

Electronic Supporting Information

Enhancing Thermoelectric Performance of Donor-Acceptor Conjugated Polymers through Dopant Miscibility: A Comparative Study of Fluorinated Substituents and Side Chain Lengths

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S.1 Materials

All necessary reagents and solvents were obtained commercially and used without further purification. Tributyl(4-(2-butyloctyl)thiophen-2-yl)stannane,^[1] tributyl(4-(2-hexyldecyl)thiophen-2-yl)stannane,^[1] tributyl(4-(2-octyldodecyl)thiophen-2-yl)stannane,^[1] 4,7-dibromobenzo[c][1,2,5]thiadiazole,^[2] 2,5-bis(trimethylstannyl)thiophene,^[3] and (3,4-difluorothiophene-2,5-diyl)bis(trimethylstannane)^[4] were prepared following established procedures as described in the literature.

S.2 Monomer synthesis

S.2.1 Synthesis of 4,7-bis(4-(2-butyloctyl)thiophen-2-yl)benzo[c][1,2,5]thiadiazole (1a)

A chemical reaction was conducted utilizing 5.8 g (10.0 mmol) of tributyl(4-(2-butyloctyl)thiophen-2-yl)stannane and 1.04 g (3.5 mmol) of 4,7-dibromobenzo[c][1,2,5]thiadiazole. This reaction was executed within a microwave reaction vessel and featured the inclusion of Pd(PPh₃)₄ (10 mg) as the catalyst, with toluene (8 mL) serving as the solvent. The reaction mixture underwent heating to 200°C over a duration of 2 hours. Upon the completion of the reaction, the mixture was transferred into a 250 mL round-bottom flask, and the solvent was eliminated via rotary evaporation. Subsequently, the resultant product was subjected to purification through column chromatography, utilizing hexane as the mobile phase. This procedure culminated in the formation of a red, viscous gel, yielding a final mass of 3.24 g, corresponding to a 47.5% yield. ¹H NMR (400 MHz, CDCl₃): δ 7.97-7.96 (d, *J* = 1.2 Hz, 2H), 7.85 (s, 2H), 7.03 (s, 2H), 2.66-2.64 (d, *J* = 6.8 Hz, 4H), 1.74-1.73 (m, 2H), 1.34-1.30 (m, 32H), 0.94-0.90 (m, 12H). ¹³C NMR (100 MHz, CDCl₃): δ 152.5, 142.9, 138.7, 129.4, 125.8, 125.3, 122.3, 38.7, 34.9, 33.2, 32.9, 31.8, 29.6, 28.8, 26.5, 23.0, 22.6, 14.1, 14.0.

S.2.2 Synthesis of 4,7-bis(4-(2-hexyldecyl)thiophen-2-yl)benzo[c][1,2,5]thiadiazole (1b)

A chemical reaction was conducted, starting with 5.42 g (9.0 mmol) of Tributyl(4-(2-hexyldecyl)thiophen-2-yl)stannane and 0.88 g (3.0 mmol) of 4,7-dibromobenzo[c][1,2,5]thiadiazole. This reaction took place within a microwave reaction vessel, utilizing Pd(PPh₃)₄ (10 mg) as the catalyst and toluene (8 mL) as the solvent. The reaction mixture was subjected to heating at 200°C for a duration of 2 hr. Upon completion

of the reaction, the mixture was transferred to a 250 mL round-bottom flask, and the solvent was eliminated through rotary evaporation. Subsequently, the resulting product underwent purification via column chromatography, employing hexane as the mobile phase. This process resulted in the production of a red, viscous gel with a mass of 3.07 g, representing a yield of 45.6%. ^1H NMR (400 MHz, CDCl_3): δ 7.97-7.96 (d, $J = 1.2$ Hz, 2H), 7.85 (s, 2H), 7.03 (d, $J = 1.2$ Hz, 2H), 2.66-2.64 (d, $J = 6.8$ Hz, 4H), 1.73-1.72 (m, 2H), 1.33-1.28 (m, 48H), 0.91-0.87 (m, 12H). ^{13}C NMR (100 MHz, CDCl_3): δ 152.5, 142.9, 138.7, 129.4, 125.8, 125.3, 122.3, 38.8, 34.9, 33.2, 31.8, 29.9, 29.6, 29.5, 29.3, 26.5, 22.6, 14.0.

