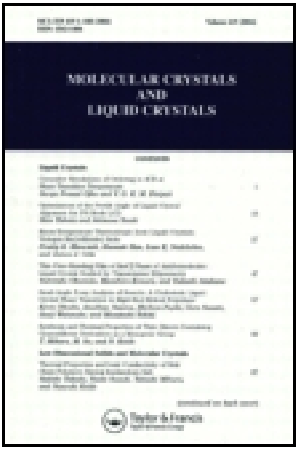

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Electroluminescent Properties of Heterocyclic Conjugated Random Copolymers Containing Phenothiazine and Carbazole Moieties

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Electroluminescent properties of polymer light-emitting diode (PLED)s fabricated from heterocyclic conjugated random copolymers, poly(PZV-ran-CZV), containing (N-2-ethylhexyl)-3,6-phenothiazinylene vinylene (PZV), (N-2-ethylhexyl)-3,6-carbazolylene vinylene (CZV) and phenylene vinylene (PV) unit as a light emitting layer were investigated. PLEDs made with corresponding alternating copolymers, poly(PZV-PV) and poly(CZV-PV), as a light emitting layer were also studied and

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exhibited EL maximum at 570 nm and 500 nm, respectively. Single-layered PLEDs made with poly(PZV-ran-CZV) copolymers, however, showed EL maxima shifted to longer wavelength. Double-layered PLED made with poly(PZV-ran-CZV) copolymer as a light emitting layer and Alq_3 *as an electron transporting layer exhibited enhanced EL emission compared to that of single-layered PLED.*

Keywords: carbazolylene vinylene; heterocyclic conjugated copolymer; Horner-Emmons reaction; phenothiazinylene vinylene; polymer light-emitting diode

INTRODUCTION

Research on conjugated polymers has been motivated by their promising application in polymer light emitting diodes (PLEDs), since R. H. Friend *et al*. reported PLED made from poly(*p*-phenylenevinylene) (PPV). To date, it has been generally accepted that the electronic properties and chemical nanostructures of conjugated polymers have played a key role on the emission color, quantum efficiency, and luminance/voltage profile of PLEDs [1,2]. In a PLED, the conjugated polymer sandwiched between two electrodes must act as a hole transport, electron transport, and emission layer at a same time for efficient operation of PLEDs. However, a few types of conjugated polymers have been satisfied with the requirement mentioned above. Difficulties associated with fabricating high efficient PLEDs arise principally due to unbalanced injection rate of holes and electrons from both opposite electrodes, resulting in low recombination efficiency of injected holes and electrons in polymer emissive layer. Thus, one approach to further improve electroluminescent properties such as electroluminescent efficiencies, fine-tuning of balanced rate of carrier (electron and hole) injection/transporting and recombination efficiency in PLEDs can be to construct multi-layered PLEDs consisting of hole injection layer (HIL), hole transporting layer (HTL), emitting layer (EML), etc [3–6]. Aromatic amines such as *N,N′*-diphenyl-*N,N′*-bis (3-methylphenyl)-(1,1′-biphenyl)-4,4′-diamine and carbazole have been reported to have hole transporting capability [7,8]. On the other hand, organic compounds such as 2-(4-biphenylyl)-5-(4′-*tert*-butylphenyl)-1,3,4-oxadiazole and anthracene derivatives were found to have electron transporting capability [9–11].

In this work, we describe electroluminescent properties of conjugated random copolymers with hole transporting phenothiazine and carbazole moieties in the main chain. Single- and multi-layered PLEDs were fabricated by spin-coating the conjugated random copolymer as a light-emitting layer on ITO glass and their electroluminescent properties were studied.

