DOI: 10.1002/asia.201100753

A Facile and Versatile Approach to Efficient Luminescent Materials for Applications in Organic Light-Emitting Diodes

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Design and synthesis of luminophors with high emission efficiency is a hot research topic due to their practical applications in optoelectronics such as organic light-emitting diodes (OLEDs).[1] To respond to the demand, scientists have prepared a variety of luminogenic materials. Whereas these materials emit intensely in solution, they become weakly fluorescent or even non-luminescent in the solid state. In the solid state, the molecules are located in the immediate vicinity. The aromatic rings of the neighboring fluorophores, especially those with disc-like shapes such as pyrene and anthracene, experience strong π–π stacking interactions, which promote the formation of aggregates with ordered or random structures.[2] The excited states of the aggregates often decay via nonradiative pathways, which are notoriously known as aggregation-caused quenching (ACQ) of light emission in the condensed phase. This notorious ACQ effect has prevented many lead compounds carrying chromophore groups that were identified by solution-based screening processes in the laboratory from finding real-world applications and becomes a thorny problem for the development of efficient OLEDs. Various chemical, physical, and engineering approaches and processes have been proposed to alleviate the ACQ effect but have met with only limited success. The difficulty lies in the fact that aggregate formation is an intrinsic process when luminogenic molecules are located in close vicinity in the aggregate phase.[3]

Our group observed a phenomenon of aggregation-induced emission (AIE) that is exactly opposite to the ACQ effect. Instead of quenching, aggregate formation has turned a series of propeller-like luminogens to emit intensely in the solid state.[4,5] This AIE phenomenon is rationalized to be caused by the restriction of intramolecular rotation (IMR). In the solution state, the active rotation of aromatic rings effectively deactivates the excited state through a rotational energy relaxation channel, thus rendering the luminogens non-emissive. In the aggregate state, the IMR process is impeded, thereby blocking the nonradiative decay pathway and converting the luminogens into strong emitters.[6] Among the AIE luminogens, tetraphenylethene (TPE) possesses a simple chemical structure[7] but shows an outstanding AIE effect. TPE and its derivatives have found an array of high-technological applications in environmental and biological sciences.[8] However, TPE itself is not a good light-emitting material for the construction of efficient OLEDs. An electroluminescence (EL) device fabricated using TPE as emitting layer shows inferior performances with maximum luminance and maximum current efficiency of merely 1800 cd m^{-2} and 0.45 cd A^{-1}, respectively, presumably due to TPE's high tendency to crystallize during the film fabrication process as well as its low thermal stability.[7a] Linking two or three TPE units through a silicon atom affords luminogens with enhanced thermal stability and emission efficiency in the solid state. However, these luminogens exhibit limited improvement in EL performance.[9] Delightfully, recent studies show that TPE can be easily attached to some ACQ chromophores, thus generating new AIE luminogens with efficient solid-state emissions and outstanding device performances. Representative examples are given by mono-substituted TPE derivatives (TPEArs) that are non-emissive in solution but exhibit high fluorescence quantum yield (Φ_F), up to unity, in the solid state (Scheme 1).[10] We envision that such strategy is versatile to solve the ACQ problem of traditional luminophors and, at the same time, to create high-performance solid-state emitters for OLEDs. To further verify the applicability of such synthetic tools, we prepared new luminogens consisting of TPE cores with different ACQ chromophores at both ends (Scheme 2) and in-

