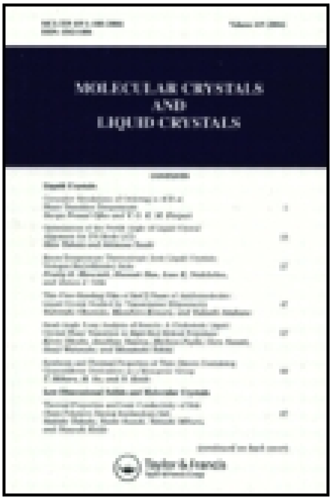

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Synthesis and Properties of Polymers Containing Charge Transport Pendant Group

Hui Wang [a], Jeong-Tak Ryu [a], Young Hwan Shin [b] & Younghwan Kwon [b]

[a] College of Information and Communication Engineering, Daegu University, Gyeongsan, Gyeongbuk, Korea

[b] Department of Chemical Engineering, Daegu University, Gyeongsan, Gyeongbuk, Korea

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Synthesis and Properties of Polymers Containing Charge Transport Pendant Group

Hui Wang[1], Jeong-Tak Ryu[1], Young Hwan Shin[2], and Younghwan Kwon[2]
[1]College of Information and Communication Engineering, Daegu University, Gyeongsan, Gyeongbuk, Korea
[2]Department of Chemical Engineering, Daegu University, Gyeongsan, Gyeongbuk, Korea

*Non-conjugated polymers containing charge transporting pendant groups were synthesized and characterized. Poly(*N*-(4-vinylphenyl)-carbazole) (PPCZ) with a hole transporting carbazole group and poly(2-methylphenyl-5-(4-vinylphenyl)-1,3,4-oxadiazole) (PPOXD) with an electron transporting 1,3,4-oxadiazole group were synthesized by free radical polymerization of corresponding monomers, respectively, *N*-(4-vinylphenyl)-carbazole (VPCZ) and 2-methylphenyl-5-(4-vinylphenyl)-1,3,4-oxadiazole (VPOXD). Poly(vinyl carbazole) (PVK), well-known hole transporting material, was also prepared for comparison. These polymers with moderate molecular weight and molecular weight distribution were found to be all solution-processable. Thermal, optical and electrochemical properties of these polymers were investigated for an attempt to apply for charge transporting layer and/or host matrix in emitting layer in OLEDs. It appeared that photophysical properties such as ionization potential and electron affinity were dependent on respective pendant groups in polymers. High band gap energy levels (3.19 eV ~ 3.44 eV) could make polymers a promising candidate for host matrix in OLEDs.*

Keywords: 1,3,4-oxadiazole; carbazole; charge transport; non-conjugated polymer; OLED; PVK

INTRODUCTION

Organic light-emitting diodes (OLEDs) have been paid tremendous attention for flat panel displays (FPDs), due to their attractive advantages of low driving voltage, wide viewing angle, ease of fabrication by

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Address correspondence to Younghwan Kwon, Department of Chemical Engineering, Daegu University, Gyeongsan, Gyeongbuk 712-714, Korea. E-mail: y_kwon@daegu.ac.kr

solution process with large area, and capability of tuning the color emission by selecting proper emitting materials [1–3].

Conjugated polymers have been extensively developed, since electroluminescence of poly(p-phenylene vinylene) (PPV) was first reported in 1990 [4]. Since then, PPV, polyphenylene (PPP), polyfluorene (PF), polythiophene (PTh), conjugated polycarbazole (PCZ) and their derivatives have been widely investigated as promising candidate materials for OLEDs, because the modification of their chemical architectures could be easily achieved via various polymerization techniques to satisfy the requirement for improved performance of OLEDs. In addition, conjugated polymers synthesized have been favored by combining the nature of the monomers, tuning the physical, optical, electrochemical properties and transferring the energy between different band gaps of the monomers. To achieve stable and efficient OLEDs for commercialization, molecular design and synthesis of conjugated polymers have been still investigated.

