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Graphical abstracts



Benzoselenadiazole-based Donor-Acceptor Small Molecule: Synthesis, Aggregation-Induced Emission and Electroluminescence

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Abstract: Several tetraphenylethene and triphenylamine decorated donor-acceptor (D-A) benzoselenadiazole derivatives with aggregation-induced emission (AIE) features were synthesized and characterized. The photophysical, thermal, electrochemical and electroluminescent (EL) properties, as well as the AIE property of the resulting luminogens are discussed. All the luminogens are soluble in common organic solvents and show high thermal stability with a T_d of 399-460 °C. The absolute photoluminescence quantum yield in the solid state ranged from 14.2% to 36.5%. The D-A structures of TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA enable them to have aggregation-caused quenching (ACQ) plus aggregation-induced emission enhancement (AIEE) effects dominated by the twisted intramolecular charge-transfer (TICT) and restriction of intramolecular rotations (RIR), respectively. The best device performances were obtained from TPA-Se-TPE with a maximal luminous efficiency of 2.91 cd/A and the maximal brightness is 5855 cd/m². This is the first report of benzoselenadiazole-based fluorophore with an AIE feature for organic electronics application.

Keywords: orgainc light-emitting diode; aggregation-induced emission; benzoselenadiazole; tetraphenylethene; triphenylamine

1. Introduction

Organic light-emitting diodes (OLEDs) have attracted considerable attention due to their potential applications in flat-panel displays and solid-state lighting [1-9], with tremendous advances such as thin-film formation, high contrast, low weight, fast response, wide-view angle and low power consumption. Organic optoelectronic functional molecules are usually used as films or aggregates [10-12]. However, traditional luminophores generally suffer from the aggregation-caused quenching (ACQ) effect: they are highly emissive in dilute solution but become weakly or nonemissive in high concentration or condensed phases like aggregates or solid state due to their planar conjugated structures. The ACQ effect has reduced the performance of traditional luminophores when used in optoelectronic devices. In order to alleviate this negative effect, various approaches have been adopted, such as physical doping or chemical modification with bulky groups [13-20]. Nevertheless, it is difficult to precisely control the doping concentration and avoid the phase separation between the host and the guest during usage in the physical doping system. Whereas, in the latter chemical modification case, tedious synthetic procedures and a lengthened operating time are also involved. In other words, these methods met with limited success. The discovery of aggregation-induced emission (AIE) molecular systems which exhibit either no or weak luminescence in solution but exhibit strong luminescence in the aggregated state supplies a novel method to solve the problem of ACQ [21, 22].

Among the reported AIE fluorophores, tetraphenylethene (TPE) is a classical luminogen with intriguing AIE characteristic [23, 24]. TPE and TPE-based materials have received the most attention owing to their well-defined AIE properties, high solid-state fluorescence quantum yields and ease of synthesis, which has been widely used in optic- and electro-active materials due to its electron-donating property and

propeller-like molecular structure [25]. The triphenylamine (TPA) moiety is well known for its luminescence properties, electron-donating ability, and hole transporting capability. Recently, much work incorporating the TPA moiety into the construction of new AIE luminogens (AIEgens) has been conducted [26-28]. The three phenyl rotors and the nonplanar structure of TPA make it an ideal building block for AIEgens. The rotational motions of the phenyl rings in TPA aid in dissipating excited-state energy in the solution state and assist in restriction of intramolecular rotations (RIR) in the solid state. Its twisted structure helps reduce the formation of destructive intermolecular π - π stacking interactions.

The benzoselenadiazole is an excellent acceptor with good electron-withdrawing ability, which has a higher electron affinity than benzothiadiazole, thus using benzoselenadiazole as an acceptor is conductive to promoting intramolecular charge-transfer (ICT) process and constructing D-A materials for OLEDs [29-31]. In this manuscript, we have synthesized three donor-acceptor benzoselenadiazole based small molecules by Suzuki coupling reaction. The photophysical, thermal, electrochemical and electroluminescent (EL) properties, as well as the AIE property of the resulting luminogens are discussed. All luminogens show high thermal stability with a T_d nearly or above 400 °C. The best device performances were obtained from TPA-Se-TPE with a maximal luminous efficiency of 2.91 cd A⁻¹ and the maximal brightness is 5855 cd m⁻². This is the first report of a benzoselenadiazole-based fluorophore with an AIE feature which was successfully applied to OLEDs.

