Inner Side-Chain Engineering of Non-Fullerene Acceptors Enables Efficient Thick-Film All-Polymer Solar Cells

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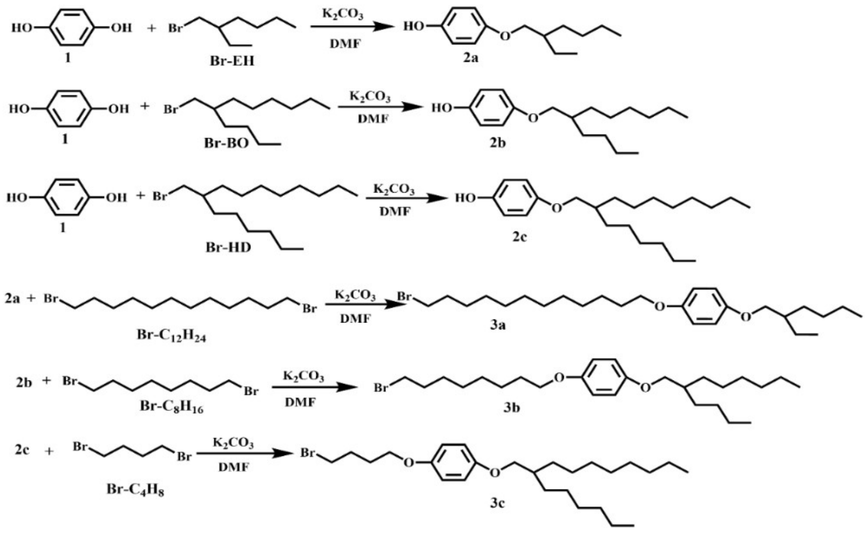
1.Materials and synthesis

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 compounds **3a-3c**.

Synthesis of compound 2a

Hydroquinone (compound **1**) (5 g, 0.055 mmol) and potassium carbonate (18.82 g, 0.165 mmol) were added to a 250 mL two-necked flask, then 120 mL of N, N-dimethylformamide (DMF) was added, nitrogen was pumped and vented 25 times, and the reaction was stirred in an oil bath at 50 °C for 1 h. Brominated isooctane (compound Br-EH) (10.62 g, 0.055 mmol) was added to the flask at this temperature to the flask and then warmed to 85 °C with stirring overnight. The mixture was filtered to remove potassium carbonate and concentrated under reduced pressure to remove a large amount of solvent, then the concentrate was poured into 200 mL of water and extracted with dichloromethane (DCM), then dried and concentrated by rotary evaporation, and the crude product was purified by separating the impurities from the by-products on a silica gel column using dichloromethane and petroleum ether as the eluents (v/v, 1:1) to give compound **2a** (4.71 g) as a colorless liquid. ^{1}H NMR (400MHz, $CDCl_3$) δ 6 .80-6.73 (m, 4H), 3.80-3.75 (m, 2H), 1.50-1.40 (m, 1H), 1.39-1.24 (m, 4H), 0.91 (q, J=7.3, 6.9 Hz, 11H).

Synthesis of compound 2b

To a 250 mL double-necked flask was added Compound **1** (5.0 g, 0.055 mmol), potassium carbonate (18.82 g, 0.165 mmol) and N, N-dimethylformamide (120 mL), and stirred in an oil bath under nitrogen atmosphere at 50 °C for 1 h. The flask was charged with Compound Br-BO (2-butyl-1-bromooctane) (13.70g, 0.055 mmol), and the temperature was raised to 85 °C with stirring overnight. The reaction mixture was filtered, and then concentrated under reduced pressure to remove a large amount of solvent, the concentrate was poured into 200 mL of water and washed and extracted three times, the combined organic phases were dried and filtered and concentrated, and then purified by silica gel column with dichloromethane and petroleum ether as eluents (v/v, 1:1) to obtain Compound **2b** (3.97 g), which was a colorless liquid.^{1}H NMR (400 MHz, $CDCl_3$) δ 6.84-6.69 (m, 4H), 3.77 (d, J=5.7 Hz, 2H), 1.73 (h, J=5.9 Hz, 1H), 1.47-1.17 (m, 17H), 0.89 (q, J=6.7 Hz, 6H).

