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Synthesis and Electro-Optical Properties of Carbazole-Substituted Pyrene Derivatives

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Mono and dicarbazole-substituted pyrene derivatives, 9*H*-carbazol-9-ylpyrene (MCzP) and 1,6di(9*H*-carbazol-9-yl)pyrene (DCzP), with dual-purpose function as a blue emitting and charge transporting layer in organic light emitting diodes, were synthesized and characterized. These series of molecules consisted of an electron donating (D) carbazole and an electron accepting (A) pyrene in D-A and D-A-D shapes. Non-doped blue electroluminescent devices with the configurations of ITO (150 nm)/ α -NPD (30 nm)/DCzP (40 nm)/LiF (1 nm)/Al (150 nm) (D1) and ITO (150 nm)/2-TNATA (15 nm)/ α -NPD (20 nm)/DCzP (40 nm)/BCP (15 nm)/Alq₃ (10 nm)/LiF (1 nm)/Al (120 nm) (D2) were fabricated. D1 and D2 devices showed blue emission at 492 nm and 488 nm, and maximum luminance of 840 and 7560 cd/m² obtained at 13 V and 15 V, respectively.

Keywords: Carbazole, Pyrene Derivative, Organic Light Emitting Diode, Electron Donating, Electron Accepting.

1. INTRODUCTION

Since organic light emitting diodes (OLEDs) were developed by Tang and Van Slyke in 1987, intense researches have been progressed due to their potential application to full color flat panel displays.¹⁻⁴ OLEDs have shown special advantages such as self-emission, high luminous efficiency, high contrast ratio, fast response time, and wide color gamut, as compared with other displays. Therefore, π -conjugated molecules have been extensively studied because of their potential applications in OLEDs. The combination of molecules has allowed their optical and electrochemical properties to be tuned over a wide range, which can be achieved by appropriate chemical way of selecting and modifying the molecular structures.

Polyaromatic compounds such as naphthalenes, pyrenes, carbazoles, and anthracenes have been widely studied as emitting, hole transporting, and electron transporting materials in OLEDs.^{5–11} Among the polyaromatic compounds, pyrenes exhibit good properties such as strong π electron delocalization, blue emission, and high carrier mobility.

 matebunds,
and photovoltaic cells,^{18, 19} because of not only their excellent photoconductivity and relatively intense luminescence, but increased thermal stability and glass-state durability of them.
Although the research progress on organic molecular materials has allowed the OLED devices to achieve high luminescence and high efficiency, continuous research and

However, studies on using them as the emitting layer for OLEDs have been very limited due to their strong stacking

tendency to form excimer or exciplex, inducing low quan-

tum efficiency. It also causes a problem that pyrene deriva-

tives are to easily crystallize. Since microcrystallization of

the materials has harmful effects on the OLED device sta-

bility, amorphous stability is a principal requirement for

materials used in OLEDs. Chemical modification by incor-

porating bulky side chains on pyrenes facilities to alleviate

those drawbacks. Along this line, it was reported to have an

enhanced amorphous stability of the pyrenes by being mod-

ified it into 9-alkyloxyphenyl-9-pyrenefluorene,¹² and an

efficient blue emission.13 Meanwhile, the carbazole deriva-

tive, as one of heteroaromatic compounds with nitrogen

atom, has been extensively investigated as an electron

donor in optoelectronic devices such as organic and poly-

meric light emitting diodes,^{14–16} field effect transistors,¹⁷

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development of blue emitting materials, especially, are still demanded.²⁰⁻²⁵ In particular, dual-purpose blue emitters intended to serve blue emitting as well as charge transporting properties are becoming important, because they can make it possible to simplify the layered structures of OLED devices.^{26, 27}

Recently, we have been interested in applying pyrene molecules with carbazole substituents as a key dualpurpose material for blue emission and charge transport in OLEDs. Thus, we have synthesized two derivatives of the pyrenes, and studied their use in OLEDs. We report the synthesis of 9*H*-carbazol-9-ylpyrene (MCzP) and 1,6di(9*H*-carbazol-9-yl)pyrene (DCzP) whose structures are based on the pyrenes substituted with asymmetric monocarbazole and symmetric di-carbazole moieties, respectively, and discuss their photophysical and electro-optical properties.

