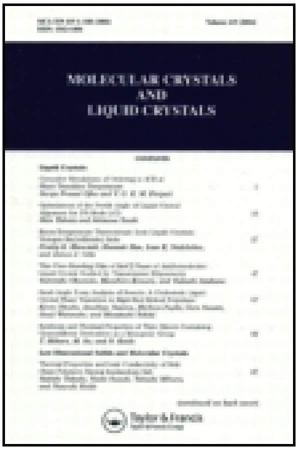

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Green and Red Electrophosphorescent Devices Consisting of Cabazole/Triarylamine-Based Polymers Doped with Iridium Complexes

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*Two types of polymers were synthesized such as poly[*N*-(2-ethylhexyl)carbazole-*alt*-*N*-(4-aminophenyl)carbazole] (PECAC) and poly[*N*-(2-ethylhexyloxy-phenyl) carbazole-*alt*-*N*-(4-aminophenyl)carbazole] (PEPCAC). These polymers are designed to have carbazole groups with wide band gap and a triarylamine moiety with a hole transporting property. Green and red phosphorescent polymer light emitting diodes (PhPLEDs) were fabricated with an emitting layer consisting of either PECAC or PEPCAC as a host matrix with suitable iridium complexed, Ir(ppy)$_3$ as a green or IR-PIQCH as a red dopant, respectively. Red PhPLEDs doped with IR-PIQCH exhibited maximum EL emission peaks at 620 nm with higher luminescence and lower driving voltage, due to effective energy transfer from host*

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polymers to IR-PIQCH red dopant. Based on HOMO and LUMO levels of the polymers and Ir(ppy)$_3$, however, the synthesized polymers could not be served as suitable host polymers for Ir(ppy)$_3$ green dopant. Ir(ppy)$_3$ doped PhPLEDs exhibited red shift and wide range of EL emission.

Keywords: carbazole; dopant; host; phosphorescence; PLED; triarylamine

INTRODUCTION

Increasing interest has been paid to carbazole-based polymers because of their well-known optical properties and hole transporting ability in organic light emitting diodes (OLEDs). Such good properties for series of carbazole materials are due to the electron-donating capabilities with its nitrogen atom [1–4]. Among them, polyvinylcarbazole (PVK) was widely used and investigated as non-conjugated vinyl polymer with carbazole attached in the side chain [5]. Recently, conjugated polymers with carbazole as main chain built-unit have been studied extensively. Polymers incorporated of 3,6-carbazole into varieties of aromatic and heterocyclic comonomers have been synthesized. For instance, the synthesis of well-defined copolymers of 3,6-carbazole with phenylenes, thiophenes or fluorenes has led to a significant improvement in the performance of these polymeric materials and to a better understanding of their structure-property relationships [6–8]. Considering good hole-transporting property and wide band gap energy for the conjugated polycarbazole, it can be used as a host segment in copolymerization with various comonomers and used as host-matrix materials in OLEDs.

Triarylamine derivatives with low molecular weight are the most common class of hole transporting materials, such as *N,N′*-diphenyl-*N,N′*-bis(3-methylphenyl)-1,1′-biphenyl-4,4′-diaimine (TPD). Whereas, TPD shows low glass transition temperature at 60°C that causes crystallization and thermal breakdown during the operation of OLEDs. So, *N,N′*-diphenyl-*N,N′*-bis(α-naphthyl)benzidine (NPB) has been widely used instead of TPD [9–10]. Further, high T_g polymers containing TPD unit were synthesized. Normally, triarylamine-based hole-transporting materials were usually synthesized by Ullmann coupling [10], Pd-catalyzed amination reaction [11], and Suzuki coupling reaction and showed high hole-mobility with high T_g [12–13].

