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Fused tetraphenylethylene-triphenylamine for efficient hole transporting materials in perovskite solar cells

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Fused tetraphenylethylene-based hole transporting material shows higher power conversion efficiency and better stability compared with that of non-fused counterpart, and former molecule even outperforms the conventional spiro-OMeTAD.

During last decade, perovskite solar cells (PSCs) have caused great attention as one of the next generation photovoltaics because of their high photoelectric conversion efficiency and easy preparation process.¹⁻³ The recorded PCE over 25% have been achieved.⁴ PSCs without any hole transporting materials (HTMs) achieved an efficiency of around 16%,5-7 which is much lower compared with standard devices using HTMs. Due to the stiff price and low hole mobility the conventional 2,2',7,7'-tetrakis(N,N-di-pof methoxyphenylamino)-9,9'-spirobifluorene (spiro-OMeTAD), lots of alternatives have been developed, including small organic molecules, polymers, inorganic semiconductors.^{8, 9} Among them, small molecules have attracted much attention for their simple and well-defined structure, insignificant batch-to-batch variation compared with polymers. Lots of tetraphenylethylene-based smallmolecular HTMs with high mobility and suitable HOMO level have been proved efficient candidates for spiro-OMeTAD, such as TAE-4,10 DBC-OMeDPA,11 DMZ,12 TPE,13 KR216 (Bifluo-OMeTAD),14, 15 EtheneTTPA (TAE-1),^{16, 17} pm-TPE-4DPA,¹⁸ W2,¹⁹ illustrating their great application potential. The molecular structures of these reported tetraphenylethylene-based HTMs is also illustrated in Scheme S1.

A planar molecular conformation and enhanced π -conjugation have been proved as efficient way to facilitate intermolecular

[†] Footnotes relating to the title and/or authors should appear here.

charge transport and adjust their HOMO level with perovskite materials. We further introduce fused tetraphenylethylene as reference compound, termed **TPE** in this work,¹³ was also prepared, which had been proved as an efficient HTM for Tin-Based PSCs. The molecular structures of the investigated compounds are illustrated in Fig. 1. **F-TPE** is sequential synthesized via Scholl reaction and Suzuki-Miyaura cross-coupling reactions, as provided in ESI.



Fig. 1 Molecular structures of tetraphenylethylene-triphenylamine molecule and fused counterpart.

The optimized molecular geometries, electronic density distribution of LUMO and HOMO, electrostatic surface potential map and hole reorganization energy for TPE and F-TPE are explored by time dependent density functional theory (TDDFT) using Gaussian 16 at B3LYP under the B3LYP/6-311G* level. As illustrated in Fig. 2 and Fig. S1, F-TPE owns superior planar structure compared with TPE from optimized molecular geometries (smaller dihedral angles), allowing for better close molecular packing.²⁰ The orbital density of HOMO of F-TPE is fully delocalized evenly over the whole molecules, illustrating superior conjugation and promoting carrier transport. Therefore, F-TPE is more conducive to offset the disparity between the HOMO and valence-band maximum of perovskite.²¹⁻²² The electrostatic surface potential (ESP) illustrates more electronegative character of the fused tetraphenylethylene core, while the most electropositive unit is delocalized on the whole nonfused counterpart. Therefore, fused tetraphenylethylene molecule possess a dipole in the structure, which may be also anticipated to a

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close molecular packing.^{23, 24} On the other hand, the calculated hole reorganization energies (λ_{hole}) of **TPE** and **F-TPE** is 0.121 and 0.085 eV, respectively, illustrating stronger driving forces of fused tetraphenylethylene molecule for charge transfer. Therefore, tetraphenylethylene-based molecule would exhibit higher hole mobility after being fused.



Fig. 2 Optimized geometrical structures and electrostatic surface potential map for **TPE** and **F-TPE**.

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Fig. 3a shows the UV-vis absorption and emission properties of TPE and F-TPE in CH₂Cl₂ solution, the corresponding characteristic data are summarized in Table 1. The UV-vis spectra of TPE exhibits an absorption peak (λ_{max}) at 362 nm. Interestingly, when tetraphenylethylene core of TPE is fused with single bond, which shows an obviously bathochromic spectra responding, which is consistent with TDDFT calculated absorption spectra (Fig. S2) and the electron cloud result. The λ_{max} of fused tetraphenylethylenebased F-TPE rises to 388 nm. Therefore, fused core owns stronger conjugation ability with triphenylamine groups than non-fused counterpart, which is beneficial for localized aromatic π - π * excitation migration from core to terminal moieties. On the other hand, though F-TPE shows slightly stronger absorption in visible light than TPE, which is not large enough to compete significantly with perovskite light harvesting layer in PSCs, which has been extendedly proved in lots of literatures.⁸ The photoluminescence results of TPE and F-TPE display a maximum peak at 551 and 528 nm, respectively, with the Stokes shifts of 189 and 140 nm. It is noted that the Stokes shift of fused tetraphenylethylene based molecule is smaller than non-fused one, which can be resulted from higher planarity after being fused, also illustrating smaller conformational changes in excited state. The optical band gap (E_a) of TPE and F-TPE estimated from normalized UV-vis and PL spectrum are 2.79 and 2.71 eV, respectively.²⁵



Fig. 3 (a) The optical absorption and emission spectrum, and (b) energy level diagram of the device with TPE and F-TPE as HTM.

