Synthesis and Properties of Poly[di(1-naphthyl)-4-tolylamine] as a Hole Transport Material

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ABSTRACT: A novel hole-transporting poly[di(1-naphthyl)-4-tolylamine:DNTA] (PDNTA) has been developed. PDNTA was easily prepared by oxidative coupling polymerization of DNTA using FeCl₃ as an oxidant. This polymerization produced regiocontrolled PDNTA with high molecular weights. PDNTA exhibited strong UV–vis absorption bands at 267 and 370 nm in chloroform solution. Their photoluminescence spectra showed a maximum peak band around 445–448 nm in the blue region. The organic light-emitting device (OLED) prepared by spin-casting PDNTA solution onto ITO-coated glass substrate in conjunction with Alq and LiF/Al, as an electron-transporting light-emissive layer and a metal cathode, respectively, showed a maximum luminescence of 25 000 cd/m² at 12.5 V.

Introduction

Organic light-emitting diodes (OLEDs) are promising devices for use in full-color flat-panel displays and offer a number of advantages over conventional displays, such as low-voltage driving requirements, high brightness and contrast, simplicity, and thin-film devices.^{1–3}

Triarylamine derivatives, which are easily oxidized to form stable radical cations, are well-known as holetransport materials.⁴ Triarylamine units form covalent bonds with one another, or attach to a polymer backbone through substituents, leading to a wide variety of lowmolecular-weight compounds and polymers. When lowmolecular-weight compounds involving triarylamine units are used, spontaneous crystallization generally occurs, limiting the applicability toward OLEDs, due to electric shorts that occur in the device under these conditions. The process of the device fabrication generally requires vapor deposition, which creates an additional challenge in the application of these devises, as vapor fabrication results in an unfavorable process for a large area OLED display. On the other hand, when triarylamine units insert into or attach to a polymer backbone, an amorphous material results, which is easily processed from a solution using coating techniques.

Polymeric triarylamines are prepared using coppermediated Ullman reactions,^{5,6} palladium-^{7–9} or nickelcatalyzed coupling reactions,^{10,11} and nucleophilic substitution reactions;¹² however, these synthetic methods are not straightforward and require multiple steps in order to prepare the monomers.

Recently, Ogino et al. reported oxidative coupling polymerization of 4-methyltriphenylamine using FeCl₃ as an oxidant for the synthesis of poly(arylamine).¹³ This is a straightforward method to prepare polymeric triarylamines without introducing extra leaving and nucleophilic groups on monomers, and the structure of the resulting polymer is very simple, the poly(triarylamine) structure. However, the obtained polymers had low molecular weights, and properties of OLED for this polymer have not been reported.

1-Alkoxynaphthalenes are susceptible to a coupling reaction in the presence of Lewis acids, and the successful oxidative coupling polymerization of di(1-naphthoxy) compounds by using various oxidants has been reported.^{14–16} Thus, di(1-naphthylamino) compounds would be more suitable monomers compared to 4methyltriphenylamine for the synthesis of poly(arylamine)s with high molecular weights. Furthermore, the simple synthetic method for the synthesis of regiocontrolled poly(2,6-dihydroxynaphthalene) (PDHN) by Cu(II)-amine-catalyzed oxidative coupling polymerization of 2,6-dihydroxynaphthalene, in which PDHN shows a large dihedral angle between neighboring naphthalene rings, was very recently reported.¹⁷ The interaction between polymer chains was reduced when these kink structures were utilized; this is thought to inhibit the formation of charge-transfer complexes at the interface between a hole-transporting layer (HTL) and an electron-transporting layer (ETL).18

These findings prompted the development of another thermally stable, hole-transporting polymer, based on the oxidative coupling of naphthalene rings.

Herein, the successful synthesis of regiocontrolled poly[di(1-naphthyl)-4-tolylamine:DNTA] (PDNTA) by oxidative coupling polymerization of a novel triarylamine monomer DNTA is reported. The luminescence properties of PDNTA prepared by spin-casting a chloroform solution of PDNTA onto an indium—tin oxide (ITO)-coated glass substrate to form double-layered polymer light-emitting diodes (PLEDs) are also reported.

Experimental Section

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Materials. Nitrobenzene was distilled under nitrogen before use. Other reagents and solvents were purchased and used as received.