EXPERIMENTAL

Monomer Synthesis

N-(2-ethylhexyl)-3,6-diformylphenothiazine (DFPZ) [12]

Phosphoryl chloride (122.7 g, 0.8 mol) was added dropwise over a period of 30 min to a mixture of *N*-(2-ethylhexyl)phenothiazine (EHPZ) (12.36 g, 0.04 mol) and *N*,*N*-dimethylformamide (DMF) (58.5 g, 0.8 mol) in 1,2-dichloroethane (15 ml) in an ice bath. The solution was refluxed for 72 h. DFPZ was obtained as liquid in 69% yield. ^{1}H-NMR ($CDCl_3$), δ: 9.73 (*m*, 2H, aldehydic hydrogen), 6.88, 7.56 (*m*, 6H, Ar H), 3.73 (*t*, 2H, $>N{-}CH_2{-}$), 1.17–1.85 (*m*, 9H, $-CH_2-$, $>CH-$), 0.76–0.82 (*m*, 6H, $-CH_3$).

N-(2-ethylhexyl)-3,6-diformylcarbazole (DFCZ) [13]

Phosphoryl chloride (122.7 g, 0.8 mol) was added dropwise over a period of 30 min to a mixture of *N*-(2-ethylhexyl)carbazole (EHCZ) (11.18 g, 0.04 mol) and DMF (58.5 g, 0.8 mol) in 1,2-dichloroethane (15 ml) in an ice bath. The solution was refluxed for 72 h. DFCZ was obtained as viscous liquid in 65 % yield. ^{1}H-NMR ($CDCl_3$), δ : 10.11 (2H, aldehydic hydrogen), 7.49–8.62 (6H, Ar H), 4.23 (2H, $>N{-}CH_2{-}$), 2.05 (1H, $-CH_2-$, $>CH-$), 1.26–1.38 (8H, $-CH_2-$), 0.84–0.97 (6H, $-CH_3$).

1,4-Bis(diethoxyphosphinylmethyl)benzene (PHBZ) [12]

PHBZ was synthesized by reacting α,α'-dichloro-*p*-xylene (33 g, 0.2 mol) with triethylphosphite (33 g, 0.2 mol) at 120°C for 12 hr. The unreacted triethylphosphite was removed under reduced pressure. The product was obtained as colorless liquid and yield was 93%. ^{1}H-NMR ($CDCl_3$), δ: 7.33(s, 4H, Ar H), 4.00(s, 8H, $P{-}OCH_2{-}$), 3.13(d, 4H, $Ar{-}CH_2{-}P$), 1.25(t, 12H, $-CH_3$), IR (KBr), cm^{-1}: 3021(Ar CH; weak signal), 2952(aliphatic CH), 1515(Ar C=C), 1243($P{=}O$).

Polymerization

Poly(N-2-ethylhexyl-3,6-phenothiazinylene vinylene-alt-1,4-phenylene vinylene) [poly(PZV-PV)] [12]

To a solution of DFPZ (1.85 g, 0.005 mol) and PHBZ (1.90 g, 0.005 mol) in anhydrous tetrahydrofuran (THF) (40 ml) was added dropwise a solution of potassium *tert*-butoxide (^{t}BuOK) (4.73 g of purity 95%, 0.04 mol) in dry THF (20 ml) at room temperature under nitrogen atmosphere. After the addition of ^{t}BuOK the solution was stirred for 7 hr to give a polymer product as precipitate. The resulting mixture was concentrated under reduced pressure, and the residue was washed with methanol and water. After filtration and vacuum drying the poly(PZV-PV) was obtained as orange color powder. IR

(KBr), cm^{-1}: 3019(Ar CH), 2923(aliphatic CH), 2853(>N$-$CH$_2$$-$), 1579, 1508(Ar C=C), 956(trans vinylene).

Poly(N-2-ethylhexyl-3,6-carbazolylene vinylene-alt-1,4-phenylene vinylene) [poly(CZV-PV)]

Poly(CZV-PV) were synthesized by a synthetic method similar to that of poly(PZV-PV) using DFCZ and PHBZ. IR (KBr), cm^{-1}: 3007(Ar CH), 2915(aliphatic CH), 2853(>N$-$CH$_2$$-$), 1561, 1470(Ar C=C), 960(trans vinylene).