2. EXPERIMENTAL DETAILS

2.1. Materials

Thin layer chromatography (TLC) analyses were carried out on a pre-coated 0.2 mm HPTLC silica gel 60 plate (Merck, Darmstadt). Flash column chromatography was performed with silica gel (Merck silica gel 60, 70– 230 mesh). 1-Bromopyrene, 9H-carbazole, potassium carbonate, sodium sulfate, copper powder, pyrene, bromine, toluene, were all purchased from Aldrich Chemical Co.

4, 4', 4"- Tris(N-(2-naphthyl)-N-phenyl-amino)-triphenylamine (2-TNATA) and 2,9-dimethyl-4,7-diphenyl-1,10phenanthroline (BCP) were obtained from Doosan Electro-Materials Co., Ltd. (Korea). Tris (8-hydroxyquinolinato) aluminum (Alq₃) and bis(naphthylphenylamino)biphenyl (α -NPD) were purchased from SFC Co., Ltd. (Korea). All organic compounds for fabrication of OLEDs were sublimation grade and used without further purification. The glass substrate with patterned indium-tin oxide (ITO, 20 Ω/\Box) for OLED devices was purchased from SUNIC Co. (Korea).

2.2. Synthesis of 9H-carbazol-9-ylpyrene (MCzP)

1-Bromopyrene (2.00 g, 7.11 mmol), 9*H*-carbazole (1.78 g, 10.67 mmol), potassium carbonate (2.94 g, 21.27 mmol) and copper powder (0.14 g, 2.20 mmol) were added in a three-neck flask and slowly stirred in nitrobenzene (30 mL). The resulting solution was refluxed for 24 h at 180 °C. The mixture was allowed to cool to room temperature and extracted with ethyl acetate and DI water, then dried over Na₂SO₄ and concentrated. The residue was purified by silica gel chromatography using 2:1 EtOAc/hexane as an eluent, yielding compound MCzP as a white solid.

MCzP: Yield: 38%, ¹H-NMR (DMSO, 400 MHz) δ (*ppm*); 8.59 (d, J = 8.0 Hz, 2H), 8.45 (d, J = 8.0 Hz, 2H),

8.31 (d, *J* = 8.0 Hz, 2H), 8.21 (d, *J* = 9.2 Hz, 2H), 7.69 (d, *J* = 9.2 Hz, 2H), 7.35 (t, 3H), 7.05 (m, 4H).

2.3. Synthesis of 1,6-di(9*H*-carbazol-9-yl)pyrene (DCzP)

In the first step, 1,6-dibromopyrene (DBP) was synthesized as follows. Bromine (11.53 ml, 225 mmol) was added drop wise in the solution of pyrene (10.00 g, 49.4 mmol) in carbon tetrachloride (100 mL). The resulting solution was reacted for 12 h at room temperature. Extracted materials after the reaction were recrystallized in the toluene to give rise to DBP.^{28, 29}

In the second step, DBP (2.00 g, 5.56 mmol), 9*H*-carbazole (2.45 mg, 13.9 mmol), potassium carbonate (2.30 g, 16.64 mmol) and copper powder (0.11 g, 1.73 mmol) were added in a three-neck flask and slowly stirred in nitrobenzene (30 mL). The resulting solution was refluxed for 30 h at 180 °C. The mixture was allowed to cool to room temperature, and was extracted with ethyl acetate and DI water, then dried over Na_2SO_4 and concentrated. The residue was purified by silica gel chromatography using 2:1 EtOAc/hexane as an eluent, yielding compound DCzP as a white solid.