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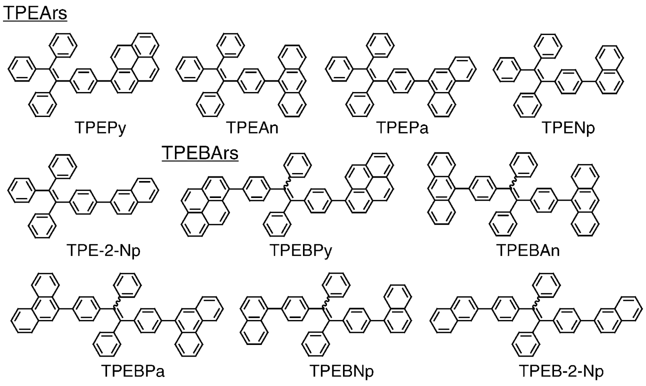
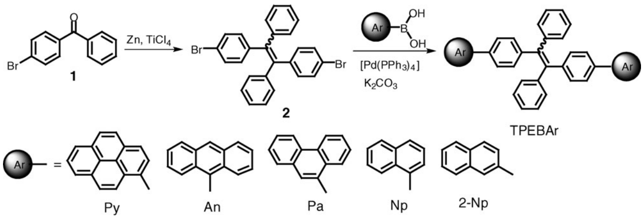
Supporting information for this article is available on the WWW under http://dx.doi.org/10.1002/asia.201100753.



Scheme 1. Chemical structures of TPE derivatives with various ACQ chromophores.

vestigated their optical and thermal properties as well as applications in OLEDs.



Scheme 2. Synthetic route to disubstituted TPE derivatives.

TPEArs were synthesized according to the literature method.[10b] Whereas their AIE characteristics and electronic and crystal structures have been presented in our recently published paper, their thermal and EL properties were investigated in this study. Their disubstituted counterparts (TPEBArs) were prepared according to the synthetic route shown in Scheme 2. A mixture of *Z*- and *E*-isomers (approximately 1:1 ratio) of 1,2-bis(4-bromophenyl)-1,2-diphenylethene (**2**) was prepared by a McMurry coupling reaction of 4-bromobenzophenone (**1**). Suzuki coupling of **2** with the corresponding arylboronic acids catalyzed by [Pd(PPh$_3$)$_4$] in basic medium afforded the target products in moderate yields (52–78 %). Similar to **2**, these molecules were also synthesized in a stereorandom fashion. The detailed synthetic procedure and characterization data are given in the Supporting Information. These luminogens are soluble in common organic solvents, such as tetrahydrofuran (THF), dichloromethane, chloroform, and toluene, but insoluble in ethanol and water.

Figure 1a shows the absorption spectra of TPEBArs in THF. The prepared TPEBArs have an absorption maximum in the range from 325 to 388 nm and a spectral profile that varies largely with the type of the ACQ chromophore. All the TPEBArs display a weak photoluminescence (PL) with hardly discernible peaks in dilute THF solution (10 μM). The determined Φ_F values, using 9,10-diphenylanthracene (Φ_F = 90 % in cyclohexane) as standard, fall in the range of 0.13–0.37 %, which are slightly higher than those of TPEArs (0.02–0.22 %). Such low Φ_F values reveal that they are practically weak emitters in the solution state. The Φ_F values of TPEBArs are much lower than those of their ACQ components such as anthracene (36 %) and pyrene (32 %),[11] thus demonstrating that the TPE unit works as an emission quencher in the solution state. The multiple phenyl rings of the TPE unit in an isolated molecule can undergo active IMR with little restraint in dilute solutions, which effectively consumes the energy of the excited state, thus rendering the dye molecules weakly emissive. Although the incorporation of an additional ACQ chromophore in TPEArs has generated TPEBArs with slightly enhanced emission efficiency, their Φ_F values are still low as the emission quenching effect of the IMR process of the TPE unit is so efficient in the solution state.

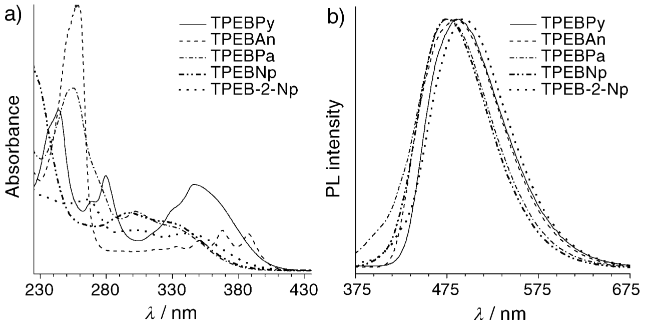


Figure 1. a) Absorption and b) PL spectra of TPEBArs in THF solution (a) and in the solid state as thin films (b).