Di(1-naphthyl)-4-tolylamine (DNTA). To a three-necked flask equipped with a reflux condenser, 1-bromonaphthalene (6.83 g, 33 mmol), p-toluidine (1.6 g, 15 mmol), Pd₂(dba)₃ (0.34 g, 0.33 mmol), P(t-Bu)3 (0.357 g, 1.32 mmol), t-BuONa (4.44 g, 46.2 mmol), and toluene (30 mL) were added and stirred at 80 °C until p-toluidine disappeared in TLC analysis. The precipitate was removed, and the filtrate was concentrated on a rotary evaporator. The residue was extracted with ethyl acetate, and the organic layer was washed with brine. This was dried over MgSO₄, concentrated under reduced pressure, and purified by silica gel column chromatography using a mixture of dichloromethane and hexane (1:1 in volume) as an eluent to give a brown solid. The product was recrystallized from a methanol-isopropyl alcohol (1:3 in volume) mixture, affording a white solid. Yield was 4.1 g (76%); mp = 155-157°C.

IR (KBr) ν : 3046 (Ar C–H), 2915, 2857 (C–H), 1592, 1569, 1504 (C=C), 1392 (C–N), 779 cm⁻¹ (naphthalene-H). ¹H NMR (300 MHz CDCl₃) δ : 8.03 (d, 2H), 7.86 (d, 2H), 7.66 (d, 2H), 7.43 (t, 2H), 7.33 (t, 4H), 7.17 (d, 2H), 6.93 (d, 2H), 6.65 (d, 2H), 2.25 (s, 3H). ¹³C NMR (75 MHz, CDCl₃) δ : 148.26, 145.29, 135.27, 130.27, 130.12, 129.53, 128.36, 126.05, 126.01, 125.90, 125.21, 124.43, 124.33, 121.03 (ArC), and 20.70 (CH₃). Anal. Calcd for C₂₇H₂₁N: C, 90.21%; H, 5.89%; N, 3.90%. Found: C, 90.35%; H, 6.05%; N, 3.62%.

Polymer Synthesis. A typical example of the polymerization is as follows: In a 50 mL round-bottomed flask fitted with a three-way stopcock were placed DNTA (0.178 g, 0.5 mmol), FeCl₃ (0.202 g, 1.25 mmol), and nitrobenzene (1 mL) under nitrogen. The solution was stirred at room temperature for 24 h and poured into a mixture of methanol containing 10% hydrochloric acid. The precipitate was collected, washed with aqueous ammonium hydroxide, dissolved in chloroform, and filtered. The filtrate was poured into methanol to precipitate the polymer. This was dried in vacuo at 100 °C for 24 h. Yield was 0.147 g (83%).

IR (KBr) ν : 3066, 3039 (Ar C–H), 2919, 2857 (C–H), 1612, 1577, 1504 (C=C), 1376 (C–N), 759 cm⁻¹ (naphthalene-H). ¹H NMR (300 MHz, CDCl₃) δ : 8.30–8.20 (m, 2H), 7.56–7.26 (m, 10H), 7.04–6.93 (m, 2H), 6.87–6.81 (m, 2H), 2.27 (s, 3H). ¹³C NMR (75 MHz, CDCl₃) δ : 148.41, 144.94, 135.64, 134.59, 130.33, 130.20, 129.68, 128.36, 127.27, 126.00, 125.96, 124.72, 124.29, 121.06 (ArC), and 20.70 (CH₃). Anal. Calcd for (C₂₇H₁₉N)_{*n*}: C, 90.73%; H, 5.35%; N, 3.92%. Found: C, 90.56%; H, 5.80%; N, 3.63%.

Measurement. FT-IR spectra were measured on a Horiba FT-720 spectrophotometer. NMR spectra were recorded on a Bruker DPX-300 spectrometer. Number- and weight-average molecular weights were measured by a gel permeation chromatography on a Jasco Gulliver 1500 system equipped with a polystyrene gel column (Plgel 5 μ m Mixed-C) eluted with CHCl₃ at a flow rate of 1.0 mL/min calibrated by polystyrene standard. The cyclic voltammograms were measured at room temperature in a typical three electrode with a working (Pt wire), a reference (Ag/AgCl), and a counter electrode (Pt spiral) under a nitrogen atmosphere at a sweeping rate of 10 mV/s (Hokuto Denko HSV-100). A 0.1 M solution of tetrabutylammonium perchlorate (TBAP) in anhydrous acetonitrile was used as an electrolyte. Thermal analysis was performed on a Seiko EXSTAR 6000 TG/DTA 6300 thermal analyzer at a heating rate of 10 °C/min for thermogravimetry (TG) and a Seiko EXSTAR 6000 DSC 6200 at a heating rate of 10 °C/min for differential scanning calorimetry (DSC) under nitrogen. The electron affinity was measured by combination of photoelectron spectrometer (Riken Keiki, AC-1) and UV-vis absorption spectroscopy.

