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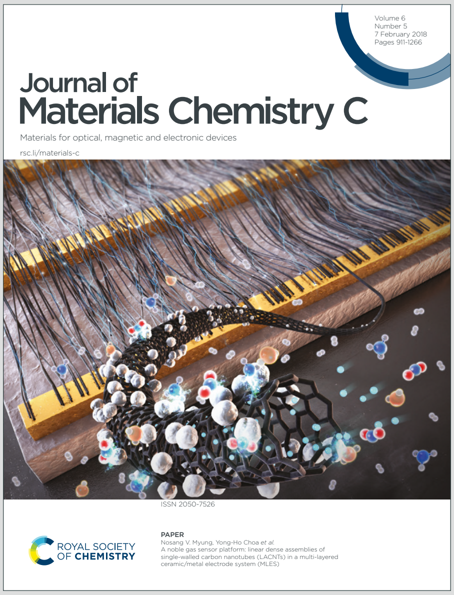

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PAPER
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Tetraphenylethylene Substituted Thienothiophene and Dithienothiophene Derivatives: Synthesis, Optical Properties and OLED Devices

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Abstract

Thieno[3,2-*b*]thiophene (TT) and dithieno[3,2-*b*;3,2-*d*]thiophene (DTT) have drawn an immense attention in the field of electronics and optoelectronics. In this work, first examples of TTs (**TPE2-TT**, **TPE3-TT**) and DTT (**TPE2-DTT**), having tetraphenylethylene (TPE) at the peripherals for aggregation induced emission (AIE) applications at OLEDs, were designed and synthesized by Suzuki coupling reaction. Their properties were investigated through experimental and computational studies, as well as X-ray diffraction and DFT-optimization. **TPE2-TT** produced an excellent device performance with a maximum luminance of 11620 cd m^{-2}, a maximum current efficiency of 6.17 cd A^{-1} and maximum external quantum efficiency of 2.43%. Moreover, these synthetically easy accessible molecules had high thermal stability, which make them perfectly suitable for optical applications.

Introduction

Thieno[3,2-*b*]thiophenes (TT) have rigid structures with an extended π-conjugation, which make them suitable for adjusting the band gap of organic materials and increasing their intermolecular interactions in solid state.[1-5] They are electron-rich molecules, making them promising materials to construct conjugated and low band gap organic semiconductors. Fusing one more thiophene to TT produces dithieno[3,2-*b*;2,3-*d*]thiophene (DTT), having three thiophene units, which have also been widely used to build promising organic materials due to their electron rich, flat and rigid delocalized systems. Organic materials made of TTs and DTTs display interesting properties in conducting and semiconducting devices, such as organic light-emitting diodes (OLEDs), organic solar cells (OSCs) and organic field-effect transmitters (OFETs).[6-9]

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Geometrical arrangement of organic materials in solid state has an important impact on their fluorescence and electroluminescence properties.[10-12] An important organization in solid state, called aggregation-induced emission (AIE), pioneered by Tang et al., has been extensively studied for the past 18 years.[13] Tetraphenylethylene (TPE) is a typical moiety, having a simple structure and providing excellent AIE property. It is known as a non-emissive chromophore in solution and highly emissive in solid state due to its twisted structure, which causes intramolecular restriction.[14-19]

As a continuation of our studies on design, syntheses and properties of TTs and DTTs for electronic and optoelectronic applications,[20-24] two TTs and a DTT, possessing peripheral TPE units to provide the materials with AIE, were synthesized. Their optoelectronic properties, along with their applications as active layers for OLEDs, were investigated. As the TTs and DTTs developed by our group are versatile compounds for further functionalization, we introduced TPEs at their various peripheral positions, such as the TTs having two and three TPEs, **TPE2-TT** and **TPE3-TT**, respectively, and a DTT, **TPE2-DTT**, having two peripheral TPEs. While the methoxy groups made the main skeletons, TTs and DTT, electron richer, hindrance between phenyl and bulky TPE moieties provided the molecules with non-planer structures; suitable for AIE studies. Thus, although several electron-transporting, hole-blocking, solar cell and light emitting materials based on thienothiophene (TT) and dithienothiophene (DTT) groups have been studied, to the best of our knowledge, TT, DTT and TPE combinations for OLED application have not been reported yet.[20-32]

Result and Discussion

Design and Synthesis

The TTs (**3**, **4**) and DTT **6** were synthesized according to our previously reported methods[18-22] (Fig. 1). Their precursor monoketones (**1**, **2**) were prepared in 79 and 81% yields, respectively, and the precursor diketone **5** in 65% yield. Their ring closure reactions in refluxing chlorobenzene gave the TTs **3** and **4** in 84 and 86% yields, respectively, and the DTT **6** in 65%. The TTs and DTT were then brominated with NBS to obtain dibromo-TTs (**7**, **8**) and DTT **9** (Scheme 1). Their Suzuki coupling with TPE-borolane **10** produced the target fluorescent materials **TPE2-TT** (76%), **TPE3-TT** (73%) and **TPE2-DTT** (69%).

