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### New Hole Transporting Materials Based on Tetraphenylbenzene and Aromatic Amine Derivatives for OLEDs

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# New Hole Transporting Materials Based on Tetraphenylbenzene and Aromatic Amine Derivatives for OLEDs

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*Three different new hole transport compounds, namely, 2-[1-{4-(N,N-diphenylamino)phenyl}-3,4,5,6-tetraphenylbenzene-2-yl]5-N,N-diphenylaminopyridine (DPAP-TB), 2-[1-{4-(N-phenylnaphthalene-1-amino)phenyl}-3,4,5,6-tetraphenylbenzene-2-yl]5-(N-phenylnaphthalen-1-amino)pyridine (1-PNAP-TB) and 2-[1{4-(N-phenylnaphthalen-1-amino)phenyl}-3,4-,5,6-tetraphenylbenzene-2-yl]-5-(N-phenylnaphthalen-2-amino)pyridine (2-PNAP-TB) were synthesized. Optical and electronic properties were examined by UV-Visible absorption spectrum and cyclic voltammetry. Luminance efficiencies and external quantum efficiencies of DPAP-TB, 1-PNAP-TB and 2-PNAP-TB were 3.72, 2.82, 3.30 cd/A, and 1.29, 0.97, 1.12% at 10 mA/cm<sup>2</sup>, respectively. Synthesized materials exhibited high T<sub>g</sub> in the range of 118 to 133°C. These values are higher than that of NPB which is commonly used as a hole transporting material.*

**Keywords** Hole transporting materials; organic light-emitting diode

## Introduction

Organic light-emitting diodes (OLEDs) have been the subject of intensive investigation in the past few years because of their potential application as emissive elements for flat panel displays [1,2]. Generally, multi-layered devices comprised of charge transport and emitting layers exhibit higher electroluminescence (EL) efficiency than single-layer devices. This is because multi-layered devices balance hole and electron carriers, and then the exciton is formed in the middle of emitting layer [3]. Among multi-layers of OLED, charge transporting materials have played important roles of balancing hole and electron carriers, and hole transporting layer (HTL) and electron transporting layer (ETL) are used [4].

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For high performance HTL material, it needs to get high mobility as well as high glass transition temperature ( $T_g$ ) for long life time. In order to achieve two points, tetraphenyl moiety and aromatic amine moiety were introduced in new HTL compounds. Tetraphenyl moiety has twisted multi-phenyl groups and it can increase  $T_g$  based on multi-aromatic groups. Aromatic amine can support high mobility because of electron donating effect.

Three materials, 2-[1-{4-(N, N-diphenylamino)phenyl}-3, 4, 5, 6-tetraphenylbenzene-2-yl]5-N, N-diphenylaminopyridine (DPAP-TB), 2-[1-{4-(N-phenylnaphthalene-1-amino)phenyl}-3, 4, 5, 6-tetraphenylbenzene-2-yl]5-(N-phenylnaphthalen-1-amino)pyridine (1-PNAP-TB) and 2-[1{4-(N-phenylnaphthalen-1-amino)phenyl}-3, 4-,5, 6-tetraphenylbenzene-2-yl]-5-(N-phenylnaphthalen-2-amino)pyridine (2-PNAP-TB) include aromatic amine groups as an electron donor moiety. In particular, we analyzed dependence of the EL device characteristics on the position at which the naphthylphenyl amine or diphenylamine group is substituted. Then we compared EL properties with those of NPB, a commercialized HTL compound. The electronic and optical properties of these materials were characterized by cyclic voltammetry (CV), UV-visible and photoluminescence (PL) spectroscopies. Moreover, multilayer EL devices were fabricated using these materials as hole transporting materials [5].