2. Experimentals

2.1. Materials

N-phenyl-N-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)benzenamine (7) was prepared according to the reported procedures [32]. All reactions were

performed under nitrogen. Tetrahydrofuran (THF) was distilled under normal pressure from sodium benzophenone ketyl under nitrogen immediately prior to use. Other solvents and chemicals were used without further purification unless otherwise stated.

Synthesis of 4,7-Dibromo-2,1,3-benzothiadiazole (1)[30]: A mixture of 2,1,3benzothiadiazole (13.6 g, 0.1 mol) in 45% hydrobromic acid (30 mL) was heated under reflux with stirring while 48 g (0.3 mol, 15.3 mL) of bromine was added slowly. The reaction mixture became a suspension of solid in hydrobromic acid and 15 mL of hydrobromic acid was added, then the mixture was refluxed for another 2.5 h. After cooling to room temperature, the precipitate formed and collected, washed with water, then recrystallized from chloroform to give yellow needle crystals (26.8 g, 91%). mp 186-188 °C. ¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.73 (s, 2H). ¹³C NMR (100 MHz, CDCl₃) δ (ppm): 153.34, 132.67, 114.31. Anal. Calcd for C₆H₂Br₂N₂S: C, 24.51; H, 0.69; N, 9.53. Found: C, 24.12; H, 0.72; N, 9.66.

Synthesis of 4,7-Dibromo-2,1,3-benzoselenadiazole (3)[30]: To a suspension of 4,7-dibromo-2,1,3-benzothiadiazole (1) (5.88 g, 20 mmol) in ethanol (190 mL), sodium borohydride (14 g, 0.37 mol) was added portionwise at 0 °C, then the mixture was stirred for 20 h at room temperature. After evaporation of the volatile solvent and water (200 mL) was added, the mixture was extracted with dichloromethane (3×30 mL). The extract was dried over anhydrous magnesium sulfate and evaporated to remove the solvents under vacuum. 3,6-Dibromo-1,2-phenylenediamine (2) (4.7 g) was obtained as a pale yellow solid in 87% yield. The intermediate 2 was directly used in the following procedure. A solution of selenium dioxide (1.17 g, 10.5 mmol) in hot water (22 mL) was added to a solution of 2 (2.7 g, 10 mmol) in refluxing ethanol (55 mL). The reaction mixture was heated under reflux for 2 h. After the mixture had been cooled to room temperature, filtration of the yellow precipitates and

recrystallization from ethyl acetate gave 4,7-dibromo-2,1,3-benzoselenadiazole (**3**) (2.8 g) in 82% yield as golden yellow needles. ¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.65 (s, 2H). mp 285-287 °C. ¹³C NMR (100 MHz, CDCl₃) δ (ppm): 157.2, 132.1, 116.5. Anal. Calcd for C₆H₂Br₂N₂Se: C, 21.14; H, 0.59; N, 8.22. Found: C, 21.34; H, 0.92; N, 7.98.

Synthesis of 1-bromo-4-(1,2,2-triphenylvinyl)benzene (5) [33]: n-Butyllithium (1.6 mol/L, 10 mmol) was added dropwise to a solution of diphenylmethane (1.77 g, 10.5 mmol) in anhydrous THF (50 mL) at 0 °C. The reaction mixture was stirred for 0.5 h, and then turned to red before (4-bromophenyl)phenylmethanone (3.38 g, 10 mmol) was added in one portion. The mixture was warmed to room temperature, stirred for 6 h and then was poured into water while stirring. The mixture was extracted with dichloromethane $(3 \times 50 \text{ mL})$ and the combined extracts were dried over anhydrous MgSO₄. The solvent was evaporated and the resultant crude alcohol 4 (containing excess diphenylmethane) was subjected to acid-catalyzed dehydration as follows. Compound 4 was dissolved in toluene (50 mL) in a 100 mL Schlenk flask fitted with a Dean-Stark trap. A catalytic amount of *p*-toluenesulfonic acid (342 mg, 1.8 mmol) was added and the mixture was refluxed for 3 h. After cooled to room temperature, the toluene layer was washed with 10% aqueous NaHCO₃ solution (2 \times 25 mL) and dried over anhydrous MgSO₄. The solvent was evaporated under reduced pressure the crude product was purified with column chromatography on silica gel with petroleum ether/ dichloromethane (15:1) as eluent to afford a residue of 5 (1.2 g) in yield of 63%. mp 216-218 °C. ¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.25–7.20 (d, J = 8.06 Hz, 2H), 7.17–7.08 (m, 9H), 7.07–6.99 (m, 6H), 6.94–6.87 (d, J = 8.14 Hz, 2H). ¹³C NMR (100 MHz, CDCl₃), δ (ppm): 148.06, 143.19, 143.14, 143.09, 141.75,

