



Fluorene derivatives for highly efficient non-doped single-layer blue organic light-emitting diodes



Xin Jiang Feng^{a,b,*}, Shao Fu Chen^{a,1}, Yong Ni^{a,1}, Man Shing Wong^{b,*}, Maggie M.K. Lam^{c,2}, Kok Wai Cheah^{c,*}, Guo Qiao Lai^{a,*}

^a Key Laboratory of Organosilicon Chemistry and Material Technology of Ministry of Education, Hangzhou Normal University, Hangzhou 310012, PR China

^b Department of Chemistry and Institute of Advanced Materials, Hong Kong Baptist University, Kowloon Tong, Hong Kong Special Administrative Region

^c Department of Physics and Institute of Advanced Materials, Hong Kong Baptist University, Kowloon Tong, Hong Kong Special Administrative Region

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ABSTRACT

Diphenylamino- and triazole-encapped fluorene derivatives which show a wide energy band gap, a high fluorescence quantum yield and high stability have been synthesized and characterized. Single-layer electroluminescent devices of these fluorene derivatives exhibited efficient deep blue to greenish blue emission at low driving voltage. The single-layer OLED of PhN-OF(1)-TAZ shows a maximum current efficiency of 1.54 cd/A at 20 mA cm⁻² with external quantum efficiency (EQE) of 2.0% and CIE coordinates of (0.153, 0.088) in deep blue region, while the single-layer device of oligothiénylfluorene PhN-OFOT-TAZ shows a maximum brightness of 7524 cd/m² and a maximum current efficiency of 2.9 cd/A with CIE coordinates of (0.20, 0.40) in greenish blue.

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1. Introduction

Organic light-emitting diodes (OLEDs) have been recently realized in consumer electronics for practical applications in small-size active matrix OLED displays and solid-state lightings due to the success of materials development and device engineering [1–6]. Among the three principal colors necessary for full color OLED display applications, blue-emitting materials and devices are still needed to be improved the most in terms of efficiency, lifetime and color purity owing to the far superior stabilities

and device performances of green and red emitters. Besides, blue-emitting materials can also be the host for dopant emitters because of its intrinsic large energy band-gap. However, only a handful of deep-blue OLEDs based on organic emitters achieved high external quantum efficiency (EQE) up to date [7–12]. Hence, the development of blue emitters for highly efficient and stable full color OLED applications still remains a challenge because these materials require possessing a high fluorescence yield, a wide energy band gap, a high thermal stability and good thin film morphology [13].

Some materials having both electron-rich and electron-deficient moieties show abilities of electron-transporting and hole-transporting. They can effectively stabilize exciton and balance the charges in the emitting layer, and thus improve device performance in OLEDs [14–18], in spite of their shortcomings [19–27]. Nevertheless, such materials with wide band gap, highly efficient and stable blue emission are still to be developed [28–33].

Oligofluorenes non-conjugated with highly electron-rich triphenylamines and electron-deficient triazole (TAZ)

* Corresponding authors. Address: Key Laboratory of Organosilicon Chemistry and Material Technology of Ministry of Education, Hangzhou Normal University, Hangzhou 310012, PR China. Tel./fax: +86 571 28862271 (X.J. Feng), fax: +852 34117348 (X.J. Feng, M.S. Wong), fax: +852 34115813 (K.W. Cheah).

E-mail addresses: xjfeng@hznu.edu.cn (X.J. Feng), mswong@hkbu.edu.hk (M.S. Wong), kwcheah@hkbu.edu.hk (K.W. Cheah), gqlai@hznu.edu.cn (G.Q. Lai).

¹ Fax: +86 571 28862271.

² Fax: +852 34115813.

moieties have been demonstrated to exhibit high thermal stability and good OLED device performance in our previous study [29]. However, significant red-shift was observed for the oligomer with higher conjugation and similar phenomenon was reported by other studies, which was due to the oxidation of 9-position on fluorine [34,35]. So a comparatively small molecule is of advantage over large ones in device fabrication by sublimation method. On the other hand, donor–acceptor oligofluorenes conjugated with diphenylamino and triazole groups have shown high fluorescence quantum yields and excellent multiphoton absorption properties [36]; and we expect good electroluminescence of these conjugated materials with the advantage in intramolecular charge transfer over the corresponding non-conjugated ones. Furthermore, introduction of thienyl ring into a molecule can reduce the energy gap effectively and thus tune the color in device [37,38].

In this contribution, we reported herein the synthesis and electroluminescence performance of three diphenylamino- and triazole-conjugated oligofluorene derivatives, PhN-OF(*n*)-TAZ (*n* = 1, 2) and oligothiénylfluorene PhN-OFOT-TAZ as a non-doped blue emitter in single-layer OLEDs. It has been found that these materials exhibit high quantum yield and thermal stability. The single-layer OLED of PhN-OF(1)-TAZ shows a maximum current efficiency of 1.54 cd/A at 20 mA cm⁻² (external quantum efficiency (EQE) of 2.0%) with CIE coordinates of (0.153, 0.088) in deep blue region, while the OLED of PhN-OF(2)-TAZ shows a maximum current efficiency of 1.8 cd/A (EQE of 1.1%) and a maximum brightness of 5349 cd/m² with CIE coordinates of (0.16, 0.23) in blue region. Furthermore, the single-layer device of PhN-OFOT-TAZ shows a maximum brightness of 7524 cd/m² and a maximum current efficiency of 2.9 cd/A (EQE of 2.0%) with CIE coordinates of (0.20, 0.40) in sky blue to greenish blue. These results are among the best performance reported for blue emission of small molecule OLEDs in non-doped single-layer devices [29,39–41].