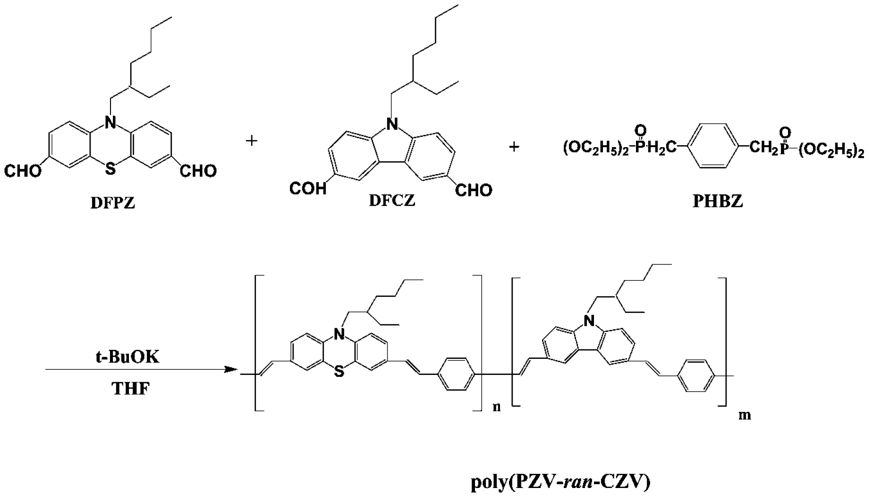
Poly[(N-2-ethylhexyl-3,6-phenothiazinylene vinylene-alt-1,4-phenylene vinylene)-ran-(N-2-ethylhexyl-3,6-carbazolylene vinylene-alt-1,4-phenylene vinylene)] [poly(PZV-ran-CZV)]

Heterocyclic copolymers, poly(PZV-*ran*-CZV)s containing *N*-2-ethylhexyl-3,6-phenothiazinylene vinylene(PZV), *N*-2-ethylhexyl-3,6-carbazolylene vinylene(CZV), and phenylene vinylene(PV) unit in different ratio were synthesized from DFPZ, DFCZ, and PHBZ with different feed ratio of comonomers. A brief synthetic scheme of the heterocyclic conjugated copolymers is shown in Scheme 1.

Fabrication of PLEDs

Single-layered PLEDs

Single-layered PLEDs were fabricated by using these conjugated copolymers as emitting materials. Indium-tin oxide (ITO) coated glass



SCHEME 1 Synthetic route to heterocyclic conjugated copolymers.

with a sheet resistance of 30 Ω/□ and a thickness of 1.08 mm was cut into 2.0 × 2.0 cm^2, and electrode area was prepared by photoetching technique. It was sequentially cleaned in an ultrasonic bath of acetone, methanol, and mixture of isopropyl alcohol and water (1:1 by vol.) solution. Filtered solutions of conjugated polymer in 1,1,2,2-tetrachloroethane were cast on patterned ITO glass by spin coating and the solvent was evaporated to give emitting layers. Thin Al film as cathode was vacuum deposited using Thermal Evaporator System (KVT-420, Korea Vacuum Co.) on top of the emitting layer by thermal method (below 2.0×10^{-5} torr) at a rate of 20 Å/sec to a thickness of about 1800 Å to give single-layered PLEDs (ITO/conjugated polymer/Al).

Multi-layered PLEDs

Double-layered PLEDs were fabricated with Alq_3 as an additional electron transporting layer between the emissive layer and the metal cathode. For ITO/conjugated polymer/Alq_3/Al double-layered PLED, after spin-coating and drying of polymeric emissive layer, vacuum deposition of Alq_3 was performed on top of the emissive layer at a rate of 3–5 Å/sec until 250 Å thickness, followed by vacuum deposition of Al metal with a thickness of 1800 Å to give the double-layered PLED. The area of Al electrode deposited was approximately 0.4 cm^2.