140.63, 134.72, 132.62, 131.21, 130.78, 130.69, 127.62, 127.54, 126.37, 126.23. Anal. Calcd for C₂₆H₁₉Br: C, 75.92; H, 4.66. Found: C, 76.14; H, 4.52.

4,4,5,5-tetramethyl-2-(4-(1,2,2-triphenylvinyl)phenyl)-1,3,2-**Synthesis** of dioxaborolane (6)[34]: To a solution of compound 5 (3.22 g, 10 mmol) in anhydrous THF (15 mL), n-Butyllithium (1.6 mol/L, 7.5 mL, 12 mmol) was added dropwise at -78 °C. The reaction mixture was stirred for 0.5 h before isopropoxyboronic acid pinacol ester (2.5 mL, 12 mmol) was added in one portion. The mixture was warmed to room temperature and stirred for 12 h. Then the mixture was poured into water while stirring. The organic phase was separated and the aqueous phase was extracted with dichloromethane (3×30 mL). The combined organic layers were dried over MgSO₄ and evaporated to remove the solvents under vacuum. The residue was purified by column chromatography (petroleum ether/dichloromethane = 8/1) to provide 6 (1.6 g) as white solid in 50% yield. mp 203-205 °C. ¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.54–7.52(d, J = 8.04 Hz, 2H), 7.09–7.07(m, 8H), 7.04–6.98 (m, 9H). ¹³C NMR (100 MHz, CDCl₃), δ (ppm): 148.26, 143.49, 143.44, 143.39, 141.75, 140.73, 134.92, 132.82, 131.30, 130.98, 130.89, 127.71, 127.65, 126.66, 126.53, 84.11, 22.99. Anal. Calcd for C₃₂H₃₁BO₂: C, 83.84; H, 6.82. Found: C, 83.06; H, 7.03.

Synthesis of TPE-Se-TPE: $Pd(PPh_3)_4$ (143 mg) was added to a mixture of **3** (340 mg, 1.0 mmol), **6** (1.14 g, 2.5 mmol), THF (50.0 mL) and 2.0 M K₂CO₃ solution (10.0 mL). The mixture was degassed and heated under reflux with vigorously stirring for 24 h under N₂ flow protection. After cooled to room temperature, the mixture was poured into water and extracted with dichloromethane (3×30 mL). The combined organic layers were collected, dried over anhydrous MgSO₄, filtered and evaporated to remove the solvent. The resulted crude product was chromatographed on a silica gel column (petroleum ether/dichloromethane = 10/1) to give TPE-Se-TPE (565 mg)

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in a yield of 67%. ¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.68–7.66 (d, J = 8.28 Hz, 4H), 7.56 (s, 2H), 7.19–7.16 (d, J = 8.32 Hz, 4H), 7.13–7.09 (m, 26H), 7.06–7.04 (m, 4H). ¹³C NMR (100 MHz, CDCl₃) δ (ppm): 159.66, 143.80, 143.68, 143.62, 141.42, 140.57, 135.97, 134.37, 131.51, 131.38, 128.65, 127.88, 127.73, 127.65, 126.59, 126.51. Anal. Calcd. for C₅₈H₄₀N₂Se: C, 82.55; H, 4.78; N, 3.32; Found: C, 82.59; H, 7.75; N, 3.39. MALDI-TOF (m/z): calcd for TPE-Se-TPE: 844.236, found 844.239.