## Experimental

### Syntheses

**{(4-Bromophenyl)ethynyl}trimethylsilane (2).** To a mixture of 1-bromo-4-iodobenzene (**1**) (3.0 g, 10.06 mmol), dichlorobis(triphenylphosphine)palladium (II) (28 mg, 0.04 mmol), and copper iodide (18 mg, 0.09 mmol) in triethylamine (30 ml) was added trimethylsilylacetylene (1.7 ml, 11.94 mmol), and stirred at 48°C for 2 hours. Solvent was evaporated under reduced pressure, and the residue was chromatographed on silica gel with hexane to give **2** (2.42 g, 95%) in a white solid.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  = 0.25 (s, 9H), 7.30(d, 2H), 7.40 (d, 2H). Anal. Calcd for  $\text{C}_{11}\text{H}_{13}\text{BrSi}$ : C, 52.17%; H, 5.17%. Found: C, 52.29%; H, 5.08%.

**1-Bromo-4-ethynylbenzene (3).** To a solution of **2** (2.0 g, 7.90 mmol) in methanol (100 ml) was added potassium fluoride (3.50 g, 37.19 mmol). The mixture was stirred at room temperature for 24 hours. Solvent was evaporated under reduced pressure, and the residue was extracted with dichloromethane ( $3 \times 30$  ml). The extracts were combined, and the solvent was evaporated to produce **3** (1.19 g, 83%) in a yellow solid.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  = 3.33 (s, 1H), 7.35 (d, 2H), 7.45 (d, 2H). Anal. Calcd for  $\text{C}_8\text{H}_5\text{Br}$ : C, 53.07%; H, 2.78%. Found: C, 53.18%; H, 2.90%.

**5-Bromo-2-{(4-bromophenyl)ethynyl}pyridine (4).** To a mixture of compound **3** (2.0 g, 11.05 mmol), dichlorobis(triphenylphosphine)palladium (II) (50 mg, 0.08 mmol), and copper iodide (18 mg, 0.09 mmol) in triethylamine (30 ml) was added 2,5-dibromopyridine (2.7 g, 11.39 mmol), and stirred at 48°C for 24 hours. Solvent was evaporated under reduced pressure, and the residue was chromatographed on silica gel with dichloromethane/hexane (1:1) to produce **4** (3.12 g, 84%) in a brown solid. Mp. 176°C.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  = 7.38–7.40 (d, 1H), 7.40–7.42 (m, 2H), 7.42–7.49 (m, 2H), 7.78–7.81 (m, 1H), 8.64 (d, 1H). Anal. Calcd for  $\text{C}_{13}\text{H}_7\text{NBr}_2$ : C, 46.32%; H, 2.09%; N, 4.16%. Found: C, 46.21%; H, 2.13%; N, 4.21%.

**2-{1-(4-Bromophenyl)-3, 4, 5, 6-tetraphenylbenzene-2-yl}-5-bromopyridine (5).** The mixture of **4** (800 mg, 2.37 mmol) and tetraphenylcyclopentadienone (1.0 g, 2.60 mmol)

in diphenyl ether (100 ml) was refluxed for 48 hours. Solvent was evaporated under reduced pressure, and the residue was chromatographed on silica gel with dichloromethane/hexane to afford **5** (1.16 g, 71%) in a brown solid. Mp. 331°C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz): δ = 6.70–6.90 (m, 22H), 6.80–7.10 (d, 2H), 7.29–7.30 (m, 2H), 8.20 (d, 1H). Anal. Calcd for C<sub>41</sub>H<sub>27</sub>NBr<sub>2</sub>: C, 71.01%; H, 3.93%; N, 2.02%. Found: C, 69.94%; H, 4.01%; N, 2.07%.

