailed discussion of the photocurrent action spectra is beyond the scope of the current study and will be presented in a separate report.

From the action spectra, we could suggest a possible mechanism that the PVK system provides stable confinement of triplet excitons at the Ir-complexes. This improves the triplet emission efficiency as well as the probability of dissociation upon application of an external electric field. Meanwhile, PF2/6 has less chance to emit photons or generate free charge carriers due to very fast back-transfer of the triplet from the dopant to the polymer matrix.

5. Conclusion

We have measured time-integrated UV–vis, PL, and PLE spectra of triplet emitter doped π-conjugated polymeric thin films spin-casted from the chloroform solutions bearing1 wt% of polymer and a Ir-complex mixture at room temperature. Doping concentrations of 0.1 and 1 wt% of Ir-complex were, respectively, used to prepare PVK:Ir(btp)$_2$acac, PF2/6:Ir(btp)$_2$acac, CNPPP-py*xs*:Ir(btp)$_2$acac, CNPPP-py*xs*:Ir(ppy)$_3$, and CNPPP-py*xs*:FIrpic blending films. These polymer matrices have triplet energy levels ranging from 2.1 up to 3.0 eV, as the triplet dopants have different triplet energy levels from 2.2 up to 2.7 eV, as summarized in Fig. 5. Furthermore, steady-state photocurrent measurements were performed to verify the fate of the triplet population at the Ir-complexes with PVK and PF2/6 matrices doped by various Ir-complexes.

It was found that the emission from the Ir-complex dopant was strictly dependent on the triplet level matching between the matrix and dopant. In the case of PL at room temperature, efficient dopant emission might be ensured when the triplet level of dopant is about 1 eV lower than that of the matrix. Additionally, the charge carrier photogeneration QY difference between PVK and PF2/6 systems revealed a clear exciton confinement effect on the Ir-complex for the PVK system. Meanwhile, the PF2/6 system is governed by fast dissipation of the triplet exciton through an efficient back-transfer mechanism.

In conclusion, back-transfer of the triplet population on the Ir-complexes, in other words, triplet exciton migration from triplet dopants back to the matrix, is the determinant factor to improve the triplet emitting efficiency and thereby realize highly efficient phosphorescent organic and polymeric LEDs. Elucidation of a subsequent pathway related to the back-transfer is expected to be crucial to improve photovoltaic device applications involving donor–acceptor pairing. [30] Therefore, the matching of triplet energy levels between matrices and dopants should be carefully designed for applying doped systems to optoelectronic application.

Acknowledgments

This work was supported by the Seoul Research & Business Development (R & BD) Program (CR070048) and the Guest Associate Professor Program, Nanoscience and Nanotechnology Center, Institute of Scientific and Industrial Research (SANKEN), Osaka University. C.I. is indebted to Dr. Sergey Bagnich for fruitful discussions.

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