Synthesis of TPA-Se-TPE: *N*-phenyl-*N*-(4-(4,4,5,5-tetramethyl-1,3,2-

dioxaborolan-2-yl)phenyl)benzenamine (**7**) (1.11 g, 3.0 mmol), **3** (1.12 g, 3.0 mmol), Pd(PPh₃)₄ (172 mg), THF (70 mL) and 2.0 M K₂CO₃ solution (15.0 mL) were added in a 150 mL two-necked round-bottomed flask. The mixture was refluxed for 24 h under argon atmosphere. After the mixture had been cooled to the room temperature, the mixture was poured into brine and extracted twice with dichloromethane. The combined organic layers were dried over MgSO₄ and the solvent was removed. The crude product was purified with column chromatography (silica gel, petroleum ether/dichloromethane (20/1) as eluent) to yield 894 mg (59%) of **8** as a light yellow solid. ¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.96–7.94 (d, *J* = 7.52 Hz, 1H), 7.83–7.81 (d, *J* = 8.68 Hz, 2H), 7.52–7.50 (d, *J* = 7.52 Hz, 1H), 7.37–7.33 (t, *J* = 7.90 Hz, 4H), 7.10–7.06 (m, 8H).

Pd(PPh₃)₄ (120 mg) was added to a mixture of **8** (1.05 g, 1.77 mmol), **6** (959 mg, 2.1 mmol), THF (50.0 mL) and 2.0 M K₂CO₃ solution (10.0 mL). The reaction mixture was degassed and heated under reflux with vigorously stirring for 24 h under N₂ flow protection. After cooling to room temperature, the mixture was poured into water and extracted with dichloromethane (3×30 mL). The combined organic layers were collected, dried over anhydrous MgSO₄, filtered and evaporated to remove the solvent. The resulted crude product was chromatographed on a silica gel column

(petroleum ether/dichloromethane = 12/1) to give TPA-Se-TPE (1.09 g) in a yield of 82%. ¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.88–7.86 (d, J = 8.72 Hz, 2H), 7.78–7.76 (d, J = 8.32 Hz, 2H), 7.69–7.64 (t, J = 9.28 Hz, 2H), 7.37–7.33 (t, J = 7.92 Hz, 4H), 7.19–7.03 (m, 23H), 7.01–6.99 (d, J = 7.96 Hz, 2H). ¹³C NMR (CDCl₃, 100 MHz) δ (ppm): 147.51, 131.52, 131.38, 130.21, 129.36, 128.62, 127.73, 127.65, 126.51, 124.89, 123.27, 122.85, 122.81, 122.07. Anal. Calcd. for C₅₀H₃₅N₃Se: C, 79.35; H, 4.66; N, 5.55; Found: C, 79.42; H, 4.73; N, 5.59. MALDI-TOF (m/z): calcd for TPA-Se-TPE: 757.2, found 757.212.

Synthesis of TPA-Se-TPA: Pd(PPh₃)₄ (143 mg) was added to a mixture of **3** (340 mg, 1.0 mmol), **7** (928 mg, 2.5 mmol), THF (60.0 mL) and 2.0 M K₂CO₃ solution (10.0 mL). The mixture was degassed and heated under reflux with vigorously stirring for 24 h under N₂ flow protection. After cooled to room temperature, the mixture was poured into water and extracted with dichloromethane (3×30 mL). The combined organic layers were collected, dried over anhydrous MgSO₄, filtered and evaporated to remove the solvent. The resulted crude product was chromatographed on a silica gel column (petroleum ether/dichloromethane = 15/1) to give TPA-Se-TPA (509 mg) in a yield of 76%. ¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.80–7.78 (d, *J* = 8.64 Hz, 4H), 7.59 (s, 2H), 7.31–7.27 (t, *J* = 7.86 Hz, 8H), 7.21–7.18 (m, 12H), 7.07–7.04 (t, *J* = 7.28 Hz, 4H). ¹³C NMR (CDCl₃, 100 MHz) δ (ppm): 147.90, 147.56, 134.02, 131.72, 129.34, 127.91, 124.87, 123.24, 122.89. Anal. Calcd. for C₄₂H₃₀N₄Se: C, 75.33; H, 4.52; N, 8.37; Found: C, 75.38; H, 4.56; N, 8.29. MALDI-TOF (m/z): calcd for TPA-Se-TPA: 670.164, found 670.168.