Non-conjugated polymers have been also used as ancillary materials to support the high performance of conjugated polymers in actual devices. One of the most widely used of these materials was poly(*N*-vinyl carbazole) (PVK), exhibiting interesting electrooptical and charge transporting properties in OLEDs, solar cells, organic semiconductor, etc. Specifically, due to the carbazole side groups, PVK showed good hole transporting property, which was important for improving the performance of OLEDs [5–8]. In addition, phosphorescent OLEDs (PhOLEDs) fabricated with PVK as a host material for a phosphorescent green $Ir(ppy)_3$ dopant by simple solution process were reported [9–11]. It revealed that non-conjugated PVK generally showed higher electroluminescent performance than conjugated polyfluorene, due to lower triplet energy of conjugated polymers resulting in the phosphorescent quenching.

Aromatic 1,3,4-oxadiazole compounds have demonstrated to facilitate electron transport and injection from the cathode, due to their high electron affinity [12–14]. Small molecule, 2-(4-biphenyl)-5-(4-*tert*-butylphenyl)-1,3,4-oxadiazole (PBD), was used to balance hole/electron transport by increasing the rate of electron transport in OLED application [15]. Non-conjugated polymers containing aromatic pendant could be expected to effectively function as electron transport and prevention of recrystallization, when compared with small molecular PBD derivatives.

In this work, design, synthesis and characterization of non-conjugated polymers containing a hole transporting group and an electron transporting group, respectively, as a pendant group were reported. For the synthesis of polymers, two different types of monomers, *N*-(4-vinylphenyl)-carbazole (VPCZ) with a hole transporting carbazole and 2-methylphenyl-5-(4-vinylphenyl)-1,3,4-oxadiazole (VPOXD) with an electron transporting 1,3,4-oxadiazole, were prepared. Then, two non-conjugated copolymers with a hole transporting carbazole for poly(*N*-(4-vinylphenyl)-carbazole) (PPCZ) and an electron transporting 1,3,4-oxadiazole for poly(2-methylphenyl-5-(4-vinylphenyl)-1,3,4-oxadiazole) (PPOXD) were synthesized by free radical polymerization of the corresponding monomers. Thermal, optical and electrochemical properties of these polymers were investigated for an attempt to apply for charge transporting layer and/or host matrix in emitting layer in OLEDs.

EXPERIMENTAL

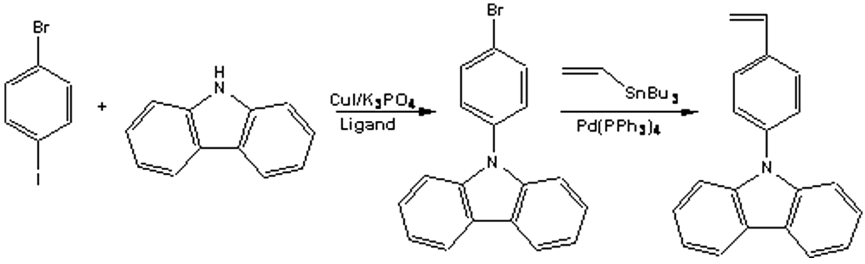
Materials

4-Iodo-bromobenzene (96%), copper (I) iodine (CuI, 98%), trans-1,2-cyclohexane- diamine (ligand) (99%), tributylvinyltin (97%), tetrakis(triphenylphosphine)palladium (0) ($Pd(PPh_3)_4$, 99%), polyphosphoric acid ($POCl_3$, 99%), *N*-vinyl carbazole (98%), benzoyl peroxide (BPO, 75%) were purchased from Aldrich Chemical Co. Reagent grade carbazole, 4-bromobenzoyl chloride, 4-methylbenzoyl hydrazide, triethylamine, and potassium phosphate (K_3PO_4) were obtained from Tokyo Kasei Co. and used as received. Solvents were reagent grades and purified prior to use.

