



Novel carbazole/indole/thiazole-based host materials with high thermal stability for efficient phosphorescent organic light-emitting diodes



Bin Jia^{a,***}, Hong Lian^b, Zheng Chen^b, Yi Chen^c, Jinhai Huang^{c,**}, Qingchen Dong^{b,*}

^a School of Basic Medical Science, Shanxi Medical University, Taiyuan 030001, PR China

^b MOE Key Laboratory of Interface Science and Engineering in Advanced Materials, Research Center of Advanced Materials Science and Technology, Taiyuan University of Technology, 79 Yingze West Street, Taiyuan 030024, PR China

^c Shanghai Taoe Chemical Technology Co., Ltd, Shanghai, PR China

ARTICLE INFO

Article history:

Received 27 July 2017

Received in revised form

22 August 2017

Accepted 28 August 2017

Available online 31 August 2017

Keywords:

Thiazole derivatives

Carbazole derivatives

Indole derivatives

Host materials

Phosphorescent organic light-emitting diodes

ABSTRACT

Two host materials **TIC1** (N-linked carbazole) and **TIC2** (C-3-linked carbazole) via changing the substitution position of carbazole, indole and thiazole moieties are reported. Their structural and physical properties were fully characterized. The thermal analysis demonstrated that both **TIC1** and **TIC2** are thermally stable. The triplet energy levels of **TIC1** and **TIC2** were determined to be 2.54 and 2.58 eV, respectively. Green and blue OLEDs with **TIC1** and **TIC2** acting as host materials were fabricated to investigate their performances in PHOLED. Both green and blue PHOLEDs based on **TIC2** show the best performance with the maximum current efficiency and maximum power efficiency of 60.0 cd/A and 48.0 lm/W (for green), 18.4 cd/A and 15.2 lm/W (for blue), respectively. The characteristic measurements of OLED devices reveal that both **TIC1** and **TIC2** are promising host materials for practical application. By comparing with the commercial host material of 4,4'-bis(carbazol-9-yl)biphenyl (CBP) and *N,N*-dicarbazoyl-3,5-benzene (mCP), the power efficiency of green and blue PHOLED was enhanced by 55.3% and 245%, respectively.

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1. Introduction

Phosphorescent organic light-emitting diodes (PHOLEDs) have attracted worldwide attention in recent years due to their appealing commercial interest in the field of flat panel display, solid-state lighting and virtual reality [1–4]. However, the commercialization of PHOLEDs is still facing the challenges of materials and life-time, thus enormous efforts have been devoted to explore novel materials including the carrier transport materials, emitting materials and host materials, *etc.* to push the development of PHOLEDs [5–12]. Among these materials, the phosphorescent host materials which serve as the recombination center for electrons and holes to generate the electronically excited states, are crucial to realize high efficiency PHOLEDs [13–16]. The following

intrinsic requirements should be met for a desired host material: firstly, the triplet energy gap (E_T) should be higher than that of the dopant to prevent reverse energy transfer from the guest back to the host; secondly, the hole-electron mobility is balanced to confine the triplet exciton within the emitting layer and reduce the driving voltage; thirdly, good thermal and morphological stability to prolong the operation lifetime of the OLED [17–23]. The carbazole derivatives exhibit suitable energy gap and high fluorescent quantum yield [24–28], and the thiazole derivatives are good hole-transport materials due to their electron donating ability [29,30]. Additionally, indole type materials which possess high triplet energy level and excellent hole transport properties, were reported as potential host material for PHOLEDs [31].

Based on the above considerations, we designed and synthesized two host materials **TIC1** and **TIC2** through different linkage mode of thiazole, indole and carbazole moieties. The blue phosphorescent OLEDs based on **TIC1** and **TIC2** were fabricated. The results of device performance measurement indicate that **TIC2** displayed the highest current efficiency and power efficiency of 13.9 cd/A and 10.9 lm/W for blue PHOLED, respectively. By

* Corresponding author.

** Corresponding author.

*** Corresponding author.

E-mail addresses: bjia2006@163.com (B. Jia), dele12@163.com (J. Huang), dongqingchen@tyut.edu.cn (Q. Dong).

comparing with the commercial host materials of 4,4'-bis(carbazol-9-yl)biphenyl (CBP) and *N,N*-dicarbazoyl-3,5-benzene (mCP), the current and power efficiency of blue PHOLED was enhanced by 80.5% and 147.7%, respectively.

2. Experimental

2.1. General information

All the solvents and chemicals that were used in the synthetic route were of reagent grades and purchased from J & K Chemical Co. and Aladdin Chemical Co. without further purification. Tetrahydrofuran (THF) was purified by distillation over sodium under a N₂ atmosphere prior to use. All reactions and manipulations were carried out under a N₂ atmosphere. All column chromatography was performed on silica gel (300–400 mesh) as the stationary phase in the column. All materials were purified further by vacuum sublimation prior to fabrication of OLED devices.

