



Synthesis, aggregation-induced emission, and electroluminescence properties of a novel emitter comprising tetraphenylethene and carbazole moieties



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ABSTRACT

In this paper, a new tetraphenylethene-based molecule with two carbazole-containing moieties, 1,2-bis(4-(9H-carbazol-9-yl)phenyl)-1,2-diphenylethylene (BCPD), has been successfully prepared by the McMurry coupling reaction of (4-(9H-carbazol-9-yl)phenyl)(phenyl)methanone (1). The structure of BCPD was fully characterized by elemental analysis, mass spectrometry, NMR spectroscopy and infrared spectroscopy. Its optical properties in both solution and solid-state were investigated systematically. The results show that BCPD has almost no emission in solution but is highly emissive in the aggregated form, demonstrating a typical aggregation-induced emission phenomenon. An extensive investigation of its thermal properties verifies that BCPD enjoys excellent thermal and morphological stabilities with T_d of 335 °C and T_g of 105 °C. Eventually, non-doped electroluminescence device by utilizing BCPD as emitting layer was fabricated. The device exhibits sky blue light with the maximum luminance and the maximum luminescent efficiency reaching 8624 cd m⁻² and 4.243 cd A⁻¹, respectively.

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1. Introduction

In the past decade, research on design and synthesis of small organic fluorescent molecules exhibiting high efficiency luminescence in the solid state has drawn considerable attention owing to their applications in organic light-emitting devices (OLEDs) [1], fluorescent probes [2], and sensors [3]. Unfortunately, most traditional fluorescent materials are commonly highly emissive in dilute solutions, but show weak or even no emission in the aggregation or solid states due to the notorious problem called aggregation-caused quenching (ACQ) [4,5]. In 2001, Tang's group discovered a new photophysical phenomenon of aggregation-induced emission (AIE), for a typical type of small organic molecules which are non- or weak emissive when dissolved in

good solvents, but give intense luminescence in aggregates and the solid film state [6]. Among the AIE-active luminogens, tetraphenylethene (TPE) as a star molecule has attracted much research interest and been selected as the ideal building block to construct AIE fluorophores for various applications because of its advantages such as striking AIE effect, blue light-emission, facile synthesis, and easy functionalization [7–18].

On the other hand, carbazole is another group frequently utilized to construct efficient emitters [19–21]. Carbazole is a typical electron donor with a large rigid plane structure, which endows carbazole and its derivatives high glass transition temperature. In addition, carbazole possesses high HOMO energy level, which is favourable to hole-transporting ability [22,23]. However, the carbazole derivatives themselves used as light-emitting materials have been subject to the ACQ effect [24]. In recent years, a new successful structural design strategy to surmount the ACQ problem is to incorporate AIE units with the ACQ chromophores resulting in new luminogens with AIE characteristics [25–31].

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In view of the above, we can meld TPE unit and carbazole unit at the molecular level to prepare functional materials with excellent solid-state emission. The studies based on combining these two units into one molecule to construct efficient luminogens further confirm our idea. Huang and co-workers synthesized and characterized a TPE-based molecule constructed by TPE and carbazole moieties, which exhibited AIE properties, good electroluminescence (EL) performance as well as high thermal and morphological stabilities [32]. Tang and co-workers synthesized three AIE-active molecules by combining TPE and carbazole moieties, which are promising bifunctional EL materials showing excellent hole-transporting and blue light-emitting properties [33]. Gong and co-workers designed and synthesized five TPE-decorated carbazole materials with AIE characteristics. OLEDs fabricated by the resulting compounds as emitting layers emitted blue-green light with a high maximum current efficiency (up to 5.5 cd A^{-1}) [34]. Odabas et al. reported two new luminogenic molecules, TPE core surrounded by *tert*-butylcarbazole and tetrahydrocarbazole, which showed high thermal stabilities and excellent AIE properties and could be used in OLEDs commendably [35]. All of the previous work demonstrates that compounds containing TPE and carbazole are prospective materials for organic EL devices.

To further develop new more efficient solid-state emitters for OLED applications, in this paper, we designed and synthesized a new compound, 1,2-bis(4-(9H-carbazol-9-yl)phenyl)-1,2-diphenylethylene (BCPD), based on tetraphenylethene as an aggregation-induced emission group and carbazole as a hole-transporting group by introducing two carbazole groups to the two diagonal benzene rings of tetraphenylethene, respectively. The synthetic route is presented in Scheme 1. The structure of this compound was characterized by elemental analysis, ^1H NMR, ^{13}C NMR, MS and IR. The thermal, electrochemical and photophysical properties of BCPD are also reported. Finally, BCPD has been successfully applied to electroluminescent device. Our results reveal that BCPD is indeed an efficient blue emission material for OLEDs device.