With insertion of PEDOT:PSS[poly(3,4-ethylenedioxythiophene):poly(styrene sulfonic acid)], which is well-known as both hole injection and transporting layer, double-layered PLED(ITO/PEDOT:PSS/conjugated polymer/Al)s were made. Triple-layered PLED(ITO/PEDOT:PSS/conjugated polymer/Alq_3/Al)s were also fabricated. After spin-coating and drying of PEDOT:PSS followed by emissive polymer layer, Alq_3 layer was vacuum deposited on top of copolymer thin film layer.

Measurements

The ^{1}H-NMR and FT-IR spectra of synthesized compounds were taken on a Varian Unity Plus 300 and Jasco FT/IR-620 spectrometer, respectively. Molecular weight was measured by Waters gel permeation chromatograph (GPC) equipped with a 410 differential refractometer, 510 HPLC pump, and Styragel HR 5E column using THF as an eluent against polystyrene standards at room temperature. Thermal analysis was performed with differential scanning calorimeter (DSC) (TA Instruments, TA4000/Auto DSC 2910 system) and thermogravimetric analyzer (TGA) (Seiko instruments Inc., TG/DTA 320) with heating rate of 20°C/min under nitrogen atmosphere. UV-visible absorption spectra of the copolymers were obtained by Shimadzu UV-2100. Electroluminescence (EL) spectra and color coordinates were measured by using

Spectroscan PR 704 (Photoresearch Inc.). Luminance with voltage was obtained by using a dc power supply connected with Model 8092 A Digital Multimeter (Hyun Chang Product Co. Ltd) and luminance meter (Minolta LS-100) equipped with close-up lens (No. 110, Φ 40.5 mm) at room temperature, respectively.

RESULTS AND DISCUSSION

Synthesis and Characterizations

Heterocyclic random copolymers, poly(PZV-*ran*-CZV)20, poly(PZV-*ran*-CZV)30 and poly(PZV-*ran*-CZV)40, containing *N*-2-ethylhexyl-3,6-phenothiazinylene vinylene (PZV), *N*-2-ethylhexyl-3,6-carbazolylene vinylene (CZV), and phenylene vinylene (PV) unit in different ratio were synthesized by Honer-Emmons reaction of DFPZ, DFCZ and PHBZ with different feed ratio of comonomers, i.e., DFPZ/DFCZ/PHBZ = 15/35/50, 25/25/50, 35/15/50, respectively. From the elemental analysis, we found that the content of PZV, CZV, and PV repeating unit within poly(PZV-*ran*-CZV)20 had 18, 32, and 50 mole%, respectively. With similarity, poly(PZV-*ran*-CZV)30 had 29 mol% of PZV, 21 mol% of CZV and 50 mol% of PV in the random copolymer, and poly(PZV-*ran*-CZV)40 had 38, 12, and 50 mol%, respectively. Alternating copolymers, poly(PZV-PV) and poly(CZV-PV), were also synthesized for comparison.

Synthesized monomers and polymers were characterized with ^{1}H-NMR and FT-IR, and their resulting spectral data were listed in experimental section. Other characterizations such as molecular weight and thermal decomposition temperature were also conducted. The molecular weights of synthesized conjugated polymers ranged from 14,000 to 18,000 g/mol. TGA analysis indicated that synthesized polymers were stable up to about 360°C under nitrogen. The good solubility in common organic solvents at room temperature is one of the important properties of the synthesized copolymers for the fabrication of PLEDs by the simple spin casting method. The observed good solubility of these poly(CZV-PV), poly(PZV-PV), and poly(PZV-*ran*-CZV) copolymers in common organic solvents seemed to be originated from the presence of heterocyclic aromatic ring in the main chain and also due to the relatively long ethylhexyl groups attached both on the phenothiazine and carbazole moieties.

