



A novel host material with high thermal stability for green electrophosphorescent device



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ABSTRACT

A new bipolar host material based on triphenylamine, fluorene and 1,2-diphenyl-1*H*-benzo[*d*]imidazole moieties, *N,N*-diphenyl-4-(9-phenyl-2-(4-(1-phenyl-1*H*-benzo[*d*]imidazol-2-yl)phenyl)-9*H*-fluoren-9-yl)aniline (DPPBIPFA), was designed and synthesized. The as-synthesized material was well characterized by ¹H and ¹³C NMR spectroscopy, high-resolution mass spectrometry and thermogravimetric analysis, respectively. The photophysical and electrochemical properties of the material were also studied. The material exhibited an excellent thermal stability ($T_d = 475$ °C), electrochemical stability and high triplet energy (2.68 eV). A green phosphorescent organic light-emitting diode (PhOLED) device based on DPPBIPFA as the host material and Ir(ppy)₃ as the dopant was fabricated, which displayed favorable electrophosphorescent properties with a turn-on voltage of 3.75 V, a maximum brightness of 1685 cd/m² and a maximum current efficiency of 4.26 cd/A.

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1. Introduction

In the past decades, organic light-emitting diodes (OLEDs) have drawn much attention due to their applications in full-color flat-panel displays and solid-state lighting sources.^{1–3} Phosphorescent organic light-emitting diodes (PhOLEDs) can theoretically achieve 100% internal quantum efficiency, which has attracted considerable attention since the first report on applying the phosphorescent host-dopant system to address the issues of concentration quenching and the triplet–triplet annihilation, etc.^{4–6} In the PhOLEDs, the phosphorescent hosts play a vital role, which serve as the recombination center for electrons and holes to generate the electronically excited states.^{7,8} Recently, bipolar hosts have aroused considerable interest because they can balance the mobility of electrons and holes, generate broad charge recombination zones, and simplify device structure, thus boosting the device performance obviously.^{5,9,10} For example, Hsu et al. in 2009 reported a phosphine-oxide-containing

bipolar host for application in a blue device with maximum power efficiency of 26.2 lm/W.¹¹ Chen et al. developed a hybrid indole/triazine bipolar host, exhibiting a high glass transition temperature of 124 °C and maximum external quantum efficiency of 17.5% for a red PhOLED.¹² Tian et al. recently designed and synthesized a spiroannulated host with a high decomposition temperature of 427 °C and a high triplet energy of 2.9 eV.¹³

An ideal host material should meet the following intrinsic requirements: (i) a triplet energy gap (E_T) larger than that of the dopant to prevent reverse energy transfer from the guest back to the host; (ii) good carrier transporting properties to balance the charge flux and reduce the driving voltage; (iii) good thermal and morphological stability to prolong the device's operational lifetime.^{14–16}

In this paper, we designed and synthesized a novel host material *N,N*-diphenyl-4-(9-phenyl-2-(4-(1-phenyl-1*H*-benzo[*d*]imidazol-2-yl)phenyl)-9*H*-fluoren-9-yl)aniline (DPPBIPFA) based on triphenylamine, carbazole and 1,2-diphenyl-1*H*-benzo[*d*]imidazole moieties, which can be used as the host matrix to fabricate the green-light PhOLED with Ir(ppy)₃ as dopant. The DPPBIPFA exhibits three important features: (i) bipolarity, resulting from the presence of the D- π -A system, (ii) a value of E_T as high as 2.68 eV, (iii) a sterically bulky and hindered structure, which can prevent the

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aggregation of guest emitters and provide excellent thermal stability (onset decomposition temperature (T_d)=475 °C). Due to the above favorable features, the material DPPBIPFA can be used as a host to fabricate green PhOLEDs with phosphorescent dopants, such as Ir(ppy)₃.^{17–19}

2. Experimental

2.1. Synthesis of *N,N*-diphenyl-4-(9-phenyl-2-(4-(1-phenyl-1*H*-benzo[d]imidazol-2-yl)phenyl)-9*H*-fluoren-9-yl)aniline (DPPBIPFA)

Solvents were dried using standard methods. All other reagents were used as received from commercial sources, unless stated otherwise.