The electrochemical properties of investigated molecules are evaluated by cyclic voltammetry (CV) Cand 10d ifferential opulse voltammetry (DPV) measurements.^{26, 27} As shown in Fig. S3a, the redox peaks of TPE and F-TPE are greatly reversible, illustrating that the molecules own superior electrochemical stability. The HOMO levels of TPE and F-TPE estimated form DPV data (Fig. S3b) are -5.26 and -5.29 eV, respectively. It can be noted that fused tetraphenylethylene based molecule owns slightly lower HOMO level compared with non-fused one, suggesting that higher opencircuit voltage (V_{oc}) of corresponding PSCs could be obtained.²⁸ As the valence band of conventional (FAPbI₃)_{0.85}(MAPbBr₃)_{0.15} perovskite locate at -5.65 eV,^{29, 30} proving the investigated molecules are favorable for hole extraction from perovskite. The LUMO levels of TPE and F-TPE calculated from Eg and HOMO levels are -2.47 and -2.58 eV respectively. The significantly positive LUMO levels of investigated molecules compared with the conduction band of $(FAPbI_3)_{0.85}(MAPbBr_3)_{0.15}$ (-3.92 eV)²⁹ could inhibit unexpected electron back transfer to counter electrode and decrease the chance of recombination. The energy level diagram with TPE and F-TPE as HTMs is outlined in Fig. 3b. Moreover, the HOMO (LUMO, Eg) level of TPE and F-TPE from TDDFT is -4.41 (-1.29, 3.12) and -4.41 (-1.34, 3.05) eV, respectively. All experimental data is consistent with TDDFT results.

To investigate the effect of the fused core on charge carrier mobility, we carried out space charge limited current (SCLC) measurements through hole-only device with an architecture of ITO/PEDOT:PSS/HTM/Au devices referring to previous reports.³⁰ The current–voltage (*J*–*V*) curves are shown in Fig. S4, the hole mobility values of **TPE** and **F-TPE** calculated according to Mott-Gurney law are 3.21×10^{-4} and 7.48×10^{-4} cm²V⁻¹s⁻¹, respectively, which is consistent with hole reorganization energies of them. It is noted that **F-TPE** owns obviously higher hole mobility than **TPE**, which could be resulted from better intermolecular π – π stacking and conjugation of fused tetraphenylethylene based molecule.³²

 Table 1
 Photophysical, electrochemical, and hole mobility properties of TPE and F-TPE.

properties of TPE and F-TPE.								
compoun d	λ_{max} [a]	λ_{PL} [a,b]	λ_{stoke}	Eg [c]	HOM O ^[d]	LUM O ^[e]	μ	λ_{hole}
	(nm)	(nm)	(nm)	(eV)	(eV)	(eV)	(cm ² V ⁻ ¹ S ⁻¹)	(eV)
TPE	362	551	189	2.7 9	-5.26	-2.47	3.21×1 0 ⁻⁴	0.12 1
F-TPE	388	528	140	2.7 1	-5.29	-2.58	7.48×1 0 ⁻⁴	0.08 5

[a] Measured in CH₂Cl₂ solution. [b] Excitation at λ_{max} . [c] E_g = 1240/ $\lambda_{intersection}$. [d] Evaluated from DPV curves.^{26, 27} [e] $E_{LUMO} = E_{HOMO} + E_g$; [f] hole reorganization energies, determined at the TDDFT/B3LYP/6-311G* level of theory.

The effect of investigated molecules on film morphology deposited on perovskite layer are analyzed by scanning electron microscopy (SEM) and atomic force microscope (AFM) measurements, as displayed in Fig. S5. it can be noted that F-TPE (RMS = 11.2 nm) exhibits smaller root-mean-square roughness

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compared with TPE (RMS = 18.7 nm). To investigated the hydrophobicity of the investigated molecules, the water-contact angles of **TPE** and **F-TPE** are recorded. As shown in Fig. S5, **TPE** and **F-TPE** exhibit water-contact angles of around 79° and 84°, respectively. Fused tetraphenylethylene based molecule owns obvious better hydrophobicity than non-fused counterpart, which may be resulted from better intermolecular π - π stacking of former one because there is no hydrophobic long chain alkyl on the structure and form better compact film (As confirmed by above AFM measurements).³³

Effect of fused core on the charge-transfer dynamics at perovskite/HTM interface is investigated by steady-state photoluminescence (TIPL) and time-resolved photoluminescence (TRPL) measurements, as illustrated in Fig. S6. In TIPL measurements (Fig. 6a), it can be noted that the quenching values of perovskite coated with **TPE** and **F-TPE** are 91% and 95%, respectively, compared with perovskite monolayer. In TRPL curves (Fig. 6b), the τ_e are around 11.26, 2.51 and 1.58 ns for perovskite and the perovskites coated with **TPE** and **F-TPE**, respectively. Therefore, fused tetraphenylethylene based molecule own obviously better hole extraction ability compared with non-fused counterpart, which can be resulted from higher hole mobility after be fused.

