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A Solution Processable D-A-D Molecule based on Thiazolothiazole for High Performance Organic Solar Cells

Qinqin Shi, Pei Cheng, Yongfang Li,* and Xiaowei Zhan*

Due to the potential advantages of low cost, light weight, flexibility, and large area fabrication, bulk heterojunction (BHJ) solar cells have attracted much attention.[1–4] The power conversion efficiencies (PCEs) of polymer solar cells (PSCs) based on blends of polymer donors and fullerene acceptors have reached 7–8%.[5–9] Although the highest PCE of solution-processed organic small molecule solar cells (OSCs) was still lower than their polymeric counterparts, the advantages of well defined molecular structure, definite molecular weight, and high purity without batch to batch variations render small molecule-based OSCs a promising field.[10–13] Among the small molecules with high photovotaic performance, the widely used donor building blocks mainly involve triphenylamine (TPA) and thiophene;[14,15] and acceptor building units include diketopyrrolopyrrole (DPP),[16,17] benzothiadiazole (BT),[18–21] squaraine,[22,23] dicyanovinyl,[24–27] perylene diimide,[28] etc.[29–31] Up to now, solution processed OSCs based on small molecules with PCEs > 4% remain rare.[17,21,32–34] Most of the small molecule-based BHJ solar cells exhibited PCEs lower than 3%.

Thiazolothiazole (TT) has a rigid and coplanar fused ring, and thereby ensures highly extended π-electron system and strong π-stacking. As a result, conjugated small molecules and polymers based on thiazolothiazole exhibited high charge carrier mobilities.[35–37] Recently, several thiazolothiazole-based copolymers were synthesized for PSC applications, and PCEs up to 5.59% were reported.[38–43] However, to our knowledge, there have been no reports on TT-based small molecules for application in organic solar cells. Here we report synthesis and characterization of a new linear, D-A-D organic small molecule with TPA as donor (D) unit, thiophene as bridge, and thiazolothiazole as acceptor unit, (TT-TTPA, **Scheme 1**). Solution processed solar cells based on blend of TT-TTPA and PC$_{71}$BM afforded a PCE as high as 3.73% after thermal annealing.

The synthetic route of compound TT-TTPA is shown in Scheme 1. Stille coupling reaction between 2,5-bis(3-dodecyl-5-trimethylstannyl-thiophen-2-yl)-thiazolo[5,4-*d*]thiazole[36]

and *N,N*-diphenyl-4-bromoaniline[44] afforded TT-TTPA. The electron-withdrawing building block thiazolothiazole was introduced to lower HOMO level (finally increase open-circuit voltage of OSC) and to extend absorption via intramolecular D-A charge transfer. TT-TTPA was fully characterized by spectroscopic methods and elemental analysis. TT-TTPA is soluble in common organic solvents such as dichlorobenzene, chloroform and THF due to the two solubilizing *n*-dodecyl substituents. The thermal property of TT-TTPA was investigated by thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). This D-A-D molecule exhibits excellent thermal stability with decomposition temperature (5% weight loss) at ca. 355 °C (Figure S1 in Supporting Information). The DSC traces for this compound do not show any peaks indicative of the phase transitions from room temperature to 250 °C, suggesting that it could be amorphous.

Figure 1a shows the normalized UV-vis absorption spectra of TT-TTPA in dilute CHCl$_3$ solution and in thin film. TT-TTPA in solution exhibits strong absorption with extinction coefficient of 2.95×10^4 L mol^{-1} cm^{-1} at 422 nm. Absorption spectrum in thin film of TT-TTPA shows red shift of 14 nm relative to that in solution, indicating that self-organization behavior somewhat exists in film. The optical band gap estimated from the absorption edge of the thin film is 2.31 eV.

The electrochemical property of TT-TTPA was investigated by cyclic voltammetry (CV) method in film on a platinum working electrode. As shown in Figure 1b, TT-TTPA exhibits one irreversible reduction peak and two quasi-reversible oxidation peaks. The onset oxidation and reduction potentials versus FeCp$_2$$^{+/0}$ are 0.59 and −1.89 V, respectively. The HOMO and LUMO energies were estimated to be −5.39 and −2.91 eV from the onset oxidation and reduction potentials, respectively, assuming the absolute energy level of FeCp$_2$$^{+/0}$ to be 4.8 eV below vacuum. The LUMO level of TT-TTPA is higher than that (−3.9 eV) of PC$_{71}$BM;[45] the LUMO gap between the donor and acceptor is large enough to guarantee photoinduced electron transfer between them. The difference between the LUMO of the acceptor and the HOMO of the donor is as large as 1.5 eV, which could lead to high open-circuit voltage (V_{oc}) of solar cells.

