Efficient thick film all-polymer solar cells enabled by incorporating ester-substituted non-fullerene based polymer acceptor

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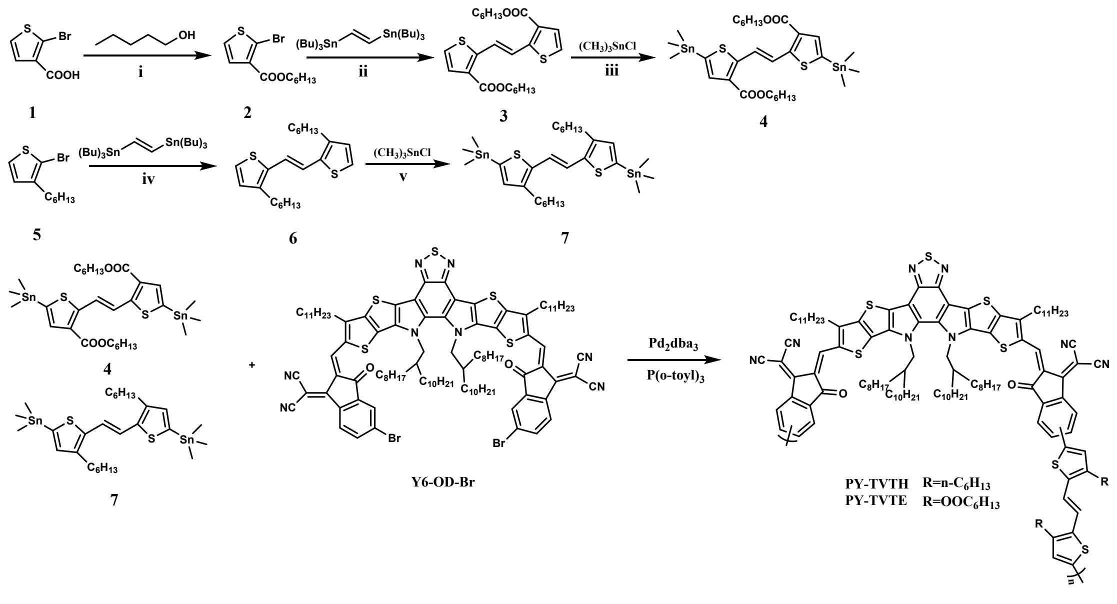
1. Materials and synthesis

All chemicals, unless otherwise specified, were purchased from commercial resources and used as received. Toluene was distilled from sodium benzophenone under nitrogen before using. The compound 1 and 5 were purchased from Woer Jiming research institute.



Scheme S1. The synthetic routes of the copolymers.

Synthesis of compound 2

2-bromothiophene-3-carboxylic acid (1) (3.00 g, 14.49 mmol), N,N′-dicyclohexylcarbodiimide (DCC) (4.48 g, 21.74 mmol), and (Dimethylamino)pyridine (DMAP) (1.06 g, 8.69 mmol) were dissolved in 20 mL dry dichloromethane, and stirred at room temperature for 15-30 minutes. Cyclohexanol (3.05 g, 21.74 mmol) was slowly added. After stirring at room temperature for 24 hours, the mixture was filtered to remove impurities, and the filtrate was poured into water and extracted with dichloromethane (DCM). The combined organic phases were dried over anhydrous $MgSO_4$ and filtered. After further concentration under reduced pressure, purification was performed by silica gel column chromatography using petroleum ether:dichloromethane (3:1) as eluent, yielding compound 2 (4.84 g, 89% yield) as a colorless liquid. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.36 (d, J = 5.8 Hz, 1H), 7.21 (d, J = 5.8 Hz, 1H), 4.28 (t, J = 6.6 Hz, 2H), 1.74 (d, J = 8.0, 6.7 Hz, 2H), 1.50 – 1.40 (m, 2H), 1.33 (pd, J = 5.5, 3.1 Hz, 4H), 0.94 – 0.86 (m, 3H).