DCzP: Yield: 27%, ¹H-NMR (CDCl₃, 400 MHz) δ (*ppm*); 8.36 (d, J = 8.0 Hz, 2H), 8.28 (m, 4H), 8.15 (d, J = 8.0 Hz, 2H), 8.06 (d, J = 9.2 Hz, 2H), 7.69 (d, J = 9.2 Hz, 2H), 7.35 (m, 8H), 7.05 (m, 4H).

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2.4. Fabrication of OLED Devices

OLED devices with two different structures were fabricated on an ITO coated glass. Under a pressure of 1×10^{-6} torr, all organic compounds such as 2-TNATA used as hole injection layer (HIL), α -NPD used as hole transport layer (HTL), DCzP used as emission layer (EML), BCP used as hole blocking layer (HBL), and Alq₃ used as electron transport layer (ETL) were deposited by thermal deposition at a rate of 1–2 Å/sec. Finally, 1.0 nm of LiF layer as an EIL and 120 nm of Al film as a cathode were vapor-deposited under the same pressure to yield OLED devices with the configurations of ITO (150 nm)/ α -NPD (30 nm)/DCzP (40 nm)/LiF (1 nm)/A1 (150 nm) (D1) and ITO (150 nm)/2-TNATA (15 nm)/ α -NPD (20 nm)/DCzP (40 nm)/BCP (15 nm)/Alq3 (10 nm)/LiF (1 nm)/Al (120 nm) (D2). The devices were sealed with a glass cap to protect from the air. The active area of the OLED was 2.0×2.0 mm².

2.5. Measurements

¹H NMR spectra of materials in CDCl₃ were recorded on a 400 MHz Bruker AM-400 spectrometer. Thermal properties of the synthesized compounds were obtained from TG/DTA SDT Q600 analyzer (TA Instrument). Mass spectral data were obtained from the Korea Basic Science

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Institute (Daegu) on a Jeol JMS 700 high resolution mass spectrometer. The ionization potential (Ip) was measured using a photoelectron spectrometer (Riken Keiki, AC-2). UV-visible absorption spectra were obtained by using a Varian Easy 50 Conc spectrophotometer. The photoluminescence (PL) spectra excited by a 150-W Xe lamp were monitored with a Jasco FP-6500 spectrofluorometer. A spectroradiometer (Photo Research, PR-650) was used to determine the electroluminescence (EL) spectra, luminance, and Commission International de l'Eclairage (CIE) coordinates of the OLED devices. The current densityvoltage characteristics were recorded by a computer linked to a source meter (Keithley 2400).

3. RESULTS AND DISCUSSION

As illustrated in Scheme 1, the two carbazole-substituted pyrene derivatives, MCzP and DCzP, were synthesized by Ullman C–N coupling reaction at the presence of copper as a catalyst and potassium carbonate as a base in a refluxing nitrobenzene under nitrogen atmosphere.³⁰ These synthesized compounds are composed of a pyrene moiety as an electron acceptor (A) and substituted carbazole as an electron donor (D). These molecules are in D–A shape, therefore, in which high charge mobility and quantum yield can be anticipated.^{31, 32} All the synthesized compounds were soluble in common organic solvents such as dichloromethane and THF.

The UV-Vis absorption and PL spectra of the synthesized materials were measured in dichloromethane and the resulting data were presented in Figure 1. The absorption bands of $\pi \rightarrow \pi^*$ transition derived mainly from the central pyrene derivatives were showed at 317 and 334 nm for MCzP and at 327 and 340 nm for DCzP. These bands were slightly shifted to longer wavelength than those



Scheme 1. Synthetic route to MCzP and DCzP.