2.2 Measurements and Characterization

¹H and ¹³C NMR spectra were recorded on a Bruker 400 spectrometer operating respectively at 400 and 100 MHz at room temperature. Chemical shifts were reported

as δ values (ppm) relative to an internal tetramethylsilane (TMS) standard. Time-offlight mass spectrometry (TOF-MS) was carried out on a Bruker-autoflex III smartbeam. Thermogravimetric analysis (TGA) was carried out on a Diamond TG/DTA instrument under a nitrogen atmosphere at a heating rate of 20 °C min⁻¹ and T_d was reported as the temperatures at 5% weight losses. UV-vis absorption spectra were measured on a HP 8453 spectrophotometer. PL spectra were recorded on an Instaspec IV CCD spectrophotometer (Oriel Co.) under 325 nm excitation of a HeCd laser. Cyclic voltammetry was carried out on a CHI660A electrochemical workstation in a solution of tetrabutylammonium hexafluorophosphate (Bu₄NPF₆) (0.1 M) in acetonitrile at a scan rate of 50 mV/s at room temperature under the protection of argon. A platinum electrode was used as the working electrode. A Pt wire was used as the counter electrode, and a calomel electrode was used as the reference electrode. The absolute photoluminescence quantum yield (Φ_{PL}) was measured with a Hamamatsu absolute PL quantum yield spectrometer equipped with an integrating sphere (Quantaurus-QY, C11347).

2.3 Theoretical Calculations

The geometries of both iridium complexes Y1 and Y2 at the gas phase were fully optimized by the density functional theory (DFT) method at the 6-31G* basis set with B3LYP functional [35, 36] without any symmetry constraints by using the Gaussian 09 software package [37].

2.4 OLEDs fabrication and measurements

The ITO-coated glass substrates were ultrasonically cleaned with deionized water, acetone, detergent, deionized water, and isopropyl alcohol. Then an MoO₃ hole injection layer (2 nm) was obtained by thermal deposition under a vacuum of 4×10^{-4} Pa. Then organic stacks of *N*,*N*'-bis(naphthalen-1-yl)-*N*,*N*'-bis(phenyl)-benzidine

(NPB, 50 nm), TPE-Se-TPE (or TPA-Se-TPE or TPA-Se-TPA) (20 nm) and 4,7diphenyl-1,10-phenanthroline (BPhen, 40 nm) served as hole transport layer, emitting layer and electron transport layer, respectively, were sequentially layered by thermal deposition under a vacuum of 4×10^{-4} Pa. Finally, LiF (1 nm)/Al (100 nm) was served as conventional bilayer cathode. The layer thickness and deposition rate were monitored and controlled *in situ* by using an oscillating quartz thickness monitor. The typical deposition rate of organic layer was 1-2 Å/s while that of Al was 3-5 Å/s. The current density (*J*) and brightness (*L*) versus voltage (*V*) data were collected using a Keithley 236 source meter and silicon photodiode. After typical encapsulation with UV epoxy and cover glass, the devices were taken out from the dry box and the luminance was calibrated by a PR-705 SpectraScan Spectrophotometer (Photo Research) with simultaneous acquisitions of the EL spectra.