In a dry flask, phenylmagnesium bromide in THF (15 ml, 12 mmol) was added dropwisely to a solution of 2-bromo-9-fluorenone (2.58 g, 10 mmol) in THF at –78 °C under a nitrogen atmosphere. The mixture was allowed to warm to room temperature followed by stirring for 12 h. After neutralization by aqueous HCl and then extraction with ethyl acetate, the organic layer was separated out and dried over anhydrous Na₂SO₄. Then the solvent was removed by using a rotary evaporator. The crude material was purified by silica gel column chromatography using a petroleum and dichloromethane mixture (*v:v*=1:1) as the eluent to obtain the desired product (**1**) as a white solid (2.3 g, 89%). ¹H NMR (CDCl₃, 400 MHz, δ /ppm): 7.67–7.69 (d, *J*=7.6 Hz, 2H), 7.33–7.41 (m, 6H), 7.24–7.29 (m, 5H), 2.51 (s, 1H).

In a flask, five drops of BF₃ ethanol solution was added dropwisely to a solution of compound (**1**) (1.1 g, 3.3 mmol) and triphenylamine (1.5 g, 6.1 mmol) in CH₂Cl₂ (15 ml) at room temperature. After stirring for 2 h, ethanol was added to quench the reaction. The solvent was evaporated and the resulting crude material was purified by silica gel column chromatography using a petroleum and dichloromethane mixture (*v:v*=3:1) as the eluent to give the desired product (**2**) as a white solid (1.0 g, 56%). ¹H NMR (CDCl₃, 400 MHz, δ /ppm): 7.71–7.73 (d, *J*=7.6 Hz, 1H), 7.61–7.63 (d, *J*=8 Hz, 1H), 7.47–7.53 (m, 2H), 7.20–7.40 (m, 12H), 6.90–7.08 (m, 10H).

In a flask, compound (**2**) (0.56 g, 1 mmol), (4-(1-phenyl-1*H*-benzo[d]imidazol-2-yl)phenyl)boronic acid (0.37 g, 1.2 mmol), K₂CO₃ (0.66 g, 4 mmol), Pd(PPh₃)₄ (30 mg, 0.03 mmol, 99.8%) were dissolved in water (4 ml) and THF (12 ml). The mixture was bubbled with nitrogen and then allowed to reflux for 6 h. Then the solution was cooled to room temperature and poured into water followed by extraction with CH₂Cl₂. The organic layer was dried over anhydrous MgSO₄ and concentrated using a rotary evaporator. The crude material was purified by silica gel column chromatography using a petroleum and dichloromethane mixture (*v:v*=1:1) to give the target compound DPPBIPFA as a white solid (0.45 g, 60%). ¹H NMR (CDCl₃, 400 MHz, δ /ppm): 7.89–7.91 (d, *J*=8 Hz, 1H), 7.79–7.81 (d, *J*=8 Hz, 1H), 7.76–7.78 (d, *J*=8 Hz, 1H), 7.59–7.64 (m, 4H), 7.49–7.55 (m, 5H), 7.41–7.42 (d, *J*=4 Hz, 1H), 7.32–7.39 (m, 4H), 7.19–7.30 (m, 11H), 7.05–7.08 (m, 6H), 6.89–7.04 (m, 5H). ¹³C NMR (CDCl₃, 101 MHz): δ 152.14, 152.00, 151.75, 147.59, 146.29, 145.86, 142.99, 142.06, 139.86, 139.51, 139.16, 137.33, 137.05, 129.94, 129.74, 129.16, 128.87, 128.61, 128.25, 128.10, 127.83, 127.50, 127.46, 126.87, 126.62, 126.22, 124.64, 124.41, 123.34, 123.01, 122.96, 122.79, 120.49, 120.26, 119.76, 110.40. HRMS (ESI, *m/z*): [M+H]⁺ calcd for C₅₆H₃₉N₃, 754.3222, found 754.3214.