Phosphorescent organic light emitting diodes (PhOLEDs) have been attracting much attention because of their high quantum efficiency. The phosphorescent molecules allow harvesting light not only from the singlet but also from the triplet excitons so that 100% internal quantum efficiency can be achieved theoretically [14]. High efficient

PhOLEDs have been reported by doping dopants from blue to red emissive phosphorescence in low molecular weight organic host. Phosphorescent polymer LEDs (PhPLEDs) are also investigated greatly with the advantage that they can be fabricated by simple solution processes, such as spin-casting, screen-printing, or inject-printing at room temperature [15]. However, PhPLEDs show lower quantum efficiency than PhOLEDs with low molecular weight organic host, because of the phase separation or aggregation of dopant. It was reported from devices of PVK doped with FIrpic that maximum external quantum efficiency of 1.3% and power efficiency of 0.8 lm/W were obtained [16]. So it is important to select the proper polymeric hosts and efficient phosphorescent dopants.

In this article, we prepared poly[*N*-(2-ethylhexyl) carbazole-*alt*-*N*-(4-aminophenyl) carbazole] (PECAC) and poly[*N*-(2-ethylhexyloxyphenyl) carbazole-*alt*-*N*-(4-aminophenyl) carbazole] (PEPCAC) by Pd-catalyzed polycondensation. From the view point of design concept, carbazole groups in these polymers were introduced both to main and side chains, as well as the polymer chains were linked by hole-transporting triarylamine groups. Photophysical properties of these polymers and the performance of PhPLEDs fabricated by doping a green, Ir(ppy)$_3$, or a red, IR-PIQCH, phosphorescent dopant in PECAC and PEPCAC hosts were investigated.

EXPERIMENTAL

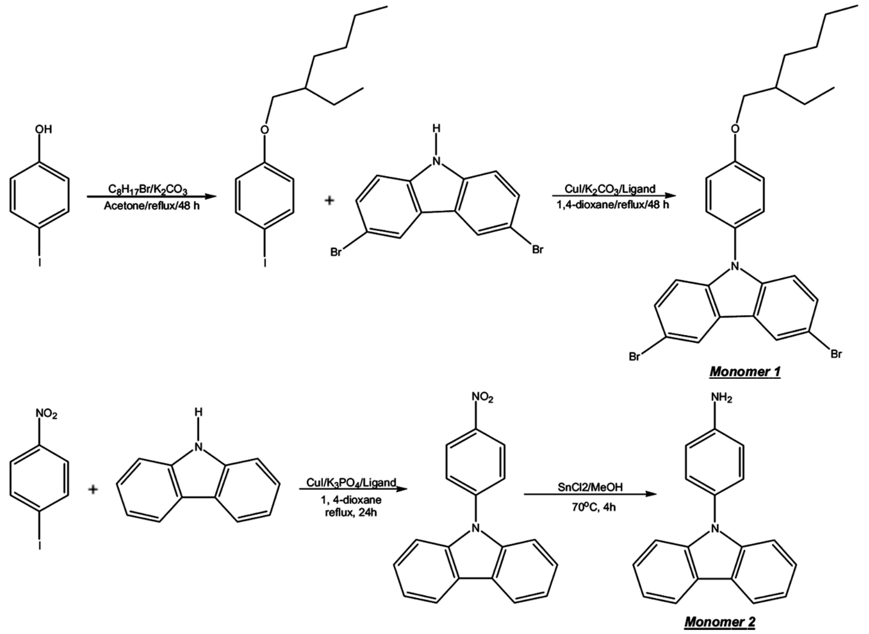
Materials

2-Ethylhexylbromide (97%), 3,6-dibromocarbazole (97%), 4-iodophenol (99%), copper (I) iodide (98%), trans-1,2-cyclohexane-diamine (ligand) (99%), tin (II) chloride ($SnCl_2$, 98%), sodium *tert*-butoxide (NaO-*t*-Bu, 97%), tris(dibenzylidene-acetone)dipalladium(0) ($Pd_2(dba)_3$), and tri-*tert*-butylphosphine ($P(t\text{-}Bu)_3$, 90%) were purchased from Aldrich Chemical Co. Reagent grade carbazole, 4-iodonitrobenzene, potassium carbonate anhydrous (K_2CO_3) and potassium phosphate tribasic (K_3PO_4) were obtained from Tokyo Kasei Co. and used as received. Solvents were reagent grades and purified prior to use. Bis(2-methyl-8-quinolinato)-4-phenlphenolate aluminum (BAlq), tri(8-hydroxyquinolate)aluminum (Alq_3) and lithium fluoride (LiF) were obtained from Tokyo Kasei Co. and used as received. Ir(ppy)$_3$ was purchased from Asung Co. IR-PIQCH was synthesized according to the procedure reported previously [17]. PEDOT was received from LG electronics Co. Aluminum (99.99%) was purchased from CERAC, USA. Indium-tin oxide (ITO) coated glass with a sheet resistance of $50\,\Omega/\square$ was obtained from Sinan SNP Co.