On the contrary, all synthesized TPEBArs are highly emissive in the solid film state. The PL spectra of their thin films are shown in Figure 1b. The TPEBArs emit light at 474–492 nm (sky blue) and their photoluminescence is bathochromically shifted from that of TPEArs (Table 1), presumably due to their higher conjugation. The associated Φ_F values measured by an integrating sphere are much higher than those in solution, reaching values up to 100 %, thus revealing that TPEBArs are completely AIE-active.

Table 1. Optical and thermal properties of TPEArs and TPEBArs.[a]

	λ_{abs} (nm) Soln	λ_{em} (nm) Film	Φ_F (%) Soln[b]	Film[c]	T_g/T_d [°C]
TPEBPy	347	488	0.37	100	142/494
TPEBAn	388	486	0.29	100	-/441
TPEBPa	332	475	0.34	100	129/448
TPEBNp	325	474	0.14	100	90/387
TPEB-2-Np	340	492	0.13	100	100/363
TPEPy	348	468	0.22	100	-/325
TPEAn	387	450	0.12	100	-/353
TPEPa	323	481	0.03	88	86/363
TPENp	321	469	0.02	83	63/311
TPE-2-Np	326	474	0.05	100	71/284

[a] Soln = solution (10 μM in THF), λ_{abs} = absorption maximum, λ_{em} = PL maximum at excitation at 350 nm. [b] Fluorescence quantum yield measured in THF solution using 9,10-diphenylanthracene (Φ_F = 90 % in cyclohexane) as standard. [c] Absolute fluorescence quantum yield measured using an integrating sphere.

To further confirm the AIE feature of TPEBArs, we added water, a non-solvent for the luminogens, to their solution in THF and investigated the PL change. Figure 2a shows the PL spectra of TPEBPy in THF/water mixtures with different water fractions (f_w) as an example. Addition of a large amount of water ($f_w \geq 70\,\%$) to the THF solution of TPEBPy causes its molecules to aggregate due to the immiscibility of the hydrophobic luminogen with the hydrophilic medium and, accordingly, increases its emission. The higher the water content, the stronger is the PL intensity. Clearly, the emission of TPEBPy is enhanced by aggregate formation. Other TPEBArs also show similar emission behaviors (Figure 2b), thus suggesting that the melding of TPE and ACQ chromophores at the molecular level has endowed the resultant molecules with a novel feature of AIE.

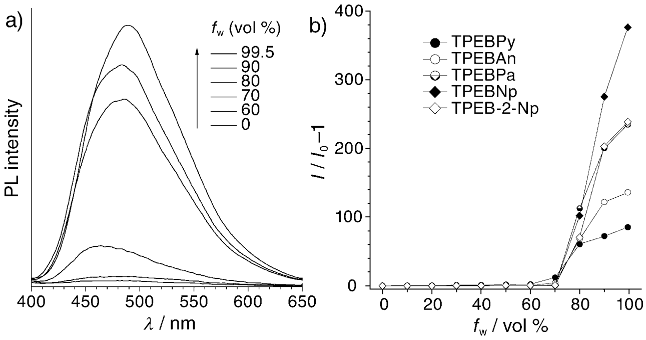


Figure 2. a) PL spectra of TPEBPy in THF/water mixtures with different water fractions (f_w). b) Plots of (I/I_0-1) values as a function of f_w in THF/water mixtures of TPEBArs, in which I_0 is the PL intensity in pure THF solution.

The thermal properties of TPEArs and TPEBArs were examined by differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA). Figure 3 shows the TGA thermograms and DSC curves of TPEBArs recorded under nitrogen. TPEBArs are thermally stable, showing high onset decomposition temperatures (T_d) at 363–494 °C. They are also morphologically stable, as revealed by their high glass transition temperatures (T_g) of 90–142 °C. Both T_d and T_g values of TPEBArs are much higher than those of TPEArs, thereby demonstrating that the thermal properties of the luminogens are readily tuned by incorporation of rigid aromatic cyclic rings.

