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Synthesis of fused phenylcarbazole phosphine oxide based high triplet energy host materials

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ABSTRACT

High triplet energy host materials based on novel fused phenylcarbazole core structure and diphenylphosphine oxide were synthesized and the physical properties of the host materials were investigated. A high triplet energy of 2.95 eV was obtained from the fused phenylcarbazole based host materials and the energy levels could be manipulated using the diphenylphosphine oxide group. The fused phenylcarbazole based high triplet energy host materials showed excellent morphological stability at high temperature.

1. Introduction

Phosphorescent organic light-emitting diodes (PHOLEDs) are better than fluorescent organic light-emitting diodes (OLEDs) in terms of quantum efficiency.[1–11] Theoretically, PHOLEDs show four times higher quantum efficiency than fluorescent OLEDs as the triplet excited state as well as the singlet excited state can contribute to the light emission. Therefore, there have been many researches about PHOLEDs. In particular, many recent researches were devoted to the development of deep blue PHOLEDs as the quantum efficiency of deep blue PHOLEDs is still not high compared with that of red or green PHOLEDs.[11–15]

The quantum efficiency of deep blue PHOLEDs is mainly determined by the host and dopant materials in the emitting layer. The host should have high triplet energy for an energy transfer to the dopant and have proper energy levels for charge injection from charge transport layers. The management of the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) of the host material as well as the triplet energy level was carried out using various core structures. carbazole type core structures were found to be useful to obtain high triplet energy and several derivatives with the carbazole core structure were developed.[5,16,17] *N*,*N*-Dicarbazolyl-3,5-benzene is one of carbazole derivatives with the high triplet energy of 2.9 eV.[5] Recently, carbazole type host materials with diphenylphosphine oxide groups were also developed and high quantum efficiency was reported in the deep blue PHOLED.[11,18] Silane type materials were also effective as high triplet energy materials for deep blue PHOLEDs even though the hole injection was difficult due to the deep HOMO level of the Si based host material.[7,8]

In this work, a fused phenylcarbazole type core structure was developed as a high triplet energy core structure with good thermal/morphological stability and the physical properties of the host materials modified with diphenylphosphine oxide were investigated. It was demonstrated that high triplet energy of 2.95 eV for deep blue PHOLED application could be obtained using the fused phenylcarbazole core structure, and stable morphological and thermal stability could be realized in the fused phenylcarbazole based host materials.

2. Experimental

2.1. Synthesis

*2.1.1. Synthesis of 9-(2-bromophenyl)-9H-carbazole (**1**).* 9-Carbazole (14.77 g, 88.3 mmol), 2-bromoiodobenzene (50 g, 176 mmol), copper iodide (8.41 g, 44.1 mmol), and potassium carbonate (24.32 g, 176 mmol) were dissolved in anhydrous xylene under nitrogen atmosphere. The reaction mixture was stirred for 12 h at 120 °C. The mixture was diluted with dichloromethane and washed with distilled water (100 mL) three times. The organic layer was dried over anhydrous $MgSO_4$ and evaporated in vacuo to give the crude product, which was purified by column chromatography using

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n-hexane as an eluent and recrystallized by methanol. The final white powdery product was obtained in 45% yield.

*2.1.2. Synthesis of 8,8-diphenyl-8H-indolo[3,2,1-de]acridine (**SPC**).* 9-(2-Bromophenyl)-9*H*-carbazole (**1**) (2.1 g, 6.6 mmol) was dissolved in 20 mL of anhydrous tetrahydrofuran under argon and cooled to −78 °C and *n*-butyllithium (2.5 M in hexanes, 3.4 mL) was added dropwise slowly. Stirring was continued for 2 h at −78 °C, followed by the addition of a solution of benzophenone (1.5 g, 8.5 mmol) in THF (10 mL) under an argon atmosphere. The resulting mixture was gradually warmed to ambient temperature and quenched by adding saturated, aqueous $NaHCO_3$ (20 mL). The mixture was extracted with dichloromethane. The combined organic layers were dried over magnesium sulfate, filtered, and evaporated under reduced pressure. A yellow powdery product was obtained. The crude residue was placed in another two-necked flask was dissolved in acetic acid (20 mL). A catalytic amount of aqueous HCl (5 mol %, 12 N) was then added and the whole solution was refluxed for 12 h. After cooling to ambient temperature, purification by silica gel chromatography using dichloromethane/*n*-hexane gave a white powder.

SPC yield 70%, mp 248 °C. T_g 84.9 °C. ^1H NMR (200 MHz, $CDCl_3$): δ 8.14–8.07 (t, 2H), 7.94–7.91 (d, 1H), 7.59–7.48 (t, 1H), 7.42–7.19 (m, 11H), 7.07–7.03 (m, 7H). ^{13}C NMR (200 MHz, $CDCl_3$): δ 146.409, 138.930, 137.862, 137.182, 132.423, 131.646, 130.966, 129.898, 128.538, 128.247, 127.470, 126.207, 123.488, 122.905, 121.934, 120.865, 118.923, 117.758, 115.329, 114.067, 112.998, 67.739, 57.347. MS (FAB) *m*/*z* 407 [$(M+H)^+$]. Anal. Calcd for $C_{31}H_{21}N$: C, 91.37; H, 5.19; N, 3.44. Found: C, 90.92; H, 5.19; N, 3.72.