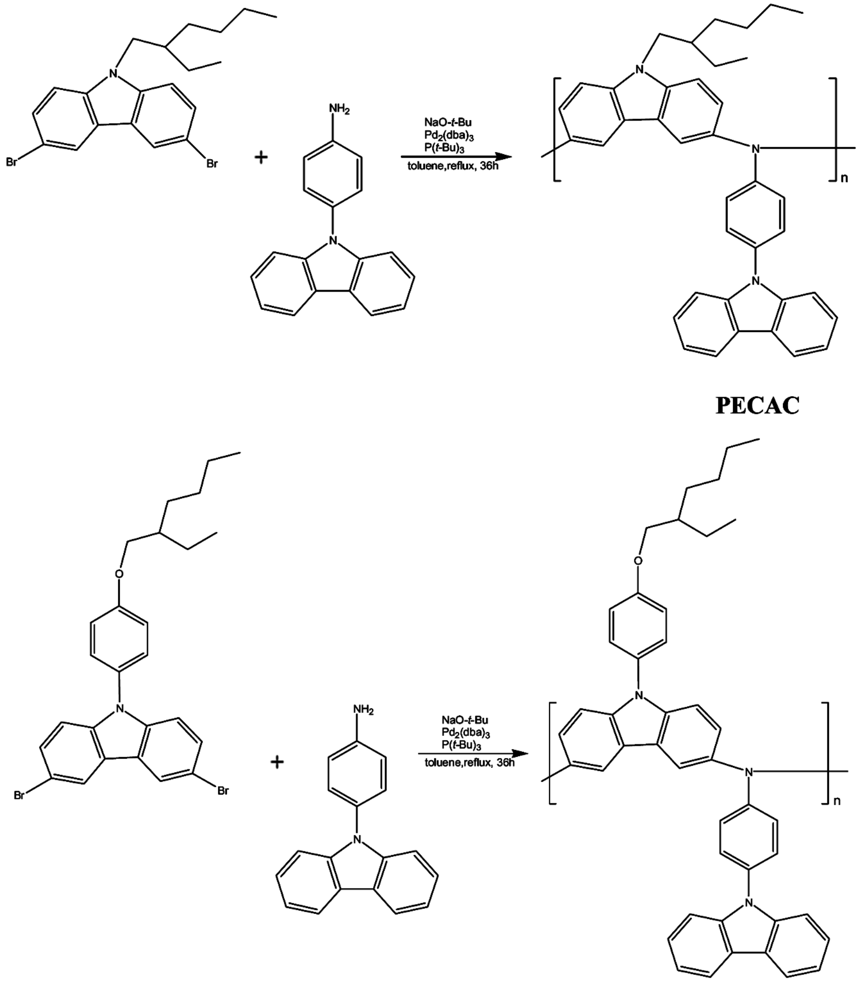
Synthesis of Polymers

PECAC was prepared according to the procedure reported previously [18]. Two monomers for PEPCAC were prepared as shown in Scheme 1. The polymerization was carried out according to reference [19], as shown in Scheme 2.