To demonstrate potential applications of TT-TTPA in OSCs, we used TT-TTPA as an electron donor and PC$_{61}$BM or PC$_{71}$BM[45,46] as an electron acceptor, and fabricated BHJ OSCs with a structure of ITO/PEDOT:PSS/TT-TTPA:PCBM/Ca/Al. **Table 1** summarizes the open circuit voltage (V_{oc}), short circuit current density (J_{sc}), fill factor (FF), and power conversion efficiency (PCE) of the PC$_{71}$BM-based devices at different donor/acceptor weight ratios. The V_{oc} of 0.9 ~ 0.94 V is insensitive to the donor/acceptor weight ratio, while J_{sc}, FF, and PCE of the devices are strongly dependent on the

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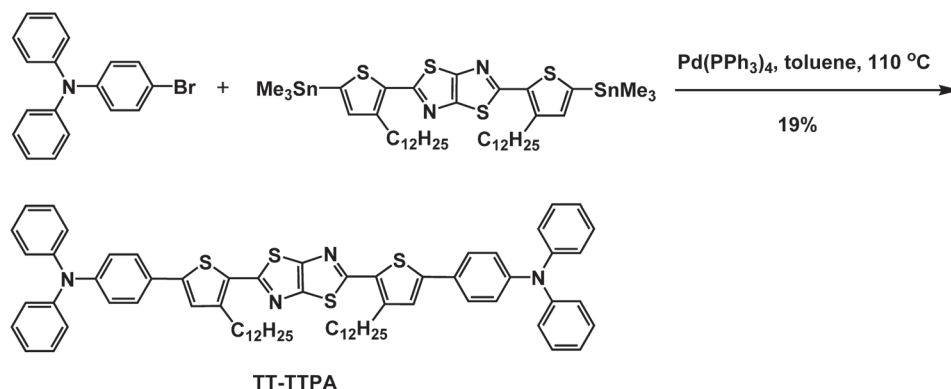
Scheme 1. Synthetic route of compound TT-TTPA.

donor to acceptor weight ratio in the active blend layers. The donor/acceptor weight ratio of 1:4 gave the best performance: V_{oc}, J_{sc}, FF and PCE reaches 0.92 V, 7.39 mA cm^{-2}, 38.1% and 2.59%, respectively. The PCE of 2.59% for the PC$_{71}$BM-based
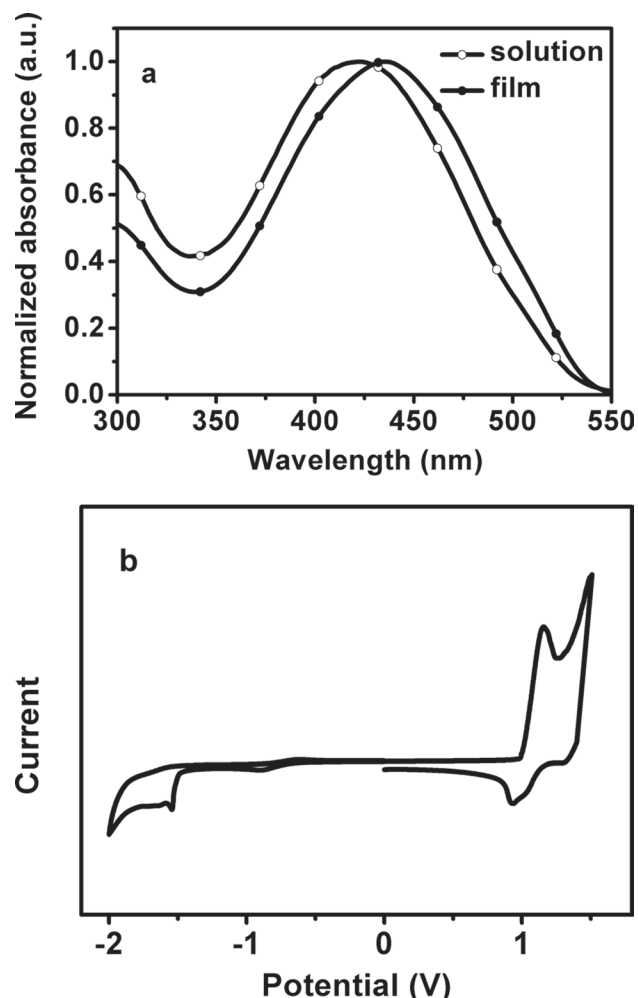
devices is higher than that (1.85%) of the PC$_{61}$BM-based devices, probably due to better absorption of PC$_{71}$BM relative to PC$_{61}$BM. Thermal annealing at 110 °C for 10 min improves J_{sc} and FF to 9.39 mA cm^{-2} and 43.7%, respectively. The improved J_{sc} and FF leads to an enhanced efficiency of 3.73%, 44% enhancement relative to that for the as-cast device (Table 1, **Figure 2**a). More than 20 devices were fabricated and their average PCEs were 3.61%. The incident photon to converted current efficiency (IPCE) and absorption spectra of the blend thin film with donor/acceptor weight ratio of 1:4 without and with annealing at 110 °C are shown in Figure 2b. Compared to that of pure TT-TTPA solid film, absorption of the blend broadens and extends to 700 nm as a result of contribution from PC$_{71}$BM. Increase in the absorbance of the blend film after annealing was also observed. The blend of TT-TTPA:PC$_{71}$BM (1:4, w/w) exhibits a broad IPCE plateau between 300 and 700 nm with a maximum of 49% at 480 nm. PC$_{71}$BM makes significant contribution to IPCE, which is consistent with the PCE data. Thermal annealing improves the IPCE maximum to 55%.

