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New limb structured blue light emitting materials for OLEDs

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ABSTRACT

We designed new three limb structured anthracene derivatives, PNA, NNA and FNA, which are composed of an anthracene core and naphthalene units at the 9,10-positions of anthracene and phenyl, naphthalene and fluorene units at the 2,3-positons of anthracene. The effect of the introduced limbs on the 2,3,9,10-positions of anthracene was studied. The doped EL devices using ADN as a host and (3% PNA or NNA or FNA) as a dopant showed similar maximum quantum efficiency of 3.9%–4.3% with high color purity of (0.15, 0.13).

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1. Introduction

Organic light emitting devices (OLEDs) have been attracting considerable attention on the basis of their potential application in flat-panel displays [1–3]. During the past several years, much progress has been made in this field [4–10]. Great efforts have been made to develop high performance materials with desirable materials for developing marketable OLEDs. Many new materials with RGB (red, green, blue) emission have been developed to meet the requirements of full-color displays.

Efficient blue-light OLEDs are of particular interest, because they are desired for use as blue light sources in full-color display applications; furthermore, they can also serve as hosts for exothermic energy transfer to lower-energy fluorophores to realize white-light OLEDs [11–18].

Anthracene derivatives have been extensively studied and developed as light emitting materials in OLEDs because of their interesting photoluminescence (PL) and electroluminescence (EL) properties. Moreover, it has been also reported that the anthracene derivatives have ambipolar transporting property, leading high efficient devices [19–23].

Recently, we reported that the whole molecule becomes highly twisted by introducing bulky substituents at the 9- and 10-positions of anthracene. Thus, the fluorescence – quenching interactions caused by aggregates can be suppressed to some extent and non-radiative energy decay can be reduced [8,9,22–26].

Here, we designed new limb-structured blue light emitting materials, which are composed of an anthracene main core, naphthalene units at the 9,10-positions of anthracene and phenyl or naphthalene or fluorene units at the 2,3-positions of anthracene. We studied the effect of the introduced limbs on the 2,3,9,10-positions of anthracene. In particular, it is expected that the incorporated limbs on the 2,3-positions are highly twisted to the anthracene main unit and result in amorphousness of the molecule and reduced the intermoleular interaction. Moreover, despite that the new designed molecular structure is composed of an aromatic unit, the unique twisted limb structure of the molecule leads to increased solubility. Thus, we expect that the new limb structured blue light emitting materials will be high efficient and stable blue light emitting materials.





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2.1. Materials

All starting materials were purchased from Aldrich and Strem and were used without further purification.

2.2. Instrument

¹H-NMR spectra were recorded using a Bruker Avance-300 MHz FT-NMR spectrometer, and chemical shifts were reported in ppm units with tetramethylsilane as internal standard. FT-IR spectra were recorded using a Bruker IFS66 spectrometer. Thermogravimetric analysis (TGA) was performed under nitrogen using a TA instruments 2050 thermogravimetric analyzer. Differential scanning calorimeter (DSC) was conducted under nitrogen using a TA instrument DSC Q10. The both samples were heated at a rate of 10 °C/min. UV–visible spectra and photoluminescence (PL) spectra were measured by Shimadsu UV-1065PC UV–visible spectrophotometer and Perkin Elmer LS50B fluorescence spectrophotometer, respectively. The electrochemical properties of the materials were measured by cyclic voltammetry using an Epsilon C3 in a 0.1 M 385