The ¹H and ¹³C NMR spectra were recorded on a Bruker AM 400 spectrometer with tetramethylsilane as the internal standard. High-resolution mass spectra were measured on a Waters LCT Premier XE spectrometer. IR spectra were recorded on the Nicolet Magna 550 Series II FTIR spectrometer using KBr pellets for solid state spectroscopy. The ultraviolet-visible (UV-Vis) absorption spectra were obtained on a Varian Cary 500 spectrophotometer. Photoluminescence (PL) spectra were recorded on a Varian-Cary fluorescence spectrophotometer. The cyclic voltammetry experiments were performed using a Versastat II electrochemical work station (Princeton applied research) using a conventional three-electrode configuration with a glassy carbon working electrode, a Pt wire counter electrode, and a regular calomel reference electrode in saturated KCl solution. The oxidation and reduction potentials were measured in dichloromethane/acetonitrile (7:3, v/v) solution containing of 0.1 M tetra-*n*-butylammonium perchlorate (TBAP) as the supporting electrolyte at a scan rate of 100 mV/s. The differential scanning calorimetry (DSC) analysis was performed on a DSC Q2000 V24.11 Build 124 instrument with a heating scan rate of 10 °C/min from 0 °C to 250 °C under nitrogen atmosphere. Thermogravimetric analysis (TGA) was carried out on the TGA instrument by measuring weight loss of samples with a heating scan rate of 10 °C/min from 50 °C to 800 °C under nitrogen.

2.2. Preparation of 1-(2-bromophenyl)-5-(dibenzo[*b,d*]thiophen-4-yl)-1H-indole (**1**)

A mixture of 5-(dibenzo[*b,d*]thiophen-4-yl)-1H-indole (2.99 g, 10 mmol) and 1-bromo-2-iodobenzene (3.11 g, 11 mmol) in 5 g of trichlorobenzene and potassium carbonate (2.76 g, 20 mmol) was added into the mixture, and then the reaction mixture was refluxed for 8 h under N₂ atmosphere. After the mixture cooled to room temperature, the reaction solvent was removed in vacuum and further purified by SiO₂ column chromatography, affording compound **1** as a white solid (2.27 g, 50%). ¹H NMR (CDCl₃, 400 MHz, δ/ppm): 8.24–8.21 (m, 1H), 8.19–8.15 (m, 1H), 8.09 (dt, *J* = 2.0, 1.2 Hz, 1H), 7.87–7.82 (m, 2H), 7.60–7.58 (m, 5H), 7.55–7.50 (m, 2H), 7.50–7.49 (m, 1H), 7.49–7.45 (m, 2H), 7.35 (d, *J* = 3.4 Hz, 1H), 6.83–6.81 (m, 1H).

2.3. Preparation of 9-(2'-(5-(dibenzo[*b,d*]thiophen-4-yl)-1H-indol-1-yl)-[1,1'-biphenyl]-4-yl)-9H-carbazole (TIC1) and 3-(2-(5-(dibenzo[*b,d*]thiophen-4-yl)-1H-indol-1-yl)phenyl)-9-phenyl-9H-carbazole (TIC2)

In a 100 ml one-neck flask, compound **1** (0.79 g, 1.74 mmol) and compound **2** (0.50 g, 1.74 mmol) were dissolved in solution of THF