To understand the influence of charge carrier transport on photovotaic performance, we used space-charge limited current (SCLC) method to measure the hole and electron mobilities in the blend. **Figure 3** shows the J–V curves for hole-only (a) and electron-only (b) devices processed as cast and annealed at 110 °C. The average hole and electron mobilities for the blend as-cast were found to be 3.66×10^{-7} cm^2 V^{-1} s^{-1} and 1.03×10^{-4} cm^2 V^{-1} s^{-1}, respectively. The percolation pathways for hole transport are poorly formed at low donor concentration (20%), leading to lower hole mobilities; while the percolation pathways for electron transport are well formed at high acceptor concentration (80%), leading to higher electron mobilities. After annealing at 110 °C for 10 min, the blend exhibited hole mobility of 1.28×10^{-6} cm^2 V^{-1} s^{-1} and electron mobility of 3.02×10^{-6} cm^2 V^{-1} s^{-1}. The X-ray diffraction (XRD) patterns of pure TT-TTPA thin films (Figure S2 in Supporting Information)

Figure 1. (a) Absorption spectra in solution and in film and (b) cyclic voltammogram of TT-TTPA film in CH$_3$CN/ 0.1 M [^{n}Bu$_4$N]$^+$[PF$_6$]$^-$ at 50 mV s^{-1}; the horizontal scale refers to an anodized Ag wire pseudo-reference electrode.

Table 1. Photovoltaic performances of BHJ solar cells based on TT-TTPA:PC$_{71}$BM under the illumination of AM 1.5 solar simulator, 100 mW cm^{-2}.

TT-TTPA:PC$_{71}$BM [w/w]	V_{oc} [V]	J_{sc} [mA cm^{-2}]	FF [%]	PCE [%]	
				best	ave
1:2	0.94	5.28	38.1	1.89	1.85
1:3	0.90	7.36	34.7	2.30	2.23
1:4	0.92	7.39	38.1	2.59	2.52
1:4$^{a)}$	0.91	9.39	43.7	3.73	3.61