solution of tetrabutyl ammonium perchlorate in acetonitrile. The organic electroluminescence (EL) devices were fabricated using successive vacuum deposition of ITO/N,N'-diphenyl-N,N'-bis-[4-(phenyl-*m*-thonyl-amino)-phenyl]-biphenyl-4,4'-diamine (DNTPD, 70 nm)/1,4-bis[(1-naphthylphenyl)-amino]biphenyl (α -NPD, 30 nm)/ (PNA or NNA or FNA, 20 nm) or (PNA or NNA or FNA doped ADN, 3%, 20 nm)/2.2'.2"-tris(8-hvdroxyguinoline) aluminium (Alg₃, 40 nm)/ LiF/Al. In these devices, ITO (indium tin oxide) and Al were the anode and the cathode. The ITO glass with a sheet resistance of about 10 Ω was etched for the anode electrode pattern and cleaned in ultrasonic baths of isopropyl alcohol and acetone. The overlap area of Al and ITO electrodes is about 4 mm². A UV zone cleaner (Jeilight Company) was used for further cleaning before vacuum deposition of the organic materials. Vacuum deposition of the organic materials was carried out under a pressure of 2×10^{-7} torr. The deposition rate for organic materials was about 0.1 nm/s. The evaporation rate and the thickness of the film were measured with a quartz oscillator. OLED performance was studied by measuring the current-voltage-luminescence (I-V-L) characteristics, EL, and PL spectra at room temperature. I-V-L characteristics and CIE color coordinates were measured with a Keithley SMU238 and Spectrascan PR650. EL spectra of the devices were measured utilizing



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Scheme 1. Synthetic route to PNA, NNA and FNA.



Fig. 1. Calculated stereostructures and frontier orbitals of PNA, NNA, and FNA.

a diode array rapid analyzer system (Professional Scientific Instrument Corp.) Fluorescence spectra of the solutions in chloroform were measured using a spectrofluorimeter (Shimadzu Corp.).

2.3. 3,4-Dibromothiophene-1,1-dioxide (1)

Trifluoroacetic acid (1101 mmol, 153 mL) was slowly added into the 30% H₂O₂ (60 mL) at 0 °C. After addition of trifluoroacetic acid, the solution of 3,4-dibromothiophene (70.40 mmol, 8.14 mL) in CH₂Cl₂ (90 mL) was added in the mixture. The mixture was stirred for 3 h at room temperature. The reaction was worked up by adding Na₂CO₃ aqueous solution at 0 °C. The organic layer was separated by methylene dichloride, and the solvent was evaporated. The crude product was recrystallized in co-solvent of CH₂Cl₂ and ethanol Yield: (12.4 g, 49%). Mp 103–104 °C. IR (KBr): 1309, 1146 cm⁻¹ (S=O), 3015 cm⁻¹ (sp² C–H). ¹H NMR (300 MHz, CDCl₃, ppm): δ 6.85 (1).

2.4. 2,3-Dibromoanthraquinone (2)

3,4-Dibromothiophene-1.1-dioxide (5.65 g, 20.62 mmol) and 1,4-naphthoquinone (16.31 g, 103.13 mmol) were dissolved in

350 mL of acetic acid. The reaction mixture was refluxed with for 48 h. The reaction was worked up by adding Na₂CO₃ aqueous solution at 0 °C. The organic layer was separated by methylene dichloride, and the solvent was evaporated. The crude product was purified by column chromatography with n-hexane/CH₂Cl₂ (1:1, v/v) as eluent. Yield: (3.2 g, 40.65%). Mp 278–279 °C. IR (KBr):

Table 1					
Physical	data	of	com	poui	nds.

Comp	$\lambda_{abs}/(nm)^a$	$\lambda_{em}/(nm)^{a}$		HOMO	LUMO	Eg	
	Solution	Solution Film		Film	(eV)	(eV)	(eV)
PNA	291, 351,	372, 394,	438	452	5.65	2.74	2.91
	369, 389, 411	416					
NNA	260, 275,	373, 392,	432	457	5.60	2.78	2.82
	318, 371, 392,	415					
	413						
FNA	260, 287, 321,	375, 394,	448	468	5.63	2.85	2.78
	372, 394, 416	414					
ADN	260, 360, 377,	374, 392,	442	461	5.66	2.65	3.01
	398	414					

1681 cm⁻¹ (C=O), 3075 cm⁻¹ (sp² C–H), 1656 cm⁻¹ (C=C). ¹H NMR (300 MHz, CDCl₃, ppm): δ 8.49 (2), 8.38–8.40 (2), 7.86–7.89 (2).