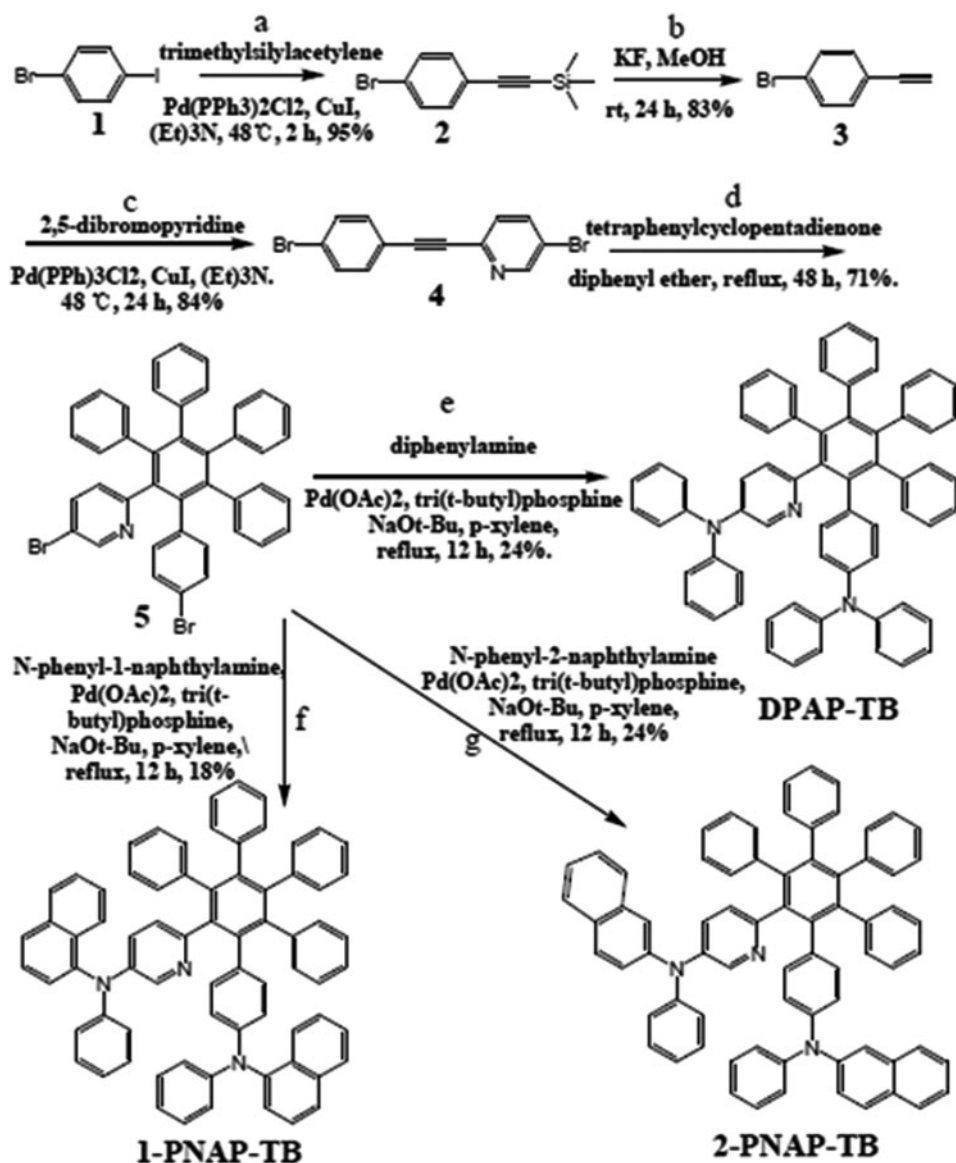
**2-[1-{4-(N, N-diphenylamino)phenyl}-3, 4, 5, 6-tetraphenylbenzene-2-yl]5-N, N-diphenylaminopyridine (DPAP-TB).** To a mixture of **5** (50 mg, 0.07 mmol), palladium (II) acetate (5 mg, 0.022 mmol), tri(t-butyl)phosphine (0.05 ml), and sodium tert-butoxide (0.10 g, 1.04 mmol) in p-xylene (10 ml) was added diphenylamine (40 mg, 0.18 mmol). The mixture was stirred at room temperature for 10 minutes, and then refluxed for 12 hours. After evaporation of the solvent under reduced pressure, the residue was chromatographed on silica gel with dichloromethane to give **DPAP-TB** (15 mg, 24%) in a white solid. Mp. 260°C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz): δ = 6.62–6.69 (d, 2H), 6.80–6.98 (m, 36H), 7.11–7.19 (m, 8H), 8.00 (d, 1H). Anal. Calcd for C<sub>65</sub>H<sub>47</sub>N<sub>3</sub>: C, 89.73%; H, 5.45%; N, 4.83%. Found: C, 86.66%; H, 5.49%; N, 4.85%.

