



# Triphenyl phosphine oxide-bridged bipolar host materials for green and red phosphorescent organic light-emitting diodes



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## ARTICLE INFO

### Article history:

Received 23 June 2015

Received in revised form

3 September 2015

Accepted 3 September 2015

Available online xxx

### Keywords:

Triphenyl phosphine oxide  
Phosphorescent organic light-emitting diode

Thermal stability  
Triplet energy level

Host materials

Electroluminescent performance

## ABSTRACT

Two wide band gap functional compounds of phenylbis(4-(spiro [fluorene-9,9'-xanthen]-2-yl)phenyl) phosphine oxide (**2SFOP**O) and (4-(9-ethyl-9H-carbazol-3-yl)phenyl)(phenyl)(4-(spiro[fluorene-9,9'-xanthen]-2-yl)phenyl)phosphine oxide (**SFOP**O-**CZ**) were designed, synthesized and characterized. Their thermal, photophysical, electrochemical properties and device applications were further investigated to correlate the chemical structure of bipolar host materials with the electroluminescent performance for phosphorescent organic light-emitting diodes (PhOLEDs). Both of them show high thermal stability with glass transition temperatures in a range of 105–122 °C and thermal decomposition temperatures at 5% weight loss in a range of 406–494 °C. The optical band gaps of compound **2SFOP**O and **SFOP**O-**CZ** in CH<sub>2</sub>Cl<sub>2</sub> solution are 3.46 and 3.35 eV, and their triplet energy levels are 2.51 eV and 2.52 eV, respectively. The high photoluminescent quantum efficiency of emissive layer of doped green device up to 50% is obtained. Employing the developed materials, efficient green and red PhOLED in simple device configurations have been demonstrated. As a result, the green PhOLEDs of compound **SFOP**O-**CZ** doped with tris(2-phenylpyridine) iridium shows electroluminescent performance with a maximum current efficiency ( $CE_{max}$ ) of 52.83 cd A<sup>-1</sup>, maximum luminance of 34,604 cd/m<sup>2</sup>, maximum power efficiency ( $PE_{max}$ ) of 39.50 lm W<sup>-1</sup> and maximum external quantum efficiency ( $EQE_{max}$ ) of 14.1%. The red PhOLED hosted by compound **2SFOP**O with bis(2-phenylpyridine)(acetylacetonato) iridium(III) as the guest exhibits a  $CE_{max}$  of 20.99 cd A<sup>-1</sup>, maximum luminance of 33,032 cd/m<sup>2</sup>,  $PE_{max}$  of 20.72 lm W<sup>-1</sup> and  $EQE_{max}$  of 14.0%. Compound **SFOP**O-**CZ** exhibits better green device performance, while compound **2SFOP**O shows better red device performance in PhOLEDs.

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## 1. Introduction

On account of the advantages of organic light-emitting diodes (OLEDs) relative to their inorganic counterparts, the academics have devoted a lot of time to study OLEDs for their wide applications in the next generation full color flat panel displays and low-

cost solid-state lighting in the past few decades [1–6]. It is generally acknowledged that the ratio of singlet and triplet excitons formed under electrical excitation is approximately 1:3 [7], and phosphorescent OLEDs (PhOLEDs) based on heavy metal complexes can elevate the internal quantum efficiency up to 100% through making full use of both singlet and triplet excitons, which surmounts the confine of 25% efficiency of fluorescent OLEDs with emission solely from singlet excitons [8–10]. Generally, in order to suppress the concentration quenching, a phosphorescent emitter is dispersed in a suitable host material to obtain a high quantum yield. After a thriving development in the electrophosphorescent devices over recent years, the challenges to synthesize ideal host materials are still underway. Ideal host materials should have excellent carrier injecting/transporting ability, high thermal stability, good morphological, and film-forming ability, high triplet energy levels ( $T_1$ ), suitable molecular orbital of highest occupied molecular

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orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) [11–23]. To meet these requirements, much work has been done to develop rational host materials with balancing charge-carrier injection and transport ability in the past ten years [24]. Especially, some effective strategies for the host materials design exhibiting reduced triplet–triplet annihilation (TTA), such as unsymmetrical configuration and special linkage model, have been developed [25–28]. The bipolar host materials with noncoplanar and unsymmetrical configuration, excellent electron and hole transporting abilities are helpful to simplify the device structure and reduce the cost of PhOLEDs.