3. Results and discussion

3.1 Synthesis and Characterization

The synthetic routines to TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA are shown in Scheme 1. The 4,7-Dibromo-2,1,3-benzoselenadiazole (**3**) was prepared in good yield over three steps starting from the readily commercially available materials. Firstly, 2,1,3-benzothiadiazole was brominized in HBr and recrystallized from chloroform to give yellow solid 4,7-Dibromo-2,1,3-benzothiadiazole (**1**) in the yield of 91%. Secondly, the compound **1** was reacted with NaBH₄ at 0 °C to obtain 3,6dibromobenzene-1,2-diamine (**2**). Thirdly, the compound **2** was added in ethanol solution containing selenium dioxide to produce the compound **3** with high yield [38]. After the lithiation of diphenylmethane, (4-bromophenyl)phenylmethanone was added to the reaction mixture to generate the resultant alcohol **4**, which underwent dehydration in the presence of PTSA to form **5** in 63% yield. Treatment of **5** with n-

butyllithium followed by reaction with isopropoxyboronic acid pinacol ester furnished compound **6** [33]. The target products TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA were synthesized by Suzuki coupling reaction of compound **3** with tetraphenylethene boronic ester (**6**) and triphenylamine boronic ester (**7**). The chemical structures of TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA were fully characterized by NMR spectroscopy, elemental analysis and time-of-flight mass spectrometry (TOF-MS). All target products are readily soluble in common organic solvents such as chloroform, dichloromethane and THF at room temperature, but are insoluble in water.

Photophysical Properties

The photophysical properties of the resultant compounds TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA in neat film were investigated. The UV-vis absorption and photoluminescence (PL) spectra of TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA at room temperature are shown in Figure 1. The UV-vis absorption spectra of the three target compounds showed two distinct absorption peaks in THF solution: 330 nm and 433 nm for TPE-Se-TPE, 329 nm and 461 nm for TPA-Se-TPE, 323 nm and 475 nm for TPA-Se-TPA. The peaks at short wavelength are attributed to the localized $\pi - \pi^*$ transition; and the other peaks at longer wavelength are due to the intramolecular charge transfer (ICT) transition between the donor and acceptor units. The compounds TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA displayed the maximum PL emission peaks in neat film at 552 nm, 615 nm and 624 nm, respectively. Compared to TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA exhibit much red-shifted emission with 63 nm and 72 nm. This suggests that the higher energy transfer between the triphenyamine unit and the benzoselenadiazole unit than that of the tetraphenylethene unit and the benzoselenadiazole unit. The absolute photoluminescence quantum yields (Φ_{PL}) for TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA in solid state are summarized in Table 1.

The values of TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA were recorded to be 14.2, 36.5 and 32.4%, respectively.

3.2 Electrochemical Properties and Theoretical Calculations

The electrochemical behavior of TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA were studied by cyclic voltammetry (CV) using ferrocene as the internal standard and the results are listed in Table 1. The oxidization peaks with onset potentials of TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA at 0.45, 0.13 and 0.09 eV, respectively, could be easily observed in the positive scan direction, which may be resulting from the oxidation of selenium atom in the benzoselenadiazole unit, where the selenium is an electron-rich atom and easy to be oxidized. The energy levels of the highest occupied molecular orbital (E_{HOMO}), determined by calculation from the empirical formula of $E_{\text{HOMO}} = -(4.8 + E_{\text{ox}})$, are -5.25, -4.93 and -4.89 eV. Unfortunately, no clear reduction waves of TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA were observed despite many attempts. The lowest unoccupied molecular orbital energy levels (E_{LUMO}) had to be calculated based on the equation of $E_{LUMO} = E_{HOMO} + E_{g}$, in which energy band gap (E_g) was estimated from the UV-vis absorption threshold. The E_{LUMO} of TPE-Se-TPE is calculated to be -2.77 eV, while those of TPA-Se-TPE and TPA-Se-TPA are -2.61 eV and -2.65 eV. To further investigate the structure-property relationship of the compounds TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA at the molecular level, the geometrical and electronic structures of the resulting compounds were performed using density functional theory, see details in theoretical calculations. The electron density distributions of both HOMO and LUMO for TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA are illustrated in Figure 2. The LUMO distrubutions of TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA were mainly located in the benzoselenadiazole moieties. While the HOMO distrubutions of TPE-Se-TPE and TPA-Se-TPA were

mainly located in the whole molecular framework, HOMO distrubutions of TPA-Se-TPE was mainly located in the triphenylamine moieties, which further indicated that there really existed obvious ICT in TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA. The calculated energies of HOMOs for TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA are -5.15 eV, -4.89 eV and -4.78 eV, respectively. The corresponding energies of LUMOs for TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA are -2.44 eV, -2.41 eV and -2.37 eV, respectively. Based on the calculated HOMO and LUMO energies for these target compounds, we found that the calculated HOMO–LUMO energy gap of TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA are 2.70 eV, 2.48 eV and 2.41 eV, which is qualitatively consistent with the experimental results by CV measurement. The results suggested that the more triphenylamine groups the energy band-gap is smaller, which is in agreement with the photophysical properties.