**2-[1-{4-(N-phenylnaphthalene-1-amino)phenyl}-3, 4, 5, 6-tetraphenylbenzene-2-yl]5-(N-phenylnaphthalen-1-amino)pyridine (1-PNAP-TB).** To a mixture of **5** (100 mg, 0.144 mmol), palladium (II) acetate (5 mg, 0.022 mmol), tri(t-butyl)phosphine (0.05 ml), and sodium tert-butoxide (30 mg, 0.31 mmol) in p-xylene (20 ml) was added N-phenyl-1-naphthylamine (100 mg, 0.46 mmol). The mixture was stirred at room temperature for 10 minutes, and then refluxed for 12 hours. After evaporation of the solvent under reduced pressure, the residue was chromatographed on silica gel with dichloromethane/hexane (4:1) to give **1-PNAP-TB** (25 mg, 18%) in a white solid. Mp. 165°C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz): δ = 6.20–6.40 (d, 2H), 6.78–6.92 (m, 28H), 7.02–7.19 (m, 10H), 7.30–7.40 (m, 4H), 7.60–7.70 (m, 4H), 7.78–7.82 (m, 2H), 7.98 (d, 1H). Anal. Calcd for C<sub>73</sub>H<sub>51</sub>N<sub>3</sub>: C, 90.37%; H, 5.30%; N, 4.33%. Found: C, 90.24%; H, 5.37%; N, 4.39%.

**2-[1{4-(N-phenylnaphthalen-1-amino)phenyl}-3, 4, 5, 6-tetraphenylbenzene-2-yl]-5-(N-phenylnaphthalen-2-amino)pyridine (2-PNAP-TB).** To a mixture of **5** (1.00 g, 1.44 mmol), palladium (II) acetate (50 mg, 0.22 mmol), tri(t-butyl)phosphine (0.2 ml), and sodium tert-butoxide (0.3 g, 3.12 mmol) in p-xylene (20 ml) was added N-phenyl-2-naphthylamine (631 mg, 2.88 mmol). The mixture was stirred at room temperature for 10 minutes, and then refluxed for 12 hours. After evaporation of the solvent under reduced pressure, the residue was chromatographed on silica gel with dichloromethane/hexane (4:1) to give **2-PNAP-TB** (343 mg, 24%) in a white solid. Mp. 167°C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz): δ = 6.70–6.76 (d, 2H), 6.78–7.00 (m, 28H), 7.02–7.22 (m, 10H), 7.24–7.34 (m, 4H), 7.42–7.49 (m, 2H), 7.59–7.68 (m, 4H), 8.09 (d, 1H). Anal. Calcd for C<sub>73</sub>H<sub>51</sub>N<sub>3</sub>: C, 90.37%; H, 5.30%; N, 4.33%. Found: C, 90.29%; H, 5.41%; N, 4.30%.

## Measurements

The optical absorption spectra were obtained by HP 8453 UV-VIS-NIR spectrometer. Perkin Elmer luminescence spectrometer LS50 (Xenon flash tube) was used for photoluminescence (PL) spectroscopy. Glass-transition temperature (*T<sub>g</sub>*) and degradation temperature (*T<sub>d</sub>*) of the compounds were measured by differential scanning calorimetry (DSC) under nitrogen atmosphere using a DSC2910 (TA instrument) and thermogravimetric analysis (TGA) using SDP-TGA2960 (TA instrument). The redox potential of the synthesized materials was measured at the scanning rate of 50 mV/s by WBCS 3000 system with cyclic voltammeter (CV), and acetonitrile (AN) containing 0.1 M tetrabutylammonium perchlorate was used as an electrolyte when measuring. Indium tin oxide (ITO) was used