Synthesis of compound 3

Compound 2 (3.97 g, 10.58 mmol), trans-1,2-bis(tri-n-butylstannyl)ethylene (2.79 g, 4.60 mmol), and $Pd(PPh_3)_4$ (150 mg, 0.13 mmol) were mixed in 25 mL of toluene under a nitrogen atmosphere. The mixture was stirred overnight at 110°C. After completion of the reaction, the crude product was cooled to room temperature and concentrated under reduced pressure. The mixture was then poured into water and extracted with dichloromethane. The combined organic phases were dried over anhydrous $MgSO_4$ and the solvent was removed under reduced pressure. Purification was performed by silica gel column chromatography using petroleum ether: dichloromethane (1:1) as eluent. Finally, compound 3 was obtained as a pale yellow solid (2.33 g, 82% yield) after recrystallization from methanol. ^1H NMR (400 MHz, Chloroform-*d*) δ 8.19 (s, 1H), 7.44 (d, $J = 5.3$ Hz, 1H), 7.13 (d, $J = 5.3$ Hz, 1H), 4.30 (t, $J = 6.6$ Hz, 2H), 1.78 (d, $J = 8.3$, 6.7 Hz, 2H), 1.46 (d, $J = 15.1$, 10.1, 6.3, 3.2 Hz, 2H), 1.34 (t, $J = 6.0$, 2.7 Hz, 4H), 0.94 – 0.86 (m, 3H).

Synthesis of compound 4

Compound 3 (0.50 g, 0.81 mmol) was dissolved in 40 mL of anhydrous tetrahydrofuran (THF) in a 100 mL two-necked flask. Under a nitrogen atmosphere at -78°C, n-butyllithium (1.14 mL, 2.84 mmol) was added dropwise to the sample. The stirring mixture was kept at this temperature for 30 minutes before being cooled to -78°C again. Trimethyltin chloride (3.25 mL, 3.25 mmol) was then added to the reaction mixture, and stirring continued for 1 hour under these conditions. The mixture was then heated to room temperature and stirred overnight. Water was added to the mixture to stop the reaction, and the mixture was extracted twice with dichloromethane. The organic phase was dried over anhydrous $MgSO_4$, and the solvent was removed under reduced pressure. The crude product was obtained by ethanol recrystallization to yield compound 4. Vacuum drying produced a yellow-green solid (0.39 g, 51.02% yield). ^1H NMR (400 MHz, Chloroform-d) δ 8.21 – 8.15 (m, 1H), 7.49 (s, 1H), 4.30 (t, J = 6.7 Hz, 2H), 1.80 (p, J = 6.8 Hz, 2H), 1.35 (d, J = 3.6 Hz, 5H), 0.94 – 0.84 (m, 4H), 0.39 (s, 9H).

Synthesis of compound 6

2-bromo-3-hexylthiophene (1.02 g, 4.1 mmol), trans-1,2-bis(tri-n-butylstannyl)ethylene (1.0 g, 1.60 mmol), and $Pd(PPh_3)_4$ (95 mg, 0.08 mmol) were mixed in 25 mL of toluene under a nitrogen atmosphere. The mixture was stirred overnight at 110°C. After completion of the reaction, the crude product was cooled to room temperature and concentrated under reduced pressure. The mixture was then poured into water and extracted with dichloromethane. The combined organic phases were dried over anhydrous $MgSO_4$, and the solvent was removed under reduced pressure. Purification was performed by silica gel column chromatography using petroleum ether as eluent. Finally, the solvent was removed under reduced pressure, yielding compound 6 as a pale yellow liquid (0.65 g, 80% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.08 (d, J = 5.1 Hz, 1H), 7.01 (s, 1H), 6.85 (d, J = 5.1 Hz, 1H), 2.71 – 2.62 (m, 2H), 1.64 – 1.53 (m, 2H), 1.42 – 1.21 (m, 6H), 0.94 – 0.82 (m, 3H).