Device Fabrication and Characterization. ITO-coated glass with a sheet resistance $14 \ \Omega/\Box$ was patterned by aqua regia. The patterned ITO-coated glass was cleaned by sequential ultrasonication in acetone, detergent, and 2-propanol for 10 min. Oxygen plasma cleaning was performed before use. The OLED device was first spin-cast on a 2.5×2.5 cm ITO glass from the polymer solution in chloroform. The spin-coated polymer layer was annealed under reduced pressure at 100 °C for 2 h and dried at room temperature for 24 h to remove



the residual chloroform. Then, tris(8-quinolinolato)aluminum-(III) (Alq) was deposited onto the surface of the polymer films. The film thickness was measured by using a DEKTAK3ST. The Al cathode (1500 Å) with ultrathin LiF layer (5 Å) was vapor deposited using a SOLCIET apparatus. The measurements were made at room temperature under air. The EL property was measured using a MCPD-7000 fluorescence spectrophotometer by blocking the incident light.

Results and Discussion

Synthesis of DNTA. The synthetic route for DNTA is outlined in Scheme 1. DNTA was obtained by the palladium-catalyzed N-arylation reaction of p-toluidine with 2 equiv of 1-bromonaphthalene. The product was purified by silica gel column chromatography, followed by recrystallization from a methanol-isopropyl alcohol mixture to give a white solid. The structure of DNTA was assigned on the basis of elemental analysis as well as IR and NMR spectroscopy. The IR spectrum of DNTA showed characteristic absorptions corresponding to the carbon-carbon double bond of the aromatic rings at 1592, 1569, and 1504 cm^{-1} , and no bands at around 3420 and 3340 cm^{-1} due to the NH₂ stretching of *p*-toluidine were observed. Figure 1 shows the ¹H NMR spectrum of DNTA, consisting of one singlet at 2.25 ppm and multiplets at 6.65-8.03 ppm that are assigned to the methyl and aromatic protons, respectively. Furthermore, the structure of DNTA was confirmed by elemental analysis.

A full assignment of the aromatic protons was required to determine the coupling position between naphthyl units. Figure 2 shows the H–H COSY spectrum for the aromatic region of DNTA. Aromatic signals at 6.65 and 6.93 ppm are connected with a singlet signal at 2.25 ppm. These signals can be assigned to the aromatic protons of the tolylamino unit. In the spectrum of 1-aminonaphthalene (Figure 3), the aromatic protons at the 8- and 5-positions appear at the most downfield (7.77 ppm) and at the second downfield (7.75 ppm), respectively.¹⁹ In contrast, the aromatic proton at the 2-position is observed the most upfield (6.71 ppm). Accordingly, the resonances at 8.03 and 7.86 ppm of DNTA are assigned to the protons on position-l and -i,



Figure 2. H-H COSY spectrum of DNTA in CDCl₃.



Figure 3. 1 H NMR spectrum of 1-aminonaphthalene in CDCl₃.

respectively. Since the signals at 6.65 and 6.93 ppm have been assigned to the protons on position-c and -b, the signal at 7.17 ppm is assigned to the proton on position-f. The proton on position-i is connected with a triplet signal at 7.43 ppm, indicating this triplet signal is due to the proton on position-j. Other doublet and triplet signals at 7.66 and 7.33 ppm are assigned the protons on position-h and -g, k, respectively. All assignments of aromatic protons are summarized in Figure 2. Further spectral evidence for DNTA was given by ¹³C NMR, DEPT45, and C–H COSY spectroscopy. There were 15 resonances due to one methyl carbon and 14 aromatic carbons. In the aromatic region, five resonances at 148.26, 145.29, 135.27, 130.27, and 130.12 ppm were derived from quaternary carbons. Figure 4 shows the C–H COSY spectrum of DNTA. The ¹³C signal at 125.21 ppm is connected at the proton of position-h. Other ¹³C signals are well connected with the corresponding protons, as shown in the inset of Figure 4.

Cyclic Voltammogram. In the first step of oxidative coupling polymerization, selective one-electron oxidation of the aromatic rings is required. Thus, the oxidation potential of the first one-electron transfer from DNTA was investigated with cyclic voltammetry. The redox potential was measured in a 10 mM acetonitrile solution containing 0.1 M TBAP in the cell equipped with a reference (Ag/AgCl), a working (Pt wire), and counter electrode (Pt spiral). Figure 5 shows the cyclic voltammogram of DNTA, which indicates a clear oxidation peak at 0.99 V. The polymerization of 1,4-dibutoxybenzene having the oxidation potential of 1.27 V vs SCE has been reported, where FeCl₃ was used as an oxidant.²⁰ Thus, FeCl₃ was selected as the oxidant for oxidative coupling polymerization of DNTA.