Monomer Synthesis

N-(4-vinylphenyl)-carbazole (VPCZ)

Scheme 1 presents the synthesis of *N*-(4-vinylphenyl)-carbazole (VPCZ) in two steps. First, a mixture of 4-iodo-bromobenzene (2 mmol), carbazole (2 mmmol) and K_3PO_4 (4 mmol) were added



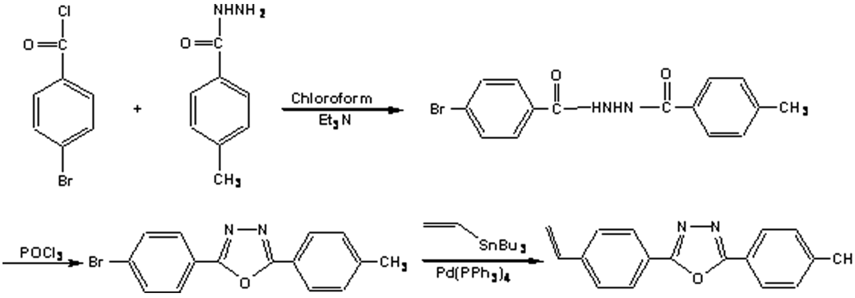
SCHEME 1 Synthetic route to vinyl *N*-phenylcarbazole (VPCZ).

to1,4-dioxane (10 ml) at room temperature. After stirring for 30 min, CuI (0.06 mmol) and trans-1,2-cyclohexanediamine (0.024 mmol) were added to the mixture. The reaction mixture was refluxed for 24 h. After cooling down to room temperature, the reaction mixture was filtered. 1, 4-Dioxane was evaporated, and ethyl acetate was added. The mixture was washed with distilled water, and dried over $MgSO_4$. The final product (compound 1) was obtained through column chromatography (eluent:hexane) with the yield of 90%.

In the second step, a mixture of compound 1 (4.7 mmol), tributylvinyltin (4.7 mmol) and $Pd(PPh_3)_4$ (0.1 mmol) was dissolved in toluene (2 ml) and heated at 70°C for 24 h. After the mixture was cooled to room temperature, the solid was filtered out. After extraction with ethyl acetate three times, the combined organic layers was washed with distilled water, dried over $MgSO_4$ and evaporated. The final product, *N*-(4-vinylphenyl)-carbazole, was obtained through column chromatography (eluent: hexane) with the yield of 60%. ^{1}H NMR (300 MHz in $CDCl_3$): δ 8.13–8.16 (d, 2 H, Ar-H), 7.23–7.78 (m, 10 H, Ar-H), 6.77–6.87 (t, 1 H, olefinic H), 5.82–5.88 (d, 1 H, olefinic H), 5.34–5.37 (d, 1 H, olefinic H). ^{13}C NMR (300 MHz in $CDCl_3$): δ 140.7, 135.9, 133.1, 128.7, 127.5, 125.9, 123.3, 120.4, 119.9, 114.7, 109.8, 109.5.

2-Methylphenyl-5-(4-vinylphenyl)-1,3,4-oxadiazole (VPOXD)

As shown in Scheme 2, 2-methylphenyl-5-(4-vinylphenyl)-1,3,4-oxadiazole (VPOXD) was prepared in three steps. 4-Bromobenzoyl chloride (10 mmol) was added dropwise to a solution of 4-methylbenzoyl hydrazide (10 mmol), triethylamine (10 mmol), and chloroform (150 ml) at room temperature. The resulting mixture was stirred for 4 h and then filtered. The solid collected was washed with water and methanol to give compound 2 with the yield of 95%. ^{1}H NMR



SCHEME 2 Synthetic route to vinyl 2-methylphenyl-5-phenyl-1,3,4-oxadiazole (VPOXD).

(300 MHz in $CDCl_3$): δ 10.54 (s, 2 H, NH), 7.74–7.87 (m, 6 H, Ar-H), 7.31–7.34 (m, 2 H, Ar-H), 2.74 (s, 3 H, CH_3). ^{13}C NMR (300 MHz in $CDCl_3$): δ 165.75, 165.06, 142.02, 131.71, 129.62, 129.57, 129.14, 129.13, 127.55, 125.78, 21.13.

Second, a mixture of compound 2 (3.6 g) and polyphosphoric acid (50 ml) was heated to reflux for 5 h. After completion of reaction, the mixture was precipitated into deionized water. The precipitate was recrystallized from ethanol and dried to give the compound 3 with the yield of 90%. ^1H NMR (300 MHz in $CDCl_3$): δ 7.99–8.04 (m, 4 H, Ar-H), 7.66–7.71 (m, 2 H, Ar-H), 7.33–7.36 (m, 2 H, Ar-H), 2.45 (s, 3 H, CH_3). ^{13}C NMR (300 MHz in $CDCl_3$): δ 164.88, 163.59, 142.49, 132.37, 129.80, 128.24, 126.88, 126.26, 122.85, 120.84, 21.68.