N-(2-Ethylhexyloxylphenyl)-3, 6-dibromocarbazole (Monomer 1). Monomer 1 was synthesized in two steps. First, 4-iodophenol (10 mmol) was dissolved in 100 ml of acetonitrile, then K_2CO_3 (20 mmol) was added. The mixture was stirred in N_2 atmosphere at 60°C. After 30 minutes, 2-ethylhexylbromide (12 mmol) was dissolved in 25 ml of acetonitrile and added to the flask dropwise. The reaction was kept for 48 hours. K_2CO_3 was filtered out, and the solvent was evaporated, then the crude product was washed with water and dried with $MgSO_4$. The pure white liquid product was obtained by silica column chromatography (eluent: hexane). (Yield: 77%). ^{1}H-NMR (300 MHz in $CDCl_3$): δ 7.33 (d, 2H, Ar–H), 6.79 (d, 2H, Ar–H), 3.79 (d, 2H, $-O-CH_2-$), 1.71 (m, 1H, –CH–), 1.29 $\sim$ 1.46 (m, 8H, $-CH_2-$), 0.91 (m, 6H, $-CH_3$); ^{13}C–NMR (300 MHz in $CDCl_3$): 158.4, 132.0, 116.2, 112.4, 70.6, 39.2, 30.4, 29.0, 23.8, 23.0, 14.1, 11.1.



SCHEME 1 Synthetic route to monomers.



SCHEME 2 Synthesis of the polymers.

In the second step, 4-iodo-(2-ethylhexyloxyl)benzene (7.5 mmole), 3,6-dibromocarbazole (9.0 mmol), K_3PO_4 (15.8 mmol), CuI (0.16 mmol) and trans-1, 2-cyclohexane-diamine (0.90 mmol) were added to 1,4-dioxane (100 ml) at room temperature. The reaction mixture was refluxed for 24 hours. After cooling the mixture to room temperature, the dissolved materials were filtered. 1,4-Dioxane was evaporated, and ethyl acetate was added. The mixture was washed with distilled water, and dried with $MgSO_4$. The final product was obtained through column chromatography (hexane: ethylacetate = 9:1v/v). White solid product was obtained (Yield: 90%). ^{1}H-NMR (300 MHz in $CDCl_3$):δ 8.18 (d, 2H, Ar–H), 7.47 (d, 2H, Ar–H), 7.36 (d, 2H, Ar–H), 7.16 (d, 2H, Ar–H), 7.08 (d, 2H, Ar–H), 3.94 (d, 2H, –O–CH_2–), 1.77 (m, 1H, –CH–), 1.26 ~ 1.57 (m, 8H, –CH_2–), 0.91 (m, 6H, –CH_3); ^{13}C–NMR (300 MHz in $CDCl_3$): 159.1, 140.3, 129.2, 128.9, 128.3, 123.6, 123.1, 115.7, 112.7, 111.5, 70.8, 39.3, 30.5, 29.1, 23.8, 23.1, 14.1, 11.1.

N-(4-aminophenyl)carbazole (Monomer 2). First, a mixture of 4-iodonitrobenzene (20 mmole), carbazole (15.24 mmmol) and K_3PO_4 (42 mmol) were added to 1,4-dioxane (100 ml) at room temperature. After stirring for 30 minutes, CuI (0.4 mmol) and trans-1,2-cyclohexanediamine (2.4 mmol) were added to the mixture. The reaction mixture was refluxed for 24 hours. After cooling the mixture to room temperature, the dissolved materials were filtered. 1,4-Dioxane was evaporated, and ethyl acetate was added. The mixture was washed with distilled water, and dried with $MgSO_4$. The final product was obtained through column chromatography (hexane: ethylacetate = 9:1 v/v) with the yield of 98%. ^{1}H-NMR (300 MHz in $CDCl_3$): δ 8.58 (d, 1H, Ar–H), 8.55 (d, 1H, Ar–H), 8.26 (m, 1H, AR–H), 8.23 (m, 1H, Ar–H), 8.01 (d, 1H, Ar–H), 7.98 (d, 1H, Ar–H), 7.60 (t, 1H, Ar–H), 7.58 (t, 1H, Ar–H), 7.51 ~ 7.45 (m, 2H, Ar–H), 7.39 ~ 7.33 (m, 2H, Ar–H); ^{13}C-NMR (300 MHz in $CDCl_3$): δ 144.35, 140.70, 131.85, 127.94, 127.34, 126.36, 124.82, 121.91, 121.33, 110.65; GC-MS m/z = 288 288 (m+).