Electro-Optic Properties of Single-Layered PLEDs

UV absorption spectra of synthesized copolymers, poly(CZV-PV), poly(PZV-*ran*-CZV) and poly(PZV-PV), are shown in Figure 1. The

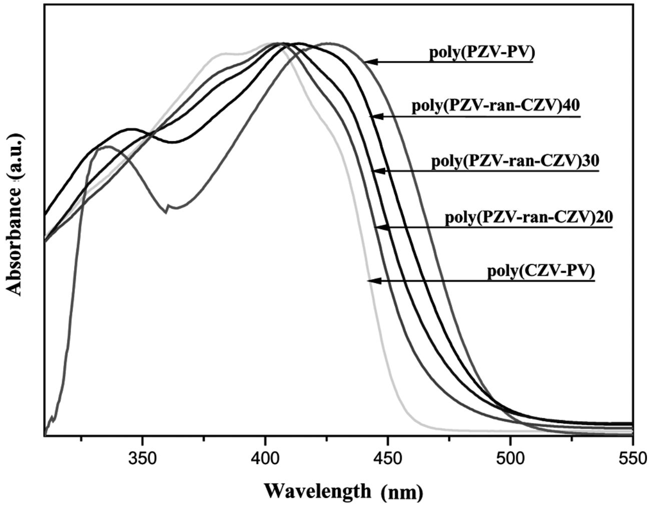


FIGURE 1 UV-visible absorption spectra of conjugated copolymers.

absorption maxima ($\lambda_{\mathrm{max,UV}}$) of the copolymer solution were varied from 401 to 425 nm, and the band gap energy estimated from the absorption edge ($\lambda_{\mathrm{edg,UV}}$) decreased with increasing the amount of the PZV unit.

PLEDs fabricated with poly(CZV-PV) and poly(PZV-PV) as light emitting layers exhibited EL maxima at 500 and 570 nm, respectively, as shown in Figure 2. Figure 3 shows emission spectra from PLEDs made with random copolymers, poly(PZV-*ran*-CZV)20, 30, and 40. PLEDs made with poly(PZV-*ran*-CZV) copolymers showed EL maxima shifted to longer wavelength than 570 nm of ITO/poly(PZV-PV)/Al PLED even though poly(PZV-*ran*-CZV) had lower PZV content than that of poly(PZV-PV). On the contrary, PLEDs fabricated with blends(1:1 by weight) of poly(PZV-CV) and poly(CZV-CV) alternating copolymers as light emitting layers exhibited EL maxima at 564 nm.

The electro-optical data such as $\lambda_{\mathrm{max,UV}}$, $\lambda_{\mathrm{edg,UV}}$, $\lambda_{\mathrm{max,EL}}$, Stoke's shift, and color coordinates measured from UV-visible absorption and EL spectrum are summarized in Table 1. Random copolymers, poly(PZV-*ran*-CZV)20, 30, and 40, showed larger Stoke's shift than that of alternating copolymers, poly(CZV-PV) and poly(PZV-PV). This large Stoke's shift in random copolymers which have various chain segments may be due to the larger possibility of excimer formation than that of alternating copolymers. Of the poly(PZV-*ran*-CZV) copolymers used, the PLED fabricated with poly(PZV-*ran*-CZV)30 copolymer exhibited the highest Stoke's shift of 190 nm. It has been well known

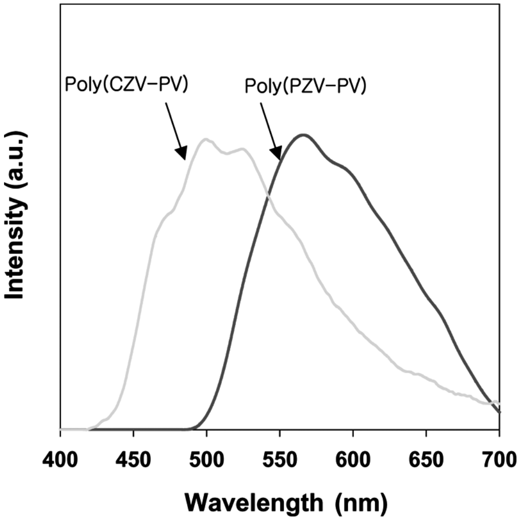


FIGURE 2 Emission spectra of PLEDs made with alternating copolymers.

that the emission at excimer formation is observed in longer wavelength than normal emission peak [14,15]. Therefore, large shift of EL emission by poly(PZV-*ran*-CZV)30 may be due to the enhanced excimer formation at this copolymer composition compared to alternating copolymers and other random polymers with different copolymer composition.