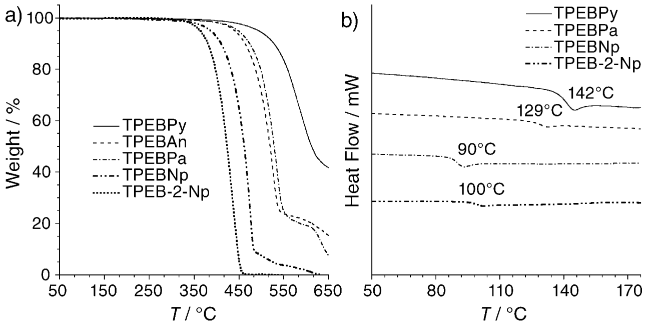


Figure 3. a) TGA thermograms and b) DSC curves of TPEBArs recorded under nitrogen at a heating rate of 10 °C min^{-1}.

The high PL efficiency and good thermal stability of TPEArs and TPEBArs in the solid state suggest that they are good candidates for the fabrication of efficient OLEDs. Multilayer OLEDs with a configuration of indium tin oxide (ITO)/*N*,*N*-bis(1-naphthyl)-*N*,*N*-diphenylbenzidine (NPB) (60 nm)/TPEArs or TPEBArs (20 nm)/2,2′,2″-(1,3,5-benzinetriyl)tris(1-phenyl-1-*H*-benzimidazole (TPBi) (10 nm)/tris(8-hydroxyquinolinolato)aluminum (Alq$_3$) (30 nm)/LiF (1 nm)/Al (100 nm) (device I) were constructed, in which TPEArs or TPEBArs functioned as light emitters, NPB worked as hole transport layer, and TPBi and Alq$_3$ served as hole blocking and electron transport layers, respectively. The device performances are summarized in Table 2. OLEDs based on TPEPy, TPEAn, TPENp, and TPE-2-Np show sky-blue EL at 480–492 nm (Figure 4a), which are slightly red-shifted from the PL of their films due to the microcavity effect. The EL of TPEPa is observed at 464 nm, which is between the PL of its crystals and film.[10b] This suggests that the TPEPa film fabricated by the vacuum deposition process is partially crystalline in nature.[7b] All OLEDs with TPEArs as emitters exhibit good EL performances (Table 2 and Figure 4b–d), which are much better than those of the TPE-based device.[7a] Among them, the TPEPy-based device shows the highest EL efficiency. It is turned on at a low voltage of 3.6 V and emits brilliantly with a maximum luminance (L_{max}) of 13400 cd m^{-2}. The maximum current efficiency ($\eta_{C,max}$), power efficiency ($\eta_{P,max}$), and external quantum efficiency ($\eta_{ext,max}$) attained by the device are 7.3 cd A^{-1},

Table 2. EL performances of TPEArs and TPEABrs.[a]

	EL (nm)	V_{on} (V)	L_{max} (cd m^{-2})	$\eta_{P,max}$ (lm W^{-1})	$\eta_{C,max}$) (cd A^{-1})	$\eta_{ext,max}$ (%)
Device I						
TPEPy	484	3.6	13400	5.6	7.3	3.0
TPEAn	486	4.0	8410	3.7	4.6	2.1
TPEPa	464	5.2	4600	1.6	2.7	1.4
TPENp	480	6.0	4120	1.1	2.4	1.3
TPE-2-Np	492	3.8	19800	4.2	7.3	2.7
TPEBPy	516	4.8	13370	2.0	5.8	2.0
TPEBAn	516	6.0	22600	2.0	5.7	2.0
TPEBPa	488	4.8	9250	3.5	5.5	2.6
TPEBNp	488	5.4	7130	2.3	4.2	1.9
TPEB-2-Np	494	4.4	13500	3.7	6.1	2.5
Device II						
TPEBPy	516	4.6	25500	2.7	6.0	2.1
TPEBAn	512	4.4	14750	3.0	5.1	1.9