2. Experimental

2.1. Synthesis of 4-*N*-phenyl-5-(4-*tert*-butylphenyl)-3-{4-[9',9'-di-*n*-butyl-7'-diphenylamino-2'-fluorenyl]phenyl}-1,2,4-triazole (PhNOF(1)-TAZ) [42]

A mixture of 9,9-di-*n*-butyl-7-diphenylamino-2-fluorenylboronic acid (**1**, 1.93 g, 3.94 mmol), **2** (1.14 g, 2.63 mmol), Pd(OAc)₂ (30 mg, 0.13 mmol), P(*o*-tolyl)₃ (80 mg, 0.26 mmol), 2 M K₂CO₃ (4 mL), toluene (20 mL) and methanol (8 mL) under nitrogen atmosphere was heated at 75 °C overnight with good magnetic stirring. After cooling to room temperature, the reaction mixture was poured into water and extracted with ethyl acetate (30 mL), followed by dichloromethane (30 mL). The combined organic layer was dried over anhydrous Na₂SO₄, evaporated to dryness. The residue was purified by silica gel column chromatography using 8:1 dichloromethane:ethyl acetate as eluent affording 1.74 g (83%) of a white solid. ¹H NMR (400 MHz, CDCl₃) 7.63–7.47 (m, 10H), 7.37 (d, *J* = 8.4 Hz, 2 H), 7.31 (d, *J* = 7.6 Hz, 2 H), 7.27 (m, 6 H), 6.99–7.25 (m, 9 H), 1.85–1.91 (m, 4 H),

1.29 (s, 9 H), 1.04–1.09 (m, 4 H), 0.63–0.71 (m, 10 H). ¹³C NMR (100 MHz, CDCl₃) 154.8, 154.4, 152.9, 152.4, 151.4, 147.9, 147.3, 142.7, 140.8, 137.9, 135.6, 135.5, 130.0, 129.7, 129.1, 129.0, 128.4, 128.0, 126.9, 125.9, 125.4, 123.9, 123.4, 122.5, 121.0, 120.5, 119.4, 119.2, 55.1, 40.0, 34.7, 31.1, 26.0, 23.0, 13.8. HRMS (MALDI-TOF) *m/z* calcd for C₅₇H₅₆N₄ 796.4505, found [*M*⁺] 796.4512.

2.2. Measurements

¹H and ¹³C NMR spectra were recorded on a Bruker AM 400 spectrometer. Mass spectrometric measurements were recorded by a HP5989 mass spectrometer. UV-vis spectra were obtained on a Varian Cary 200 spectrophotometer. Fluorescence spectra were obtained on a Perkin-Elmer LS55 luminescence spectrometer. The differential scanning calorimetry (DSC) analysis was performed under a nitrogen atmosphere on a TA Instruments DSC 2920. Thermogravimetric analysis was undertaken using a TGA instrument (PE-TGA6). To measure the fluorescence quantum yields (Φ_F), degassed solutions of the compounds in different solvents were prepared and quinine sulfate monohydrate (Φ₃₅₀ = 0.58) was used as a standard, while the absolute quantum yields Φ_F of the dyes in powder were measured by Edinburgh Photonics FLS920 fluorescence spectrometer. The concentration was adjusted so that the absorbance of the solution would be lower than 0.1. Cyclic voltammetric (CV) measurements were carried out in a conventional three-electrode cell, using a Pt button working electrode 2 mm in diameter, a platinum wire counter electrode, and a SCE reference electrode on a computer-controlled EG&G Potentiostat/Galvanostat model 283 at room temperature. Reduction CV of all compounds was performed in dichloromethane containing Bu₄NPF₆ (0.1 M) as the supporting electrolyte.

2.3. Device fabrication

Prior to the deposition of organic materials, indium–tin oxide (ITO)/glass was cleaned with a routine cleaning procedure and pretreated with oxygen plasma. Devices were fabricated under about 10⁻⁶ Torr base vacuum in a thin-film evaporation coater following a published protocol [43]. The current–voltage–luminance characteristics were

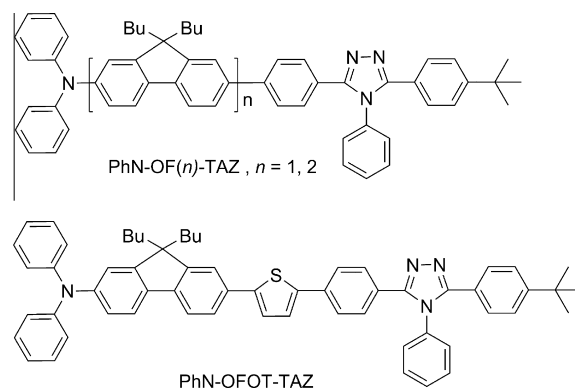


Fig. 1. Molecular structure of fluorene derivatives.

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