2,3-Dibromoanthraquinone (2.10 g, 5.74 mmol) were dissolved

in 70 mL of THF. Naphthylmagnesium bromide (10.2 mL) was added to the reaction mixture at 0-15 °C. The mixture was stirred for 3 h

at room temperature. The organic layer was extracted with diethyl

ether. The crude product was purified by column chromatography

2.5. 2,3-Dibromo-9,10-dihydroxy-9,10-dinaphthylanthracene (3)

with n-hexane/CH₂Cl₂ (1:1, v/v) as eluent. Yield: (1.99 g, 68%). Mp 226 °C. IR (KBr): 3521 cm⁻¹ (–OH), 3017–3029 cm⁻¹ (sp² C–H), 1582 cm⁻¹ (C=C). ¹H NMR(300 MHz, CDCl₃, ppm): δ 8.52(2), 8.38–8.40 (4), 7.86–7.89 (5), 6.94 (2).

2.6. 2,3-Dibromo-9,10-dinaphthylanthracene (4)

2,3-Dibromo-9,10-dihydroxy-9,10-dinaphtylanthracene (2.4 g, 3.86 mmol), NaH₂PO₄·H₂O (4.09 g, 38.56 mmol) and KI (1.92 g, 11.58 mmol) were dissolved in 50 ml of acetic acid. After the



Fig. 2. TGA thermogram and DSC thermogram of (a) PNA, (b) NNA, and (c) FNA in nitrogen atmosphere at a scan rate 10 °C/min.

reaction mixture was refluxed for 4 h, the reaction was worked up by water. The crude solid product was several time filtered by water. Yield: (2.02 g, 83%), Mp 298 °C. IR (KBr): 3011–3043 cm⁻¹ (sp² C–H), 1752–1734 cm⁻¹ (C=C). ¹H NMR (300 MHz, CDCl₃, ppm): δ 8.07–7.96 (10), 7.72–7.67 (8), 7.64–7.59 (2).

2.7. 2,3-Diphenyl-9,10-dinaphtylanthracene (PNA) (5a)

2,3-Dibromo-9,10-dinaphthalene anthracene (1 g, 1.7 mmol) and phenyl boronic acid (0.93 g, 7.64 mmol) and 2M K₂CO₃ (1.94 g) was added to the 50 mL of THF. After Pd(PPh₃)₄ (0.0038 g, 3 mol/%) was added to the mixture, the mixture was stirred at 90 °C for 48 h. After the reaction was worked up by adding water, the organic layer was extracted with chloroform. After the solvent was evaporated, the crude product was purified by column chromatography using n-hexane/ethyl acetate (5:1, v/v) as eluent. Yield: (0.86 g, 81%). Mp 342 °C. IR (KBr): 3073–3023 cm⁻¹ (sp² C–H), 1732–1715 cm⁻¹ (C=C). ¹H NMR (300 MHz, CDCl₃, ppm): δ 8.19–7.81 (10), 7.68–7.60 (4), 7.58–7.43 (4), 7.40–7.32 (2), 7.23–7.02 (10). ¹³C NMR (300 MHz, CDCl₃, ppm): δ 141.3, 138.7, 137.1, 136.4, 133.4, 132.8, 130.6, 130.2, 129.9, 129.5, 129.4, 128.3, 128.2, 127.9, 127.7, 127.1, 126.4, 126.2, 125.2. EI-MS: *m/z* 582.

2.8. 2,3,9,10-Tetranaphtylanthracene (NNA) (5b)