In the second step, a mixture of *N*-(4-nitrophenyl)carbazole (5 mmol), $SnCl_2$ (30.4 mmol) and EtOH (15 ml) was heated at 70°C for 4 h. After the mixture was cooled to room temperature, 1 M NaOH solution was added until the mixture became alkaline. After extraction with ethyl acetate, the combined organic layers was washed with brine, dried with $MgSO_4$ and evaporated. The final compound was obtained with the yield of 98%. ^{1}H-NMR (300 MHz in $CDCl_3$):δ 8.15 (d, 1H, Ar–H), 8.12 (d, 1H, Ar–H), 7.42 ~ 7.22 (m, 8H, Ar–H), 6.87 (d, 1H, Ar–H), 6.84 (d, 1H, Ar–H), 3.81 (s, 2H, Ar–NH_2); ^{13}C–NMR (300 MHz in $CDCl_3$):δ 145.89, 141.45, 128.48, 125.70, 122.92, 120.16, 119.39, 115.89, 109.75; GC-MS m/z = 258 (m+).

Device Fabrication

ITO coated glass was cut into 5.0 cm × 5.0 cm, and electrode area was prepared by photo-etching technique. It was sequentially cleaned in an ultrasonic bath of acetone, methanol, and mixture of isopropyl alcohol and water (1:1 v:v) solution. PhPLEDs were fabricated with a configuration of ITO/PEDOT/Polymers + iridium complex (10 wt%)/BAlq (30 nm)/Alq_3 (20 nm)/LiF (1 nm)/Al (120 nm). The hole injecting PEDOT layer was spin-coated with a solution of 1 wt% in isopropyl alcohol onto ITO substrate in two steps at 1000 rpm for 10 s and 1000 rpm for 20 s, and then baked on hot plate at 80°C for 10 minutes. Subsequently, an emissive layer was spin-coated with a solution (1 wt%) of polymer and iridium complex in 1,1,2,2-tetrachloroethane, after filtration using MFS filter (0.45 µm pore size), at 1200 rpm for 10 s and dried at 100°C for 10 minutes. The emissive thin films prepared had uniform surface with thickness of about 60–70 nm. Finally, hole-blocking BAlq, electron transporting Alq_3, and LiF/Al were successively deposited under pressure $< 10^{-6}$ torr. The active area of the PhPLEDs was 2 mm × 2 mm.

Characterization

NMR spectra were recorded on a Varian Unity Plus 300 with solvent of $CDCl_3$. GC Mass (GC-MS) spectra were recorded on a QP 5050 mass spectrometer. Weight and number average molecular weight were measured by gel permeation chromatograph (GPC) equipped with Styragel HR 5E column using THF as an eluent against polystyrene standards at room temperature. Thermogravimetric analysis (TGA) was performed by using a Seiko EXSTAR 6000 TG/DTA 6300 thermal analyzer at a heating of 20°C/min under N_2 atmosphere. UV-visible absorption spectra were taken with Shimadzu UV-2100. The photoluminescence (PL) spectra excited by He-Cd laser at 325 nm were monitored by Optical Multichannel Analyzer (Laser Photonics, OMA system). The ionization potential (IP) was measured by a photoelectron spectroscopy (Riken Keiki AC-2). Electroluminescence (EL) spectra were measured by using Spectroscan PR 704 (Photoresearch Inc). Current-luminescence-voltage (*I-V-L*) profiles of devices were obtained by using dc power supply connected Model 8092 A Digital Multimeter and luminance meter (Minolta LS-100).

RESULTS AND DISCUSSION

As outlined in Scheme 1, alkylation of 4-iodophenol with 2-ethylhexylbromide for the synthesis of monomer 1 was easily obtained, which could impart higher solubility of synthesized polymers in common organic solvents. For the synthesis of monomer 2, *N*-(4-nitrophenyl)carbazole was first synthesized via Ullmann coupling of

TABLE 1 Physical Property of Polymers

Polymer	Yield (%)	PDI	M_w	M_n	T_d (°C)[a]	Weight loss at 800°C
PECAC	80	1.54	5700	3700	465	36.8%
PEPCAC	81	1.70	7800	4600	464	27.8%

[a] Temperature resulting in 5% weight loss based on initial weight.