*2.1.3. Synthesis of 8-(4-(diphenylphosphoryl)phenyl)-8-phenyl-8H-indolo[3,2,1-de]acridine (**SPCPO1**).* Intermediate compound **2** was synthesized according to the same synthetic procedure as the **SPC** except that 4-bromobenzophenone was used instead of benzophenone. Into a 100 mL, two-neck flask, was placed a 8-(4-bromophenyl)-8-phenyl-8*H*-indolo[3,2,1-*de*]acridine (**2**) (2.0 g, 4.1 mmol) in THF (20 mL). The reaction flask was cooled to −78 °C and *n*-BuLi (2.5 M in hexane, 2.13 mL) was added dropwise slowly. The whole solution was stirred at this temperature for 3 h, followed by addition of a solution of chlorodiphenylphophine (1.17 g, 5.34 mmol) under argon atmosphere. The resulting mixture was gradually warmed to ambient temperature and quenched by methanol (7 mL). The mixture was extracted with dichloromethane. The combined organic layers were dried over magnesium sulfate, filtered, and evaporated under reduced pressure. A white powdery product was obtained to 1.21 g (50%). It was dissolved in dichloromethane (20 mL) and hydrogen peroxide (4 mL), which was stirred overnight at room temperature. The organic layer was separated and washed with dichloromethane and water. The extract was evaporated to dryness affording a white solid, which was further purified by column chromatography to yield 1.19 g of chemically pure **SPCPO1**.

SPCPO1 yield 50%, T_g 126.4 °C. ^1H NMR (200 MHz, $CDCl_3$): δ 8.15–8.09 (m, 3H), 7.96–7.92 (d, 1H), 7.69–7.60 (m, 4H), 7.55–7.29 (m, 12), 7.26–7.22 (m, 4H), 7.17–7.04 (m, 6H). ^{13}C NMR (200 MHz, $CDCl_3$): δ 150.488, 145.826, 138.736, 137.085, 133.492, 132.617, 132.035, 131.452, 130.966, 129.898, 129.218, 129.024, 128.538, 128.150, 127.858, 127.470, 126.790, 126.402, 126.013, 123.585, 123.002, 122.225, 121.934, 120.768, 119.117, 117.952, 115.135, 114.261, 113.970, 113.096, 57.250. MS (FAB) *m*/*z* 608 [$(M+H)^+$]. Anal. Calcd for $C_{43}H_{30}NOP$: C, 84.99; H, 4.98; N, 2.30. Found: C, 82.33; H, 4.96; N, 2.34.

*2.1.4. Synthesis of 8,8-bis(4-(diphenylphosphoryl)phenyl)-8H-indolo[3,2,1-de]acridine (**SPCPO2**).* Intermediate compound **3** was synthesized according to the same synthetic procedure as the **SPC** except that 4,4′-dibromobenzophenone was used instead of benzophenone. Into a 100 mL two-neck flask, was placed a 8,8-bis(4-bromophenyl)-8*H*-indolo[3,2,1-*de*]acridine (**3**) (2.5 g, 3.53 mmol) in THF (20 mL). The reaction flask was cooled to −78 °C and *n*-BuLi (2.5 M in hexane, 3.53 mL) was added dropwise slowly. The whole solution was stirred for 3 h followed by addition of a solution of chlorodiphenylphophine (1.95 g, 8.84 mmol) under argon atmosphere. The resulting mixture was gradually warmed to ambient temperature and quenched by methanol (7 mL). The mixture was extracted with dichloromethane. The combined organic layers were dried over magnesium sulfate, filtered, and evaporated under reduced pressure. The white powdery product was obtained to 1.7 g (62%). It was dissolved in dichloromethane (20 mL) and hydrogen peroxide (4 mL), which was stirred overnight at room temperature. The organic layer was separated and washed with dichloromethane and water. The extract was evaporated to dryness affording a white solid, which was further purified by column chromatography to yield 1.18 g of chemically pure **SPCPO2**.