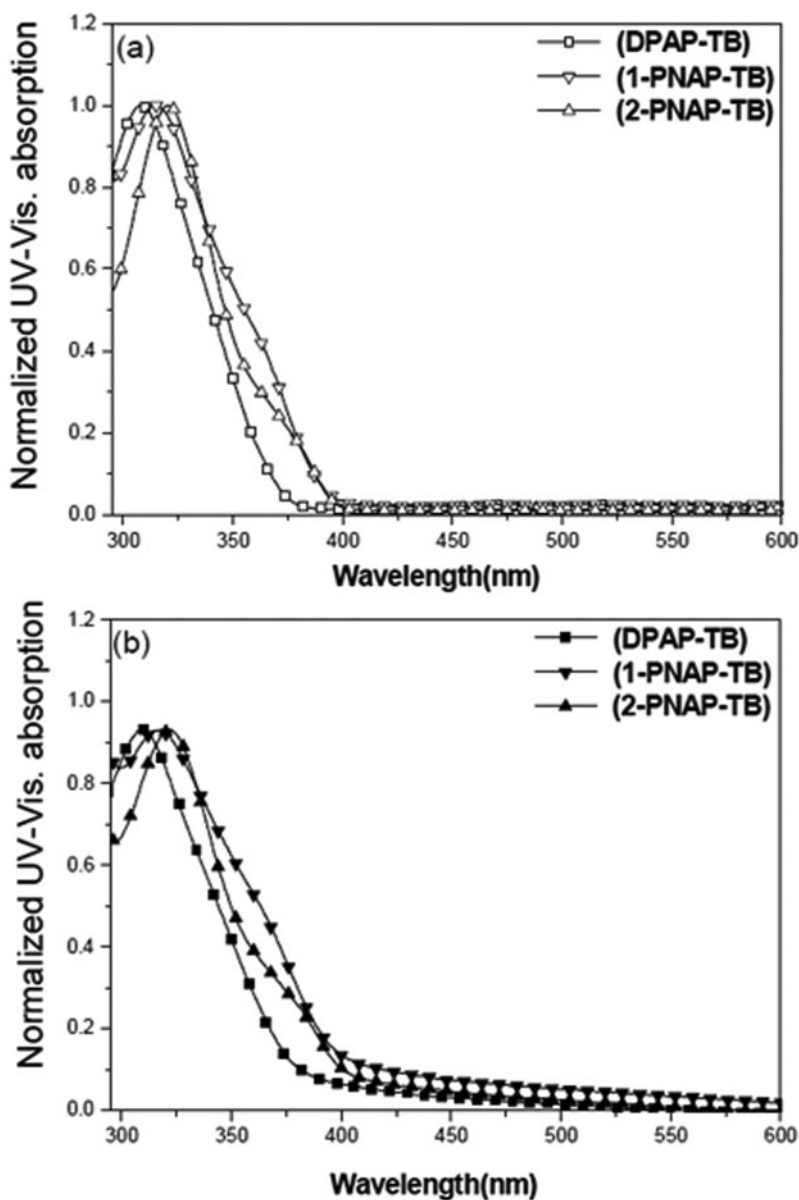


**Scheme 1.** Chemical structures and synthetic method of DPAP-TB, 1-PNAP-TB, and 2-PNAP-TB.

as a working electrode and saturated Ag/AgNO<sub>3</sub> as a reference electrode. Ferrocene was a reference compound for potential calibration. The current–voltage–luminance (I–V–L) characteristics of the fabricated EL devices were obtained by Keithley 2400 electrometer and light intensity was obtained by Minolta CS-1000 A, respectively.

### Preparation of OLED Device

EL devices of ITO/2-TNATA (60 nm)/synthesized materials or NPB (15 nm)/Alq<sub>3</sub> (70 nm)/LiF (1 nm) /Al (200 nm) were fabricated using 2-TNATA as a hole injection layer, NPB or synthesized materials as a hole transporting layer, Alq<sub>3</sub> as an electron



**Figure 1.** UV-Visible absorption spectrum: (a) toluene solution (b) evaporated film.

transporting and emitting layer, LiF as an electron injection layer. The organic layer was vacuum-deposited using thermal evaporation at a vacuum pressure of  $10^{-6}$  torr and the rate of deposition  $1 \text{ \AA/s}$  to give an emitting area of  $4 \text{ mm}^2$ , and the Al layer was continuously deposited under the same vacuum condition.