[a] Device configuration: ITO/NPB (60 nm)/TPEArs or TPEBArs (20 nm)/TPBi (10 nm)/Alq$_3$ (30 nm)/LiF (1 nm)/Al (100 nm) (device I) and ITO/NPB (60 nm)/TPEBPy or TPEBAn (20 nm)/TPBi (40 nm)/LiF (1 nm)/Al (100 nm) (device II). λ_{EL} = EL maximum, V_{on} = turn-on voltage at 1 cd m^{-2}, L_{max} = maximum luminance, $\eta_{P,max}$ = maximum power efficiency, $\eta_{C,max}$ = maximum current efficiency, and $\eta_{ext,max}$ = maximum external quantum efficiency.

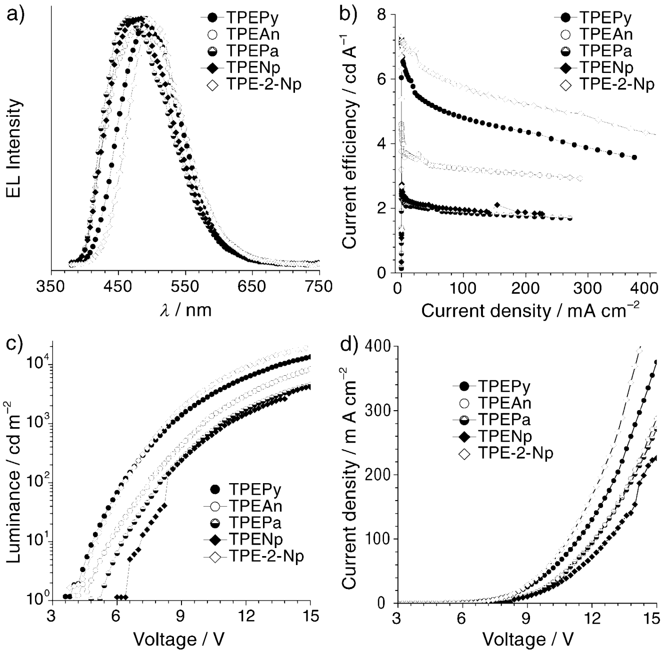


Figure 4. Performance of multilayer EL devices in which different TPEArs were incorporated, with a configuration of ITO/NPB/TPEArs/TPBi/Alq$_3$/LiF/Al. a) EL spectra. b) Current efficiency versus current density curves. c) Luminance and d) current density as a function of applied voltage.

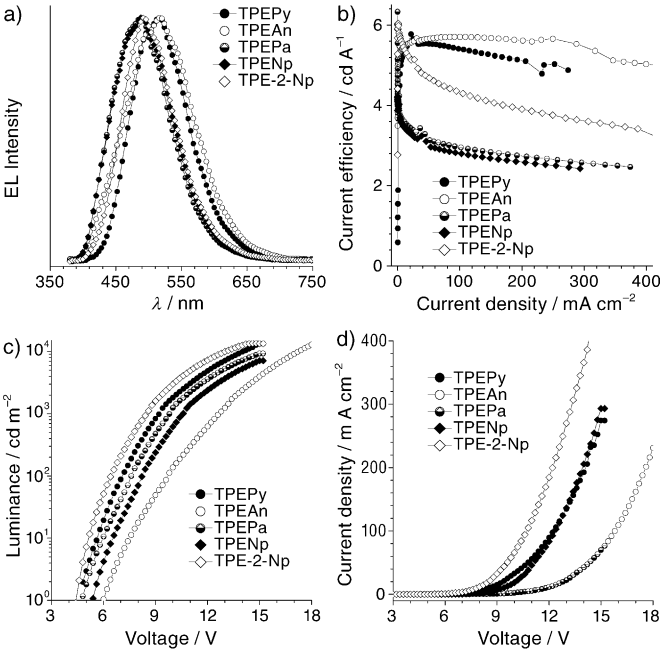


Figure 5. Performance of multilayer EL devices in which different TPEBArs were incorporated, with a configuration of ITO/NPB/TPEBArs/TPBi/Alq$_3$/LiF/Al. a) EL spectra. b) Current efficiency versus current density curves. c) Luminance and d) current density as a function of applied voltage.