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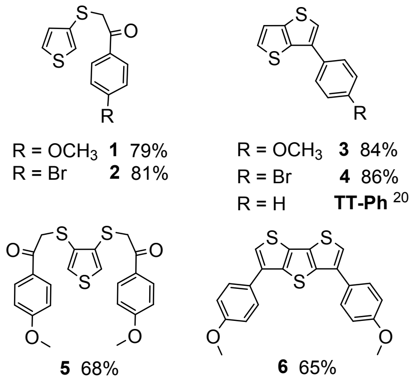
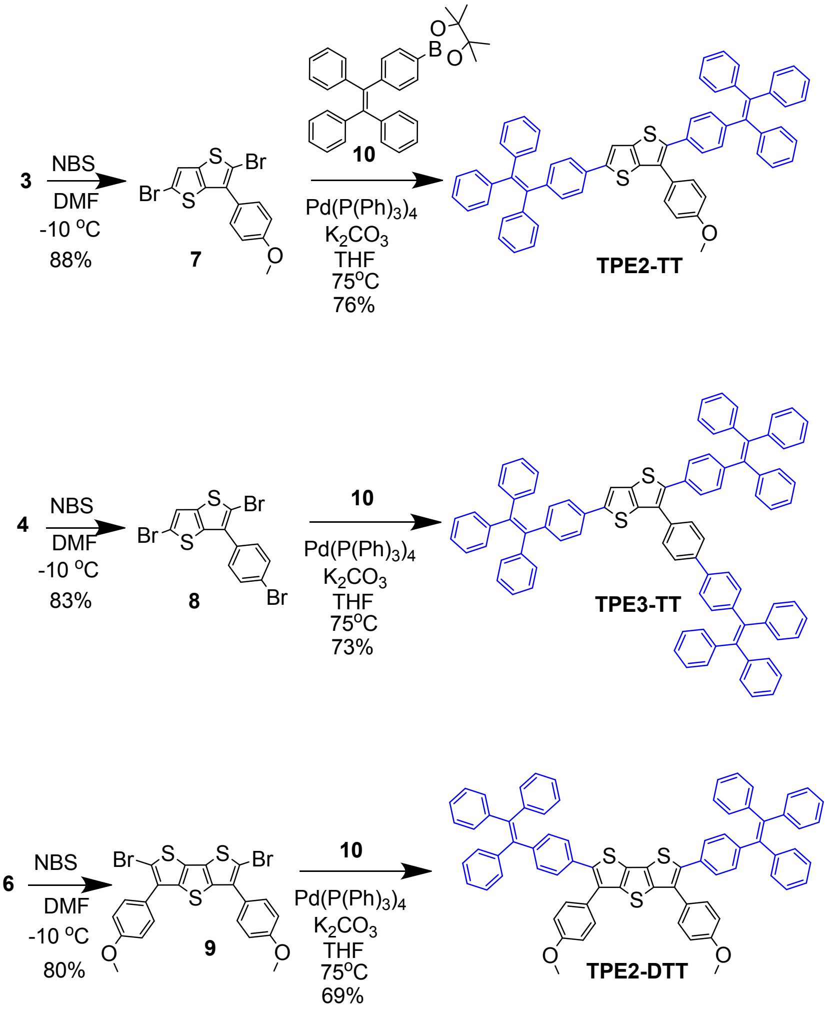


Fig. 1. Previously synthesized TTs and DTT

Scheme 1. Synthesis of **TPE2-TT**, **TPE3-TT** and **TPE2-DTT**



Photophysical Properties

Optical properties of all the compounds were then investigated (Table 1). As expected, presence of bulky TPEs at the peripherals of the TTs and DTT had a significant effect on their optical properties. While the unsubstituted TT **3**, **TT-Ph**,[22] and DTT **6** displayed π−π* absorptions around 302, 300 and 293 nm, respectively, the TPE substituted TTs (**TPE2-TT** and **TPE3-TT**) and DTT (**TPE2-DTT**) had

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these absorptions with a red shift around 390, 360 and 389 nm, respectively. The band gaps were narrowed from around 3.70 eV to between 2.80 - 2.88 eV. Most importantly, with the effect of the TPE, the non-fluorescent TTs **3**, **4** and DTT **6** became fluorescent, having emission wavelengths at 500 nm (**TPE2-TT**), 490 nm (**TPE3-TT**) and 495 nm (**TPE2-DTT**) (Fig. 2).

Aggregation-Induced Emission Properties

AIE properties of **TPE2-TT**, **TPE3-TT** and **TPE2-DTT** were studied by gradual addition of distilled water into their THF solutions (Fig. 3). Low fluorescence of **TPE2-TT** in THF did not change up to addition of 50% water, after which a gradual increase was observed until 90% of water content. While **TPE2-DTT** demonstrated a similar trend, a gradual increase of which was observed after addition of 20% water, **TPE3-TT** did not have any increase up to 90% of water, after which a dramatic increase was observed. Possible explanations could be their packing, resulting in AIE and presence of different number of OMe groups, since **TPE2-TT** with one OMe group responded to 50% of water, **TPE2-DTT** with two OMe groups responded to 20% of water, and **TPE3-TT** with no OMe group, having full hydrophobic character, responded to 90% of water. As all the synthesized compounds displayed successful aggregation induced emission, their solid-state quantum yields were then measured. The highest quantum yield was obtained with **TPE3-TT** (28%), the second one was **TPE2-TT** (21%) and the lowest was found to be **TPE2-DTT** (14%). This order indicated that as the planarity was increased a lowest quantum yield was obtained (Table 1).