Synthesis of compound 2c

Under nitrogen atmosphere, a 250 mL double-necked flask with hydroquinone (5.0 g, 0.055 mmol), potassium carbonate (18.82 g, 0.165 mmol) and DMF (120 mL) were placed in an oil bath and stirred at 50 °C for 1 h. The temperature was maintained at which time the bromoalkanes Br-HD, i.e., 1-bromo-2-hexyldecane (16.78 g, 0.055 mmol) were added to the reaction flask, and the temperature of the oil bath was elevated to 90 °C, and the reaction was carried out overnight. After completion of the reaction, the mixture was withdrawn and filtered to collect the filtrate concentrated under reduced pressure and then washed and extracted three times, the combined organic phases were dried, filtered and concentrated, and the concentrated crude product was separated and purified by silica gel column using dichloromethane/petroleum ether as eluent (v/v,1:1) to obtain the final product compound **2c** (4.78 g). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.77 (q, J= 9.1 Hz, 4H), 4.55 (s, 1H), 3.76 (d, J=5.8 Hz, 2H), 1.78-1.60 (m, 2H), 1.29 (dd, J=14.4, 7.3 Hz, 23H), 1.01-0.82 (m, 6H).

Synthesis of compound 3a

Compound **2a** (3.62 g, 16.3 mmol) and potassium carbonate (6.76 g, 49 mmol) were added to a 250 mL two-necked flask, then 80 mL DMF was added, and the air in the flask was evacuated by nitrogen pumping and venting for 25 times, and then stirred at room temperature for 1 h. To the flask was added 1,12-dibromododecane (Compound Br-$C_{12}H_{24}$) (4.23 g, 17.0 mmol) under room temperature, and the flask was stirred at room temperature for 30 min and then the reaction was stirred overnight at 90 °C in an oil bath. The mixture was filtered and concentrated under reduced pressure to remove the solvent and then washed and extracted three times, the extract was dried and filtered and then evaporated to obtain the concentrated crude product, which was purified by silica gel column using dichloromethane and petroleum ether as eluents (v/v, 1:5) to obtain compound **3a** (2.58g). ^{1}H NMR (600 MHz, $CDCl_3$) δ 6.82 (s, 4H), 3.90 (t, J=6.6 Hz, 2H), 3.82-3.72 (m, 2H), 3.41 (t, J=6.9 Hz, 2H), 1.89-1.65 (m, 5H), 1.47-1.22 (m, 24H), 0.95-0.85 (m, 6H).

Synthesis of compound 3b

A 250 mL double-necked vial with compound **2b** (3.78 g, 13.6 mmol), potassium carbonate (5.66 g, 41 mmol) and DMF (80 mL) was stirred at room temperature under nitrogen for 1 h. To the reaction vial was added 1,8-dibromooctane (compound Br-C8H16) (2.91 g, 15.1 mmol), stirred at room temperature for 0.5 h, and then the temperature was raised to 60 °C with stirring in an oil bath, and the reaction was carried out overnight. The mixture was withdrawn and filtered to collect the filtrate and concentrated under reduced pressure and then washed and extracted three times, the combined organic phases were dried and filtered and concentrated, and the concentrated crude product was separated and purified by a silica gel column using petroleum ether/dichloromethane as the eluent (v/v, 5:1) to give the final product compound **3b** (2.04 g). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.82 (s, 4H), 3.90 (t, J=6.5 Hz, 2H), 3.76 (s, 2H), 3.41 (t, J=6.8 Hz, 2H), 1.86 (p, J=6.9 Hz, 5H), 1.47-1.25 (m, 24H), 0.89 (q, J=6.8 Hz, 6H).