SPCPO2 yield 62%, T_g 153.1 °C. ^1H NMR (200 MHz, $CDCl_3$): δ 8.15–8.09 (t, 3H), 7.97–7.93 (d, 1H), 7.69–7.60 (m, 7H), 7.49–7.26 (m, 21H), 7.16–7.06 (m, 5H), 7.00–6.96 (m, 2H). ^{13}C NMR (200 MHz, $CDCl_3$): δ 149.905, 138.736, 137.571, 137.085, 133.297, 132.617, 131.452, 130.772, 129.704, 129.315, 129.024, 128.830, 128.150, 127.955, 127.276, 126.790, 126.499, 126.110, 125.772, 124.945, 123.682, 123.196, 122.419, 122.128, 121.060, 120.865, 119.409, 118.243, 115.135, 114.455, 113.970, 113.290, 57.347. MS (FAB) *m*/*z* 808 [$(M+H)^+$]. Anal. Calcd for $C_{55}H_{39}NO_2P_2$: C, 81.77; H, 4.87; N, 1.73. Found: C, 81.02; H, 5.06; N, 1.87.

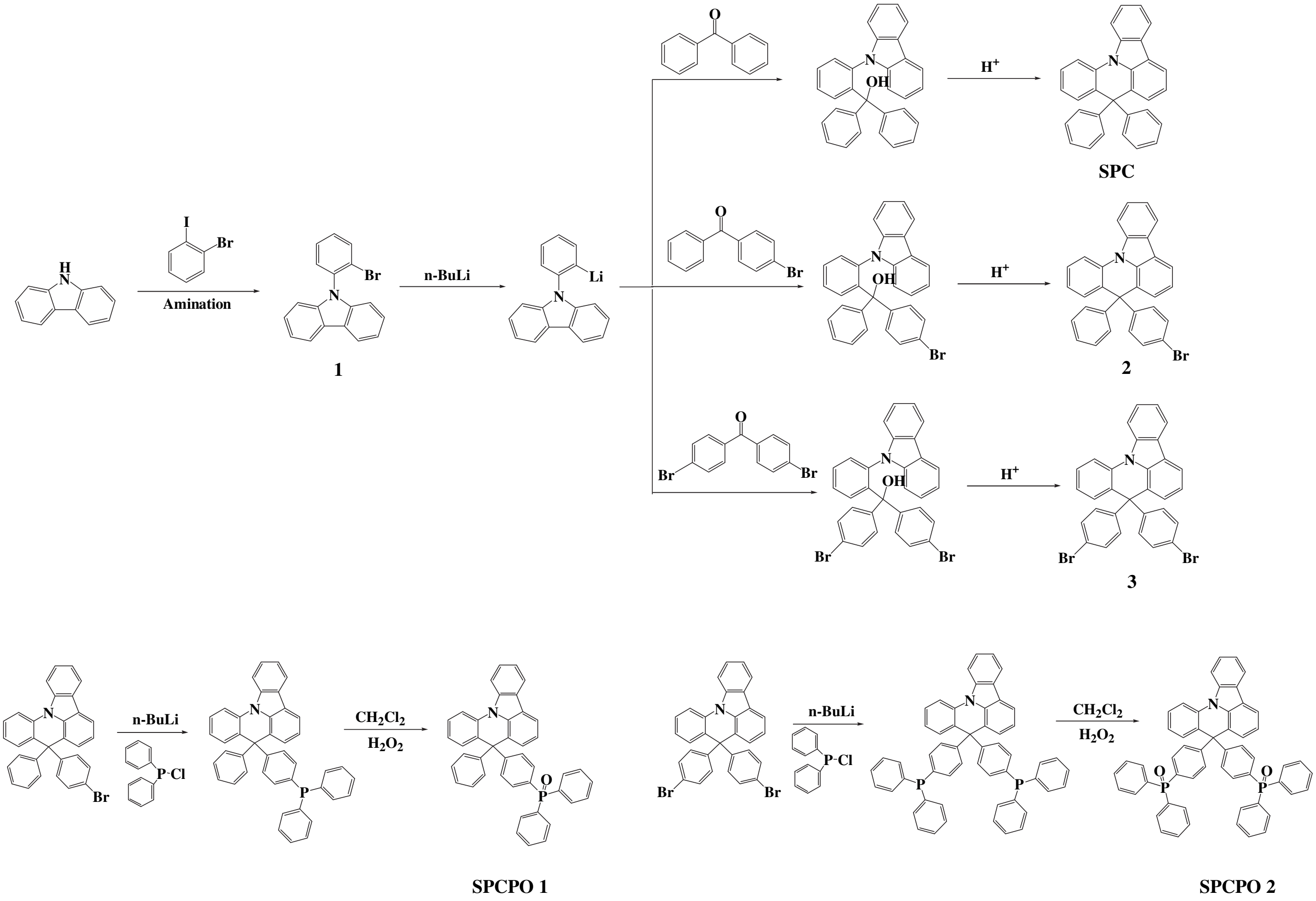
2.1.5. Measurements. The ^1H and ^{13}C nuclear magnetic resonance (NMR) were recorded on a Varian 200 (200 MHz) spectrometer and the mass spectra were recorded using a JEOL, JMS-AX505WA spectrometer in FAB mode. Ultraviolet-visible (UV–vis) and photoluminescence (PL) spectra of the **SPCPO1** and **SPCPO2** were obtained from UV–vis spectrophotometer (Shimadzu, UV-2501PC) and fluorescence spectrophotometer (HITAXHI, F-7000). The energy level of the **SPCPO1** and **SPCPO2** was measured with a cyclic voltameter. Glass transition temperature (T_g) measurements of the **SPCPO1** and **SPCPO2** were carried out using differential scanning calorimeter (Mettler DSC 822) at a heating rate of 10 °C/min under nitrogen atmosphere.