2.2. Characterization of materials

¹H and ¹³C NMR spectra were recorded using a Switzerland Bruker spectrometer relative to tetramethylsilane (TMS) as the

internal standard. Elemental analyses were performed using a Vario EL elemental analyzer. Differential scanning calorimetry (DSC) was performed using a DSC Q2000, operated at a heating rate of 10 °C/min and a cooling rate of 50 °C/min; the glass transition temperature (T_g) was determined from the second heating scan. Thermogravimetric analysis (TGA) was undertaken using a Netzsch TG 209, the thermal stability of sample was determined under a nitrogen atmosphere by measuring the weight loss while heating at a rate of 20 °C/min. UV–vis absorption spectrum was measured by using a Hitachi U-3900 spectrophotometer. The photoluminescence (PL) spectrum was obtained by using a Fluoromax-4 spectrophotometer in diluted THF solution. The low-temperature phosphorescence spectrum was obtained using a composite spectrometer incorporating a monochromator (F-7000 FL Spectrophotometer) coupled with a liquid nitrogen cooled charge-coupled device (CCD) detector (F-7000 FL Spectrophotometer). Cyclic voltammetry (CV) measurement was performed using an Autolab/PG STAT302 electrochemical analyzer operated at a scan rate of 50 mV/s through a one-compartment electrolysis cell consisting of a platinum wire as working electrode, a platinum electrode as counter electrode, and a calomel electrode as reference electrode. Tetra-butylammonium perchlorate dissolved in acetonitrile was used as a supporting electrolyte (0.1 mol/L).

2.3. OLED fabrication and measurements

The EL device was fabricated through vacuum thermal evaporation technology according to the methods modified from our previous approach. Device with an area of 3 mm×3 mm was fabricated by vacuum deposition (at 1×10^{–6} Torr) of functional layers onto indium tin oxide (ITO) glass substrate which has a sheet resistance of 25 Ω/square. The ITO glass substrate was cleaned with deionized water, acetone, ethanol in turn and dried in the oven at 120 °C. Then, the clear ITO glass substrate was treated with oxygen plasma for 8 min. The PEDOT:PSS layer was spin-coated onto the ITO substrate in air, and then allowed to anneal at 120 °C for 15 min in a glovebox. All the other organic layers were deposited at a rate of 1.0 Å/s sequentially. The cathode was completed through thermal deposition of LiF (10 Å) at a deposition rate of 0.1 Å/s; then Al metal (200 nm) was deposited through thermal evaporation at a rate of 5.0 Å/s. The voltage-current-luminance relationships of the OLEDs were recorded with a Keithley 2400 Source Meter. The EL spectrum was obtained using a Hitachi F4500 luminescence spectrometer. The brightness–current–voltage (B–I–V) characterizations were carried out with a 3645 DC power supply combined with a 1980A spot photometer and were recorded simultaneously. All measurements were done at room temperature under ambient conditions.

3. Results and discussion

3.1. Synthesis and characterization

The synthetic route of the bipolar material DPPBIPFA is illustrated in Scheme 1. Intermediate compound (**1**) was firstly synthesized from the starting materials 2-bromo-9-fluorenone and phenylmagnesium bromide by Grignard reaction, which then undergo coupling with triphenylamine to generate intermediate (**2**) with Lewis acid BF₃ as the catalyst. Finally, the target compound was synthesized by the Suzuki reaction of compound (**2**) and (4-(1-phenyl-1*H*-benzo[d]imidazol-2-yl)phenyl)boronic acid. All the new compounds were obtained with satisfactory yield and well characterized by ¹H and ¹³C NMR spectroscopy and high-resolution mass spectrometry (HRMS).

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