Table 1. Optical Data of the Compounds.

	λ_{max} [a] (nm)	λ_{onset} (nm)	λ_{em} [a] (nm)	E_{gap}^{optic} (eV)	ϕ_{solid} [b] (%)
3	302	334	-	3.70	-
TT-Ph [20]	300	329	-	3.75	-
6	293	339	-	3.65	-
TPE2-TT	390	430	500	2.88	21
TPE3-TT	360	435	490	2.85	28
TPE2-DTT	389	443	495	2.80	14

[a]Absorption and emission maxima in THF. [b]Solid state quantum yields. E_{optic} estimated from the onset of absorption spectra.

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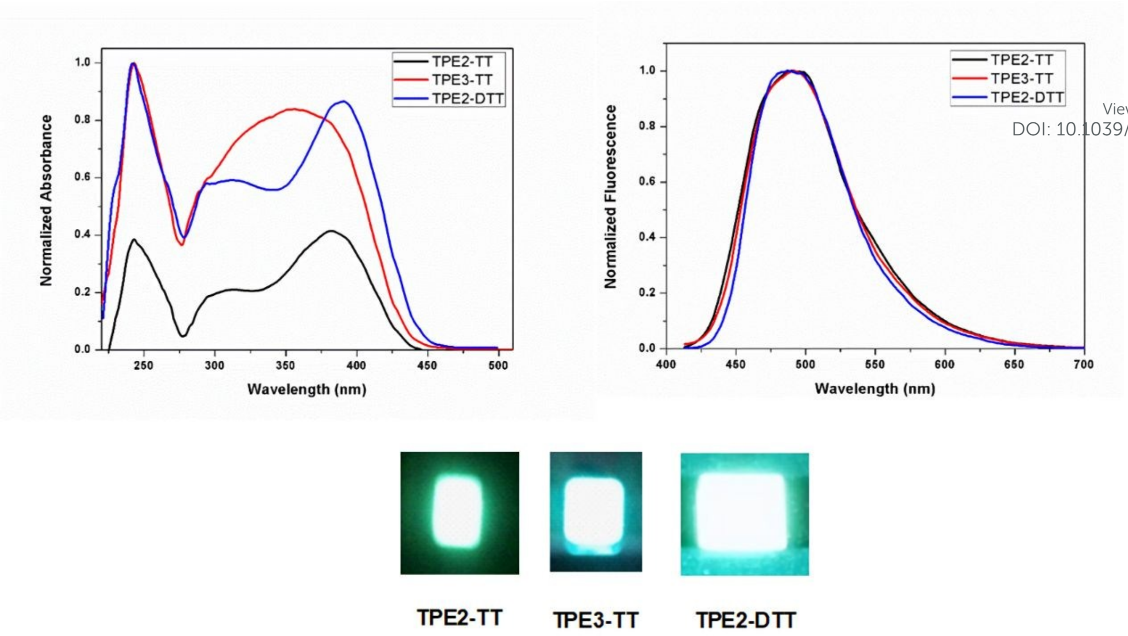

Fig. 2. Absorption and emission of **TPE2-TT**, **TPE3-TT** and **TPE2-DTT** in THF and their electroluminescence devices.

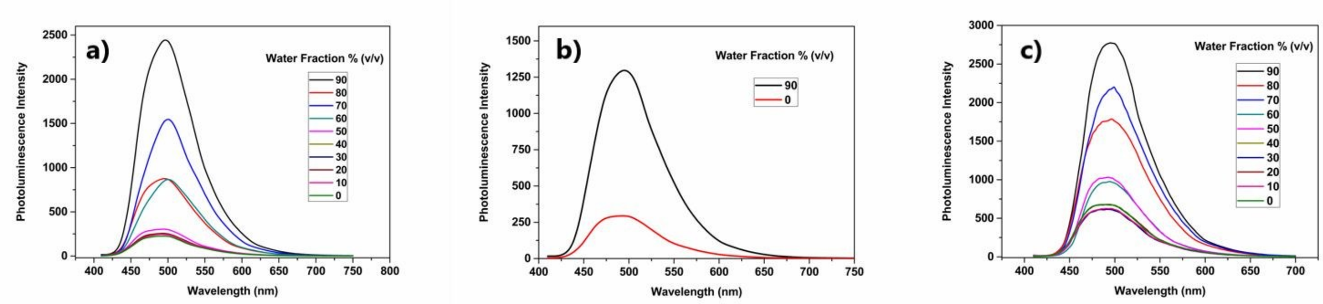

Fig. 3. Emission spectra as a function of the water fraction of compounds, a) **TPE2-TT** b) **TPE3-TT** c) **TPE2-DTT** in THF. Luminogen concentration: 10 μM. Excited at maximum absorption wavelengths in THF.

Single Crystal Structures

In order to have more insight on their solid state, attempts were made to grow single crystals, which resulted in obtaining a rod-like dark yellow single crystal of **TPE2-DTT** with a dimension of 0.50 x 0.05 x 0.03 by slow evaporation of its chloroform solution. Crystal data and the structure refinement parameters are given in Table S1. Table S1-S6 showed the selected bond lengths, bond and torsion a on angles for the compound **TPE2-DTT**. Thermal ellipsoids and side view are plotted in Fig. 4. **TPE2-DTT** was crystallized in a triclinic crystal system with P-1 space group (Fig. 4). Each unit cell contained two molecules (Z=2).