Synthesis of compound 3c

To a 250 mL double-necked flask was added compound **2c** (4.12 g, 12.3 mmol), potassium carbonate (5.11g, 37.0 mmol) and DMF (90 mL), stirred at room temperature for 1 h after nitrogen pumping and venting, stirred at 50 °C in an oil bath for 1 h, and the flask was charged with compound Br-C4H8 (1,4-dibromobutane) (3.43 g, 25.0 mmol), stirred at room temperature for 30 min, then warmed to 60 °C and stirred overnight. The reaction mixture was filtrated, then concentrated under reduced pressure to remove a large amount of solvent, and the organic phase obtained by washing and extracting the concentrate with water was dried with anhydrous magnesium sulfate, and then concentrated under reduced pressure to obtain the crude product, which was purified by a silica gel column to give compound **3c** (2.96 g). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.82 (s, 4H), 3.94 (d, J=6.0 Hz, 2H), 3.77 (d, J=5.7 Hz, 2H), 3.49 (t, J=6.7 Hz, 2H), 2.12-1.98 (m, 2H), 1.92 (dt, J=8.8, 6.0 Hz, 2H), 1.49-1.17 (m,25H), 0.88 (t, J=6.5 Hz, 6H).

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. The blended solution was spin-coated on the PEDOT:PSS layer at 3000 rpm for 30s. It is then annealed at 100°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 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 $J^{0.5}$~V curves.

(3) Atomic Force Microscopy (AFM) Characterization

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

(4) 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.

(5) Contact Angles Calculation:

The contact angles of two different solvent (water and Glycerol) on neat films of polymer donor PM6, three polymer acceptors PY-EH, PY-BO and PY-HD were used to calculate the surface tension by Wu model. The detailed calculation process is described below:

$$\gamma_{Water}(\cos\theta + 1) = \frac{4\gamma_{Water}^{d}\gamma^{d}}{\gamma_{Water}^{d} + \gamma^{d}} + \frac{4\gamma_{Water}^{p}\gamma^{p}}{\gamma_{Water}^{p} + \gamma^{p}}$$

$$\gamma_{Glycerol}(\cos\theta + 1) = \frac{4\gamma_{Glycerol}^{d}\gamma^{d}}{\gamma_{Glycerol}^{d} + \gamma^{d}} + \frac{4\gamma_{Glycerol}^{p}\gamma^{p}}{\gamma_{Glycerol}^{p} + \gamma^{p}}$$

$$\gamma = \gamma^{d} + \gamma^{p}$$

Where is the contact angle of films and γ is the surface tension of the films which is the sum of dispersion (γ_d) and polarity (γ_p) components. The γ_{Water} and γ_{Glycerol} are the surface tensions of the water and glycerol. The Compatibility between different materials can be deduced from the empirical formula of Flory-Huggins parameter.