Hole only devices with a device configuration of indium tin oxide (100 nm)/**SPCPO1** or **SPCPO2** (100 nm)/Au (100 nm) and electron only device with a device configuration of indium tin oxide (100 nm)/2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline (5 nm)/**SPCPO1** or **SPCPO2** (100 nm)/LiF (1 nm)/Al (100 nm).

3. Results and discussion

The fused phenylcarbazole unit was designed as a core structure, which has a high triplet energy and thermal/morphological stability. The fused phenylcarbazole unit can be synthesized by ring closing reaction of the 9-(2-bromophenyl)-9*H*-carbazole with diphenylketone derivatives as shown in Scheme 1. In this work, 4-bromobenzophenone and 4,4′-dibromobenzophenone were reacted with the **1** to synthesize the **SPCPO1** and **SPCPO2**. The brominated fused phenylcarbazole compounds, **2** and **3**, were modified with diphenylphosphine oxide to synthesize high triplet energy bipolar host materials as the diphenylphosphine oxide has electron transport properties compared with the hole transport type fused phenylcarbazole core unit.[11,18–20] The **SPCPO1** and **SPCPO2** could be successively synthesized by the ring closing reaction followed by the phosphonation reaction and a high purity over 99% could be obtained after purification by column chromatography.

Molecular simulation of the **SPCPO1** and **SPCPO2** was carried out to understand the molecular orbital distribution in the two host materials. Density functional theory (DFT) calculations were



Scheme 1. Synthetic scheme of **SPCPO1** and **SPCPO2**.

performed using a suite of Gaussian 03 program. The nonlocal density functional of Becke's 3-parameters employing Lee–Yang–Parr functional (B3LYP) with 6-31G* basis sets was used for the calculation. Figure 1 shows the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) distribution of the **SPCPO1** and **SPCPO2**. The HOMO was mainly distributed over the phenylcarbazole unit of the fused phenylcarbazole, while the LUMO was dispersed over the whole

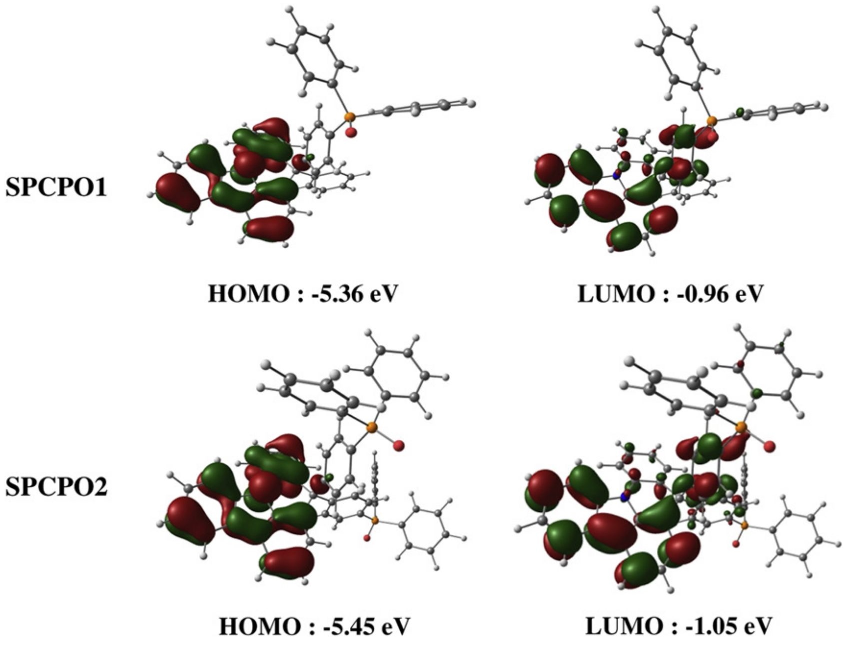


Figure 1. Simulated HOMO and LUMO distribution of the **SPCPO1** and **SPCPO2**.

fused phenylcarbazole structure. The LUMO orbital was rather dispersed in the **SPCPO1** and **SPCPO2** as the diphenylphosphine oxide group has strong electron withdrawing character. Comparing the orbital distribution of the **SPCPO1** and **SPCPO2**, the HOMO was more localized in the phenylcarbazole unit and the LUMO was more dispersed in the fused phenylcarbazole unit in the **SPCPO2**. The difference of the orbital distribution affected the simulated HOMO and LUMO levels of the host materials and 0.1 eV energy level shift downward from the vacuum level was observed in the **SPCPO2** compared with **SPCPO1** due to the additional diphenylphosphine oxide group.