The torsion angles between two thiophene rings and the adjacent phenyl rings of ΨS1-C18 = 34.87 and ΨS3-C450= 38.44 are suitable for π-conjugation.[33] C-C average bond length of 1.481 Å between thiophene and tetraphenylethylene units are similar to distances observed in the related literature.[34]

All the bond lengths and bond and torsion angles representing dithienothiophene rings (Table S1) are also in close agreement with the literature.[35,36] The solvent molecules of water were preserved in the crystal lattice of **TPE2-DTT** and contained some abundant hydrogen bonding between each other. The unit cell also contained a weak CH_3-π interaction between tetraphenylethylene units of the adjacent molecules, which is a usual behavior for molecules containing tetraphenylethylene units.[37,38] It led to a relatively loose packing (Fig. 5). X-ray diffraction patterns (snapshots) collected for **TPE2-TT** and **TPE3-TT** (Fig. S1) revealed that, contrary to the structure of **TPE2-DTT**, the two molecules formed amorphous aggregates possibly due to strong π- π aggregation.

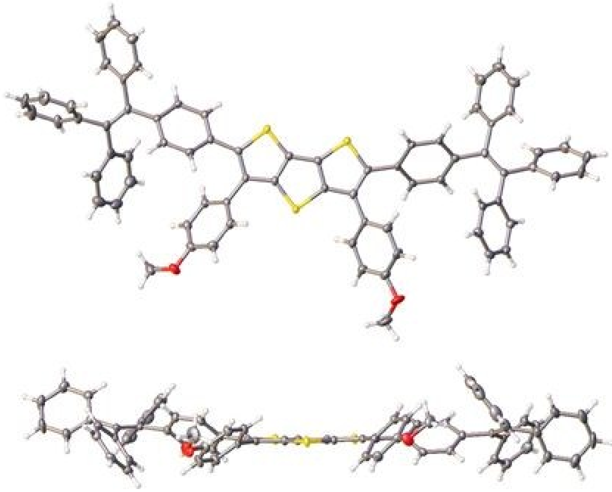

Fig. 4. X-ray crystal structures of **TPE2-DTT**

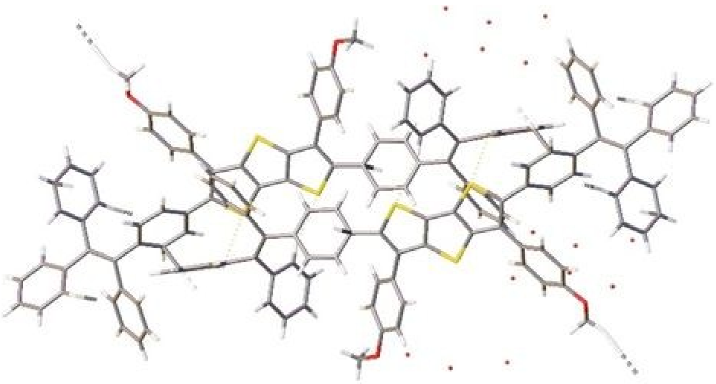

Fig. 5. Packing conformation of **TPE2-DTT**

Computational Studies

In order to have a more insight on the three molecules, density functional theory calculations were conducted at the B3LYP/6-31 G (d, p) level. Regarding **TPE2-DTT**, dihedral angles between DTT plane and TPE, DTT and methoxy substituted phenyl groups were estimated to be 38° and 49°, respectively. Similarly, in **TPE2-TT** and **TPE3-TT**, the dihedral angle between TT plane and TPE is 40°, and TT plane and methoxy substituted phenyl units have 49°, 48°, respectively. The TPE and phenyl methoxy groups are nearly perpendicular to the DTT and TT plane in all the compounds as a result of steric effect of the bulky TPE groups. Calculated geometry optimization data set is in agreement with the crystallographic analysis. The HOMOs were observed to be on TPE parts, somewhat expended over TT. The calculated HOMO-LUMO gaps for **TPE2-TT**, **TPE3-TT** and **TPE2-DTT** are 3.02, 3.07 and 3.02 eV, respectively. The HOMO of **TPE3-TT** is localized over TT and the phenyl unit attached to the **TPE3-TT**, while only a minor delocalization over phenyl group attached to **TPE2-DTT** was observed. Similarly, the LUMO is localized on DTT and the phenyl unit attached DTT and the phenyl unit attached to **TPE2-TT**. In contrast, electrons in the HOMO and LUMOs of **TPE3-TT** are localized at the same region containing, two TPE and thiophene rings. In order to have absorption bands to obtain steady-state spectra for the compounds, N states of 50 for singlets were computed. The λ_{max} values of **TPE2-TT**, **TPE3-TT** and **TPE2-DTT** were predicted to be 410.8, 403.7 and 410.1 nm, which shows low energy transitions resulting from HOMO-LUMO (95.6-96%) (Fig. 6).