The triphenyl phosphine oxide group with triangular pyramidal configuration has attracted broad attentions. In addition, this group with electron-deficient feature can polarize the molecule framework and promote electron-transporting ability of the obtained materials [28–34]. In this regard, many phosphine oxide-related host materials have been synthesized, which have shown good thermal stability, high  $T_1$  values and relatively good device performance [35–38]. To the best of our knowledge, despite the great progress that has been made in the bipolar host materials based on the phosphine oxide structure for PhOLEDs, it is still far from meeting the requirements of commercialization. Carbazole, as a kind of rigid plane biphenyl compound with wide band gap, high luminescence efficiency and high flexibility to modify the molecule skeleton, has been a key chromophore in the synthesis of host materials for PhOLEDs [39–41]. The introduction of noncoplanar spirocyclic aromatic structure into carbazole molecular skeleton can increase glass transition temperature ( $T_g$ ), restrict crystallization, weaken the formation of aggregation or excimer related species, and improve the photostability. Fortunately, Xie et al. reported the derivatives based on spiro[fluorene-9,9'-xanthene] (SFX) and their applications in PhOLEDs [42–44]. The carbazole and SFX moieties with different optoelectronic properties can finely adjust the carrier injecting/transporting ability. The introduction of the moieties of SFX and carbazole into the skeleton of triphenyl phosphine oxide with “Y” shape in space can then be implemented to finish this consideration. So in this contribution, we synthesized and characterized two wide band gap noncoplanar Y-shaped aromatic compounds named phenylbis-(4-(spiro[fluorene-9,9'-xanthene]-2-yl) phenyl) phosphine oxide (**2SFOPO**), and (4-(9-ethyl-9H-carbazol-3-yl)phenyl) (phenyl)(4-(spiro[fluorene-9,9'-xanthene]-2-yl)- phenyl)phosphine oxide (**SFOPO-CZ**) (as shown in Scheme 1). The green PhOLED device hosted by compound **SFOPO-CZ** gave a maximum external quantum efficiency ( $EQE_{max}$ ) of 14.1%, and red PhOLED device incorporating compound **2SFOPO** as the host showed a  $EQE_{max}$  as high as 14.0%.

## 2. Experimental

### 2.1. Materials and measurements

Materials purchased from commercial suppliers were used without further purification. Tetrahydrofuran and toluene were distilled over sodium/benzophenone under nitrogen atmosphere. The other common solvents were purified according to their standard methods. Both  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR spectra were measured by a Varian Mercury Plus 400 spectrometer with  $\text{CDCl}_3$  and DMSO as solvents and tetramethylsilane as internal reference. Chemical shifts were reported in ppm relative to  $\text{CDCl}_3$  and DMSO as internal standard.  $\text{CHCl}_3$  was set to be 7.26 ppm and DMSO was set to be 2.5, 3.4, and coupling constant was expressed in hertz. The signals have been designed as follows: s(singlet), d(doublet), t(triplet), m(multiplet). Matrix assistant laser desorption/ionization time-of-flight mass (MALDI-TOF-MS) spectrometry was performed with a Bruker Daltonics flex Analysis. Elemental analysis of carbon,