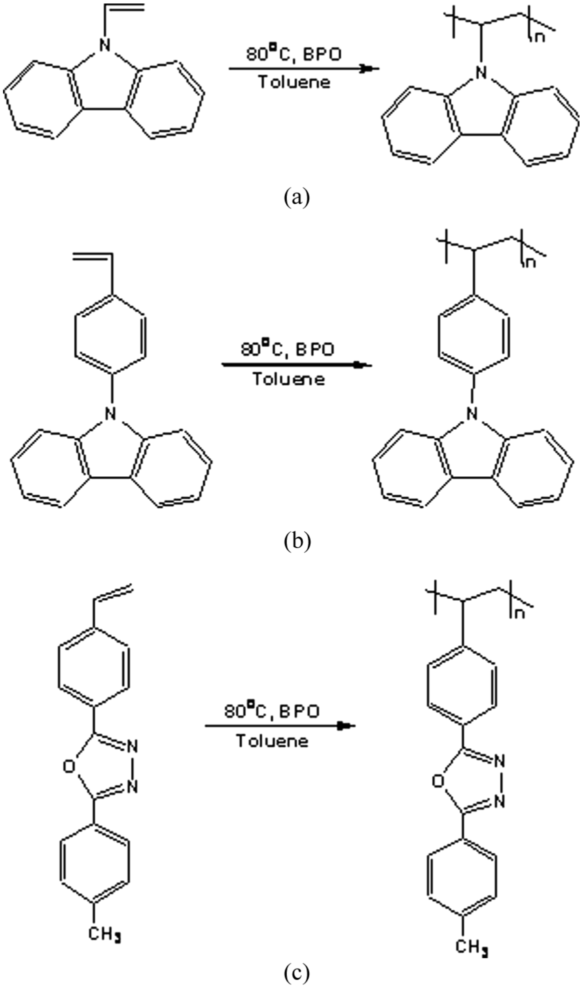
In the third step, a mixture of compound 3 (6 mmol), tributylvinyltin (6 mmol) and $Pd(PPh_3)_4$ (0.12 mmol) was dissolved in DMF (80 ml) and heated at 70°C for 24 h. After cooling down to room temperature, the mixture was poured into distilled water. After extraction with ethyl acetate, the combined organic layers was washed with distilled water, dried over $MgSO_4$ and evaporated. The final product, 2-methylphenyl-5-(4-vinylphenyl)-1,3,4-oxadiazole, was obtained through column chromatography (eluent: hexane) with the yield of 55%. ^1H NMR (300 MHz in $CDCl_3$): δ 7.93–8.16 (m, 4 H, Ar-H), 7.61–7.64 (d, 2 H, Ar-H), 7.27–7.36 (d, 2 H, Ar-H), 6.73–6.83 (t, 1 H, olefinic H), 5.87–5.92 (d, 1 H, olefinic H), 5.38–5.42 (d, 1 H, olefinic H), 2.45–2.60 (s, 3 H, $-CH_3$). ^{13}C NMR (300 MHz in $CDCl_3$): δ 164.9, 163.6, 142.5, 140.7, 135.9, 132.4, 129.8, 128.3, 127.1, 122.9, 120.9, 116.2, 21.7.

Polymer Synthesis

All the polymers were prepared with free radical polymerization by using BPO as an initiator, as presented in Scheme 3. For the synthesis of poly(*N*-vinyl carbazole) (PVK), a mixture of *N*-vinyl carbazole was dissolved in toluene (10 ml). After the mixture was stirred for 30 min in nitrogen atmosphere, the reaction mixture was heated to 80°C. Then, BPO in toluene was added dropwise. The mixture was kept at 80°C for 12 h. After cooling to room temperature, the mixture was precipitated in methanol. The polymer, PVK, was obtained by filtration and drying in vacuum at 40°C. Two other polymers, PPCZ and PPOXD, were prepared with the same procedure mentioned previously.