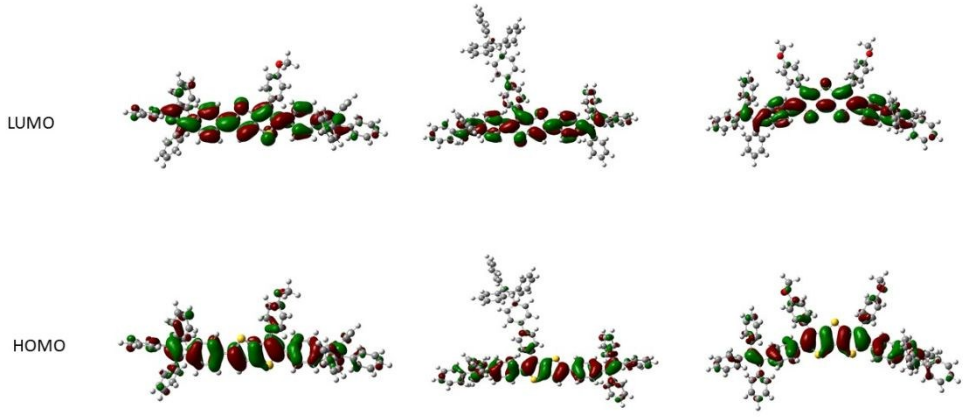

Fig. 6. HOMO and LUMO frontier orbitals of compounds **TPE2-TT**, **TPE3-TT** and **TPE2-DTT** created at the B3LYP/6-31 G (d, p) level in gas phase, respectively from left to right.

Thermal Properties

Thermal gravimetric (TGA) analysis of the three compounds indicated two gradual endothermic decompositions (Fig. 7). While the first decompositions of **TPE3-TT** and **TPE2-DTT** took place at 175 ^{o}C with around 10% and 50% weight loss, respectively, it was observed at 300 ^{o}C for **TPE2-TT**. Second decomposition temperatures were at 450 ^{o}C for **TPE2-TT** and **TPE2-DTT**, and 500 ^{o}C for **TPE3-TT**. Weight loss of **TPE3-TT** at higher temperature, compared to the other compounds, demonstrated its better thermal stability, which is a remarkable property. The high thermal properties are profitable to the preparation of stable and durable OLED fabrication.

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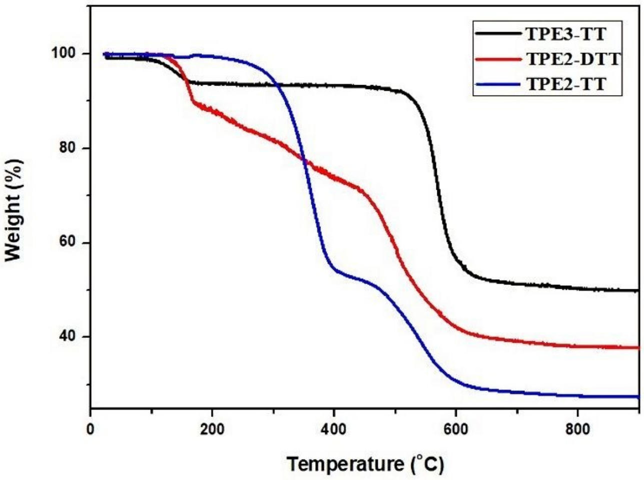

Fig. 7. TGA of the compounds

Electroluminescence Properties

To investigate the electroluminescence properties of the **TPE2-TT**, **TPE3-TT** and **TPE2-DTT**, we fabricated OLEDs with a configuration of glass/ITO (120 nm)/NPB (60 nm)/Molecules (**TPE2-TT**, **TPE3-TT** or **TPE2-DTT**) (20 nm)/TPBi (10 nm)/Alq$_3$ (30 nm)/LiF (1 nm)/Al (120 nm) (Fig. 8c). In this configuration, ITO and LiF/Al were used as anode and cathode electrodes, respectively; (NPB) was used as a hole injection layer; (TPBi) was used as a hole blocking layer; and tris-(8-hydroxyquinoline) aluminum (Alq$_3$) was used as an electron transport layer. **TPE2-TT**, **TPE3-TT** and **TPE2-DTT** were used as emitting molecules (Fig. 8).

OLEDs of the three molecules **TPE2-TT**, **TPE3-TT** and **TPE2-DTT** were fabricated. The current density-voltage-luminance (J-V-L) characteristics and efficiency-current density curves are depicted in Fig. 9. Fig. 8a shows normalized EL spectra of the fabricated devices and Table 2 summarizes the device performances. While the devices of **TPE2-TT** and **TPE2-DTT** displaced a low turn-on voltage of 3.2 and 3.7V, respectively, the device with **TPE3-TT** had higher turn-on voltage of 5.0 V. Among the three devices, **TPE2-TT** showed the best device performance, i.e. the highest luminance (11620 cd/m^2) and the efficiency (LE: 6.17 cd/A and EQE: %2.34). According to the CIE coordinates, the devices of **TPE2-TT** and **TPE2-DTT** had cloudy sky and **TPE3-TT** slightly blue emitting (Fig. 8b). The EL spectrum of **TPE2-DTT** has a red shift compared to **TPE2-TT** and **TPE3-TT**, which could be due to the extended conjugation of **TPE2-DTT**.