2,3-Dibromo-9,10-dinaphthalene anthracene (1 g, 1.7 mmol) and 2-naphthalene boronic acid (1.3 g, 7.64 mmol) and 2M K₂CO₃ (1.94 g) was added to the 50 mL of THF. After Pd(PPh₃)₄ (0.0038 g, 3 mol/%) was added to the mixture, the mixture was stirred at 90 °C for 48 h. After the reaction was worked up by adding water, the organic layer was extracted with chloroform. After the solvent was evaporated, the crude product was purified by column chromatography using n-hexane/ethyl acetate (5:1, v/v) as eluent. Yield: (0.82 g, 84%). Mp 328 °C. IR (KBr): 3072–3020 cm⁻¹ (sp² C–H), 1712–1701 cm⁻¹ (C=C). ¹H NMR (300 MHz, CDCl₃, ppm): δ 8.09 (4), 7.98–7.82 (6), 7.76–7.74 (6), 7.69–7.43 (8), 7.4 (2), 7.26–7.23 (6), 7.03 (2). ¹³C NMR (300 MHz, CDCl₃, ppm): δ 139.1, 133.5, 129.5, 128.9, 128.3, 128.2 (2), 128.1 (2), 127.9, 127.5, 127.1, 127.0, 126.4, 126.2, 125.8, 125.7, 125.3. EI-MS: *m/z* 682.

2.9. 2,3-bis(9,9-dimethyl-9H-fluoren-2-yl)-9,10-dinaphthylanthracene (FNA) (5c)

2,3-Dibromo-9,10-dinaphthalene anthracene (1 g, 1.7 mmol) and 2-(9,9-dimethyl-9H-fluoren-2-yl)-4,4,5,5-tetramethyl-1,3,2dioxaborolane (1.96 g, 7.64 mmol) and 2M K₂CO₃ (1.94 g) was added to the 50 mL of THF. After Pd(PPh₃)₄ (0.0038 g, 3 mol/%) was added to the mixture, the mixture was stirred at 90 °C for 48 h. After the reaction was worked up by adding water, the organic layer was extracted with chloroform. After the solvent was evaporated. the crude product was purified by column chromatography using n-hexane/ethyl acetate (5:1, v/v) as eluent. Yield (0.95 g, 88%). Mp 331 °C. IR (KBr): 3068–3022 cm⁻¹ (sp² C–H), 2786–2698 cm⁻¹ (sp³ C–H) 1788–1719 cm⁻¹ (C=C), 1375–1373 cm⁻¹ (–CH₃). ¹H NMR (300 MHz, CDCl₃, ppm): δ 7.98 (2), 7.82 (2), 7.67-7.45 (16), 7.41–7.38 (2), 7.36–7.21 (10), 7.01 (2), 1.08 (12). ¹³C NMR (300 MHz, CDCl₃, ppm): 153.6, 152.7, 140.5, 139.2, 136.5, 137.4, 137.1, 136.4, 133.6, 132.8, 130.5, 129.4, 128.5, 128.2, 128.0, 127.6, 127.1, 126.8, 126.6, 126.0, 125.1, 125.0, 122.1, 119.8, 119.4, 46.5, 26.6. EI-MS: *m*/*z* 814.

3. Results and discussion

The synthetic scheme of the three limb structured anthracene derivatives is shown in Scheme 1.

2,3-Dibromoanthraquinone was obtained by oxidation and the Diels—Alder reaction following degradation of SO₂. 2,3-Dibromo-9,10-dihydroxy-9,10-dinaphthylanthracene was synthesized by reaction of 2-naphthyl magnesium bromide and 2,3-dibromoanthraquinone. 2,3-Dibromo-9,10-dinaphthylanthracene obtained through reduction of 2,3-dibromo-9,10-dihydroxy-9,10-dinaphthylanthracene was reacted with phenyl boronic acid, 2-naphthyl boronic acid, and 9,9-diethylfluoren-2-ylboronic acid to afford 2,3-diphenyl-9,10-dinaphthylanthracene (PNA), 2,3,9,10-tetranaphthylanthracene (NNA), and 2,3-difluorenyl-9,10-naphthylanthracene (FNA), respectively.

The structures of new limb structured materials were confirmed by various spectroscopic methods such as FT-NMR, IR and mass spectroscopies. Theoretical calculations using Spartan08 software in order to fully optimize the molecular structure, were carried out for the characterization of 3-dimensional structures and the energy densities of the HOMO and LUMO states of each materials. Fig. 1 shows the stereostructures and the energy densities of the HOMO and LUMO states of materials derived from the calculations.