$$\chi \propto \left(\sqrt{\gamma_A} - \sqrt{\gamma_B}\right)^2$$

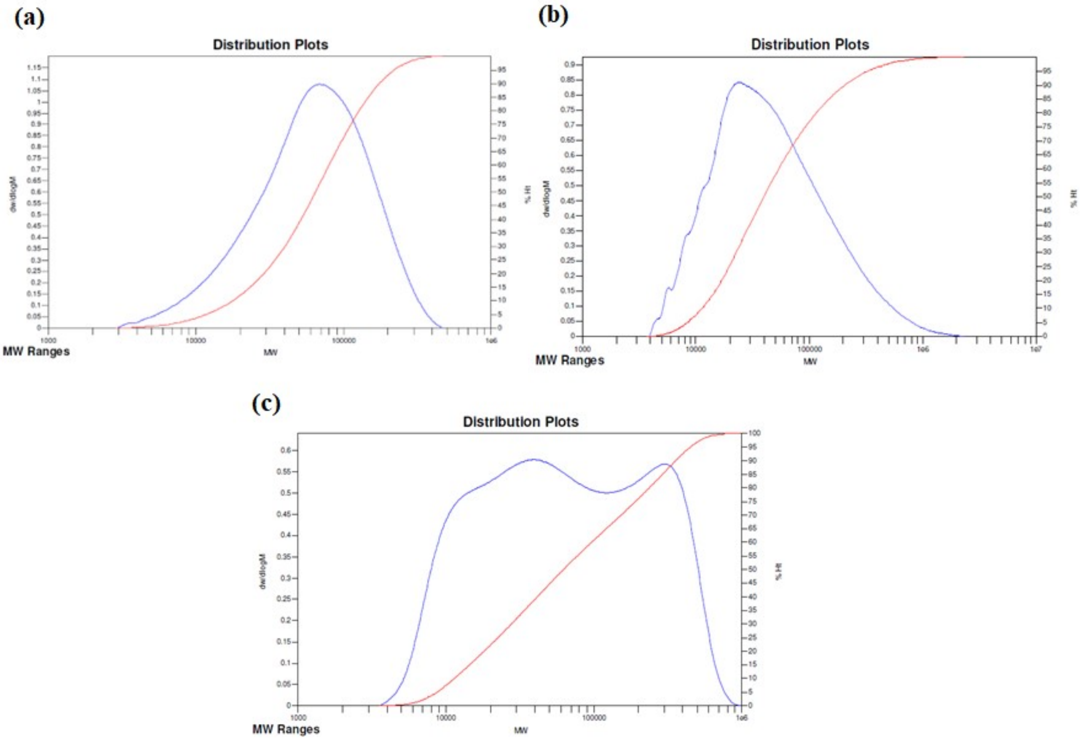


Figure S1. GPC plots of the polymer acceptors of (a) **PY-EH,** (b) **PY-BO** and (c) **PY-HD**.

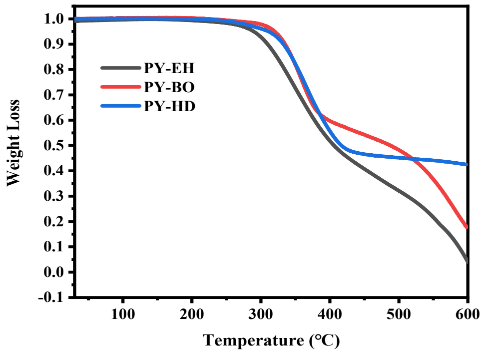


Figure S2. TGA plots of the polymer acceptors of **PY-EH, PY-BO** and **PY-HD** with a heating rate of 20 °C/min under the inert atmosphere.

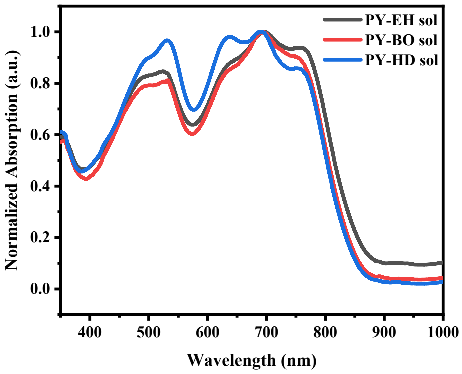


Figure S3. The normalized UV–vis absorption spectra of the polymer acceptors of **PY-EH, PY-BO** and **PY-HD** in diluted chloroform solutions.

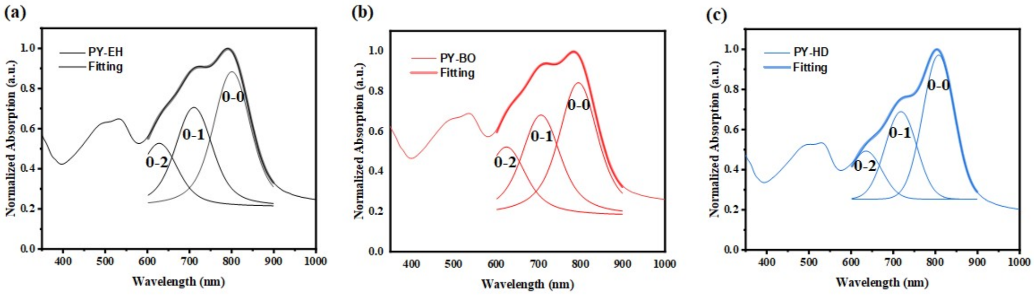


Figure S4. Theoretical absorption spectra with Gaussian curves for these three polymer acceptors.