Table 2: OLED performances of **TPE2-TT**, **TPE3-TT** and **TPE2-DTT**

	Turn-on [a] Voltage	EQE [b] (%)	λ_{EL} [c] (nm)	CIE Coordinates [d] (x, y)	L [e] (cd/m^2)	LE [f] (cd/A)
TPE2-TT	3.2	2.34	486	0.230, 0.456	11620	6.17
TPE3-TT	5.0	1.89	488	0.200, 0.383	1547	4.45
TPE2-DTT	3.7	1.62	513	0.230, 0.515	2590	3.78

[a] Turn on voltage, recorded at the luminance of 1 cd m^{-2}. [b] External quantum efficiency. [c] Maximum electro-luminescence wavelength. [d] Chromaticity coordinates according to the CIE 1931 diagram. [e] Maximum luminance. [f] Luminous efficiency.

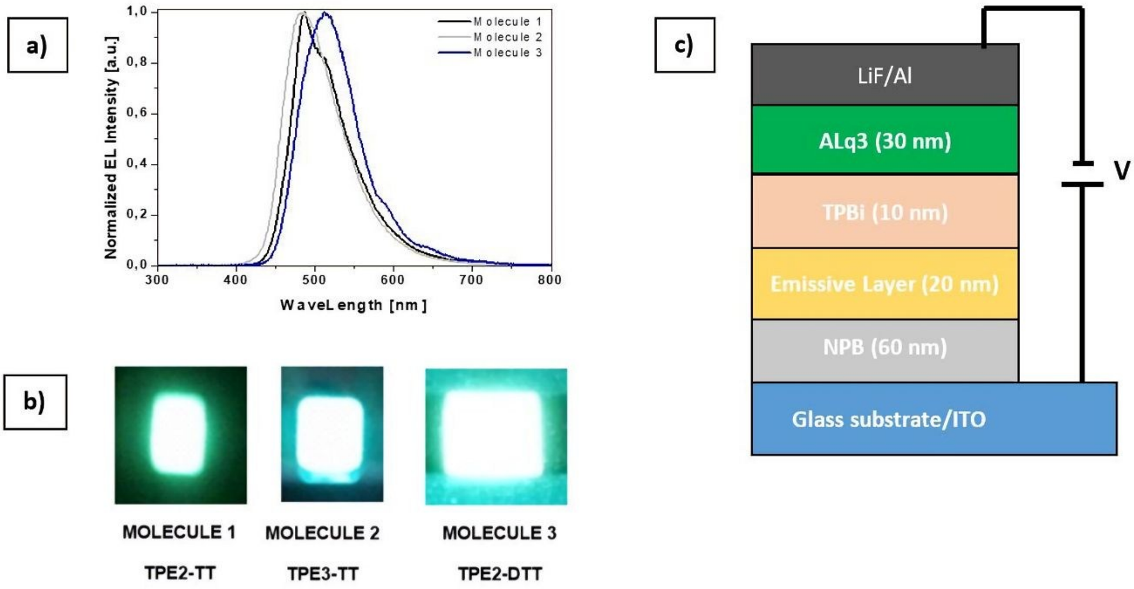

Fig. 8. a) Normalized Electro luminance Intensity- wavelength, b) device photographs and c) schematic illustration of the device configuration of the **TPE2-TT** (Molecule 1), **TPE3-TT** (Molecule 2) and **TPE2-DTT** (Molecule 3).

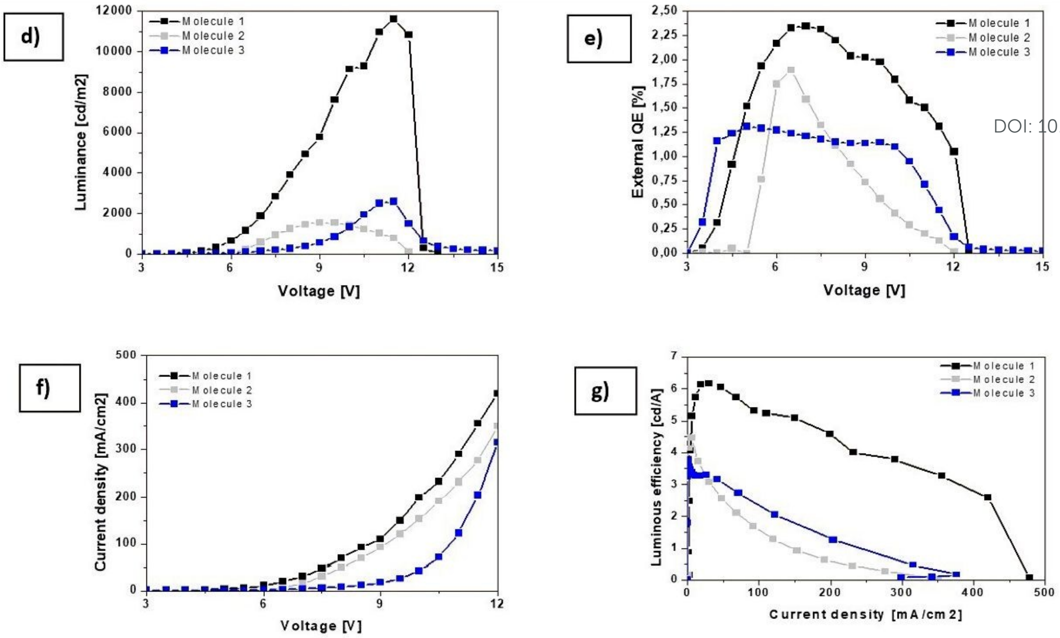

Fig. 9. d) Luminance-voltage, e) external quantum yield-current density, f) current density-voltage, g) luminous efficiency-current density characteristics of the **TPE2-TT** (Molecule 1), **TPE3-TT** (Molecule 2) and **TPE2-DTT** (Molecule 3).