S.2.3 Synthesis of 4,7-bis(4-(2-octyldodecyl)thiophen-2-yl)benzo[c][1,2,5]thiadiazole (1c)

A chemical reaction was carried out, employing 3.13 g (4.8 mmol) of Tributyl(4-(2-octyldodecyl)thiophen-2-yl)stannane and 0.47 g (1.6 mmol) of 4,7-dibromobenzo[c][1,2,5]thiadiazole. This reaction was conducted within a microwave reaction vessel and included $\text{Pd}(\text{PPh}_3)_4$ (10 mg) as the catalyst, with toluene (8 mL) serving as the solvent. The reaction mixture was heated to 200°C and maintained at this temperature for a duration of 2 hours. Following the completion of the reaction, the mixture was transferred into a 250 mL round-bottom flask, and the solvent was removed through rotary evaporation. Subsequently, the resulting product underwent purification via column chromatography, utilizing hexane as the mobile phase. This process resulted in the production of a red, viscous gel, yielding 1.79 g, which represents a 43.5% yield. ^1H NMR (400 MHz, CDCl_3): δ 7.97-7.96 (d, $J = 1.6$ Hz, 2H), 7.85 (s, 2H), 7.03 (s, 2H), 2.66-2.64 (d, $J = 6.8$ Hz, 4H), 1.73-1.72 (d, 2H), 1.33-1.27 (m, 64H), 0.91-0.87 (m, 12H). ^{13}C NMR (100 MHz, CDCl_3): δ 152.5, 142.9, 138.7, 129.4, 125.8, 125.3, 122.3, 38.8, 35.0, 33.2, 31.8, 29.9, 29.6, 29.6, 29.3, 26.5, 22.6, 14.0.

S.2.4 Synthesis of 4,7-bis(5-bromo-4-(2-butyloctyl)thiophen-2-yl)benzo[c][1,2,5]thiadiazole (M1a)

In a 100 mL round-bottom flask, 1.08 g (1.7 mmol) of 4,7-bis(4-(2-butyloctyl)thiophen-2-yl)benzo[c][1,2,5]thiadiazole was combined with 50 mL of chloroform (CHCl_3). The mixture was stirred until a homogeneous solution was achieved. Subsequently, N-Bromosuccinimide (NBS) (0.60 g, 3.4 mmol) was gradually added to the flask. The reaction vessel was sealed with a rubber stopper, and the reaction proceeded overnight. Upon completion, the reaction was quenched by the addition of deionized water. The resulting mixture underwent extraction

using a combination of chloroform and deionized water. The organic layer was separated and dried with anhydrous magnesium sulfate to remove residual water content. The solvent was then removed via rotary evaporation. The resulting product was subjected to purification through column chromatography, employing hexane as the mobile phase. This process resulted in the isolation of a red, viscous gel with a yield of 0.68 g, corresponding to a 50.5% yield. ^1H NMR (400 MHz, CDCl_3): δ 7.75-7.74 (d, J = 3.2 Hz, 2H), 2.60-2.58 (d, J = 1.2 Hz, 4H), 1.77 (s, 2H), 1.57-1.30 (m, 32H), 1.00-0.91 (m, 12H). ^{13}C NMR (100 MHz, CDCl_3): δ 152.1, 142.1, 138.1, 128.5, 125.2, 124.7, 112.1, 38.4, 34.1, 33.2, 32.9, 31.8, 29.6, 28.6, 26.4, 22.9, 22.6, 14.0.

S.2.5 Synthesis of 4,7-bis(5-bromo-4-(2-hexyldecyl)thiophen-2-yl)benzo[c][1,2,5]thiadiazole (M1b)