UV–vis and PL measurements of the **SPCPO1** and **SPCPO2** were carried out to study the light absorption and emission properties of the host materials. UV–vis and PL spectra of host materials are shown in Figure 2. The **SPCPO1** and **SPCPO2** showed similar UV–vis absorption spectra and two main absorption peaks assigned to the absorption of the fused phenylcarbazole unit were observed at 293 nm and 350 nm. The emission peaks of the **SPCPO1** and **SPCPO2** appeared at 367 nm and 368 nm, respectively. The HOMO and LUMO were mostly distributed over the fused phenylcarbazole unit with little contribution of the diphenylphosphine oxide group. Therefore, UV–vis light was absorbed by the fused phenylcarbazole core and light emission was also induced from the fused phenylcarbazole core by UV–vis excitation. As the light absorption and emission happened in the fused phenylcarbazole core structure, the **SPCPO1** and **SPCPO2** showed similar UV–vis and emission spectra in spite of different chemical structures. The bandgap of the **SPCPO1** and **SPCPO2** could be calculated from the absorption edge of the UV–vis absorption spectra and the bandgaps were 3.41 eV and 3.40 eV, respectively. Low temperature PL measurement of the **SPCPO1** and **SPCPO2** was also carried out to obtain the triplet energy of the host materials. The triplet energy of the two host materials was the same and it was 2.95 eV. It has been known that the triplet energy of the carbazole core is 3.02 eV and there was only 0.07 eV decrease of the triplet energy in the **SPCPO1** and **SPCPO2**.[21] The high triplet energy of the fused phenylcarbazole based host materials can be explained by the distorted chemical structure of spiro connecting unit. The fused phenylcarbazole has a sp^3 carbon atom between the phenylcarbazole and diphenyl unit. The sp^3 carbon separates the phenylcarbazole and phenyl units and does not extend the conjugation of the phenylcarbazole group. Therefore, the high triplet energy of the phenylcarbazole unit could be maintained in the **SPCPO1** and **SPCPO2** host materials. The 0.07 eV reduction of the triplet energy is originated from the geometrical structure of the phenyl unit in the phenylcarbazole core. Figure 3 shows the geometrical structure of the **SPCPO1** and 3-(diphenylphosphoryl)-9-phenyl-9*H*-carbazole. The triplet energy of the PPO1 was 3.02 eV. The angle between the phenyl unit and carbazole is 28.8° in the fused phenylcarbazole, while it was 56.3° of common phenylcarbazole group. The reduced angle between the phenyl and carbazole planes of the fused phenylcarbazole slightly increases the conjugation between the phenyl group in the phenylcarbazole and carbazole unit, resulting in the reduced triplet energy in **SPCPO1**. However, the triplet energy of two host materials was high enough for application as host materials in deep blue PHOLEDs as the triplet energy of common deep blue emitting phosphorescent dopant is around 2.8 eV.

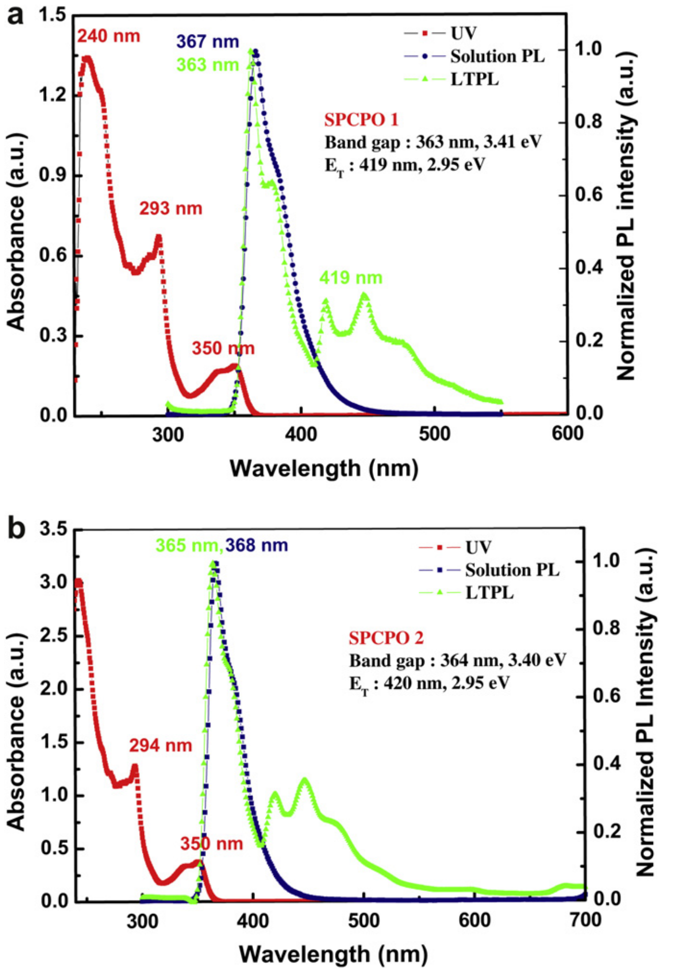


Figure 2. Uv–vis absorption, photoluminescence and low temperature photoluminescence spectra of the **SPCPO1** (a) and **SPCPO2** (b).