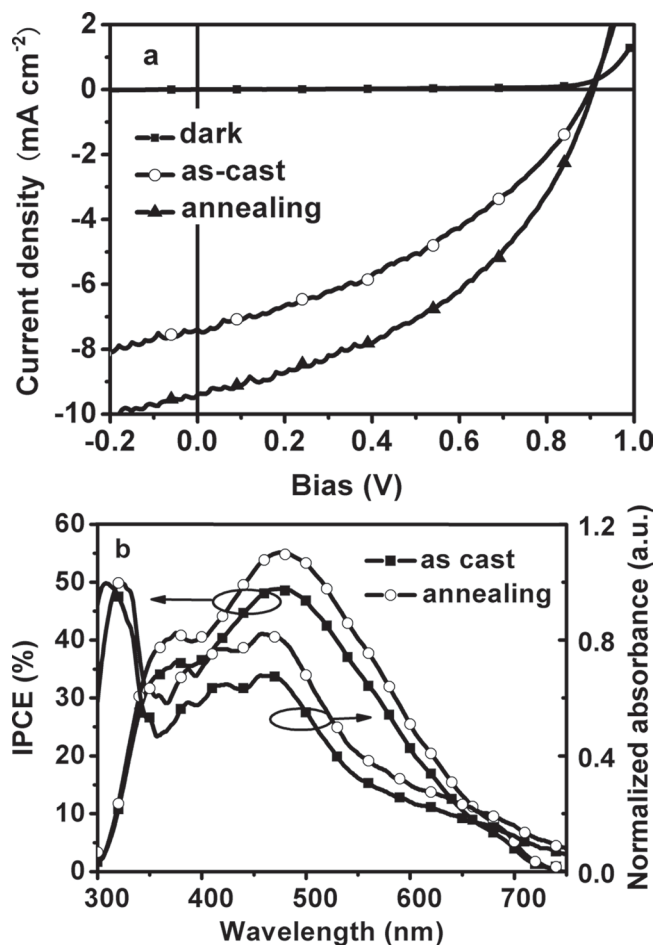$^{a)}$Thermal annealing at 110 °C for 10 min.

Figure 2. As-cast and annealed (a) *J*–*V* curves and (b) IPCE spectra of a device with the structure ITO/PEDOT:PSS/TT-TTPA:PC$_{71}$BM (1:4, w/w)/Ca/Al in dark and under the illumination of an AM 1.5 solar simulator, 100 mW cm^{-2} (left) and corresponding absorption spectra (right).

Figure 3. Field dependence of the current for hole-only (a) and electron-only (b) OSC devices as cast and annealed at 110 °C for 10 min.

show that the broad peak at $2\theta = 23.5°$ is enhanced after annealing at 110 °C for 10 min. This result indicates that after annealing more ordered thin film structure could be obtained as a result of enhanced π–π stacking. Thus, thermal annealing leads to hole mobility enhancement by a factor of 3.5. However, the increase in order of the donor phase probably causes decrease in order of the acceptor phase, which leads to electron mobility reduction by a factor of 34. The more balanced hole and electron transport is beneficial to enhancement in J_{sc} and FF.

The active layer morphology was examined by atomic force microscope (AFM) technique in tapping mode. **Figure 4** shows the actual phase separation state of blend films of TT-TTPA:PC$_{71}$BM (1:4, w/w) as cast and after annealing. The blend films exhibit a typical cluster structure with many aggregated domains and a root-mean-square (RMS) roughness of 0.376 nm. The phase separation sizes estimated by cross-section profiles are about 10-20 nm, which is within exciton diffusion range. This perfect phase separation is beneficial to charge separation and enhanced efficiency of the OSC. After thermal
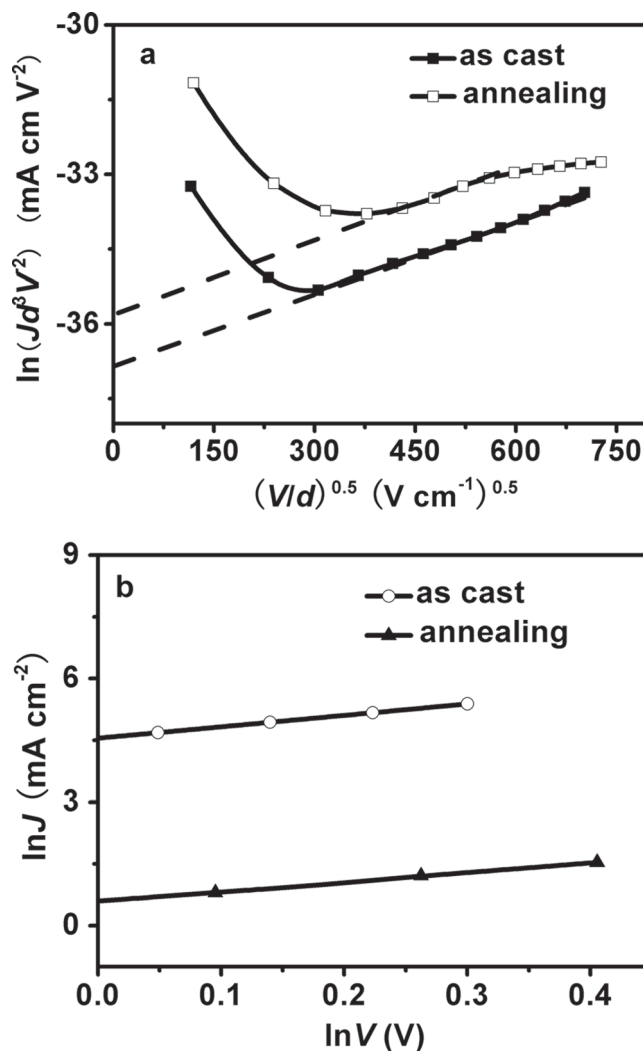
annealing, RMS roughness of the blend films decrease to 0.368 nm. The more uniform and smooth films lead to improved FF.