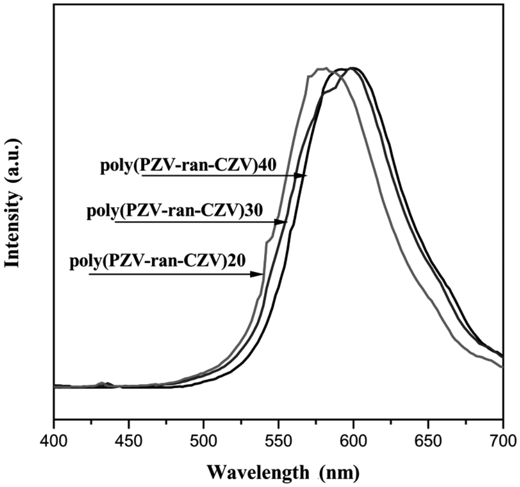


FIGURE 3 Emission spectra of PLEDs made with random copolymers.

TABLE 1 Electro-optical Properties of Heterocyclic Conjugated Copolymers

Conjugated copolymers	Content of PZV (mol%)	$\lambda_{\mathrm{max,UV}}$ (nm/eV)	$\lambda_{\mathrm{edg,UV}}$[2] (nm/eV)	$\lambda_{\mathrm{max,EL}}$ (nm/eV)	Stoke's Shift[3] (nm)	Color coordinates (x,y)	Emission color
Poly(CZV-PV)	0	401/3.08	468/2.65	500/2.48	99	0.28, 0.44	Green
Poly(PZV-*ran*-CZV)20	18[1]	405/3.06	488/2.54	582/2.13	177	0.51, 0.48	Yellow
Poly(PZV-*ran*-CZV)30	29[1]	408/3.04	498/2.49	598/2.07	190	0.52, 0.47	Yellowish orange
Poly(PZV-*ran*-CZV)40	38[1]	414/3.00	503/2.47	600/2.07	186	0.53, 0.47	Yellowish orange
Poly(PZV-PV)	50	425/2.92	506/2.45	566/2.19	141	0.48, 0.51	Yellow

[1] Calculated from elemental analysis, [2] onset point of UV-visible absorption spectrum, and [3] $\lambda_{\mathrm{max,EL}} - \lambda_{\mathrm{max,UV}}$.

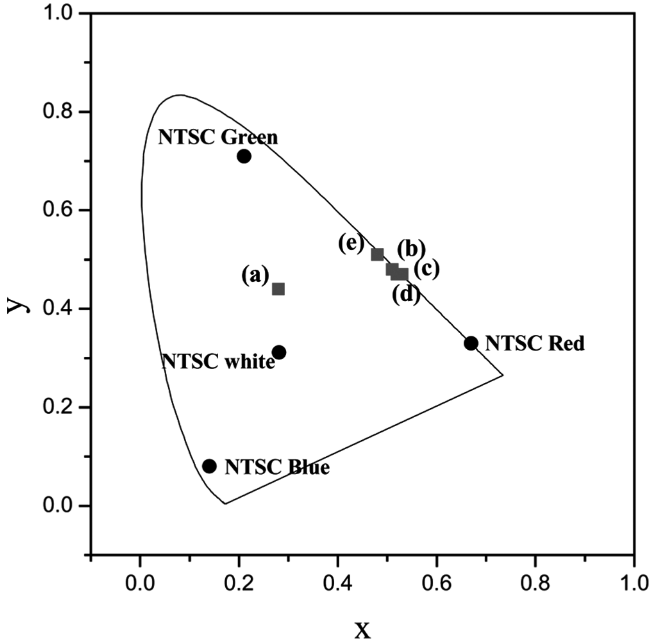


FIGURE 4 Color coordinates of (a) poly(CZV-PV), (b) poly(PZV-*ran*-CZV)20, (c) poly(PZV-*ran*-CZV)30, (d) poly(PZV-*ran*-CZV)40, and (e) poly(PZV-PV) in CIE 1931 chromaticity diagram.