Synthesis of compound 7

Compound 6 (0.6 g, 3.1 mmol) was dissolved in 20 mL of anhydrous tetrahydrofuran (THF) in a 50 mL two-necked flask. Under a nitrogen atmosphere at -78°C, lithium diisopropylamide (LDA) (1.56 mL, 3.54 mmol) was added dropwise to the sample. The stirring mixture was kept at this temperature for 30 minutes before being cooled to -78°C again. Trimethyltin chloride (4.3 mL, 4.35 mmol) was then added to the reaction mixture, and stirring continued for 1 hour under these conditions. The mixture was then heated to room temperature and stirred overnight. Water was added to the mixture to stop the reaction, and the mixture was extracted twice with dichloromethane. The organic phase was dried over anhydrous $MgSO_4$, and the solvent was removed under reduced pressure. The crude product was obtained by ethanol recrystallization to yield compound 7. Vacuum drying produced a green solid (0.27 g, 65.3% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.00 (d, J = 1.6 Hz, 1H), 6.92 (s, 1H), 2.69 – 2.62 (m, 2H), 1.65 – 1.55 (m, 3H), 1.42 – 1.21 (m, 8H), 0.93 – 0.83 (m, 4H), 0.36 (s, 9H).

Synthesis of the copolymer of PY-TVTE

Compound 4 (0.025 mmol) and Y6-OD-Br (0.025 mmol) were separately dissolved in 4 mL of toluene. Then, a combination catalyst consisting of 5% bis(acetylacetonato)dibenzylidene palladium(0) and 50% triphenylphosphine was added. The mixture was purged with nitrogen for 10 minutes, and the reaction mixture was stirred at 130°C for 72 hours. The polymer was obtained when the cooled solution precipitated in methanol. Subsequently, sequential extraction with methanol and hexane was performed using a Soxhlet extractor. The final product was obtained by precipitating the chloroform solution in methanol (yield: 83%). M_n = 33.1K; polydispersity = 2.81.

Synthesis of the copolymer of PY-TVTH

Compound 7 (0.025 mmol) and Y6-OD-Br (0.025 mmol) were separately dissolved in 4 mL of toluene. Then, a combination catalyst consisting of 5% bis(benzylidene)acetone palladium(0) and 50% triphenylphosphine was added. The mixture was purged with nitrogen for 10 minutes, and the reaction mixture was stirred at 130°C for 72 hours. The polymer was obtained when the cooled solution precipitated in methanol. Subsequently, sequential extraction with methanol and hexane was performed using a Soxhlet extractor. The final product was obtained by precipitating the chloroform solution in methanol (yield: 71%). M_n = 24.3K; polydispersity = 2.50.

2. Characterization of materials

^{1}H NMR spectra were recorded on a Bruker AVANCE 300 MHz spectrometer using $CDCl_3$ as the solvent. The molecular weight of polymers was determined by gel permeation chromatography (GPC) relative to polystyrene standards with chloroform as the eluent. Thermal gravimetric analysis (TGA) was performed on a Perkin-Elmer Pyris 1 thermogravimetric analyzer. UV–vis absorption measurements were carried out on a Hitachi (model U-3010) UV–vis spectrophotometer. Cyclic voltammetric (CV) measurements were carried out in a conventional three-electrode cell using a platinum plate as the working electrode, a platinum wire as the counter electrode, and an Ag/Ag^+ electrode as the reference electrode on a Zahner IM6e Electrochemical workstation in a tetrabutylammonium hexafluorophosphate (Bu_4NPF_6) (0.1 M) acetonitrile solution at a scan rate of 20 mV s^{-1}.