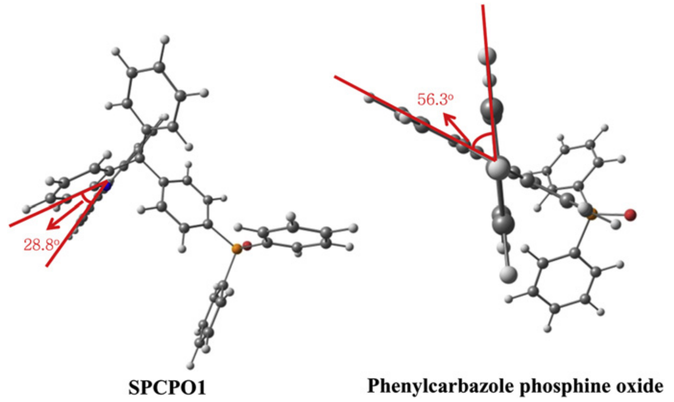


Figure 3. Geometrical structure of **SPCPO1** compared with phenylcarbazolephosphine oxide (3-(diphenylphosphoryl)-9-phenyl-9*H*-carbazole). The angle between the phenyl plane and carbazole plane is shown.

The HOMO levels of **SPCPO1** and **SPCPO2** were measured with CV and the LUMO levels were calculated from the HOMO and absorption edge of UV–vis spectra. The HOMO levels of the **SPCPO1** and **SPCPO2** were −6.08 eV and −6.11 eV, respectively. The HOMO level of the **SPCPO2** was shifted by 0.03 eV compared with that of the **SPCPO1**. The HOMO level shift of 0.03 eV is due to the additional diphenylphosphine oxide group with electron withdrawing character. The LUMO level was also shifted by 0.04 eV by the phosphine oxide group. All physical properties of the **SPCPO1** and **SPCPO2** are summarized in Table 1.

The fused phenylcarbazole core has a rigid structure as the rotation of the phenyl group in the phenylcarbazole is limited by the spiro structure. The rigidity of the fused phenylcarbazole core may improve the T_g of the host materials. In addition, the diphenylphosphine group can also improve the T_g of the host materials.[20] Figure 4 shows DSC thermograms of **SPCPO1** and **SPCPO2**

Table 1
Physical properties of **SPCPO1** and **SPCPO2**

	Optical analysis		Thermal analysis		Electrical analysis			
	Absorption (nm)	Emission (nm)	T_g (°C)	T_m (°C)	HOMO (eV)	LUMO (eV)	Bandgap (eV)	E_T (eV)
SPCPO1	293, 350	367	126	—	−6.08	−2.67	3.41	2.95
SPCPO2	294, 350	368	153	—	−6.11	−2.71	3.40	2.95

compared with that of the fused phenylcarbazole core. The fused phenylcarbazole core showed a T_g of 85 °C and T_m of 248 °C. The T_g of the **SPCPO1** and **SPCPO2** was greatly improved by the diphenylphosphine oxide group and the T_gs of the **SPCPO1** and **SPCPO2** were 127 °C and 153 °C, respectively. T_m was not observed up to 400 °C. The diphenylphosphine oxide group hinders the close packing of the fused phenylcarbazole core, suppressing the crystallization of the **SPCPO1** and **SPCPO2**. Therefore, it is expected that the **SPCPO1** and **SPCPO2** form a stable amorphous morphology.