The limbs attached at the 2,3,9,10-positions of anthracene are highly twisted toward the anthracene backbone at angles of 88.73–80.40° for the 9,10-positions and 122.82–123.54° for the 2,3-positions. From this calculation, it is expected that the newly obtained limb-structured materials have non-coplanar structures that bear bulky substituents. These substituents disrupt the intermolecular interaction and suppress the problematic recrystallization,



Fig. 3. Energy level diagrams of devices using PNA as emitting material and ADN-3% PNA as emitting material.

which would eventually reduce self-aggregation, and they thus which in turn, improve the morphological stability of the thin film of OLED device.

The absorption spectra of the compounds in chloroform solution reveal the characteristic vibronic patterns from the $\pi - \pi^*$ transitions of the isolated anthracene group (λ_{max} are 360, 378, and 397 nm). The absorption spectra of PNA, NNA and FNA in film as well as in solution are almost similar to those of 9,10-dinaphthylanthracene (ADN) while the absorption edges are slightly red-shift compared with ADN.

The absorption and emission spectra of PNA, NNA, and FNA in solution and solid films are summarized in Table 1.

A slight emission red-shift is observed for the film states, which may be accounted for by the different dielectric constants of the media^[20]. The PL emission of PNA, NNA, and FNA display blue emission and exhibit high PL quantum yield in solution; $68 \pm 10\%$, $65 \pm 10\%$, and $59 \pm 10\%$, respectively.

The relevant energy levels for these materials are listed in Table 1. The band gap energies were estimated from the onset of the absorption spectra in dilute solution. The results indicate that PNA, NNA, and FNA have low band-gap energies compared with that of ADN. Although the similar energy band gap, band gap energy order is PNA > NNA > FNA. From the results, it is suggested that the introduction of aromatic substituents at the 2,3-positions leads to



Fig. 4. Current density- voltage-luminescence and current density efficiency characteristics of the devices (a) using PNA, (b) using NNA, (c) using FNA, (d) using ADN-3% PNA, (e) using ADN-3% NNA (f) using ADN-3% FNA.

Table 2	
Key device performance parameters and EL em	hission characteristics

Host	Dopant	wt%	V	А	J (mA/cm ²)	LE (Cd/A)	PE (lm/W)	EQE	L (Cd/m ²)	CIE	
										x	у
PNA	X		5.17	0.0004	10	2.96	1.80	2.74	295.7	0.15	0.14
NNA	Х		4.92	0.0004	10	2.98	1.90	2.68	297.6	0.15	0.14
FNA	Х		4.99	0.0004	10	4.33	2.73	2.81	433.1	0.16	0.22
ADN	PNA	3	5.59	0.0004	10	3.72	2.09	3.62	372.4	0.15	0.13
ADN	NNA	3	5.60	0.0004	10	4.44	2.49	4.16	444.2	0.15	0.14
ADN	FNA	3	5.78	0.0004	10	4.64	2.52	4.32	463.7	0.15	0.14

increased conjugation length and decreased band gap, although they render the molecules non-coplanar amorphous due to steric hindrance. The experimental results are consistent with frontier orbitals of the materials. (Fig. 1) In particular, FNA has expanded conjugation through fluorene.

The electrochemical behaviors of PNA, NNA, and FNA were investigated by cyclic voltammetry (CV). The HOMO, LUMO, and band gap (E_g) are summarized in Table 1. The oxidation peak potentials are high, $E_{ox} = 1.2$ for PNA, $E_{ox} = 1.25$ for NNA, and $E_{ox} = 1.23$ for FNA. The HOMO levels of PNA, NNA, and FNA are 5.65 eV, 5.6 eV, and 5.63 eV, respectively.

Thermal properties of the obtained materials were evaluated via a thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) (Fig. 2).

The TGA (10 °C/min) showed that the three materials were highly stable in nitrogen, with a thermal decomposition temperature (T_d) of 382, 410, and 410 nm for PNA, NNA, and FNA, respectively. In the DSC, T_g was not observed for PNA and NNA, while FNA has a T_g of 158 °C. Additionally, the materials showed melting temperatures higher than 310 °C. These results clearly reveal that the materials possess good thermal stability, a very desirable characteristics for OLEDs stability.