3. Device Fabrication and Characterization

(1) Device Fabrication

A typical architecture was fabricated with ITO/PEDOT:PSS/active layer/ PNDIT-F3N/Ag. The ITO-coated glass substrates were sequentially ultrasonicated in soap water, deionized water, acetone, and isopropyl alcohol for at least 15 min, and ultimately dried in an oven overnight. The ITO-coated glass substrates were treated by uv-ozone for 10 min. Filter the PEDOT:PSS aqueous solution (Baytron P 4083, from HCStarck) through a 0.45 mm filter, and pre-coat it on the pre-cleaned ITO glass at 5000 rpm for 30 seconds, and then heat the ITO substrate in the air at 150°C annealing for 0.5 h. The PM6: acceptors (D:A=1:1, 10 mg mL^{-1} in total) was dissolved in chloroform, adding 1-chloronaphthalene (CN) (0.5%, v/v) as additive). The blended solution was spin-coated on the PEDOT:PSS layer at 3000 rpm for 30s. It is then annealed at 110°C for 10 minutes. Then PNDIT-F3N methanol solution with a concentration of 0.3 mg mL-1 was deposited on the active layer at a speed of 3000 rpm for 30 seconds to provide a PDINO cathode modification layer. After cooling to room temperature, the sample was transferred to the evaporation chamber. Under the pressure of 1×10^{-5} Pa, about 100 nm of Ag electrode was evaporated and deposited. The device area is 4.0 mm^2. The active area of the devices is 4.0 mm^2. Current density-voltage (*J*-*V*) characteristics were measured by a Keithley 2400 Source Measure Unit, in N_2 atmosphere under an AM 1.5G solar simulator with an irradiation light intensity of 100mw·cm^{-2}. The external quantum efficiency (EQE) of the devices was measured by using a QEX10 solar cell EQE measurement system (PV measurements.Inc.). The light intensity at each wavelength was calibrated with a standard single-crystal Si photovoltaic cell.

(2) Space-Charge-Limited Current (SCLC)

The current density–voltage (J–V) characteristics of the hole or electron only devices are fitted by the Mott–Gurney law:

$$J = (9/8)\varepsilon_r\varepsilon_0\mu(V^2/L^3)$$

where J is the current density, ε_r is the dielectric permittivity of the active layer, ε_0 is the vacuum permittivity, L is the thickness of the active layer, μ is the mobility. $V=V_{app} - V_{bi}$, where V_{app} is the applied voltage, V_{bi} is the offset voltage (V_{bi} is 0 V here). The mobility can be calculated from the slope of the $J^{0.5}\sim V$ curves.

(3) Grazing Incidence Wide-Angle X-ray Scattering (GIWAXS) Characterization

GIWAXS measurements were performed at beamline 7.3.3 at the Advanced Light Source. Samples were prepared on Si substrates using identical blend solutions as those used in devices. The 10 keV X-ray beam was incident at a grazing angle of 0.12°-0.16°, selected to maximize the scattering intensity from the samples. The scattered x-rays were detected using a Dectris Pilatus 2M photon counting detector.

(4) Atomic force microscopy (AFM) Characterization

AFM images were investigated on a Dimension Icon AFM (Bruker) in a tapping mode.

(5) Transmission Electron Microscopy (TEM) Characterization

TEM images were performed on a JEOL JEM-1400 transmission electron microscope. TEM samples were prepared as follows: First, the active layer was spin cast on the top of ITO/PEDOT:PSS substrates; Then, the active layer film was peeled off and floated onto the surface of deionized water; Finally, the floated films were picked up on a carbon film 200 mesh copper grid for TEM measurements.

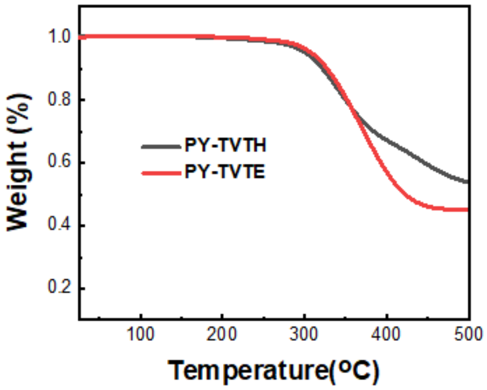


Figure S1. TGA plots of the polymer acceptors of **PY-TVTH** and **PY-TVTE** with a heating rate of 20 °C/min under the inert atmosphere.