## Results and Discussion

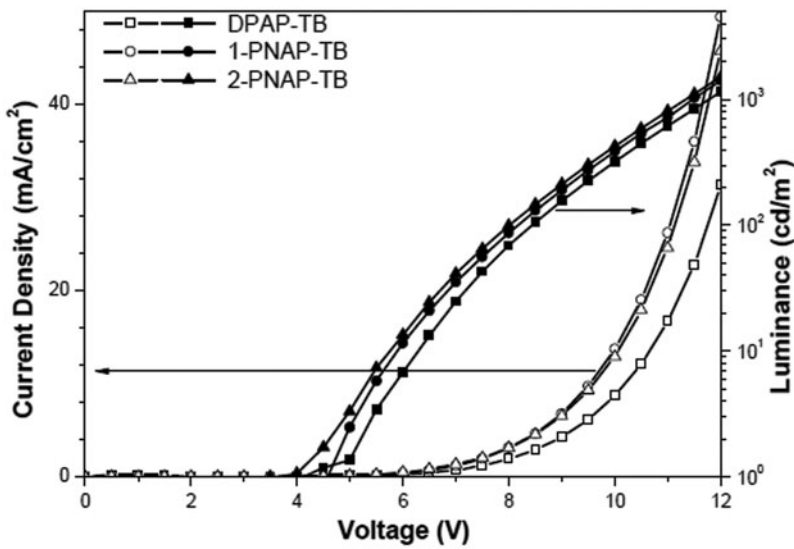
Scheme 1 shows the chemical structures of the synthesized hole transporting materials. UV-visible absorption (UV-Vis.) of the synthesized materials are described and summarized in Fig. 1 and Table 1. As shown in Fig. 1 and Table 1, UV-Vis. spectra

**Table 1.** Optical, electrical, and thermal properties of synthesized materials

Compounds	UV-Vis. <sub>max</sub> (nm)		HOMO (eV)	LUMO (eV)	E <sub>g</sub> (eV)	T <sub>g</sub> (°C) <sup>a</sup>	T <sub>d</sub> (°C) <sup>b</sup>
	Solution	Film					
DPAP-TB	309	309	5.27	1.97	3.30	118	394
1-PNAP-TB	314	316	5.31	2.18	3.13	133	396
2-PNAP-TB	320	322	5.25	2.16	3.09	125	400
NPB	339	348	5.34	2.40	2.94	104	378

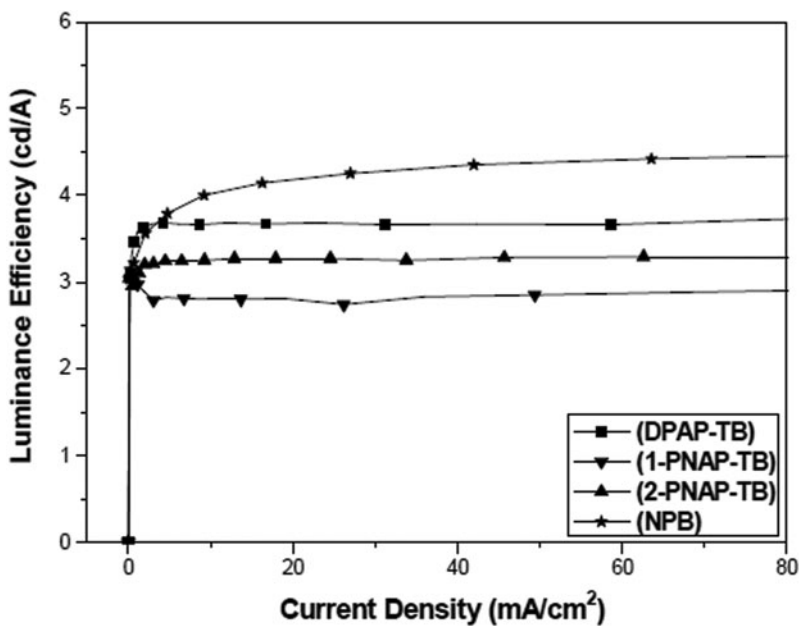
<sup>a,b</sup>: measured by DSC and TGA at a heating rate of 10°C/min.