Polymer Synthesis. Oxidative coupling polymerization of DNTA was carried out in the presence of FeCl₃ in nitrobenzene at room temperature (Scheme 2). The results are summarized in Table 1. The polymerization proceeded smoothly in a homogeneous state at room temperature for 24 h, giving poly(DNTA) (PDNTA) with



Figure 4. C-H COSY spectrum of DNTA in CDCl₃.

a high molecular weight. Oxidative coupling polymerization requires theoretically 2 equiv of FeCl₃ based on the monomer; however, the excess amount of FeCl₃ generally increases the rate of the polymerization. In contrast, unfavorable gelation occurs due to the extra oxidation of polymers. Therefore, the amount of FeCl₃ is an important factor for oxidative coupling polymerization. The polymer with a number-average molecular weight (M_n) of 2900 was obtained in 70% for 24 h polymerization when 2.2 equiv of FeCl₃ was used (run 5). Increasing the amount of FeCl₃ up to 2.5 equiv gave the polymer with M_n value of 13 000 in 83% yield (run 4). A small portion of polymers for runs 6 and 8 was insoluble in chloroform. Thus, GPCs were measured for soluble parts of polymers. Further increase of the oxidant decreased the M_n value of the polymer (run 6) and finally gave only gel material (run 7).

Polymer Characterization. The structure of PDN-TA was identified by NMR spectroscopy and elemental analysis. The IR spectrum exhibited characteristic absorptions at 3050 and 1600 cm⁻¹ due to aromatic C-H and C=C stretching, respectively. Elemental analysis also supported the formation of the expected polymer. The microstructure of PDNTA was investigated by NMR spectroscopy (¹H, ¹³C, DEPT45). In the 300 MHz ¹H NMR spectrum, resonances are observed



Figure 5. Cyclic voltammogram of DNTA

Scheme 2. Oxidative Coupling Polymerization of





PDNTA

Table 1. Oxidative Coupling Polymerization of DNTA^a

run	FeCl ₃ [equiv]	time (h)	concn [mol/L]	yield [%]	$M_{\rm n}{}^b$	$M_{\rm w}/M_{\rm n}{}^b$
1	2.5	3	0.5	54	1700	2.0
2	2.5	5	0.5	48	1770	2.0
3	2.5	12	0.5	83	3300	2.6
4	2.5	24	0.5	83	13000	5.3
5	2.2	24	0.5	70	2900	2.0
6	3.0	24	0.5	76	8000	23.0
7 ^c	3.5	24	0.5	0		
8	2.5	48	0.5	88	7900	16.1
9	2.5	24	0.25	69	3200	2.4

^{*a*} Polymerization was carried out by FeCl₃ as an oxidant in nitrobenzene at room temperature. ^{*b*} Determined by GPC (CHCl₃), polystyrene standard. ^{*c*} Gelation occurred.



Figure 6. ¹H NMR spectrum of PDNTA (run 4) in CDCl₃.

at 2.27 ppm (3H) and 6.83–8.26 ppm (16H) due to methyl and aromatic protons, respectively (Figure 6).

Further spectral evidence for the proposed structure of PDNTA was provided by 75 MHz ¹³C NMR and DEPT45 spectroscopy. There were 15 intense resonances due to 1 methyl carbon and 14 aromatic carbons. Aromatic regions of ¹³C NMR and DEPT45 spectra and



Figure 7. Aromatic regions in NMR spectra of DNTA and PDNTA (run 4): (a) 13 C NMR spectrum of DNTA; (b) 13 C NMR spectrum of PDNTA; (c) DEPT45 spectrum of PDNTA in CDCl₃.

assignment of PDNTA are depicted in Figure 7. The DEPT45 spectrum shows that the signals at 148.41, 144.94, 135.64, 134.59, 130.33, and 130.20 ppm are the quaternary carbons. The position-h resonance of 125.21 ppm in DNTA shifts to 134.59 ppm in PDNTA, and then this new signal disappears in the DEPT45 spectrum of PDNTA. These findings clearly indicate the coupling took place selectively at the h-position of unsubstituted *N*-naphthyl rings, giving regiocontrolled PDNTA.

The polymer was soluble at room temperature in organic solvents such as chloroform, 1,1,2,2-tetrachloroethane, and tetrahydrofuran. A pinhole-free thin transparent film was obtained by spin-coating from a chloroform solution.