In summary, we demonstrate the first example on thiazolothiazole-based small molecules for high-performance OSCs. The linear D-A-D small molecule TT-TTPA exhibits excellent thermal stability and relatively low HOMO energy level. Through tuning the donor to acceptor weight ratios and thermal annealing at 110 °C, the TT-TTPA:PC$_{71}$BM (1:4, w/w) blend film exhibited perfect phase separation and balanced hole and electron mobilities, which leads to the best device performance: V_{oc} of 0.91 V, J_{sc} of 9.39 mA cm^{-2}, FF of 43.7% and PCE of 3.73%. The PCE of 3.73% is among the top reported for solution processed BHJ OSCs based on small molecules. Optimization of the device structures and post-treatments (e.g. solvent annealing or additive addition) can be expected to substantially increase the PCE of the OSCs. This preliminary work demonstrates that TT-based small molecules are promising donor materials for solution processed BHJ OSCs.
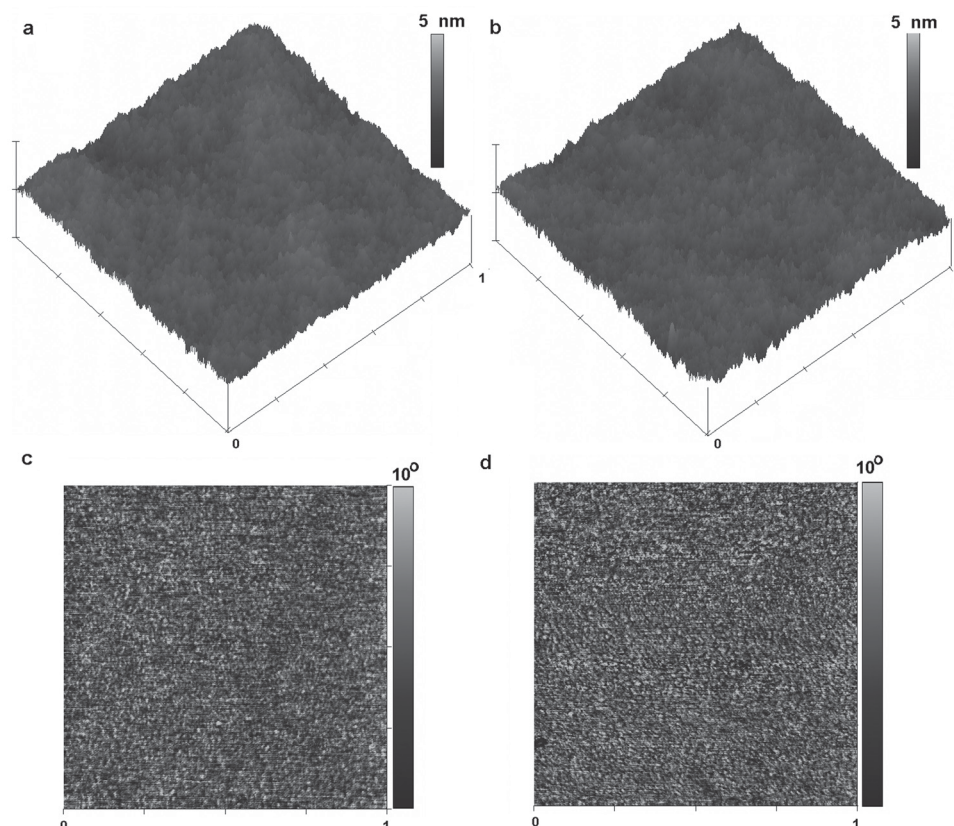
Figure 4. AFM height images (top) and phase images (bottom) of TT-TTPA:PC$_{71}$BM (1:4, w/w) blend films as cast (left) and annealed at 110 °C for 10 min (right).