of 2-[1-{4-(N, N-diphenylamino)phenyl}-3, 4, 5, 6-tetraphenylbenzene-2-yl]5-N, N-diphenylaminopyridine (DPAP-TB), 2-[1-{4-(N-phenylnaphthalene-1-amino)phenyl}-3, 4, 5, 6-tetraphenylbenzene-2-yl]5-(N-phenylnaphthalen-1-amino)pyridine(1-PNAP-TB), and 2-[1{4-(N-phenylnaphthalen-1-amino)phenyl}-3, 4,5, 6-tetraphenylbenzene-2-yl]-5-(N-phenylnaphthalen-2-amino)pyridine(2-PNAP-TB) in solution showed maximum absorption wavelengths of 309 nm, 314 nm and 320 nm, respectively. Similarly UV-Vis. spectra of DPAP-TB, 1-PNAP-TB and 2-PNAP-TB in film showed maximum absorption wavelengths of 309 nm, 316 nm and 322 nm, respectively. UV-Vis. spectra between solution and film showed a small difference of 2 nm. This indicates that the behavior of molecules in film state is not very different from that in the solution state and there is not special packing effect in all three compounds [6]. In both solution state and film state, UV-Vis. maximum values of 1-PNAP-TB and 2-PNAP-TB have red shift compared to DPAP-TB. This is because 1-PNAP-TB and 2-PNAP-TB has more conjugation length of naphthalene moiety than that of DPAP-TB [7]. All three compounds do not show any

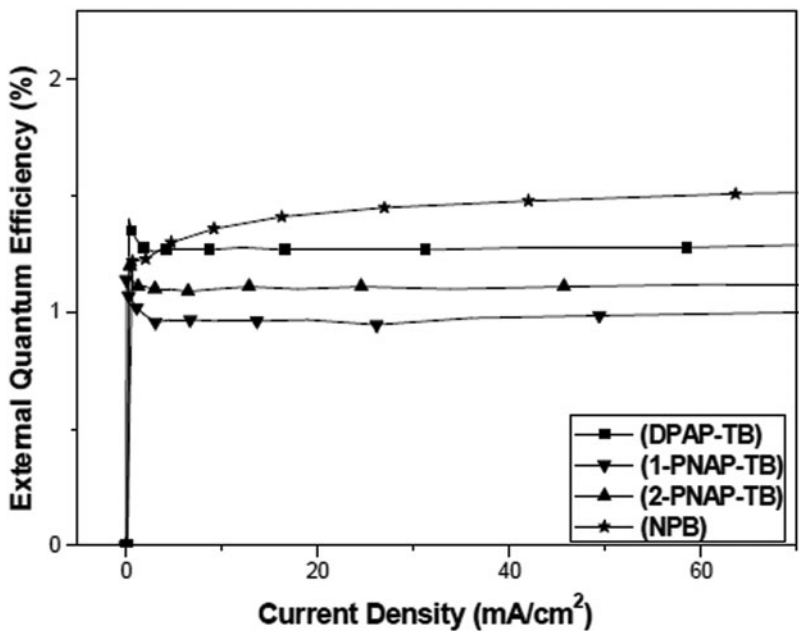


**Figure 2.** Current-Voltage-Luminance (I-V-L) characteristics of the fabricated EL.





**Figure 3.** Luminance efficiency of devices: ITO/2-TNATA (60 nm)/synthesized materials or NPB(15 nm)/Alq3 (70 nm)/LiF (1 nm)/Al (200 nm).



**Figure 4.** External quantum efficiency of devices: ITO/2-TNATA (60 nm)/synthesized materials or NPB(15 nm)/Alq3 (70 nm)/LiF (1 nm)/Al (200 nm).