Characterization

NMR spectra were recorded on a Varian Unity Plus 300 with $CDCl_3$ as a solvent. Molecular weights and molecular weight distributions of



SCHEME 3 Synthesis of polymers, (a) PVK, (b) PPCZ, and (c) PPOXD, by free radical polymerization.

polymers were measured by using Waters gel permeation chromatograph (GPC) equipped with Styragel HR 5E column with tetrahydrofuran (THF) as an eluent against polystyrene standards at room temperature. Thermal analysis was performed on a Seiko EXSTAR 6000 TG/DTA 6300 thermal analyzer at a heating of 20°C/min for thermogravimetry analyzer (TGA). UV-Visible absorption spectra were taken by Shimadzu UV-2100. 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).

RESULTS AND DISCUSSION

Synthesis and Structural Characterization

For the synthesis of polymers, two monomers, VPCZ and VPOXD, were synthesized as outlined in Schemes 1 and 2, respectively. The structures of intermediates and monomers were identified by using ^{1}H NMR and ^{13}C NMR measurements. From ^{1}H NMR spectrum of VPCZ in Figure 1(a), quantitative assignment of VPCZ was confirmed by comparing the signal intensity ratio of olefinic hydrogen (***1*** or ***2***) to aromatic hydrogen (***9***) peaks. Structure of VPOXD was also identified

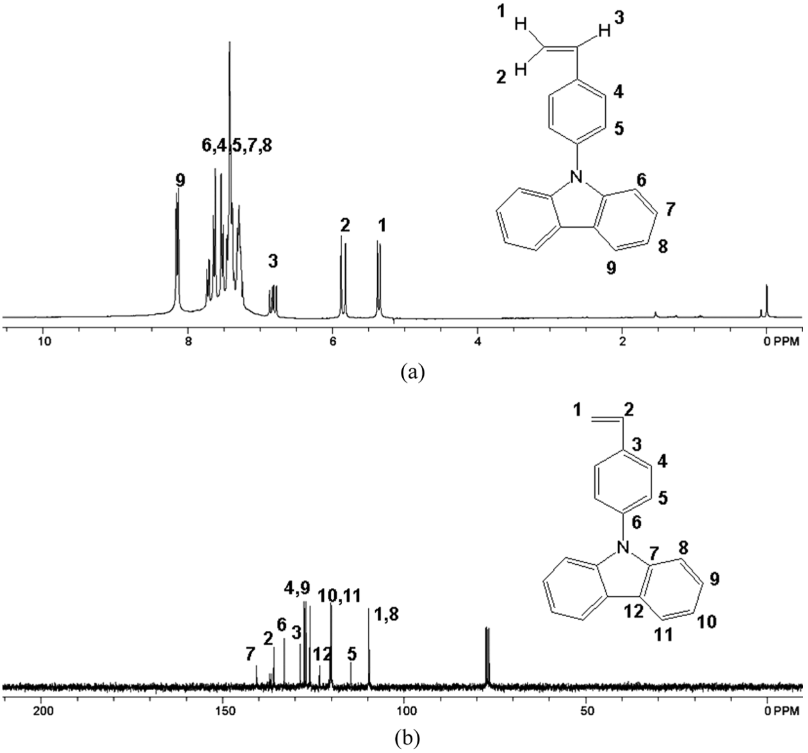


FIGURE 1 (a) ^{1}H NMR and (b) ^{13}C NMR spectra of vinyl *N*-phenylcarbazole (VPCZ).

quantitatively by comparing the signal intensity ratio of olefinic hydrogen (***1*** or ***2***) to methyl (***8***) peaks.