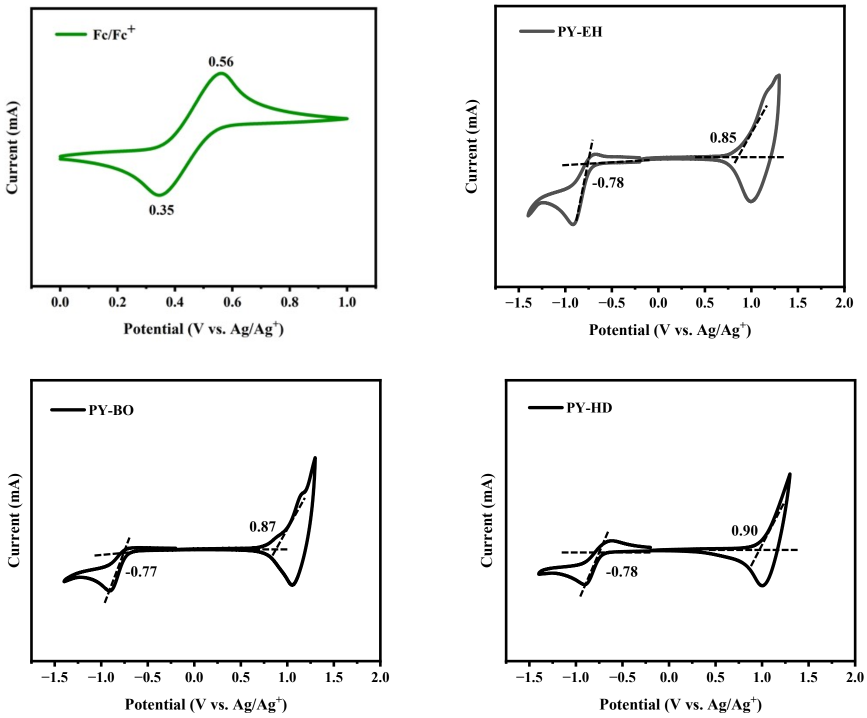


Figure S5. The electrochemical CV curves of the polymer acceptors of **PY-EH, PY-**

BO and **PY-HD** in Bu_4NPF_6 (0.1 M) acetonitrile solution at a scan rate of 20 mV s^{-1}.