(20 ml) and 2 M K₂CO₃ (20 ml), followed by catalytic amount of Pd(dppf)Cl₂ under a nitrogen atmosphere. The mixture was heated to reflux for 6 h. The completion of the reaction was verified by spot TLC. The resulting mixture was extracted by dichloromethane for 3 times, then the organic phase was dried over anhydrous sodium sulfate and concentrated by a rotary evaporator. The crude material was purified by silica gel column chromatography using a petroleum and dichloromethane mixture (v:v = 4:1) as the eluent to obtain the desired product 9-(2'-(5-(dibenzo[*b,d*]thiophen-4-yl)-1H-indol-1-yl)-[1,1'-biphenyl]-4-yl)-9H-carbazole (**TIC1**) as a white solid (0.52 g, 43%). ¹H NMR (CDCl₃, 400 MHz, δ/ppm): 8.12 (dd, *J* = 6.8, 1.8 Hz, 1H), 8.06 (dd, *J* = 6.8, 2.2 Hz, 1H), 8.02 (d, *J* = 7.7 Hz, 2H), 7.94 (d, *J* = 1.3 Hz, 1H), 7.70–7.66 (m, 2H), 7.60–7.57 (m, 1H), 7.55–7.52 (m, 2H), 7.49–7.43 (m, 3H), 7.42–7.40 (m, 1H), 7.40–7.35 (m, 2H), 7.32–7.29 (m, 2H), 7.26–7.23 (m, 2H), 7.22 (s, 2H), 7.20 (s, 2H), 7.18–7.13 (m, 2H), 7.05 (d, *J* = 3.2 Hz, 1H), 6.61 (d, *J* = 3.2 Hz, 1H); ¹³C NMR (CDCl₃, 101 MHz, δ/ppm): 140.72, 139.79, 139.06, 137.69, 136.83, 136.13, 135.83, 132.75, 131.27, 129.89, 129.61, 128.87, 128.28, 127.21, 126.99, 126.59, 125.95, 125.03, 124.22, 123.33, 122.67, 121.68, 120.54, 120.20, 119.83, 110.98, 109.75, 103.75; HRMS (ESI, *m/z*): [M+H]⁺ calculated for C₄₄H₂₈N₂S, 617.2051, found 617.2053. IR (KBr): 1333 (ν_{C-N}) cm⁻¹, 1595, 1523, 1485, 1452 (ν_{C=C}) cm⁻¹, 3051 (ν_{C-H}) cm⁻¹; T_{decomp} (°C): 355.3 °C for **TIC1**.

The procedure for the preparation of target production (3-(2-(5-(dibenzo[*b,d*]thiophen-4-yl)-1H-indol-1-yl)phenyl)-9-phenyl-9H-carbazole) (**TIC2**) was similar to that of target production (**TIC1**). The resulting product (**TIC2**) was obtained as a white solid (0.55 g, 47%). ¹H NMR (CDCl₃, 400 MHz, δ/ppm): 8.12 (dd, *J* = 6.0, 3.0 Hz, 1H), 8.06 (dd, *J* = 6.4, 2.6 Hz, 1H), 7.87 (d, *J* = 6.9 Hz, 2H), 7.81 (d, *J* = 1.6 Hz, 1H), 7.75–7.71 (m, 1H), 7.69 (dd, *J* = 7.5, 1.7 Hz, 1H), 7.58 (dd, *J* = 7.3, 1.8 Hz, 1H), 7.51–7.44 (m, 9H), 7.42–7.34 (m, 5H), 7.31 (d, *J* = 3.7 Hz, 2H), 7.12 (d, *J* = 8.5 Hz, 1H), 6.94 (dd, *J* = 8.6, 1.7 Hz, 1H), 6.86 (d, *J* = 3.2 Hz, 1H), 6.42 (d, *J* = 3.2 Hz, 1H); ¹³C NMR (CDCl₃, 101 MHz, δ/ppm): 141.12, 140.30, 139.87, 136.87, 136.03, 132.58, 131.95, 130.28, 129.83, 128.76, 128.21, 127.81, 127.27, 126.79, 126.14, 125.03, 124.21, 123.42, 122.64, 121.72, 120.41, 120.07, 119.68, 110.88, 109.85, 109.62, 103.26; HRMS (ESI, *m/z*): [M+H]⁺ calculated for C₄₄H₂₈N₂S, 617.2051, found 617.2051. IR (KBr): 1334 (ν_{C-N}) cm⁻¹, 1591, 1510, 1493, 1448 (ν_{C=C}) cm⁻¹, 3053 (ν_{C-H}) cm⁻¹; T_{decomp} (°C): 349.9 °C for **TIC2**.

2.4. Fabrication and measurement of OLEDs

The OLED devices were fabricated through vacuum thermal evaporation technology according to the method modified from our previous approach [32]. Device with an area of 3 mm × 3 mm was fabricated by vacuum deposition (at 1 × 10⁻⁶ Torr) of functional organic layers on the indium-tin-oxide (ITO)/glass substrate which has a sheet resistance of 25 Ω/square. The ITO/glass substrate was cleaned sequentially by detergent, de-ionized water, acetone, ethanol and then dried in the oven at 120 °C. Afterwards, the clean ITO glass substrate was treated with oxygen plasma for 8 min.

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 (1 nm) 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 EL spectra were measured by a Spectrascan PR655 photometer. The current-voltage-luminance characteristics (I-V-L) were measured by a computer-controlled Keithley 2400 source-meter integrated with a TOPCOM BM-7 luminance meter. Current efficiency and power efficiency were calculated from the plot of I-V-L. All samples were characterized immediately after thin films deposition without encapsulation at room temperature. AFM was measured to investigate the surface morphology of the thin films of **CBP**, **mCP**, **TIC1** and **TIC2**.

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