As shown in Scheme 3, the polymers were prepared by free radical polymerization of corresponding monomers using BPO as the initiator and toluene as the solvent. All the polymers exhibited high solubility in common organic solvents such as chloroform, benzene, toluene, 1,1,2,2-tetrachloroethane and tetrahydrofuran, and showed good film formation by spin-coating process. Number-average molecular weight ($\overline{M}_n$), molecular weight distribution ($\overline{M}_w/\overline{M}_n$), and thermal stability data of the polymers are summarized in Table 1. The number-average molecular weight of polymers was measured to be 8,000 g/mole, 21,500 g/mole, and 23,300 g/mole with molecular weight distributions in the range of 1.35 ~ 1.47 for PVK, PPCZ and PPOXD, respectively. Thermal stability of the polymers was characterized by using TGA. TGA thermogram in Figure 3 showed that thermal decomposition temperature (T_d) at 5 wt% loss based on the initial weight of polymers were 296°C for PVK, 292°C for PPCZ, and 275°C for PPOXD, respectively, suggesting relatively good thermal stability of the polymers. PPOXD with 1,3,4-oxadiazole group exhibited less thermal stability than PVK and PPCZ having carbazole group.

Optical and Electrochemical Properties

Figure 4(a) and (b) present UV-Visible absorption and PL emission spectra of the polymers in solution. The related optical data of the polymers are listed in Table 1. As can be seen in Figure 4(a), PVK exhibited UV-Visible absorption maximum peak ($\lambda_{max,UV}$) at 296 nm with the shoulder peaks at 330 nm and 343 nm in solution. Similarly,

TABLE 1 Physical, Optical and Electrochemical Properties of Polymers

Polymers	$\overline{M}_n$ (g/mole)[a]	($\overline{M}_w/\overline{M}_n$)	T_d (°C)[b]	$\lambda_{max,UV}$ (nm)	$\lambda_{max,PL}$ (nm)	Φ_F[c]	Band gap (eV)[d]	HOMO (eV)[e]	LUMO (eV)[f]
PVK	8,000	1.35	296	296, 330, 343	373	0.11	3.41	−5.70	−2.29
PPCZ	21,500	1.47	292	290, 325, 336	351, 359	0.17	3.19	−5.30	−2.11
PPOXD	23,300	1.35	275	292	344, 354	0.24	3.44	−5.52	−2.08

[a] Measured by using GPC with polystyrene standard calibration.

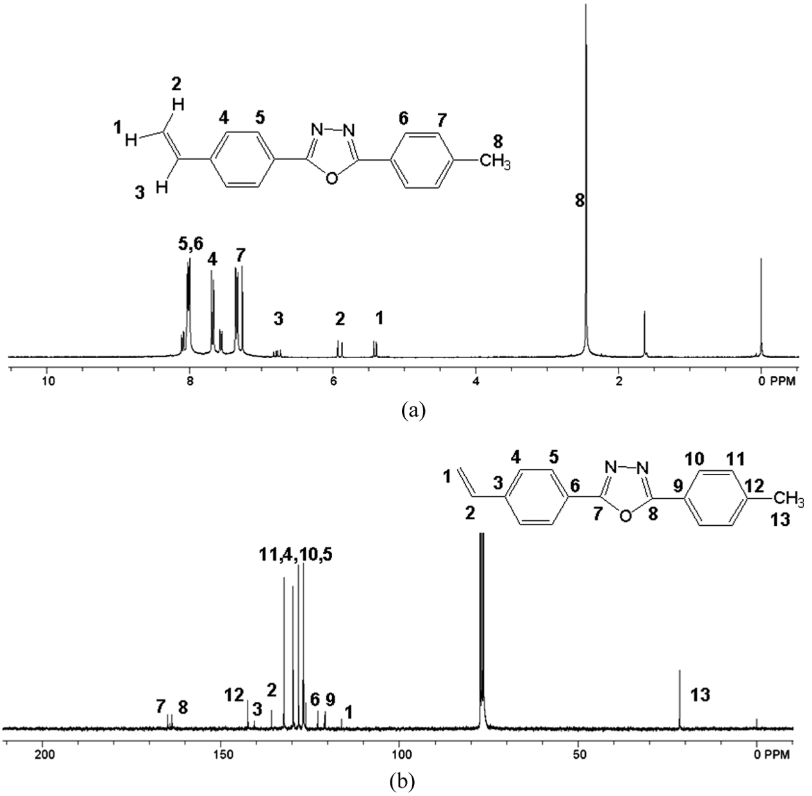
[b] Temperature at 5 wt% loss based on initial weight.

[c] Quantum yield in THF.