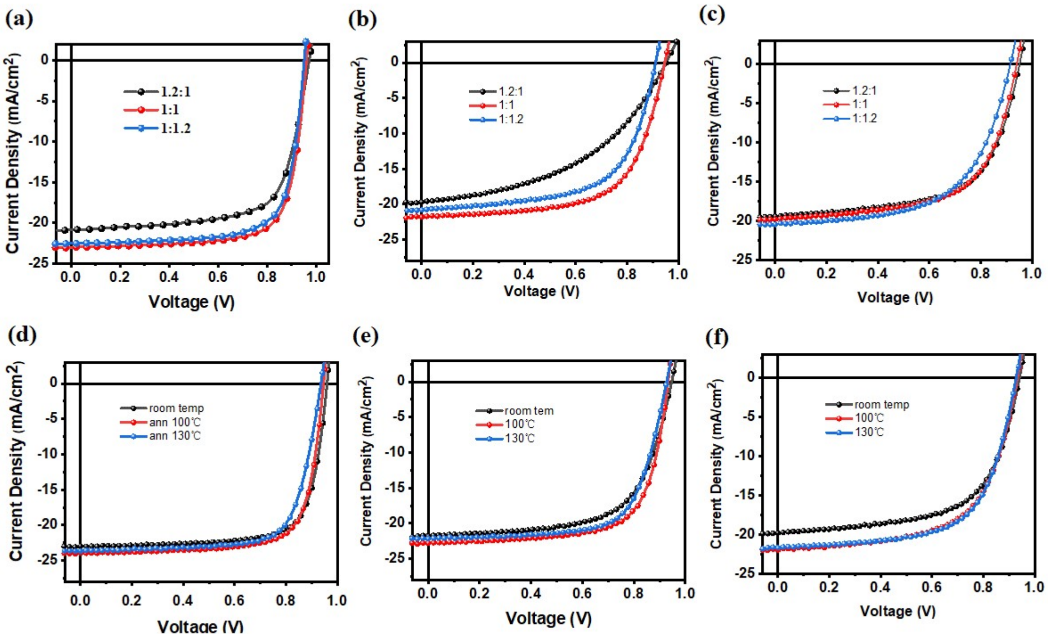


Figure S6. Photovoltaic performance of the all-PSCs based on **PM6: PY-EH**, **PM6: PY-BO** and **PM6: PY-HD** with different device fabrication conditions: (a-c) donor:acceptor weight ratios; (d-f) thermal annealing temperatures.

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

PM6:PY-EH	V_{oc} (V)	J_{sc} (mA/cm^2)	FF (%)	PCE$_{max}$ (PCE$_{ave}$ %)
1.2:1	0.97	21.33	68.71	14.22 (13.79)
1:1	0.96	22.97	72.23	15.92 (15.59)
1:1.2	0.95	22.48	71.14	15.23 (15.06)

Table S2 Photovoltaic performance of the devices based on **PM6:PY-EH** 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.	0.96	22.97	72.23	15.92 (15.59)
100°C	0.95	23.89	72.50	16.38 (16.17)
130°C	0.93	23.61	70.82	15.60 (15.44)

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

PM6:PY-BO	V_{oc} (V)	J_{sc} (mA/cm^2)	FF (%)	PCE$_{max}$ (PCE$_{ave}$ %)
1.2:1	0.95	19.67	43.42	8.11 (7.65)
1:1	0.95	21.78	62.09	12.81 (12.50)
1:1.2	0.92	20.83	62.74	12.02 (11.67)

Table S4 Photovoltaic performance of the devices based on **PM6:PY-BO** 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.	0.95	21.78	62.09	12.81 (12.36)
100°C	0.94	22.71	69.34	14.72 (14.49)
130°C	0.93	22.23	66.82	13.77 (13.47)

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

PM6:PY-HD	V_{oc} (V)	J_{sc} (mA/cm^2)	FF (%)	PCE$_{max}$ (PCE$_{ave}$ %)
1.2:1	0.95	19.42	62.38	11.51 (11.27)
1:1	0.94	19.79	63.01	11.71 (11.43)
1:1.2	0.92	20.35	61.14	11.46 (11.18)

Table S6 Photovoltaic performance of the devices based on **PM6:PY-HD** 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.	0.94	19.79	63.01	11.71 (11.43)
100°C	0.93	21.95	64.12	13.09 (12.88)
130°C	0.93	21.68	63.88	12.85 (12.50)