4-iodonitrobenzene and carbazole with a high yield. Then, the reduction of *N*-(4-nitrophenyl)-carbazole with $SnCl_2$ catalyst in methanol gave monomer 2. Synthesized copolymers are designed to have blue emitting carbazole moieties both in main chains and side chains, as well as the molecular chains are liked by triarylamine units, as depicted in Scheme 2. Results on physical properties of the synthesized polymers such as molecular weights, molecular weight distributions and decomposition temperature (T_d) were summarized in Table 1.

Maximum UV-Vis absorption and PL emission peaks of PECAC and PEPCAC were measured both in solution and solid state, as can be seen in Figure 1. In their dilute $CHCl_3$ solution, maximum absorption peaks of PECAC and PEPCAC were observed at 312 nm and 320 nm; in their film state, they were observed at 336 nm and 346 nm, respectively. With excitation at their $\lambda_{max,UV}$ in either solution or solid state,

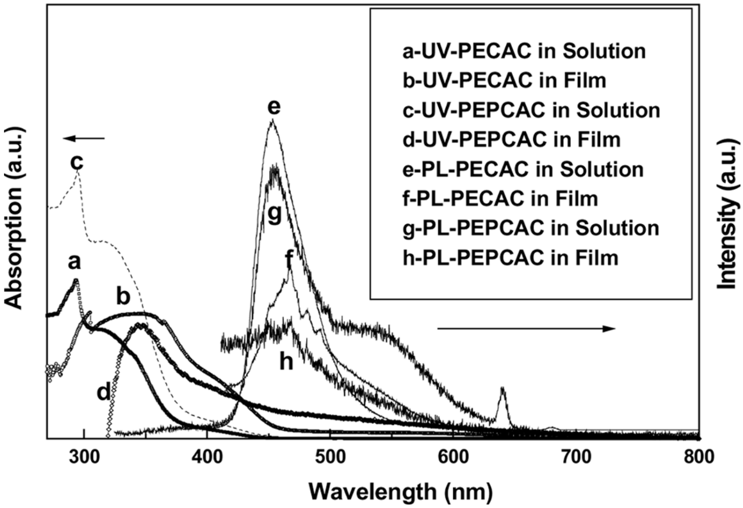


FIGURE 1 UV-Vis absorption and PL emission spectra of polymers.

PL emission of two polymers was in the range of blue emission. In dilute $CHCl_3$ solution, maximum PL emission peak ($\lambda_{max,PL}$) of PECAC and PEPCAC was observed at 453 nm and 455 nm; in their film state, they were observed at 466 nm and 467 nm. Compared with PL spectra in solution, red shift of PL emission spectra in the solid state of PECAC and PEPCAC was observed, indicating an aggregation of polymer chains in the solid state.

The optical and electrochemical properties of the polymers are listed in Table 2. The band gap energies of the synthesized polymers were calculated from UV-Vis absorption and PL emission spectra. The band gap energies of PECAC and PEPCAC were measured to be 2.97 eV and 3.25 eV, respectively. The wide band gap energy of polymers is required to be qualified as good host materials in an emitting layer. Structural characteristics of materials play a crucial role in adjusting the band gap, and wider band gap for PEPCAC means less conjugation in the molecular chains, which might be originated from the fact that substitution of 2-ethylhexyloxylphenyl groups on nitrogen atom increased the dihedral angle between the aryl unit and carbazole to decrease the degree of planarity and conjugation of the system [20]. The polymers showed HOMO levels of −5.01 eV and −5.17 eV, higher than those of low molecular weight analogs such as TPD (−5.5 eV) or NPB (−5.7 eV) [19]. Such energy levels of the polymers may provide a closer match to the work function of ITO anode (4.7 eV) or PEDOT layer (5.0 eV) when they are used as hole transporting materials in PLEDs.