**Table 2.** EL performance of the multilayered devices with the synthesized compounds at 10 mA/cm<sup>2</sup>

hole transporting materials	EL <sub>max</sub> (nm)	L. E. (cd/A) <sup>a</sup>	E.Q.E (%) <sup>b</sup>	CIE (x,y) <sup>c</sup>
DPAP-TB	520	3.72	1.29	0.327, 0.544
1-PNAP-TB	520	2.82	0.97	0.336, 0.547
2-PNAP-TB	526	3.30	1.12	0.350, 0.555
NPB	531	3.95	1.35	0.345, 0.537

<sup>a</sup>L. E.: luminance efficiency, <sup>b</sup>E.Q.E: external quantum efficiency, <sup>c</sup>C.I.E.: Commission Internationale de l'Eclairage.

absorption in the visible light area, and thus are suitable as hole transporting layer (HTL) or hole injection layer (HIL).

Based on UV-Vis. and cyclic voltammetry (CV) results, highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) levels were calculated as shown in Table 1 [8]. LUMO values of DPAP-TB, 1-PNAP-TB and 2-PNAP-TB were −1.97 eV, −2.18 eV and −2.16 eV. All three compounds showed higher LUMO levels than the LUMO value of NPB which is the commercialized as HTL. Through a higher LUMO level, electron blocking to hole transporting layer is available when electron is injected to emitting layer. Based on electron blocking effect, luminescence in HTL layer can be prevented and EML layer would be only allowed to be emitting. HOMO values of DPAP-TB, 1-PNAP-TB and 2-PNAP-TB were −5.27 eV, −5.31 eV and −5.25 eV.

Non-doped OLED devices were fabricated by using the synthesized materials or NPB(15 nm) as an hole transport layer. Device configuration is ITO/2-TNATA (60 nm)/synthesized materials or NPB(15 nm)/Alq3 (70 nm)/LiF (1 nm)/Al (200 nm). In Fig. 2, current-voltage-luminance (I-V-L) curve according to the applied voltage was described. All three compounds showed the typical rectifying characteristics and turn-on voltage was around 5V. The EL performances of devices were described and summarized in Fig. 3, Fig. 4, and Table 2. Luminance efficiency and external quantum efficiency of the devices were 3.72, 2.82, 3.30 cd/A, and 1.29, 0.97, 1.12%, respectively when DPAP-TB, 1-PNAP-TB and 2-PNAP-TB were used as a HTL in devices. These are comparable efficiencies compared to 3.95 cd/A and 1.35% of NPB, a commercialized HTL material. The reason for slightly decreased efficiency seems to be caused by the fact that the energy gap between the HOMO value of the three compounds and the HOMO value of Alq3 emitting layer was greater than 5.34 eV of NPB. In three compounds, DPAP-TB showed the highest efficiency value and it can be explained by the high LUMO level effect. Among three synthesized new compounds, DPAP-TB showed the highest efficiency. It can't be explained by clear reason, but it may be due to the improved charge balance in this device configuration. TGA and DSC were measured to identify thermal properties of synthesized materials. Glass transition temperature ( $T_g$ ) and initial thermal degradation temperature ( $T_d$ ) of the materials are summarized in Table 1. Synthesized materials exhibited high  $T_g$  in the range of 118 to 133°C. These values are higher than that of NPB which is commonly used as a hole transporting material [9].

## Conclusions

UV-Vis. spectra in film state of the three compounds of DPAP-TB, 1-PNAP-TB and 2-PNAP-TB showed maximum values of 309 nm, 316 nm, and 322 nm. The HOMO and

LUMO values of three synthesized compounds are -5.27, -1.97 eV (DPAP-TB), -5.31, -2.18 eV (1-PNAP-TB), and -5.25, -2.16 eV (2-PNAP-TB). Luminance and external quantum efficiencies of nondoped EL devices of DPAP-TB, 1-PNAP-TB and 2-PNAP-TB were 3.72, 2.82, 3.30 cd/A, and 1.29, 0.97, 1.12%, respectively. The  $T_g$  of the synthesized materials are more than 10°C higher than that of NPB.

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