Experimental Section

2,5-Bis(5-triphenylamino-3-dodecylthiophen-2-yl)-thiazolo[5,4-d]thiazole (TT-TTPA): To a three-necked round bottom flask were added 2,5-bis(3-dodecyl-5-trimethylstannyl-thiophen-2-yl)-thiazolo[5,4-*d*]thiazole (194 mg, 0.2 mmol) and *N,N*-diphenyl-4-bromoaniline (258 mg, 0.8 mmol), and anhydrous toluene (20 mL). The mixture was deoxygenated with nitrogen for 30 min. Pd(PPh$_3$)$_4$ (23 mg, 0.02 mmol) was added under nitrogen. The mixture was refluxed for two days and then cooled down to room temperature. A solution of KF (1 g) in water (10 mL) was added and stirred at room temperature for 2.5 h to remove the tin impurity. Water (100 mL) was added and the mixture was extracted with CHCl$_3$ (2 × 100 mL). The organic phase was dried over anhydrous MgSO$_4$. After removing the solvent, the residue was purified by column chromatography on silica gel using petroleum ether/CHCl$_3$ (10:1) as eluent yielding a red solid (43 mg, 19%). ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.51 (d, *J* = 8.4 Hz, 4H), 7.29 (t, *J* = 7.8 Hz, 8H), 7.15 (d, *J* = 8.4 Hz, 8H), 7.11 (s, 2H), 7.08 (m, 8H), 2.95 (t, *J* = 7.6 Hz, 4H), 1.75 (m, 4H), 1.45 (m, 4H), 1.36 (m, 4H), 1.26 (m, 28H), 0.87 (m, 6H). ^{13}C NMR (100 MHz, CDCl$_3$): δ 161.09, 149.97, 147.99, 147.35, 145.26, 144.09, 130.29, 129.46, 127.27, 126.58, 125.65, 124.88, 123.48, 123.16, 32.05, 30.63, 29.95, 29.82, 29.79, 29.63, 29.49, 22.82, 14.26. MS (MALDI): *m/z* 1129 (M$^+$). Anal. Calcd for C$_{72}$H$_{80}$N$_4$S$_4$: C, 76.55; H, 7.14; N, 4.96. Found: C, 76.39; H, 7.29; N, 4.63%. UV (CHCl$_3$), λ$_{max}$ (ε$_{max}$): 422 nm (2.95 × 10^4 L mol^{-1} cm^{-1}).

Fabrication and characterization of photovoltaic cells: Photovoltaic cells were fabricated with ITO as positive electrode and Ca/Al as negative electrode. The patterned indium tin oxide (ITO) glass (sheet resistance = 30 Ω □$^{-1}$) was pre-cleaned in an ultrasonic bath of acetone and isopropanol, and treated in ultraviolet-ozone chamber (Jelight Company, USA) for 30 min. A thin layer (35 nm) of poly(3,4-ethylenedioxythiophene):poly(styrene sulfonate) (PEDOT:PSS, Baytron P VP AI 4083, Germany) was spin-coated onto the ITO glass and baked at 150 °C for 20 min. A dichlorobenzene solution of blend of compound TT-TTPA with PC$_{61}$BM or PC$_{71}$BM (1:2 or 1:3 or 1:4, w/w) was subsequently spin-coated on PEDOT:PSS layer to form a photosensitive layer. The thickness (ca. 70 nm) of the photosensitive layer was measured by Ambios Technology XP-2 profilometer. A calcium (ca. 15 nm) and aluminium layer (ca. 60 nm) was then evaporated onto the surface of the photosensitive layer under vacuum (ca. 10^{-4} Pa) to form the negative electrode. The active area of the device was 4 mm^2. *J*–*V* curve was measured with a computer-controlled Keithley 236 Source Measure Unit. A xenon lamp coupled with AM1.5 solar spectrum filters was used as the light source, and the optical power at the sample was 100 mW cm^{-2}. The IPCE spectrum was measured by Stanford Research Systems model SR830 DSP lock-in amplifier coupled with WDG3 monochromator and 500 W xenon lamp.

Hole- or electron-only diodes were fabricated using the architectures: ITO/PEDOT:PSS/TT-TTPA:PC$_{71}$BM/Au for holes and Al/TT-TTPA:PC$_{71}$BM/Al for electrons. Mobilities were extracted by fitting the current density–voltage curves using the Mott–Gurney relationship (space charge limited current).[47]

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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