The thermal properties of PDNTA were examined by thermogravimetry (TG) and differential scanning calorimetry (DSC). The typical trace for PDNTA is shown in Figure 8. The polymer shows an excellent thermal stability, and the weight loss of polymer is less than 1% even at 455 °C under a nitrogen atmosphere. The DSC trace exhibited no endothermic peak and baseline shift due to melting point and glass transition, respectively.

UV–vis Absorption and Photoluminescence Properties. Figure 9 shows optical data of PDNTA. The UV–vis absorption spectrum of PDNTA in CHCl₃ solution has peaks at 267 and 370 nm with an onset at 427 nm, corresponding to an energy gap of 2.89 eV. The UV–vis absorption spectra of PDNTA and DNTA are quite similar, and the onset positions of both compounds are almost the same, indicating a small extension of π -conjugation in PDNTA. This suggests the dihedral angle between neighboring naphthalene rings may be



Figure 8. TGA curve of PDNTA (run 4) in N₂.



Figure 9. Absorption spectra of DNTA (\bigcirc) and PDNTA (run 4) (\blacktriangle) in chloroform solution (1 × 10⁻⁵ M). PL spectrum of PDNTA (run 4) (\Box) in chloroform solution (1 × 10⁻⁶ M).

very large because of the steric repulsion of the naphthalene rings, which decrease the interaction between polymer main chains. On the other hand, the photoluminescence spectrum of the PDNTA solution in CHCl₃ shows that PDNTA emits bright blue light between 400 and 540 nm (maximum emission at 448 nm).

Device Fabrication and Electroluminescence Properties. To investigate the hole-transporting properties of our new polymer, a double-layer device consisting of PDNTA and Alq was fabricated. The device was prepared by first spin-casting a chloroform solution of PDNTA onto an ITO-coated glass substrate and then vacuum deposition of Alq as an ETL. Finally, the Al cathode (1500 Å) with ultrathin LiF layer (5 Å) was deposited on the Alq layer in a vacuum. The PDNTA film cast from chloroform was consistently pinhole-free and had an average thickness of 400 Å. The Alq layer was deposited to a thickness of approximately 600 Å.

The EL spectrum of the device is shown in Figure 10. The EL from the device emitted bright green luminescence ($\lambda_{max} = 520$ nm) when a positive voltage was applied to the electrode. The EL spectrum was in agreement with the photoluminescence (PL) spectrum of Alq. Tails in a longer wavelength region, which are often observed in the case of the charge-transfer complex formation at the interface between HTL and ETL,^{18,21} were not observed. This could be due to the nonplanar molecular structure due to large dihedral angle between adjoining naphthalene rings of PDNTA, which prevents the molecules in the HTL from packing closely with the molecules in the EML at the interface. The band diagram of the EL device is shown in Figure 11. The result shows that the electron-hole recombination takes place at the Alq emitting layer. Since the electron



Figure 10. EL spectrum measured for the ITO/PDNTA (run 4)/Alq/LiF/Al device.



Figure 11. Band diagram of ITO/PDNTA (run 4)/Alq/LiF/Al device.



Figure 12. Voltage vs current density (\bigcirc , right axis) and luminance (\blacktriangle , left axis) of the devices ITO/PDNTA (run 4)/Alq/LiF/Al.

affinity of PDNTA is about 2.26 eV and that of Alq is about 2.88 eV, there is an electron-transporting barrier of about 0.62 eV at the PDNTA/Alq interface. In contrast, the barrier at the PDNTA/Alq interface for the transport of hole is only 0.43 eV. Therefore, it is quite natural that the recombination region is at the Alq emitting layer. The voltage–luminance and voltage– current density characteristics of device are shown in Figure 12. The maximum luminescence reached 25 000 cd/m² at 12.5 V. At the maximum luminescence, the luminous efficiency of this device was 0.64 lm/W.

Conclusions

Regiocontrolled PDNTA was prepared by oxidative coupling polymerization of DNTA using FeCl_3 , which produced polymers with M_n s up to 13 000. The structure

of PDNTA, derived from DNTA, was characterized by ¹H and ¹³C NMR spectroscopy and was determined to have a 4,4'-linkage. The polymer showed high thermal stability and good film processability. The bright blue emission obtained from the PL spectrum revealed that PDNTA lied in the blue region. The OLED device prepared using spin-cast film from PDNTA on an ITOcoated glass substrate in conjunction with Alq, functioned as an excellent hole-transporting material in OLEDs, and showed the maximum luminescence of 25 000 cd/m² at 12.5 V. The synthesis of DNTA and PDNTA is very easy and advantageous compared to that of the conventional hole-transporting polymers.

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