Conclusion

In conclusion, three novel AIE active optic materials **TPE2-TT**, **TPE3-TT** and **TPE2-DTT** containing thienothiophene (TT), dithienothiophene (DTT) and tetraphenylethylene (TPE) units were synthesized through an easy-accessible synthetic methodology with high yields of 76, 73 and 69%, respectively. Changes on the positions of the TPE units at the peripherals of TTs and DTT had a dramatic effect on their optical and AIE properties. Effects of structural tuning on AIE were explained by X-Ray analysis, computational study and device fabrication with high EQE and maximum luminescence of 2.34% and 11620 cd/m^2, respectively. The designed molecules displayed high thermal stability up to around 500 ^{o}C. Our work provides a new strategy to develop promising electroluminescence materials having the closest CIE coordinates to blue emission based on TT and DTT cores for organic material chemistry.

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Experimental

Materials

All the reagents were purchased from Aldrich and Acros, used without further purification. All the solvents used in the syntheses were technical grade and freshly distilled prior to use. The solvents used in spectroscopic measurements were spectroscopic grade. Flash chromatography was performed with ≤ 0.063 μm Silica Gel. ^{1}H and ^{13}C NMR spectra were recorded on a Varian 500 and 126 MHz, respectively, spectrometer. Proton and carbon chemical shifts are reported in parts per million downfield from tetramethyl silane, TMS.

Synthesis

3-(4-Methoxy-phenyl)-2,5-bis-(4-triphenylvinyl-phenyl)-thieno[3,2-*b*]thiophene (TPE2-TT):

To the mixture of **7** (500 mg, 1.237 mmol) and **10** (1190 mg, 2.600mmol) dissolved in THF (30 mL) and degassed for 15 minutes with N_2 was added K_2CO_3 (5 mL, 2 M) and $Pd(PPh_3)_4$ (0.123 mmol). The mixture was then saturated with N_2 and the sealed reaction flask was stirred at 75 °C for 48 h, after which the crude product was filtered through celite, extracted with sodium carbonate, dried over sodium sulfate, filtered and the solvent was evaporated under reduced pressure. The crude product was purified by column chromatography eluting with *n*-hexane: CH_2Cl_2 (4:1) to obtain the title compound **TPE2-TT** (853 mg, 76%) as a yellow powder, mp. 115-116 ° C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 (s, 1H), 7.38 (d, J = 8.4 Hz, 2H), 7.36 – 7.34 (m, 2H), 7.12 (m, 34H), 6.96 (d, J = 8.4 Hz, 2H), 6.90 (d, J = 8.8 Hz, 2H), 3.87 (s, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 158.95, 158.92, 158.87, 144.83, 143.68, 143.61, 143.58, 143.53, 143.36, 143.29, 143.05, 141.36, 140.92, 140.49, 140.30, 138.80, 137.02, 134.10, 132.76, 132.63, 131.95, 131.45, 131.43, 131.40, 131.38, 131.34, 131.33, 130.08, 128.42, 127.85, 127.76, 127.71, 127.69, 127.66, 127.60, 126.64, 126.57, 126.55, 126.50, 124.80, 115.44, 114.14, 55.24; HRMS (m/z): $[M+1]^+$ calcd. for $C_{66}H_{46}OS_2$, 907.30628; found, 907.30352.

3-(4'-Triphenylvinyl-biphenyl-4-yl)-2,5-bis-(4-triphenylvinyl-phenyl)-thieno[3,2-*b*]thiophene (TPE3-TT):

To the mixture of **3** (500 mg, 1.103 mmol) and **6** (1517 mg, 3.310 mmol) dissolved in THF (30 mL) and degassed for 10 minutes with N_2 was added K_2CO_3 (5 mL, 2 M) and $Pd(PPh_3)_4$ (0.1103 mmol). The mixture was then saturated with N_2 and the sealed reaction flask was stirred at 75 °C for 48 h, after which the crude product was filtered through celite, extracted with sodium carbonate, dried over sodium sulfate, filtered and the solvent was evaporated under reduced pressure. The crude product was purified by column chromatography eluting with *n*-hexane: CH_2Cl_2 (3:1) to obtain the title compound **TPE3-TT** (973 mg, 73%) as a yellow powder, mp. 181-182 ° C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.54 (d, *J* = 8.46 Hz, 2H), 7.43 (m, 5H), 7.37 (d, *J* = 8.27 Hz, 2H), 7.12 (m, 51 H), 6.95 (d,

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J = 8.43 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.752, 143.758, 143.678, 143.652, 143.625, 143.591, 143.557, 143.515, 143.331, 143.230, 143.207, 142.987, 141.421, 141.409, 141.379, 141.217, 140.612, 140.512, 140.425, 140.267, 139.524, 138.204, 137.335, 137.254, 134.085, 132.555, 131.947, 131.860, 131.476, 131.430, 131.412, 131.366, 131.325, 130.002, 129.240, 129.180, 128.539, 127.837, 127.777, 127.741, 127.708, 127.679, 127.639, 127.457, 126.631, 126.558, 126.517, 126.448, 126.451, 126.020, 124.825, 115.381; HRMS (m/z): $[M + 1]^+$ calcd. for $C_{90}H_{62}S_2$, 1207.43657; found, 1207.43245.