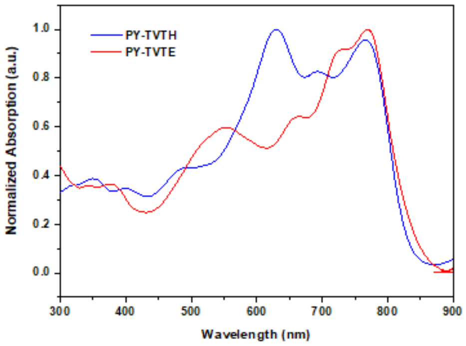


Figure S2. The normalized UV–vis absorption spectra of the polymer donor of **PM6** and the polymer acceptors of **PY-TVTH/PY-TVTE** in solutions.

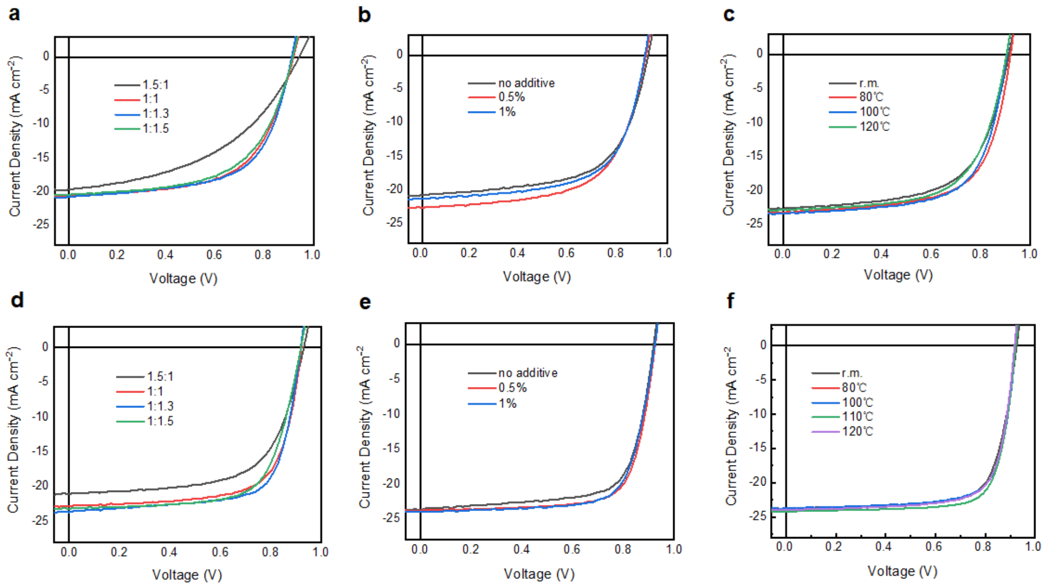


Figure S3. Photovoltaic performance of the all-PSCs based on **PM6: PY-TVTH** and **PM6: PY-TVTE** with different device fabrication conditions: (a), (d) donor:acceptor weight ratios; (b), (e) DIO additive; (c), (f) thermal annealing temperature.

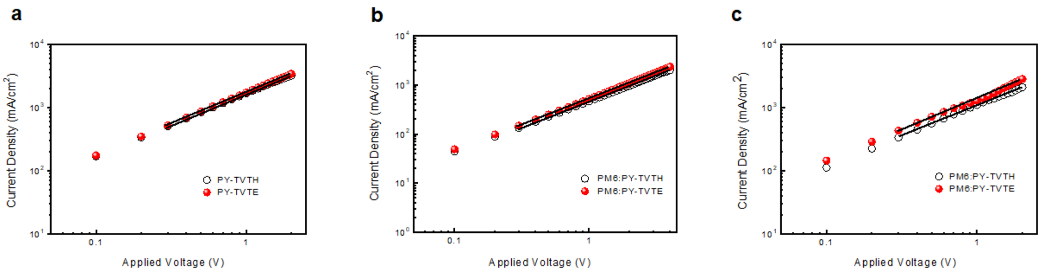


Figure S4. *J-V* characteristics of a) hole-only devices based on **PY-TVTH** and **PY-**

TVTE neat films, b) hole-only devices based on the **PM6: PY-TVTH** and **PM6: PY-TVTE** blend films, c) electron-only devices based on the **PM6: PY-TVTH** and **PM6: PY-TVTE** blend films.