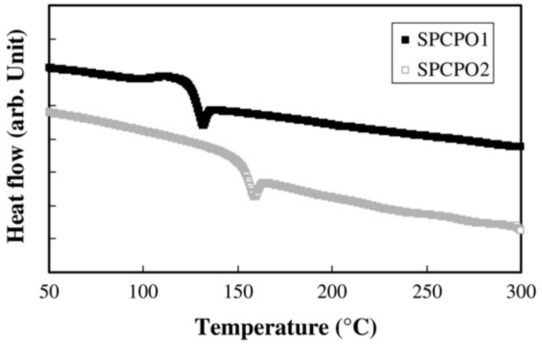
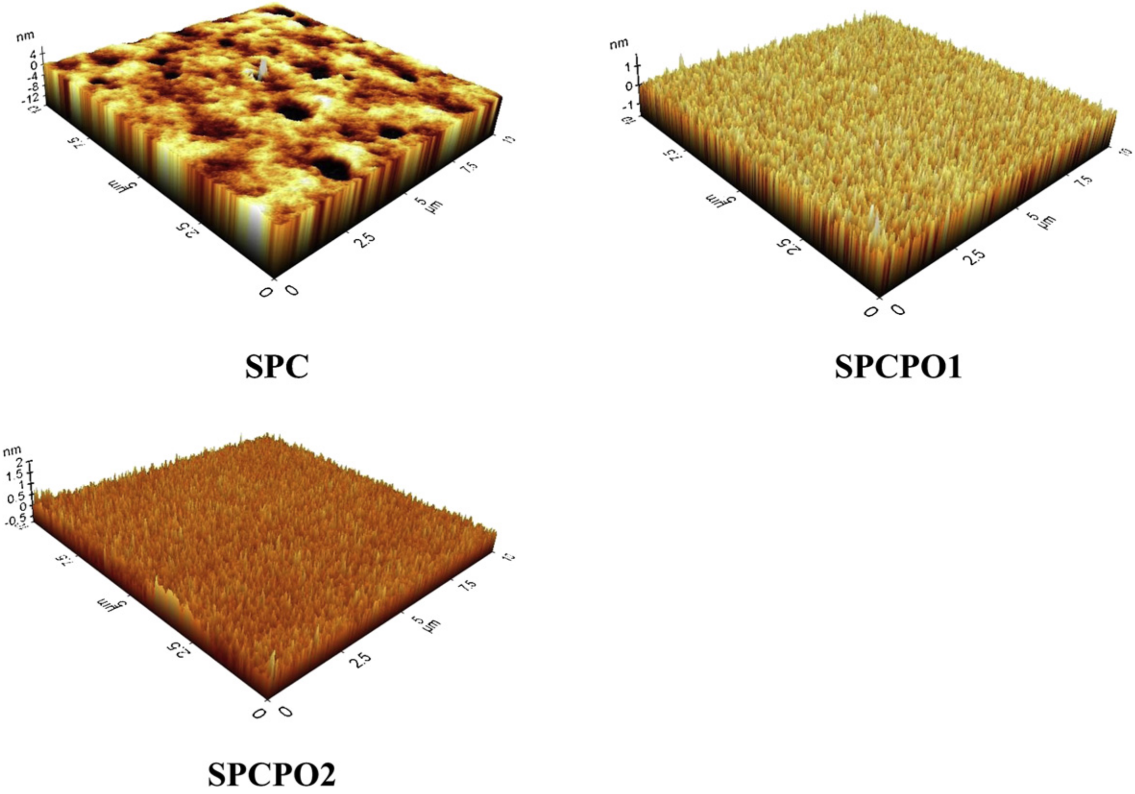


Figure 4. Differential scanning calorimetric thermogram of the **SPCPO1** and **SPCPO2**.

The surface morphology of the **SPCPO1** and **SPCPO2** on silicon wafer was measured with AFM and AFM pictures of the **SPCPO1** and **SPCPO2** compared with **SPC** are shown in Figure 5. The **SPCPO1** and **SPCPO2** showed smooth surface morphology without any aggregation or crystallization and the surface roughness of the **SPCPO1** and **SPCPO2** was 0.29 nm and 0.17 nm, while the fused phenylcarbazole core (**SPC**) showed aggregated morphology with a surface roughness of 0.40 nm. The fused phenylcarbazole core showed rather aggregated morphology due to the reduced dihedral angle as shown in Figure 3. The **SPCPO2** showed better surface roughness than the **SPCPO1**, indicating that the additional diphenylphosphine oxide group stabilized the surface morphology of the fused phenylcarbazole. The smooth surface morphology of the **SPCPO1** and **SPCPO2** was kept stable and the same AFM data were obtained even after storage for more than a week. The high T_g and rigid molecular structure stabilized the surface morphology of the **SPCPO1** and **SPCPO2**.

The morphological stability of the **SPCPO1** and **SPCPO2** was compared with a phenylcarbazole based phosphine oxide compound, 3,6-bis(diphenylphosphoryl)-9-phenyl-9*H*-carbazole (**PPO2**). Thin films of the organic materials were thermally treated at 140 °C for 5 min and the morphology of the thermally treated thin films was analyzed with an optical microscope. Figure 6 shows optical microscope images of **SPC**, **PPO2**, **SPCPO1**, and **SPCPO2**. The **SPC** was significantly crystallized after thermal treatment and aggregated morphology was observed. Crystallization of the **PPO2** was



SPC

SPCPO1

SPCPO2

Figure 5. Atomic force microscopic images of the fused phenylcarbazole core, **SPCPO1** and **SPCPO2**.