5.6 lm W^{-1}, and 3.0 %, respectively. The TPE-2-Np-based device also shows a good performance, with values of 19800 cd m^{-2} (L_{max}), 7.3 cd A^{-1} ($\eta_{C,max}$), 4.2 lm W^{-1} ($\eta_{P,max}$), and 2.7 % ($\eta_{ext,max}$).

Similar good EL performances were obtained from devices fabricated with TPEBArs (Table 2 and Figure 5). These devices display emissions at 488–516 nm, which are slightly red-shifted as compared to those of devices based on TPEArs. The TPEBPy-based device is turned on at 4.8 V and exhibits an L_{max} of 13370 cd m^{-2}, a $\eta_{C,max}$ of 5.8 cd A^{-1}, a $\eta_{P,max}$ of 2.0 lm W^{-1}, and a $\eta_{ext,max}$ of 2.0 %. Even better performances were observed in the device based on TPEB-2-Np, as it possesses a low turn-on voltage of 4.4 V and a strong radiation, with an L_{max} of 13500 cd m^{-2} and $\eta_{C,max}$, $\eta_{P,max}$, and $\eta_{ext,max}$ values of 6.1 cd A^{-1}, 3.7 lm W^{-1}, and 2.5 %, respectively. As the EL of TPEBPy and TPEBAn was observed at 516 nm, close to that of Alq$_3$, two additional devices with a configuration of ITO/NPB (60 nm)/TPEBPy or TPEBAn (20 nm)/TPBi (40 nm)/LiF (1 nm)/Al (100 nm) (device II) were fabricated to examine whether the emissions in device I originated from the Alq$_3$ layer. The EL maximum of both devices was still observed at approximately 516 nm, thus indicating that the EL originates from the radiative decay of TPEBPy and TPEBAn in devices I and II. TPEBPy even shows a slightly improved performance in device II (Figure S1 in the Supporting Information), in which a lower turn-on voltage of 4.6 V and higher L_{max} and $\eta_{C,max}$ values of 25500 cd m^{-2} and 6.0 cd A^{-1} were attained.

In this work, a series of efficient solid-state emitters were constructed from TPE and conventional ACQ chromophores and their thermal and optical properties were investigated. These luminogens are almost non-fluorescent in solution but become strong emitters as nanoparticles in poor solvents or as thin films in the solid state, in which Φ_F values reach up to 100%. They are morphologically and thermally stable with high T_g and T_d values of up to 142 and 494 °C, respectively. The application of these luminogens as light-emitting layers in OLEDs was explored. Although the device structures have not yet been optimized, excellent EL performances have been achieved, thus suggesting that these luminogens are potential materials for the construction of efficient non-doped OLEDs. These results reveal that the attachment of AIE units to ACQ chromophores is a versatile strategy to solve the ACQ problem and to create, at the same time, efficient luminescent materials in the solid state with good thermal stability, efficient solid-state PL, and promising EL properties.

Acknowledgements

We acknowledge financial support from the Research Project Competition of HKUST (RPC11SC09), the Research Grants Council of Hong Kong (604711, 603509, and HKUST2/CRF/10), the University Grants Committee of Hong Kong (AoE/P-03/08), and the National Natural Science Foundation of China (21074028 and 21104012). Z.Z. thanks the Initial Funding of Hangzhou Normal University (HSQK0085), and the Natural Science Foundation of Zhejiang Province (Y4110331).

Keywords: aggregation-induced emission • fluorescence • organic light-emitting diodes • luminescence • thin films

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Received: September 6, 2011