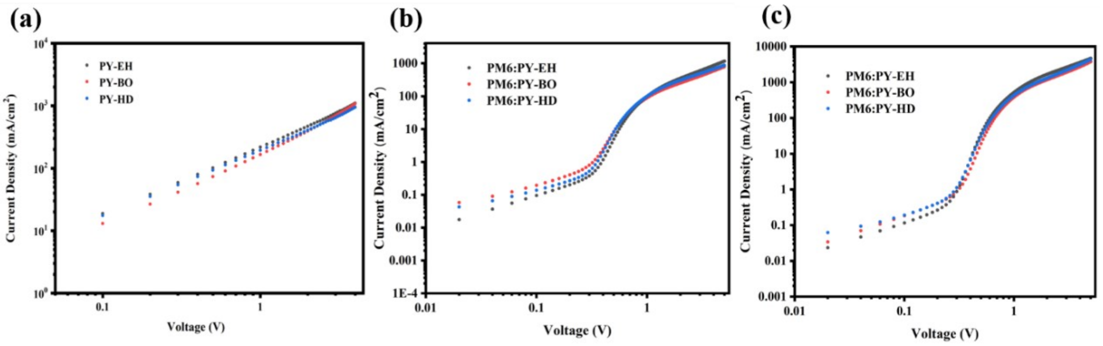


Figure S7. J-V characteristics of a) electron-only devices based on **PY-EH**, **PY-BO** and **PY-HD** neat films, b) hole-only devices based on the **PM6: PY-EH**, **PM6: PY-BO** and **PM6: PY-HD** blend films, c) electron-only devices based on the **PM6: PY-**

EH, **PM6: PY-BO** and **PM6: PY-HD** blend films.

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-EH	/	2.47×10^{-3}	/
PY-BO	/	2.03×10^{-3}	/
PY-HD	/	2.11×10^{-3}	/
PM6:PY-EH	1.86×10^{-3}	2.04×10^{-3}	0.91
PM6:PY-BO	1.42×10^{-3}	1.88×10^{-3}	0.76
PM6:PY-HD	1.50×10^{-3}	1.97×10^{-3}	0.76

Table S8 The parameters of **PM6:PY-EH**, **PM6:PY-BO** and **PM6:PY-HD** based all-PSCs.

Active laymer	$J_{ph,sc}$ (mA/cm^2)	J_{sat} (mA/cm^2)	P_{diss} (%)
PM6:PY-EH	23.81	24.35	97.8%
PM6:PY-BO	22.45	23.29	96.4%
PM6:PY-HD	21.38	22.67	94.3%

Table S9. Contact angle of water and glycerol and surface tension of **PY-EH**, **PY-BO**, **PY-HD** and **PM6** films.

Neat film	θ_{water} [°]	$\theta_{\mathrm{glycerol}}$ [°]	γ [mN m^{-1}]	χ
PY-EH	94.8	79.5	26.55	0.00342
PY-BO	96.0	81.3	24.99	0.00904
PY-HD	97.4	82.9	23.97	0.0392
PM6	99.7	83.3	25.95	

Table S10. PCE values based on reported All-PSCs and the photovoltaic performance based on the different film thickness.[1-9]

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]
41	PM6:PY-TVTH	250	12.45	[9]
42	PM6:PY-TVTH	350	11.11	[9]
43	PM6:PY-TVTH	400	10.21	[9]
44	PM6:PY-TVTE	250	14.33	[9]

45	PM6:PY-TVTE	350	13.97	[9]
46	PM6:PY-TVTE	400	13.24	[9]
47	PM6:PY-EH	250	15.76	this work
48	PM6:PY-EH	410	14.24	this work
49	PM6:PY-BO	250	13.81	this work
50	PM6:PY-BO	410	12.38	this work
51	PM6:PY-HD	250	11.72	this work
52	PM6:PY-HD	410	10.20	this work

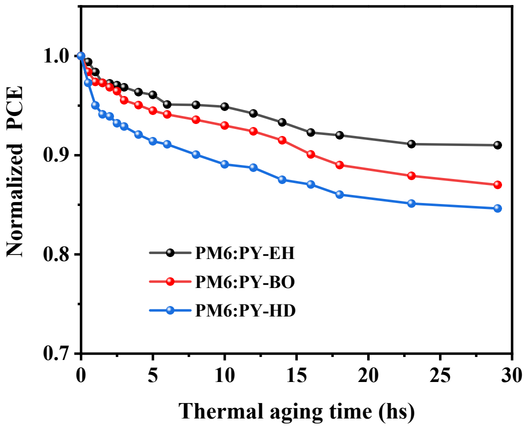


Figure S8. The thermal stability for these polymer acceptors based photovoltaic devices, which are heated at 110°C. All devices were encapsulated in a glovebox prior to testing.

Reference

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