In order to investigate the electroluminescent performance with the synthesized polymers as host matrix, PhPLEDs were fabricated with the configuration of ITO/PEDOT/Polymers + iridium complex (10 wt%)/BAlq (30 nm)/Alq_3 (20 nm)/LiF (1 nm)/Al (120 nm). The energy

TABLE 2 Optical and Electrochemical Properties of the Polymers

	Solution		Films				
Polymer	$\lambda_{max,UV}$ (nm)	$\lambda_{max,PL}$ (nm)	$\lambda_{max,UV}$ (nm)	$\lambda_{max,PL}$ (nm)	Band gap (eV)[a]	HOMO (eV)[b]	LUMO (eV)[c]
PECAC	312	453	336	466	2.97	−5.01	−2.04
PEPCAC	320	455	346	467	3.25	−5.17	−1.92

[a] Calculated from the crosspoint of UV-Vis and PL spectrum.
[b] Measured by a RIKENKeiki AC-2.
[c] Estimated from the HOMO and band gap.

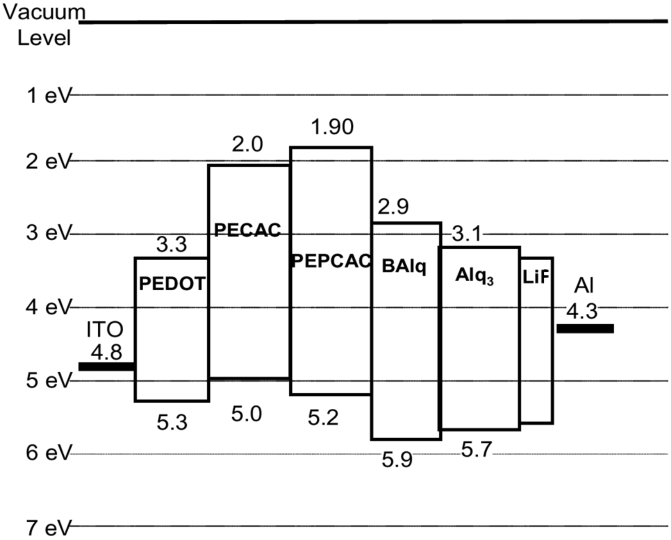


FIGURE 2 Schematic view of energy levels of devices.

diagram of the devices is shown in Figure 2. Figures 3 (a) and (b) showed the current–voltage (*I–V*) and luminescence–voltage (*L–V*) curves of PhPLEDs, respectively. For devices doped with Ir(ppy)$_3$, the maximum luminescence and driving voltage were 453 cd/cm^2 and 8 V for PECAC, and 77 cd/cm^2 and 14 V for PEPCAC. In the case of IR-PIQCH doped PhPLEDs, the maximum luminescence and driving voltage were 576 cd/cm^2 and 6 V for PECAC, and 59 cd/cm^2 and 12 V for PEPCAC, respectively. It appeared that, due to matching of HOMO energy levels, the devices with PECAC as a host matrix in PhPLEDs showed lower turn-on voltage.

Figure 4 presents electroluminescencent spectra of PhPLEDs. The maximum emission wavelengths of Ir(ppy)$_3$ doped devices were measured at 546 nm. However, emission by the triplet state of Ir(ppy)$_3$ has been reported to be 515 nm [21]. This difference in the maximum emission wavelength of Ir(ppy)$_3$ and Ir(ppy)$_3$-doped devices accounts for the inefficient band gap energy of the host materials to the guest Ir(ppy)$_3$ dopant, especially lower HOMO level of host polymers (-5.0 for PECAC and -5.2 eV for PEPCAC) than -5.5 eV of Ir(ppy)$_3$. In IR-PIQCH doped devices, on the other hand, the effective energy transfer from host polymers to IR-PIQCH red dopant was observed, showing that the maximum EL emission at 620 nm was matched to IR-PIQCH triplet emission.