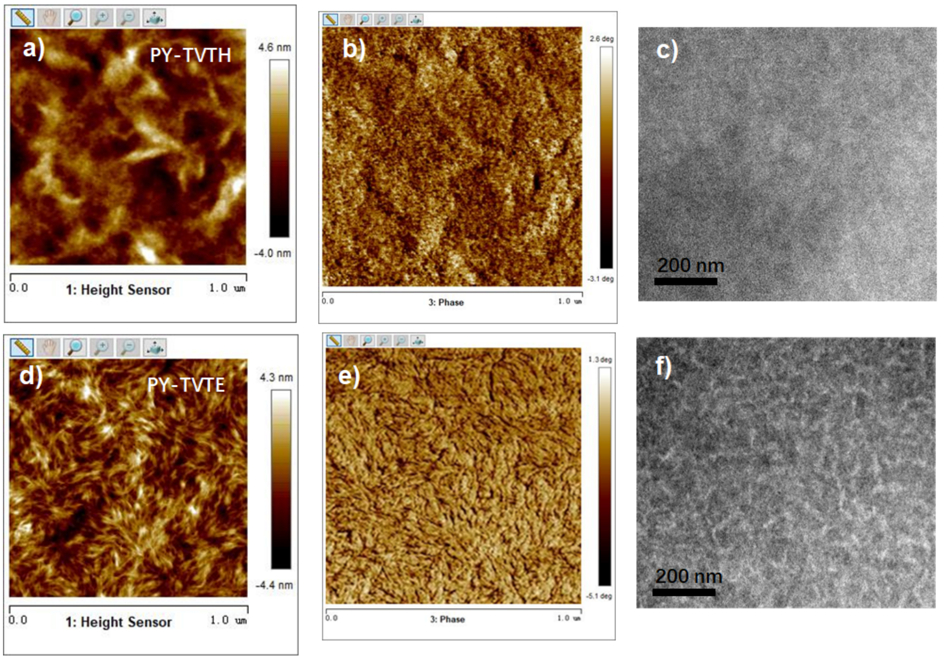


Figure S5. AFM of **PY-TVTH** neat film: a) height images, b) phase images, **PY-TVTE** neat film: d) height images, e) phase images, TEM images of c) **PY-TVTH** neat film, f) **PY-TVTE** neat film.

Table S1 Photovoltaic performance of the devices based on **PM6:PY-TVTH** with different weight ratios under the illumination of AM1.5G, 100 mW/cm^2.

PM6:PY-TVTH	V_{oc} (V)	J_{sc} (mA/cm^2)	FF (%)	PCE$_{max}$ (PCE$_{ave}$ %)
1.5:1	0.94	19.68	43.61	8.07 (8.04)
1:1	0.92	20.57	60.72	11.50 (11.44)
1:1.3	0.92	20.79	62.64	11.98 (11.93)
1:1.5	0.92	20.44	61.49	11.54 (11.49)

Table S2 Photovoltaic performance of the devices based on **PM6:PY-TVTH** with different CN under the illumination of AM1.5G, 100 mW/cm^2.

CN (%)	V_{oc} (V)	J_{sc} (mA/cm^2)	FF (%)	PCE$_{max}$ (PCE$_{ave}$ %)
0%	0.92	20.79	62.64	11.98 (11.93)
0.5%	0.91	22.68	63.03	13.04 (12.88)
1%	0.91	21.31	62.80	12.15 (12.02)

Table S3 Photovoltaic performance of the devices based on **PM6:PY-TVTH** with different temperature of thermal annealing under the illumination of AM1.5G, 100 mW/cm^2.