[d] Calculated from the crosspoint of UV-Vis and PL spectra.

[e] Measured by a RIKEN Keiki AC-2.

[f] Estimated from the HOMO and band gap.



(a)

(b)

FIGURE 2 (a) ^{1}H NMR and (b) ^{13}C NMR spectra of vinyl 2-methylphenyl-5-phenyl-1,3,4-oxadiazole (VPOXD).

PPCZ also displayed main UV-Visible absorption peak centered at 290 nm with shoulder at 325 nm and 336 nm in solution. These peaks were considered to be attributed to π-π^* transition along carbazole groups [16]. UV-Visible absorption of PPOXD was observed at 292 nm in solution, originating from π-π^* transition along 1,3,4-oxadiazole groups [16].

With excitation at their $\lambda_{\mathrm{max,UV}}$ in solution, PL emission spectra of polymers are shown in Figure 4(b). The PL emission data are also summarized in Table 1. PL emission maxima ($\lambda_{\mathrm{max,PL}}$) of PPCZ and PPOXD in solution were measured at 351 nm and 354 nm, respectively, with quantum yields of 0.17 and 0.24. PVK as a reference showed 373 nm of $\lambda_{\mathrm{max,PL}}$ with 0.11 of quantum yield. It was generally reported that the possibility of Förster energy transfer from a host matrix to a phosphorescent dopant increased with increasing the

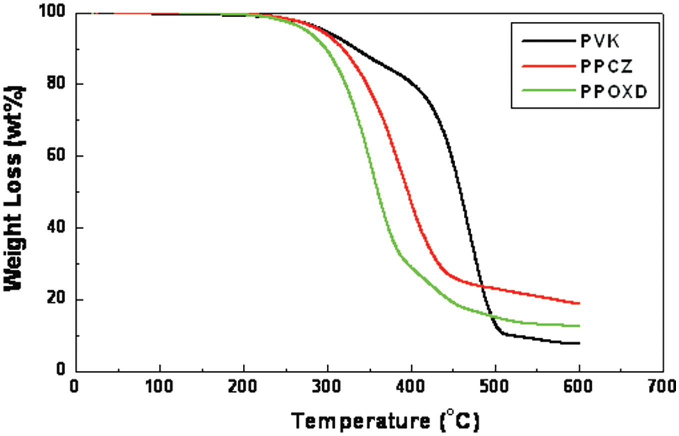
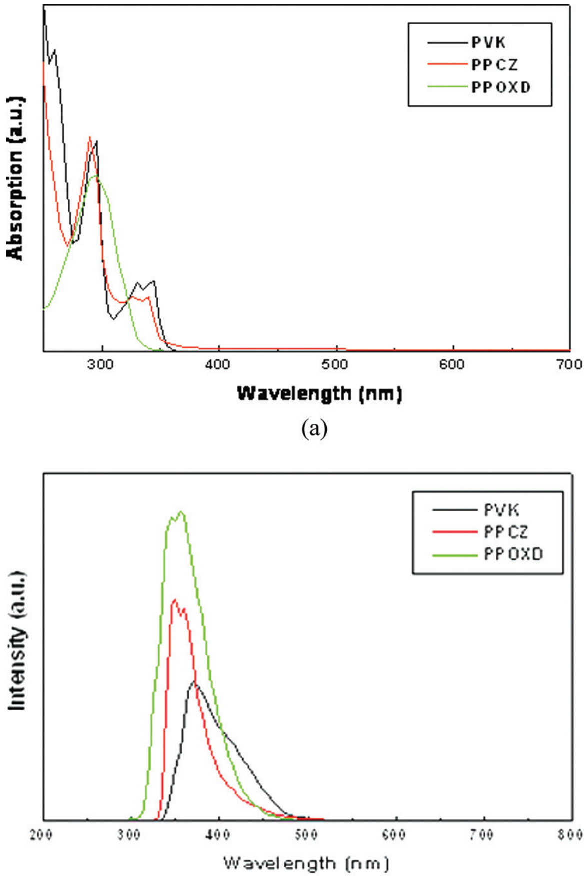


FIGURE 3 TGA thermograms of polymers.