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Fig. 1. Normalized optical absorption and PL spectra of the synthesized compounds.

of the pyrene (313 and 329 nm)³³ in dichloromethane because of increased conjugation length through substituted carbazole moieties on the pyrene ring. Additional absorption bands around 350 and 375 nm of MCzP and DCzP might be originated from the absorption of carbazole moieties incorporated to the pyrene group.³⁴ In dichloromethane, MCzP and DCzP exhibited blue emission at 426 and 438 nm, respectively, different from those of the pyrene ($\lambda_{em, max} = 356, 400, \text{ and } 410 \text{ nm}$).³³ This red-shifted emission in PL spectra might be also explained by the same reason as mentioned in absorption spectrum. The fluorescence quantum yield (Φ), measured in THF using quinone sulfate as a standard material ($\Phi = 1$),^{35, 36} was found to be 0.69 and 0.78 for MCzP and DCzP, respectively. We also synthesized 9,9',9"-(pyrene-1,3,6trivl)tris(9H-carbazole) (TCzP), consisting of a pyrene substituted with three carbazole groups. Mass analysis (FAB-MS) results showed TCzP as a main product with a small amount of DCzP contamination, due to difficulties in the purification. Crude TCzP product exhibited yellowgreen emission under the irradiation of UV lamp emitting *i*-line (365 nm).

For application to OLEDs, thermal stability of materials is one of prerequisites for the device stability and life time. Thermal properties of MCzP and DCzP were measured by using thermogravimetric analysis (TGA) and differential scanning calorimetry analysis (DSC). By the thermogravimetric analysis, it was ascertained that the two compounds were very stable in nitrogen atmosphere, with the thermal decomposition temperature (T_d) at 403 and 426 °C, for the MCzP and DCzP, respectively. It means that the compounds are stable enough to endure a temperature of the vacuum vapor deposition.

Ionization potential was investigated by photoelectron spectrometer (Riken Keiki, AC-2) and was measured to be 5.96 eV for MCzP and 5.56 eV for DCzP, respectively. Optical band gap energy was estimated from UV-Vis absorption edge using the theory reported by Burrows et al.,³⁷ and then electron affinity was calculated. Although



Fig. 2. Energy band diagram of MCzP, DCzP and TCzP.

the ionization potential and electron affinity of TCzP were not considered in this paper due to the purity of the material, the crude TCzP exhibited a yellow green emission around 541 nm, as shown in Figure 2. The ionization potential and electron affinity were decreased with increasing the number of the electron donating carbazole group substituted on the pyrene. For example, the ionization potential was changed from 5.95 eV to 5.56 eV, and the electron affinity was decreased from 2.72 eV to 2.58 eV. It appears that the electron donating carbazole substituents on the pyrene play a role in decreasing both the ionization potential and electron affinity of these molecules. It is also interesting to notice that the optical band gap energy of DCzP were substantially lower than that of MCzP, which might be more increased electron delocalization by accompanying with the number of the electron donating carbazole substituents on the pyrene. As the results, MCzP and DCzP showed blue emissions at 426 nm and 438 nm, respectively, and TCzP exhibited a yellow green emission around 541 nm.

A quantum chemical calculation method, the CAChe WS pro 7.5 for Windows using the PM3 method was employed to simulate the HOMOs and LUMOs of the MCzP, DCzP, and TCzP, and their resulting HOMO and LUMO energy structure are shown in Figure 3. The calculated results showed that HOMO is dominated by the pyrene with a small contribution from the carbazole units, and the LUMO is entirely dominated by the π^* orbital of the pyrene ring. The energy levels of HOMO for the MCzP, DCzP, and TCzP molecules were 6.28 eV, 6.05 eV and 5.67 eV, respectively, and the LUMO values were calculated to be 3.20 eV, 3.08 eV and 2.74 eV, respectively. The results well fit with above mentioned experimental results in which the energy band gap decreased with the adding of the carbazole moieties, and the electron affinity decreased as shown in Figure 2. The optical, thermal and electrochemical properties of the synthesized compounds are listed in Table I.

We selected DCzP to investigate electroluminescent (EL) performance because of its better quantum yield and suitable band gap energy. The double-layer OLED device



Fig. 3. The calculated HOMO and LUMO for MCzP, DCzP and TCzP. Occupied orbitals corresponding to HOMO are displayed as blue and green color, while empty orbitals corresponding to LUMO are red and yellow. Blue and green color for HOMO indicate positive and negative (± 0.01 a.u.) values of the wave function, respectively. The case of red and yellow color for LUMO is the same with those of HOMO.