Temperature (°C)	V_{oc} (V)	J_{sc} (mA/cm^2)	FF (%)	PCE$_{max}$ (PCE$_{ave}$ %)
Room temp.	0.91	22.68	63.03	13.04 (12.88)
80°C	0.91	23.04	63.74	13.37 (13.28)
100°C	0.90	23.42	64.11	13.51 (13.49)
110°C	0.90	22.87	62.55	12.88 (12.76)

Table S4 Photovoltaic performance of the devices based on **PM6:PY-TVTE** with different weight ratios under the illumination of AM1.5G, 100 mW/cm^2.

PM6:PY-TVTH	V_{oc} (V)	J_{sc} (mA/cm^2)	FF (%)	PCE$_{max}$ (PCE$_{ave}$ %)
1.5:1	0.93	21.04	63.91	12.53 (12.50)
1:1	0.93	22.76	68.09	14.34 (14.29)
1:1.3	0.92	23.74	69.44	15.18 (15.04)
1:1.5	0.92	23.15	67.82	14.44 (14.26)

Table S5 Photovoltaic performance of the devices based on **PM6:PY-TVTE** with different CN under the illumination of AM1.5G, 100 mW/cm^2.

CN (%)	V_{oc} (V)	J_{sc} (mA/cm^2)	FF (%)	PCE$_{max}$ (PCE$_{ave}$ %)
0%	0.92	23.74	69.44	15.18 (15.04)
0.5%	0.92	23.84	73.23	16.06 (16.01)
1%	0.92	23.91	72.16	15.87 (15.78)

Table S6 Photovoltaic performance of the devices based on **PM6:PY-TVTE** with different temperature of thermal annealing under the illumination of AM1.5G, 100 mW/cm^2.

Temperature (°C)	V_{oc} (V)	J_{sc} (mA/cm^2)	FF (%)	PCE$_{max}$ (PCE$_{ave}$ %)
Room temp.	0.92	23.84	73.23	16.06 (16.01)
80°C	0.92	23.94	74.11	16.28 (16.23)
100°C	0.92	23.70	73.52	16.03 (15.85)
110°C	0.91	24.15	74.94	16.48 (16.42)
120°C	0.91	23.93	73.69	16.12 (15.97)

Table S7. The hole and electron mobility data and the ratio μ_h/μ_e of neat and blend films.

Polymers	μ_h (cm^2 V^{-1} s^{-1})	μ_e (cm^2 V^{-1} s^{-1})	μ_h/μ_e
PY-TVTH	/	1.84×10^{-3}	/
PY-TVTE	/	2.04×10^{-3}	/
PM6:PY-TVTH	1.03×10^{-3}	1.50×10^{-3}	0.69
PM6:PY-TVTE	1.37×10^{-3}	1.61×10^{-3}	0.85

Table S8. Contact angle of water and glycerol and surface tension of **PY-TVTH**, **PY-TVTE** and **PM6** films.

Neat film	θ_{water} [°]	$\theta_{glycerol}$ [°]	γ [mN m^{-1}]	χ
PY-TVTH	85.44	76.12	24.55	0.068
PY-TVTE	89.66	73.86	30.04	0.621
PM6	100.03	85.94	22.02	

Table S9 Photovoltaic performance of the devices based on **PM6:PY-TVTH** and **PM6:PY-TVTE**, respectively, with different active layer thickness under the illumination of AM1.5G, 100 mW/cm^2.

D/A blend [a)]	Active layer thickness (nm)	V_{oc} (V)	J_{sc} (mA/cm^2)	FF (%)	PCE (%)
PM6:PY-TVTH	90	0.91	23.40	63.22	13.47
	110	0.90	23.42	64.11	13.51
	130	0.90	23.46	62.13	13.10
	150	0.90	23.12	61.95	12.90
	250	0.90	22.65	61.06	12.45
	350	0.90	21.14	58.34	11.11
	400	0.90	20.37	55.65	10.21
PM6:PY-TVTE	90	0.92	23.76	73.39	16.01
	110	0.91	24.15	74.94	16.48
	130	0.91	24.44	73.60	16.37
	150	0.91	24.18	72.25	15.85
	250	0.90	23.05	68.87	14.33
	350	0.90	22.94	67.84	13.97
	400	0.90	22.67	65.02	13.24

a) Fabricated with 0.5% CN and annealing at 110 °C.