3,5-Bis-(4-methoxy-phenyl)-2,6-bis-(4-triphenylvinyl-phenyl)-dithieno[3,2-b;2',3'-d]thiophene (TPE2-DTT):

To the mixture of **3** (200 mg, 0.353 mmol) and **6** (340 mg, 0.741 mmol) dissolved in THF (25 mL) and degassed for 10 minutes with N_2 was added K_2CO_3 (2.5 mL, 2 M) and $Pd(PPh_3)_4$ (0.0353 mmol). The mixture was then saturated with N_2 and the sealed reaction flask was stirred at 75 ^{o}C for 48 h, after which the crude product was filtered through celite, extracted with sodium carbonate, dried over sodium sulfate, filtered and the solvent was evaporated under reduced pressure. The crude product was purified by column chromatography eluting with *n*-hexane: CH_2Cl_2 (3:1) to obtain the title compound **TPE2-DTT** (256 mg, 69%) as a yellow powder, dec. 236 ^{o}C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32 (d, J = 8.7 Hz, 4H), 7.14 (m, 21H), 7.6 (m, 13H), 6.95 (d, J = 8.1 Hz, 4H), 6.87 (d, J = 8.7 Hz, 4H), 3.85 (s, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.01, 143.64, 143.54, 143.33, 131.45, 131.37, 131.35, 131.30, 130.12, 128.55, 128.31, 127.70, 127.67, 127.63, 127.23, 126.57, 126.54, 126.48, 114.30, 55.22; HRMS (m/z): M^+ calcd. for $C_{74}H_{52}O_2S_3$, 1068.31239; found, 1068.31294.

Conflicts of interest

There are no conflicts to declare.

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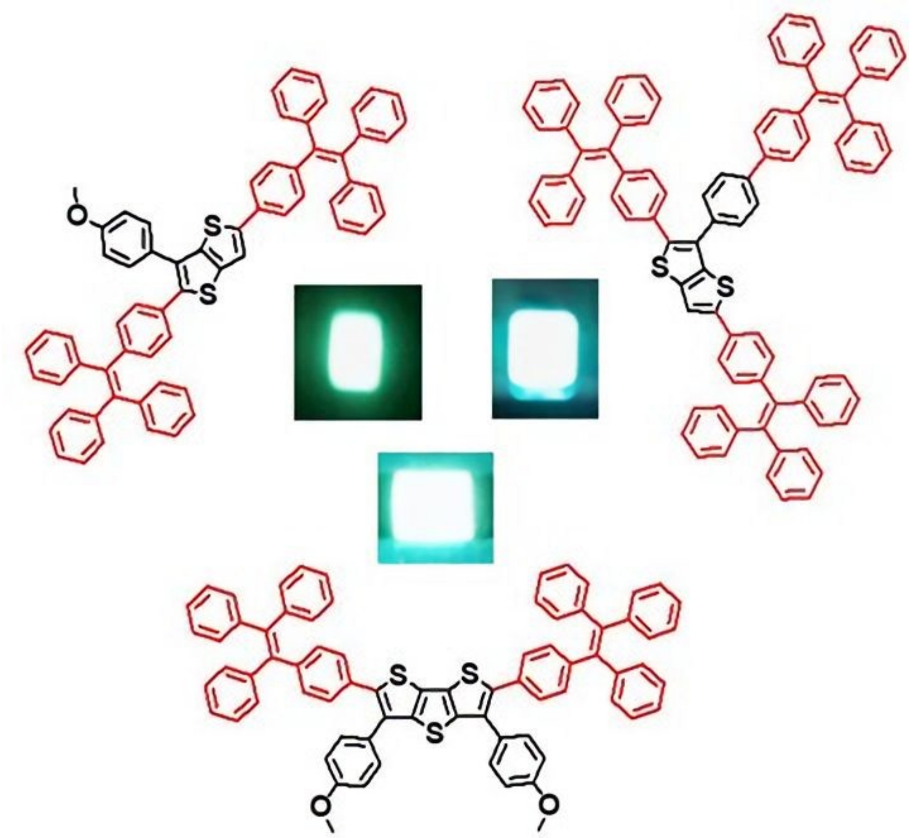
Text and Graphic for the table of contents

Tetraphenylethylene Substituted Thieno[3,2-*b*]thiophene and Dithieno[3,2-*b*;2',3'-*d*]thiophene Derivatives; Synthesis, Optical Properties and OLED Devices

Recep Isci, Emine Tekin, Kerem Kaya, Selin Piravadili Mucur, Sultan Funda Gorkem, and Turan Ozturk



TPE units at the peripherals of TTs and DTT affects structure, properties and OLED fabrications of three synthetically easily accessible novel conjugated compounds for material chemistry.