degree of spectral overlap between PL emission spectrum of the host matrix and UV-Visible absorption spectrum of the phosphorescent dopant. A green phosphorescent Ir(ppy)$_3$ film was reported to show strong absorption at 290 nm originated from the ligand-centered π-π^* transition with weaker absorptions at 340–380 nm and 460 nm attributed to the singlet metal-to-ligand charge transfer (^{1}MLCT) and triplet metal-to-ligand charge transfer (^{3}MLCT) transition, respectively [17]. The PL emission band (300 nm $\sim$ 500 nm) of polymers could be expected to overlap with the absorption band of the MLCT transitions in Ir(ppy)$_3$, implying an expected energy transfer from polymers to Ir(ppy)$_3$. Similar result was reported with non-conjugated random copolymers containing carbazole and 1,3,4-oxadiazole pendant groups [18]. Stokes shifts of polymers in solution were calculated to be 77 nm for PVK, 61 nm for PPCZ, and 62 nm for PPOXD, indicating structural differences between the ground and excited states of the polymers.

Table 1 presents the electrochemical data of the polymers. Highest occupied molecular orbital (HOMO) levels were obtained from ionization potential values measured by using a RIKEN Keiki AC-2 instrument. HOMO energy levels of polymers were measured to be -5.70 eV for PVK, -5.30 eV for PPCZ, and -5.52 eV for PPOXD. Lowest unoccupied molecular orbital (LUMO) levels were estimated from HOMO and band gap energy levels of the polymers. LUMO energy levels of PVK, PPCZ and PPOXD were estimated to be -2.29 eV, -2.11 eV, and -2.08 eV, respectively. From LUMO energy level of the polymers, PPOXD showed enhanced electron affinity, compared to PVK and



(a)

(b)

FIGURE 4 (a) UV-Visible absorption and (b) PL emission spectra of polymers.

PPCZ, presumably due to the incorporation of electron-affinitive aromatic 1,3,4-oxadiazole side groups in PPOXD. The band gap energy of the polymers was estimated from the wavelength at the crossover point in UV-Visible absorption and PL emission spectra. Large band gap energy levels were found to be 3.41 eV for PVK, 3.19 eV for PPCZ, and 3.44 eV for PPOXD, respectively. It appeared that the band gap energy of PPCZ was lower than PVK. This small reduction of band gap energy in PPCZ in comparison with PVK could be caused by incorporation of an additional aromatic phenyl side groups extending π-system in PPCZ.

The method for fabricating multi-layered polymer light-emitting diode (PLED) involved solution processes such as solution-casting, inkjet printing, and spin-coating. The problem of this method was dissolution of the former deposited film by spin-coating on top of the polymer film, when organic solvent was used continuously. It was reported that polymeric thin films containing carbazole pendant groups could be linked by electropolymerization or chemical oxidation at all 3,6-positions of carbazole units, resulting in inter- or intramolecular insoluble crosslinked thin films [19,20]. Therefore, the synthesized polymers with large band gap energy and charge transporting groups could exhibit a potential to be used as charge transporting as well as host matrix in emitting layer in PLED, coupled with multiple sing-coating process after post crosslinking of charge transporting pendant groups.

CONCLUSIONS

We have successfully synthesized and characterized new non-conjugated polymers, PPCZ and PPOXD, containing the hole transporting carbazole and the electron transporting 1,3,4-oxadiazole pendant groups, respectively, from corresponding monomers, VPCZ and VPOXD. Thermal and optical properties of these polymers were characterized for an attempt to apply for PLEDs, and compared with those of PVK. The polymers showed number average molecular weights in the range of $8{,}000 \sim 23{,}300$ g/mole and $1.35 \sim 1.47$ of molecular weight distributions. They were all solution-processable, due to their good solubility in common organic solvents. The decomposition temperature of PPCZ under inert atmosphere was higher than PPOXD. UV-Visible absorption and PL emission peaks of polymers showed characteristic dependence on respective pendant groups. PPCZ exhibited lower ionization potential and PPOXD showed higher electron affinity, due to their opposite pendant properties. Further performance study of phosphorescent polymer light-emitting devices can be done by applying these polymers as charge transporting and/or host matrix in the emitting layer.

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