Color coordinates of single layer PLED (ITO/conjugated polymer/Al)s were compared with those of standard red, green, blue, and white color established by NTSC (national television system committee) [16] in Figure 4. The emission color could be determined from these color coordinates as measured by Spectroscan PR-704 (Photoresearch Inc.) and Kelly's map [17] in CIE (Commission Internationale de l'Eclairage) 1931 chromaticity diagram [18]. The EL light from poly(CZV-PV), poly(PZV-*ran*-CZV)20, 30, 40, and poly(PZV-PV) showed green, yellow, yellowish orange, yellowish orange, and yellow, respectively. Three random copolymers, poly(PZV-*ran*-CZV)20, 30, and 40, showed similar value of CIE coordinates and their EL emission in yellowish orange region. Emission maxima($\lambda_{\mathrm{max,EL}}$) of these copolymers showed small amount of bathochromic shift with increasing PZV unit in the main chain.

Emission Properties of Double-Layered PLEDs

We also fabricated multi-layered PLEDs using poly(PZV-*ran*-CZV)30 as an emitting layer with the insertion of PEDOT:PSS as a hole injection/transporting layer, Alq_3 as an electron transporting layer, or both of PEDOT:PSS and Alq_3 layers. The double-layered PLED, ITO/poly(PZV-*ran*-CZV)30/Alq_3/Al, exhibited enhanced EL emission[220 nit at 20 V] compared to those of ITO/poly(PZV-*ran*-CZV)30/Al[50 nit at 20 V], double-layered ITO/PEDOT:PSS/poly(PZV-*ran*-CZV)30/Al[40 nit at 20 V], and triple-layered ITO/PEDOT:PSS/poly(PZV-*ran*-CZV)30/Alq_3/Al[70 nit at 20 V]. From the comparison of double-layered ITO/poly(PZV-*ran*-CZV)30/Alq_3/Al and single-layered ITO/poly(PZV-*ran*-CZV)30/Al, the enhanced luminance by insertion of Alq_3 as an electron injection/transporting layer in the double-layered PLED was attributed to the balanced rate of electron/hole injection and effective charge confinement in the interface of poly(PZV-*ran*-CZV)30/$Alq_{3.}$

Results on decreased luminance in double-layered ITO/PEDOT:PSS/poly(PZV-*ran*-CZV)30/Al PLED indicated that inserting PEDOT:PSS as a hole injection/transporting layer adversely disrupted the balance of electron and hole injection in the PLED. Therefore, it can be concluded that heterocyclic conjugated copolymers synthesized have hole transporting property under the induced electric field. This may be due to the presence of phenothiazine and carbazole moieties in these polymers since the aromatic amines were well known to be a hole transport agent in the EL devices.

CONCLUSIONS

Horner-Emmons reaction was found to be a useful synthetic method for the preparation of EL copolymers with designed microstructure. Of the synthesized copolymers, PLED fabricated with poly(PZV-*ran*-CZV)30 copolymer exhibited larger Stoke's shift compared to those of other PLEDs. This might be due to the enhanced excimer formation of poly(PZV-*ran*-CZV) copolymer at 29 mol % of PZV repeating units in the polymer chain. Double-layered ITO/poly(PZV-*ran*-CZV)30/Alq_3/Al PLED exhibited better improved EL emission than those of other multi-layered PLEDs. These results were attributed to the balanced rate of electron/hole injection and effective charge confinement in the interface of poly(PZV-*ran*-CZV)30/Alq_3. It could be concluded, therefore, that synthesized heterocyclic copolymers have good hole transporting and electron blocking capability.

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