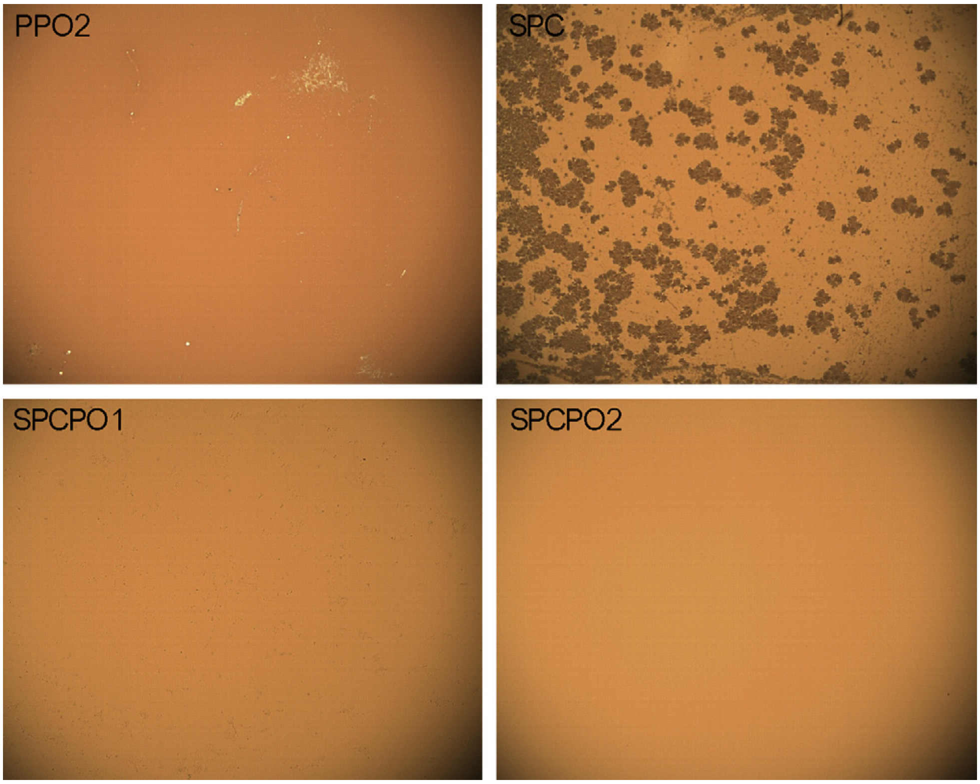


Figure 6. Optical microscope images of **SPC**, **PPO2 SPCPO1**, and **SPCPO2** after thermal treatment at 140 °C for 5 min.

also observed after thermal treatment, while **SPCPO1** and **SPCPO2** did not show any crystallization of the deposited film. The amorphous morphology of the material was kept stable after thermal treatment at 140 °C. In particular, there was no change of the morphology of the **SPCPO2** even after thermal treatment at 140 °C for more than 1 h. Therefore, the fused phenylcarbazole based compounds, **SPCPO1** and **SPCPO2**, showed much better morphological stability than a phenylcarbazole based compound. The stable morphology of the **SPCPO1** and **SPCPO2** is expected to improve the device stability in device application.

Charge transport properties of the host materials are important for device application and the hole/electron transport properties of **SPCPO1** and **SPCPO2** were studied using hole and electron only devices. Figure 7 shows current density–voltage curves of hole only and electron only devices of **SPCPO1** and **SPCPO2**. The **SPCPO2** showed higher electron current density and lower hole current density than the **SPCPO1**. The high electron current density of the **SPCPO2** is due to electron deficient two diphenylphosphine oxide groups, which play a role of electron transport unit. The **SPCPO2** showed similar hole and electron current density and it is expected that the **SPCPO2** shows good device performances in device applications.

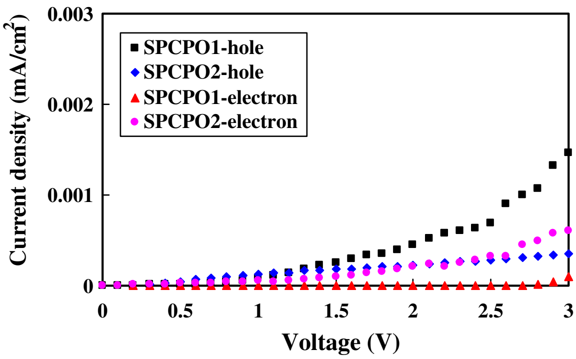


Figure 7. Current density–voltage curves of hole only and electron only devices of **SPCPO1** and **SPCPO2**.

4. Conclusions

In conclusion, high triplet energy host materials based on fused phenylcarbazole core and diphenylphosphine oxide were effectively synthesized and a high triplet energy of 2.95 eV could be obtained. The triplet energy of the **SPCPO1** and **SPCPO2** was high enough for use as hosts for deep blue phosphorescent dopant. In addition, the novel design combining the fused phenylcarbazole and diphenylphosphine oxide improved the thermal and morphological stability. Therefore, the **SPCPO1** and **SPCPO2** are promising as host materials for deep blue phosphorescent organic light-emitting diodes.

Supplementary data

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.tet.2010.07.008.

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