In order to explore the molecular structure variation of HTMs on the photovoltaic properties, the conventional PSCs with an architecture of FTO/bl-TiO₂/mp-TiO₂/perovskite/HTM/Au using **TPE** or **F-TPE** as HTMs are assembled referring to our previous reports.²⁶ The detailed device fabrication process is illustrated in ESI. Each groups of the PSCs without or with dopants in HTMs include 8 independent samples. The photovoltaic performance of devices is recorded under AM 1.5 illumination (100 mW cm⁻²), and the detailed parameters are listed in Table S1.



Fig. 4 (a) *J-V* curves of the best devices employing dopant-free and doped HTMs, (b). stability test for the devices with doped HTMs.

Table 2	Photovoltaic	parameters	of	best-performi	ng RSCs with
dopant-f	ree and doped	TPE, F-TPE,	as w	ell as ପobed ସହ	W6º0Me7AD!

	НТМ	Voc	J _{sc} (mA	FF	PCE (%)	
		(V)	cm⁻²)	(%)		
Dopant-	TPE	1.03	17.21	62	10.98	
free	F-TPE	1.05	18.21	69	13.11	
Doped	TPE	1.07	20.87	71	15.84	
	F-TPE	1.11	21.35	77	18.30	
	spiro-OMeTAD	1.09	22.31	75	18.16	

The current-voltage (J-V) curves and parameters of best-performing PSCs with different HTMs are shown in Fig. 4 and Table 2. The best device with dopant-free F-TPE illustrates a PCE of 13.11%, together with an V_{oc} of 1.05 V, a short-circuit current (J_{sc}) of 18.21 mA cm⁻² and a fill factor (FF) of 0.69. Interestingly, when the HTM layer is substituted by non-fused dopant-free TPE, the device exhibits a lower PCE of 10.98%. When TPE and F-TPE are doped with conventional TBP and Li-TFSI, the device performance displays obviously improvement, which is identical with most of reported HTMs.⁸ The best-performing device employing doped F-TPE illustrates a promisingly PCE of 18.30%, which is also obviously higher than doped TPE (PCE = 15.84%). It can be noted that F-TPEbased based devices exhibit better performance regardless of concentration or whether dopants exist, as illustrated in Table S1 and Fig. S7. The enhanced of $V_{\rm oc}$ of the devices based on fused tetraphenylethylene based molecule could be resulted from lower HOMO level compared with non-fused counterpart, leading to enhancing hole-electron separation efficiency at perovskite/HTM interface, as shown in TIPL and TRPL study. The V_{oc} variation of the devices employing fused or non-fused tetraphenylethylene based molecule also fully coincide with CV data. Moreover, due to fused tetraphenylethylene based molecule owns higher hole mobility because of better planar configuration and better π conjugation between core and terminal groups compared with non-fused one, which enable tetraphenylethylene based molecule based devices display higher FF and J_{sc}.

As a reference, doped spiro-OMeTAD shows a PCE of 18.16% (Fig. S8), which is even behind F-TPE under the same condition. Therefore, we believe that the PCE of PSCs employing F-TPE as HTMs exceeding 20% might be attainable through extensive optimization of the device fabrication and perovskite preparation.^{34,} ³⁵ The incident photo-to-current conversion efficiency (IPCE) spectra of the best-performing PSCs with doped TPE or F-TPE was also measured to confirm the photocurrent, as shown in Fig. S10. It can be noted that F-TPE exhibit obviously higher values than TPE in the range of 400-750 nm, which is consistent with the J_{sc} results obtained from J-V measurements. Therefore, the results further prove that the fused HTM owns superior hole extraction and transfer ability. As shown in Fig. S11 and Table S2, the hysteresis behaviors of the best devices employing doped TPE or F-TPE are also investigated through different scan directions. It can be noted that the fused tetraphenylethylene based molecule exhibits minor hysteresis than that of non-fused counterpart, which can be resulted from its high hole mobility.²¹

Finally, the long-term stability was also measured, the devices with doped HTMs are stored at room temperature without encapsulation in a dark drying oven (~20% relative humidity). As

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shown in Fig. 4b, it can be noted that the PSC with **F-TPE** owns obviously better stability compared with that of **TPE**, which may be due to better hydrophobicity after tetraphenylethylene was fused.³⁶ Moreover, F-TPE-based devices show similar long-term stability with that of spiro-OMETAD (Fig. S9 and Fig. 4b).

In summary, we have developed a novel facile fused tetraphenylethylene-based HTM, which has lower HOMO level and higher hole mobility compared with a reference non-fused counterpart. The PSCs with fused tetraphenylethylene-based HTM shows better photovoltaic properties regardless of the existence of dopants than that of non-fused one, which even outperforms the conventional spiro-OMeTAD. The results further prove the tetraphenylethylene with a fused structure can be a promising unit for developing new HTMs for efficient and stable PSCs.

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