Non-doped and doped OLED devices were fabricated using the synthesized materials as the emitting layer in the following structure: ITO/*N*,*N*'-diphenyl-*N*,*N*'-bis-[4-(phenyl-*m*-thonyl-amino)-phenyl]-biphenyl-4,4'-diamine (DNTPD, 70 nm)/1,4-bis[(1-naphthylphenyl)-amino]biphenyl (α -NPD, 30 nm)/(PNA or NNA or FNA, 20 nm) or (PNA or NNA or FNA doped ADN, 3%, 20 nm)/2,2,'2"-tris(8-hydroxyquinoline) aluminium (Alq3, 40 nm)/LiF/Al. In these devices, ITO (indium tin oxide) and Al were the anode and the cathode, respectively. DNTPD was the hole injection layer (HIL); α -NPD was the hole transporting layer (HTL); Alq3 was the electron

Fig. 5. EL spectra of the devices.

transporting layer (ETL) and the newly prepared limb-type anthracene derivatives were used as the emitting layer (EML) (Fig. 3).

Current density-voltage-luminescence, current density-efficiency characteristics of the devices are shown in Fig. 4.

Key device performance parameters and EL emission characteristics are summarized in Table 2 and Fig. 5.

The non-doped device using PNA exhibits the maximum external quantum efficiency of 2.74% (2.95 cd/A) with color purity of (0.15, 0.13) and the non-doped device using NNA exhibits maximum quantum efficiency of 2.67% (2.97 cd/A) with color purity of (0.15, 0.14) while the non-doped device using FNA shows maximum quantum efficiency of 2.8% (4.33 cd/A) with color purity of (0.16, 0.22). The non-doped devices using FNA with naphthalene and fluorene limbs showed slightly lower color purity than those of PNA with naphthalene and phenyl limbs or NNA with naphthalene limbs while PNA and NNA showed similar quantum efficiency and color purity. These results can be explained by the extended conjugation of FNA. However, the newly designed limb structured anthracene derivatives have better color purity than those of anthracene derivatives with 2,6 substituents [27]. The results indirectly show that the limb structured anthracene derivatives have a twisted structure because of effective steric hindrance of the limbs at the 2,3-positions^[25]. Furthermore, the non-doped devices using PNA and NNA showed higher color purity than that of reported ADN without the substituents at the 2,3-positions. It appears that the 3-dimensionally highly twisted limb structured PNA and NNA can not be easily stacked, leading to inhibited intermolecular interaction.

The doped device using ADN as a host and (3% PNA or NNA or FNA) as a dopant showed similar maximum quantum efficiency of (3.9%-4.3%) with high color purity of (0.15, 0.13) regardless of the dopant. In particular, FNA with naphthalene and fluorene limbs showed maximum quantum efficiency of 4.3% (4.6 cd/A) with high color purity of (0.15, 0.13).

4. Conclusion

We designed three new limb structured anthracene derivatives. PNA, NNA, and FNA, which are composed of an anthracene core and naphthalene units at the 9,10-positions of anthracene and phenyl, naphthyl or fluorene units at the 2,3-position of anthracene. The theoretical calculations supports that the synthesized materials have 3-dimensionally highly twisted non-coplanar structures due to steric hindrance of the introduced limbs. The maximum PL emission intensity was obtained by excitation at corresponding limbs at the 2,3,9,10-positions of anthracene due to intramolecular energy transfer from the limbs to the anthracene core. The introduced limbs at the 2,3-positions induces steric hindrance, although FNA with fluorene substituents has slightly increased conjugation length and decreased band gap. The non-doped blue EL devices using PNA, NNA, or FNA as an emitting material showed efficiency of 2.67%–2.8% and the doped EL devices using ADN as a host and (3% PNA or NNA or FNA) as a dopant showed similar maximum quantum efficiency of (3.9%-4.3%) with high color purity of (0.15, 0.13) regardless of the dopant.

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