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Table I. Physical properties of the synthesized compounds in this study.									
Compound	$T_{\rm d}$ (°C)	$\lambda_{\rm abs,max} ({\rm nm})^a$	$\lambda_{\rm em,max} ({\rm nm})^b$	FWHM (nm) ^c	$\Phi \ (\%)^d$	HOMO (eV) ^e	LUMO (eV) ^f	Band gap energy (eV) ^g	
MCzP DCzP	403 426	334 340	426 438	54 55	0.69 0.78	5.95 5.56	2.72 2.58	3.23 2.98	

^{*a*}Maximum wavelength of absorption. ^{*b*}Maximum wavelength of PL. ^{*c*}Full width at half maximum of PL spectrum. ^{*d*}Using quinone sulfate ($\Phi = 1$) as a standard. ^{*e*}Determined by photoelectron spectrometer. ^{*f*}LUMO = HOMO – band gap energy. ^{*g*}Band gap energy was estimated from the absorption edge.

of ITO (150 nm)/a-NPD (30 nm)/DCzP (40 nm)/LiF (1 nm)/Al (150 nm) (D1) was fabricated, in which α -NPD was used as HTL and DCzP was used as EML. Also, non-doped multi-layer device with the structure of ITO (150 nm)/2-TNATA (15 nm)/α-NPD (20 nm)/DCzP (40 nm)/BCP (15 nm)/Alq₃ (10 nm)/LiF (1 nm)/Al (120 nm) (D2) was fabricated. The energy diagram of the OLED devices is illustrated in Figure 4. When the bias potential was applied to the electrode, the D1 and D2 emitted a bright blue luminescence with emission maxima at 492 nm (CIE coordinates of x = 0.17, y = 0.21) and 488 nm (x = 0.17, y = 0.27) as shown in Figure 5, respectively. These emission maxima were red-shifted by 57 nm and 53 nm, respectively, compared to the PL emission maximum of DCzP. The red-shifted EL emission can be attributed to its aggregation-induced emission from the



Fig. 4. Energy diagram of the OLED device D1 (a) and D2 (b).



Fig. 5. Normalized EL spectra derived from D1 (a) and D2 (b), recorded at 13 and 15 V, respectively.

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Fig. 6. Current density and luminance curves of D1 (a) and D2 (b) as a function of applied voltage.

emissive layer.³⁸ The I–V–L characteristics of D1 and D2 are shown in Figure 6. The D1 showed turn-on voltage of about 6 V and the luminance of 840 cd/m² at 13 V. It was considerable that overall EL performance of D1 was not effective for DCzP as a bifunctional molecule, presumably due to large electron and hole injection barrier from cathode and anode, respectively. On the other hand, the D2 showed a turn-on voltage at 4 V, maximum luminescence of 7560 cd/m² at 15 V and high current efficiency of 3.67 cd/A. The multilayer device exhibited significantly improved performances compared with the double-layer device, by incorporating Alq₃ as ETL and BCP as HBL. Such results indicate that additional improvement of EL performance in the double-layer OLED should be possible with further optimization of the device structure.

4. CONCLUSION

We synthesized two D–A type molecules, MCzP and DCzP, based on the electron accepting pyrene moiety with the electron donating carbazole substitutents, whose structures had asymmetrical pyrene (MCzP) with monocarbazole substituent and symmetrical pyrene (DCzP) with di-carbazole substituent. These D–A molecules might help in providing dual-purpose role as the blue emission and charge transport material in the OLEDs. Studies on absorption, emission and electrochemical behavior indicated that the substitution of the donor carbazole units on the pyrene rings was found to influence optical and electrochemical properties of these materials. Non-doped blue electroluminescent devices, D1 and D2, containing DCzP showed an Synthesis and Electro-Optical Properties of Carbazole-Substituted Pyrene Derivatives

emission at $\lambda_{\text{max}} = 492$ nm and 488 nm, respectively, confirming that DCzP was a potential bright blue emitter for OLEDs.

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