3.3 Thermal Properties

The thermal stability of these compounds was evaluated by thermogravimetric analysis (TGA) under a stream of N₂ with a scanning rate of 20 °C/min. Their TGA curves are shown in Figure 3 and their degradation temperatures (T_d) for 5% weight loss are listed in Table 1. The recorded T_d for TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA are 399, 406 and 460 °C, respectively. The data demonstrate that the resulting compounds functionalized by tetraphenylethene or triphenylamine can possess good thermal properties, which is desirable for the application in organic light-emitting diodes.

3.4 Aggregation-induced emission performances

The AIE properties of the compounds TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA were examined by studying the PL emission behaviour of their diluted mixtures in THF/water under different water fractions. The aggregates were prepared by adding

various fractions of ultra-pure water into the THF solutions. As shown in Figure 4, TPA-Se-TPE emits at 615 nm in pure THF solution $(10^{-5} \text{ mol } \text{L}^{-1})$. When the water fraction (f_w) is inferior to 40%, the emission of TPA-Se-TPE weakened with a redshift of their peaks at the same time. Specifically, more than 95% of its fluorescence intensity was quenched and a bathochromic shift of about 13 nm could be discerned at $f_{\rm w}$ of 40%. This may be attributed to the twisted intramolecular charge-transfer (TICT) effect of TPA-Se-TPE in the gradually strengthened polar mixed solvent of THF/water with a higher fraction of polar water [39-41]. Afterwards, the fluorescence intensity promptly enhanced with a blue-shift of peaks and reached the maximum value at f_w of 80% due to the aggregate formation. The mechanism of aggregation-induced emission enhancement (AIEE) may be attributed to the restriction of intramolecular rotation (RIR). The weaker emission of TPA-Se-TPE in the THF/water mixtures with f_w of 90% compared to that with f_w of 80% is probably due to the difference in aggregate morphology [42, 43]. The similar D-A structures of TPE-Se-TPE and TPA-Se-TPA also enable them to have ACQ plus AIEE effects dominated by the TICT and RIR, respectively (Figure S1, ESI[†]).

3.5 Electroluminescent Devices

To understand the electroluminescent properties of the compounds TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA, OLEDs were fabricated using them as emitting layers in the configuration: ITO/MoO₃/NPB/Emitting Layer/Bphen/LiF/Al. The EL spectra of the TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA are shown in Figure 5. The EL spectra of TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA displayed yellow to red emission with a maximal emitting peak at ca. 570 nm, 632 and 640 nm, respectively, which are identical to the corresponding solid-state PL emission. The Commission Internationale de L'Eclairage (CIE) coordinates of TPE-Se-TPE, TPA-

Se-TPE and TPA-Se-TPA are located at (0.48, 0.51), (0.63, 0.37) and (0.65, 0.34) respectively. Figure 6 shows the brightness-voltage characteristics (L-V) and the luminous efficiency-current density (LE-J) for those devices from TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA, the relevant data are presented in Table 2. For the device of TPE-Se-TPE, a turn-on voltage (V_{on}) of 4.6 V, a maximal luminous efficiency (LE^{max}) of 0.19 cd A⁻¹ at a current density of 101 mA cm⁻² and a maximum luminance (L_{max}) of 385 cd m⁻² are achieved. For the device of TPA-Se-TPA, a V_{on} of 3.5 V, a LE^{max} of 1.84 cd A^{-1} at a current density of 1.97 mA cm⁻² and an L^{max} of 2289 cd m⁻² are achieved. Among these compounds, the device of TPA-Se-TPE exhibited the highest device performance with a low V_{on} of 3.2 V, a good LE^{max} of 2.91 cd A⁻¹ at a current density of 33.9 mA cm⁻² and an L^{max} of 5855 cd m⁻². According to the current density-voltage characteristics (J-V) (Figure S2, ESI[†]), it suggested that the more triphenylamine groups in the molecule the carrier transporting is easier, which is important to the electroluminescent properties. The EL performances for these luminescent materials are in the order of TPE-Se-TPE < TPA-Se-TPA < TPA-Se-TPE, which is identical to the order of Φ_{PL} in the solid state.