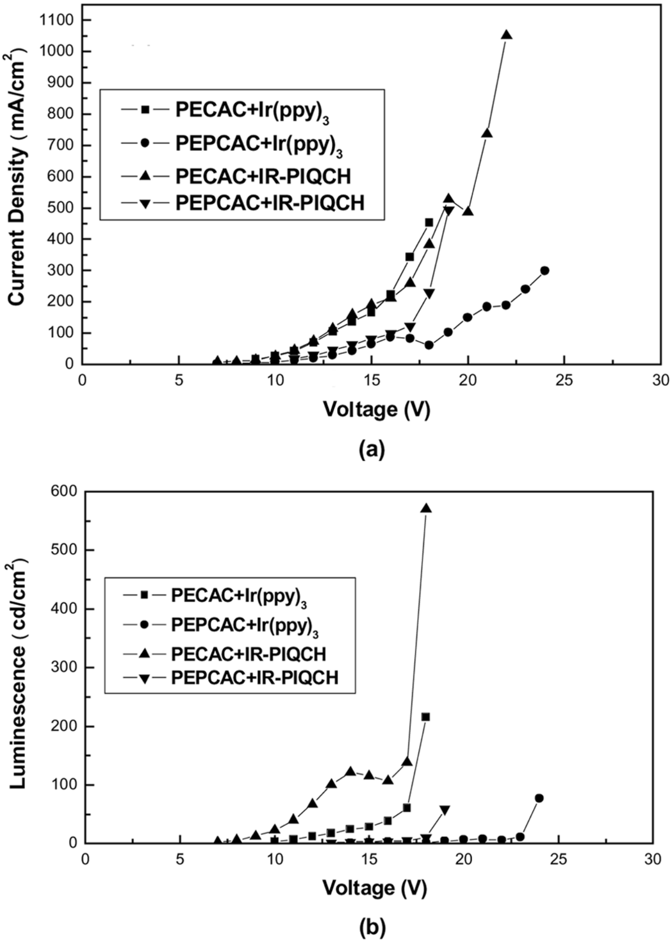


FIGURE 3 Current–voltage curves (a) and luminescence–voltage curves (b) of PhPLEDs.

CONCLUSIONS

Novel polymers such as PECAC and PEPCAC were successfully synthesized and utilized as host polymers in PhPLEDs. PhPLEDs were fabricated with the configuration of ITO/PEDOT/Polymers + iridium complex (10 wt%)/BAlq (30 nm)/Alq$_3$ (20 nm)/LiF (1 nm)/Al (120 nm). Due to insufficient band gap energy of the synthesized polymers to Ir(ppy)$_3$ green dopant, Ir(ppy)$_3$ doped PhPLEDs exhibited red shift and wide range of EL emission. On the other hand, red electrophosphorescence was observed with emission peaks

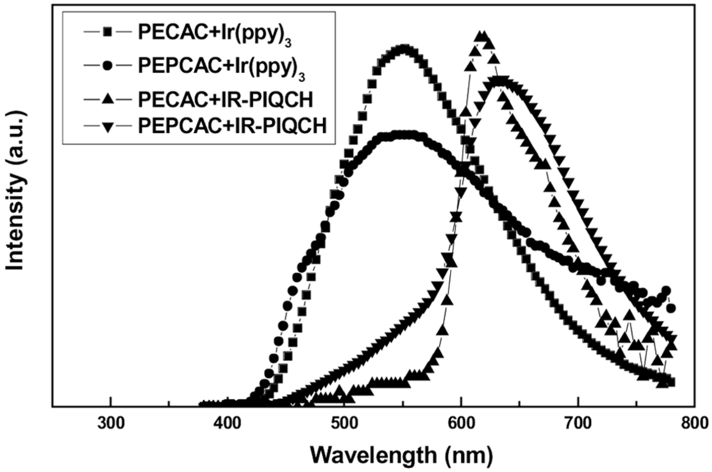


FIGURE 4 Electroluminescent spectra of PhPLEDs.

at approximately 620 nm in IR-PIQCH doped devices. Efficient energy transfer from host polymers, PECAC or PEPCAC, to IR-PIQCH red dopant confirmed that the synthesized polymers could be suitable host materials for red dopants. PhPLED doped with IR-PIQCH in PECAC as an emitting layer exhibited luminescence of 576 cd/cm^2 and driving voltage of 6 V. Further optimization of device efficiencies by utilizing PECAC host and IR-PIQCH red dopant could be a continuing study.

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