hydrogen and nitrogen was obtained by Elementar Vario MICRO elemental analyzer. UV–Vis absorption spectra were recorded on an UV-3600 SHIMADZU UV–Vis–NIR spectrophotometer. The photoluminescent emission spectra were recorded on a RF-5301PC spectro fluorophotometer with a xenon lamp as a light source. The concentration of these compounds in tetrahydrofuran solution was adjusted to be about 0.01 mg/mL or less. The thin solid films were prepared by spin-coating on quartz substrates from solution in  $\text{CDCl}_3$  at a spin rate of 3500 rpm. Time-resolved luminescence lifetime measurements were carried out by using time-correlated single-photon counting lifetime spectroscopy Edinburgh FL920 system with a semiconductor laser as the excitation source ( $\lambda_{\text{ex}} = 340 \text{ nm}$ ). Differential scanning calorimetry (DSC) measurements were carried out using a Perkin–Elmer Diamond DSC at a heating rate of  $10 \text{ }^\circ\text{C min}^{-1}$  from room temperature to  $500 \text{ }^\circ\text{C}$  under nitrogen. Thermogravimetric analysis (TGA) was measured by a SEIKO EXSTAR 6000 TG/DTA 6200 unit under nitrogen atmosphere at a heating rate of  $10 \text{ }^\circ\text{C min}^{-1}$ . Cyclic voltammetric (CV) measurements were carried out on the Chi660e system in a conventional three-electrode cell with a Pt work electrode (glass carbon), a platinum-wire counter electrode and a  $\text{Ag}/\text{Ag}^+$  reference electrode referenced against ferrocene/ferrocenium (FOC) in anhydrous dichloromethane solution of  $\text{Bu}_4\text{NPF}_6$  (0.10 M) at a sweeping rate of  $100 \text{ mV/s}$  at room temperature. The HOMO and LUMO energy levels were measured by CV and calculated according to the formula:  $\text{HOMO} = -E_{\text{oxi}} + 0.0042 - 4.8$ ,  $\text{LUMO} = -E_{\text{red}} - 0.061 - 4.8$ , where the 0.0042 and  $-0.061$  are the oxidation potential and reduction potential of FOC, respectively.  $E_{\text{oxi}}$  and  $E_{\text{red}}$  are the oxidation and reduction potentials of compounds.

### 2.2. Device fabrication

The hole injection material of  $\text{MoO}_3$ , hole transporting material of 4,4-N, N-dicarbazolylbiphenyl (CBP), electron-transporting material of 1,3,5-tris(N-phenyl- benzimidazol-2-yl)benzene (TPBI), and the phosphorescent dopants of tris(2-phenylpyridine)iridium(III) [ $\text{Ir}(\text{ppy})_3$ ] and iridium(III) bis(2-phenylquinoline)-(acetylaceton) [ $\text{Ir}(\text{pq})_2(\text{acac})$ ] are commercially available. Indium tin oxide (ITO) substrates with a sheet resistance of  $10 \text{ } \Omega$  per square were ultrasonically cleaned with detergent, de-ionized water, acetone, and isopropyl alcohol. Prior to deposition, the cleaned and dried ITO glass substrates were treated with UV-ozone for 5 min. All layers were deposited by thermal evaporation in a high vacuum system under a pressure of  $5 \times 10^{-4} \text{ Pa}$ . The emission area of the device is  $4 \times 4 \text{ mm}^2$  as defined by the overlapping area of the anode and cathode. The current–voltage–brightness characteristics were measured on a computer-controlled Keithley source measurement unit (Keithley 2400 and Keithley 2000) and a calibrated silicon photodiode. The electroluminescent (EL) spectra and Commission International de l'Eclairage (CIE) coordinates were obtained by using a spectra-scan PR655 spectrophotometer. All the measurements were carried out at room temperature under ambient conditions.

### 2.3. Synthesis of the target compounds

**3-Bromo-9-ethyl-9H-carbazole:** Under nitrogen atmosphere, a mixture of 3-bromo-9H-carbazole (13 g, 40 mmol), bromoethane (5.24 g, 40 mmol), KOH(6.72 g, 120 mmol) and 120 ml dimethyl sulphoxide were stirred at  $50 \text{ }^\circ\text{C}$  for 24 h. After cooling, the reaction mixture was quenched by the addition of water and extracted with  $\text{CH}_2\text{Cl}_2$ . The combined organic layer was washed with dilute HCl and brine several times, dried over anhydrous  $\text{MgSO}_4$ , filtered and evaporated under vacuum. The crude product was purified by recrystallization from ethanol. A white solid was obtained by

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