2. Experimental

2.1. Materials and measurements

^1H and ^{13}C NMR spectra were measured on a Bruker 600 MHz spectrometer. High-resolution mass spectra (HRMS) were measured on an Autoflex III (MALDI-TOF-MS). Elemental analyses were performed on an Element Analysis System. Infrared (IR) spectra were obtained using a Shimadzu FTIR-8400S spectrometer by incorporating samples in KBr disks. UV-vis absorption spectra were recorded on a Shimadzu UV-2450 absorption spectrophotometer. Photoluminescence spectra were determined on a Shimadzu RF-5301PC fluorescence spectrometer. The fluorescence quantum yield was obtained using quinine sulfate as the reference (excited at 350 nm). Thermogravimetric analysis (TGA) was undertaken with a TA Instruments TGA 2050 thermogravimetric analyzer under nitrogen at a heating rate of $10^\circ\text{C min}^{-1}$ from 53 to 390°C . Differential scanning calorimetry (DSC) was performed on a Q2000 DSC differential scanning calorimeter under a N_2

atmosphere with a heating rate of $10^\circ\text{C min}^{-1}$ from 40 to 200°C . Cyclic voltammetry (CV) was carried out on a CHI-600C electrochemical analyzer in a three-electrode cell with a glassy carbon working electrode, a platinum-disk auxiliary electrode and an Ag/AgCl reference electrode at the scan rate of 10 mV/s . All the chemicals and reagents were obtained from commercial sources and used as received without further purification. Tetrahydrofuran (THF) was distilled from sodium and benzophenone under dry nitrogen immediately prior to use.

2.2. Device fabrication and testing

The multilayer OLEDs were fabricated by vacuum-deposition method. Organic layers were fabricated by high-vacuum (5×10^{-4} Pa) thermal evaporation onto a glass ($3 \text{ cm} \times 3 \text{ cm}$) substrate precoated with an ITO layer. Dipyrzinoquinoxaline-2,3,6,7,10,11-hexacarbonitrile (HATCN) was used as the hole injection layer; *N,N*-bis(naphthalene)-*N,N*-bis(phenyl)benzidine (NPB) was worked as the hole-transporting layer (HTL); BCPD was served as the emitting layer; 1,3,5-tri(1-phenyl-1*H*-benzo[d]imidazol-2-yl)phenyl (TPBI) was acted as the electron-transporting layer (ETL) and LiF/Al was evaporated as cathode. All organic layers were sequentially deposited. Thermal deposition rates for organic materials, LiF and Al were 0.5 A/s , 0.5 A/s and 1 A/s , respectively. The active area of the devices is 9.0 mm^2 . The EL spectra were measured on a Hitachi MPF-4 fluorescence spectrometer. The voltage current density characteristics of OLEDs were recorded on a Keithley 2400 Source Meter. The characterization of brightness current-voltage was measured with a 3645 DC power supply combined with a 1980A spot photometer and were recorded simultaneously. All measurements were done at room temperature.

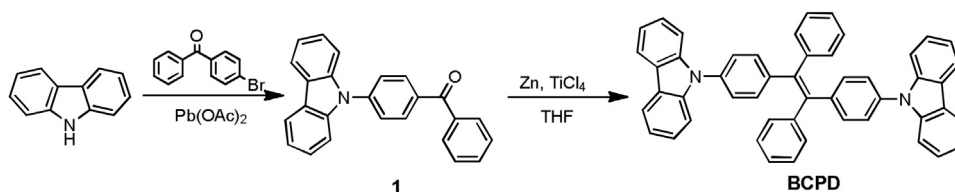
2.3. Synthesis of BCPD

2.3.1. (4-(9H-carbazol-9-yl)phenyl)(phenyl)methanone (1)

Compound 1 was synthesized according to the reported procedure [36]. ^1H NMR (600 MHz, CDCl_3 , ppm): δ 8.19–8.17 (d, $J = 7.10 \text{ Hz}$, 2H), 8.11–8.09 (d, $J = 8.10 \text{ Hz}$, 2H), 7.93–7.91 (d, $J = 6.40 \text{ Hz}$, 2H), 7.77–7.74 (d, $J = 6.50 \text{ Hz}$, 2H), 7.64 (t, $J = 6.80 \text{ Hz}$, 1H), 7.62–7.55 (d, $J = 7.30 \text{ Hz}$, 4H), 7.49–7.47 (t, $J = 6.40 \text{ Hz}$, 2H), 7.38–7.36 (t, $J = 6.90 \text{ Hz}$, 2H).

2.3.2. 1,2-bis(4-(9H-carbazol-9-yl)phenyl)-1,2-diphenylethylene (BCPD)

In a 250 mL flask equipped with a stirrer were added of zinc powder (4.47 g, 68.8 mmol) and THF (150 mL). TiCl_4 (4.3 mL, 34.4 mmol) was slowly added by a syringe at -78°C under nitrogen. The mixture was restored to room temperature and then heated to reflux for 4 h. After cooled to room temperature, compound 1 (3.00 g, 8.6 mmol) in THF solution (40 mL) was added to the mixture. The resulting mixture was heated under 70°C for 12 h. Then, the reaction was quenched with 10% K_2CO_3 aqueous solution and extracted with CH_2Cl_2 . The organic layer was collected and concentrated. The crude product was purified by silica-gel chromatography (dichloromethane/petroleum ether, $v/v = 1/1$) to



Scheme 1. Synthetic route for BCPD.

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