4. Conclusions

In summary, we have successfully synthesized three donor-acceptor luminogens, TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA, by covalently binding electrondonating TPA and TPE moieties to an electron-accepting benzoselenadiazole core via a Suzuki coupling reaction. All the luminogens are soluble in common organic solvents and show high thermal stability with a T_d nearly or above 400 °C. The absolute photoluminescence quantum yield in the solid state ranged from 14.2% to 36.5%. The D–A structures of TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA enable them to exhibit ACQ plus AIEE effects dominated by the TICT and RIR, respectively. The best device performances were obtained from TPA-Se-TPE with a V_{on} of 3.2 V, LE^{max} of 2.91 cd A⁻¹, EQE of 3.4% and L^{max} of 5855 cd m⁻². This is the first report of benzoselenadiazole-based fluorophore with an AIE feature which was successfully applied to OLEDs.

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Table 1. Photophysical, electrochemical and thermal data of TPE-Se-TPE, TPA-Se-

Compound	λ _{abs} [a] (nm)	$\lambda_{PL}[a]$ (nm)	$arPerta_{ ext{PL}}\ (\%)$	$E_{ox}[b]$ (V)	E _{red} (V)	HOMO (eV)	LUMO (eV)	Eg (eV)	<i>T</i> _d (°C)
TPE-Se-TPE	330, 433	552	14.2	0.45	-2.03	-5.25	-2.77	2.48	399
TPA-Se-TPE	329, 461	615	36.5	0.09	-2.19	-4.93	-2.61	2.32	406
TPA-Se-TPA	323, 475	624	32.4	0.13	-2.15	-4.89	-2.65	2.24	460

TPE and TPA-Se-TPA

[a] Measured in neat film at room temperature. [b] Measured in a solution of Bu_4NPF_6 (0.1 M) in acetonitrile at a scan rate of 50 mV/s at room temperature.

Table 2. The EL performances of TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA

Device	V _{on} [a] (V)	V [b] (V) (n	J [b] H $A cm^{-2}$	EQE [b] (%) ($L_{\rm max}$ cd m ⁻²) (LE_{max} cd A^{-1}) CIE (x, y)
TPE-Se-TPE	4.6	10.7	101	0.09	385	0.19	(0.477, 0.508)
TPA-Se-TPE	3.2	8.6	33.9	3.4	5855	2.91	(0.633, 0.365)
TPA-Se-TPA	3.5	4.7	1.97	2.6	2289	1.84	(0.653, 0.344)

[a] The turn-on voltage at which luminescence reach 1 cd m^{-2} . [b] Device data at maximum LE.

Figure captions

Scheme 1. Synthetic routes to TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA.

Figure 1. UV-vis absorption (a) and PL (b) spectra of TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA

Figure 2. The energies and corresponding electron density distribution of HOMO and LUMO orbitals of TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA

Figure 3. TGA curves of TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA under nitrogen atmosphere

Figure 4. (a) PL spectra of TPA-Se-TPE in THF/water mixtures with different water fractions; (b) Changes of relative PL intensity of TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA in THF/water mixtures with different water fraction.

Figure 5. EL spectra of TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA.

Figure 6. The brightness-voltage (L–V) characteristics (a) and luminous efficiency– current density (LE–*J*) characteristics (b) of TPE-Se-TPE, TPA-Se-TPE and TPA-Se-TPA. Scheme 1











Figure 4







Figure 6



Research Highlights

Benzoselenadiazole derivatives with tetraphenylethene and triphenylamine were obtained.

All luminogens displayed aggregation-induced emission features.

The device using TPA-Se-TPE as emitter exhibited a highest luminous efficiency of 2.91 cd A⁻¹.