Table S10. PCE values based on reported All-PSCs and the photovoltaic performance based on the different film thickness.[1-8]

NO.	Donor:acceptor	Blend film Thickness	PCE (%)	Reference
1	JD40:PJTVT	120	16.13	[1]
2	JD40:PJTVT	210	14.72	[1]
3	JD40:PJTVT	250	14.00	[1]
4	JD40:PJTVT	300	13.43	[1]
5	JD40:PJTET	120	10.93	[1]
6	JD40:PJTET	175	8.22	[1]
7	JD40:PJTET	185	7.46	[1]
8	PtzBI-Si:N2200 (0% DBE)	210	9.3	[2]
9	PtzBI-Si:N2200 (5% DBE)	210	8.9	[2]
10	PtzBI-Si:N2200 (10% DBE)	210	8.4	[2]
11	PtzBI-Si:N2200 (10% DBE)	370	8.5	[2]
12	PtzBI-Si:N2200 (10% DBE)	520	9.0	[2]
13	PBDB-T:PFY-3Se	110	15.1	[3]
14	PBDB-T:PFY-3Se	185	13.5	[3]
15	PBDB-T:PFY-3Se	210	12.9	[3]
16	PBDB-T:PFY-3Se	250	12.4	[3]
17	PBTA-Si:N2200	165	8.31	[4]
18	PBTA-Si:N2200	310	8.32	[4]
19	PBTA-Si:N2200	420	7.00	[4]
20	PtzBI-Si:N2200	140	9.82	[4]
21	PtzBI-Si:N2200	300	8.19	[4]
22	PtzBI-Si:N2200	430	7.34	[4]
23	PBTA-Si:PtzBI-Si:N2200(1.4:0.6:1)	155	9.36	[4]
24	PBTA-Si:PtzBI-Si:N2200(1.2:0.8:1)	170	9.50	[4]
25	PBTA-Si:PtzBI-Si:N2200(0.8:1.2:1)	120	9.07	[4]
26	PBTA-Si:PtzBI-Si:N2200(0.8:1.2:1)	150	9.56	[4]
27	PBTA-Si:PtzBI-Si:N2200(1:1:1)	270	8.81	[4]
28	PBTA-Si:PtzBI-Si:N2200(1:1:1)	350	9.17	[4]
29	PBTA-Si:PtzBI-Si:N2200(1:1:1)	420	8.34	[4]
30	PM6:PTQ10:PY-IT	138	16.52	[5]
31	PM6:PTQ10:PY-IT	205	15.27	[5]
32	PM6:PTQ10:PY-IT	306	13.91	[5]
33	PBDB-T:PJ1	305	12.1	[6]
34	PM6:L19	120	17.97	[7]
35	PM6:L19	150	17.83	[7]
36	PM6:L19	200	16.68	[7]
37	PM6:L19	300	15.50	[7]
38	PM6:PYF-T-o	75	16.1	[8]
39	PM6:PYF-T-o	185	14.2	[8]
40	PM6:PYF-T-o	310	14.2	[8]
42	PM6:PY-TVTH	250	12.45	This work

43	PM6:PY-TVTH	350	11.11	This work
44	PM6:PY-TVTH	400	10.21	This work
46	PM6:PY-TVTE	250	14.33	This work
47	PM6:PY-TVTE	350	13.97	This work
48	PM6:PY-TVTE	400	13.24	This work

Table S11 Photovoltaic performance of the devices based on **PDBT-T1:PY-TVTE**, respectively, with different active layer thickness under the illumination of AM1.5G, 100 mW/cm^2.

D/A blend	Active layer thickness (nm)	V_{oc} (V)	J_{sc} (mA/cm^2)	FF (%)	PCE (%)
PDBT-T1:PY-TVTE	110	0.92	19.47	73.9	13.24
	250	0.91	20.41	75.2	14.02
	350	0.91	19.98	71.8	13.05

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