In a 100 mL round-bottom flask, 0.89 g (1.2 mmol) of 4,7-bis(4-(2-hexyldecyl)thiophen-2-yl)benzo[c][1,2,5]thiadiazole was combined with 50 mL of chloroform (CHCl_3). The mixture was stirred until it formed a homogeneous solution. Subsequently, NBS (0.42 g, 2.4 mmol) was slowly added to the flask. The reaction vessel was sealed with a rubber stopper and allowed to proceed overnight. Once the reaction reached completion, it was quenched by the addition of deionized water. The resulting mixture underwent extraction using a mixture of chloroform and deionized water. The organic layer was separated and dried using anhydrous magnesium sulfate to remove any residual water content. The solvent was then removed via rotary evaporation. The resulting product was purified through column chromatography, with hexane as the mobile phase. This process yielded a red, viscous gel with a mass of 0.54 g, representing a 60.4% yield. ^1H NMR (400 MHz, CDCl_3): δ 7.78-7.76 (d, J = 1.6 Hz, 2H), 2.60-2.58 (d, J = 7.2 Hz, 4H), 1.77 (s, 2H), 1.34-1.28 (m, 48H), 0.89-0.86 (m, 12H). ^{13}C NMR (100 MHz, CDCl_3): δ 152.1, 142.1, 138.1, 128.5, 125.2, 124.7, 112.1, 38.4, 34.1, 33.2, 31.8, 29.9, 29.6, 29.5, 29.2, 26.4, 22.5, 14.0.

S.2.6 Synthesis of 4,7-bis(5-bromo-4-(2-octyldodecyl)thiophen-2-yl)benzo[c][1,2,5]thiadiazole (M1c)

In a 100 mL round-bottom flask, 1.55 g (1.8 mmol) of 4,7-bis(4-(2-octyldodecyl)thiophen-2-yl)benzo[c][1,2,5]thiadiazole was combined with 50 mL of chloroform (CHCl_3). The mixture was stirred until it formed a homogeneous solution. Subsequently, NBS (0.64 g, 3.6 mmol) was slowly added to the flask. The reaction vessel was sealed with a rubber stopper and

allowed to proceed overnight. Once the reaction reached completion, it was quenched by adding deionized water. The resulting mixture underwent extraction using a mixture of chloroform and deionized water. The organic layer was separated and dried using anhydrous magnesium sulfate to eliminate any residual water content. The solvent was then removed via rotary evaporation. The resulting product was purified through column chromatography, employing hexane as the mobile phase. This process yielded a red, viscous gel with a mass of 0.98 g, corresponding to a yield of 53.8%. ^1H NMR (400 MHz, CDCl_3): δ 7.77-7.76 (d, $J = 4$ Hz, 2H), 2.60-2.58 (d, $J = 7.2$ Hz, 4H), 1.77 (s, 2H), 1.58-1.26 (m, 64H), 0.88-0.86 (m, 12H). ^{13}C NMR (100 MHz, CDCl_3): δ 152.3, 142.2, 138.3, 128.7, 125.3, 124.8, 112.2, 38.5, 34.3, 33.4, 31.9, 30.0, 29.7, 29.6, 29.6, 29.3, 26.5, 22.6, 14.1.

S.3 Polymer Synthesis

S.3.1 Synthesis of PC12BTH

In a 10 ml microwave reaction vial, monomer 4,7-bis(5-bromo-4-(2-butyloctyl)thiophen-2-yl)benzo[c][1,2,5]thiadiazole (95.38 mg, 0.12 mmol) and monomer 2,5-bis(trimethylstannyl)thiophene (49.17 mg, 0.12 mmol) were combined along with $\text{Pd}(\text{PPh}_3)_4$ catalyst (11.55 mg, 0.01 mmol). To this mixture, 4.3 ml of anhydrous toluene and 0.43 ml of DMF were added as the solvent, resulting in a solution with a concentration of 0.025 M. The reaction mixture was sealed using a PTFE cap and placed inside a microwave reactor, operating at 150°C for a duration of 3.5 hours. After the reaction was completed, the resulting conjugated polymer was slowly dripped into methanol for reprecipitation. The solid product was then collected through filtration using a fritted funnel. The obtained conjugated polymer PC12BTH underwent purification using a Soxhlet extractor, successively employing methanol, acetone, hexane, dichloromethane (DCM), and chloroform (CHCl_3). The final yield of the dark blue solid, identified as PC12BTH, amounted to 91.6 mg, corresponding to a yield of 63.4%. ^1H NMR (400 MHz, CDCl_3): δ 8.02 (s, 2H), 7.87 (s, 2H), 2.87 (s, 4H), 1.88 (s, 2H), 1.39-1.28 (m, 32H), 0.92-0.89 (m, 12H). Elemental analyses calculated for $[\text{C}_{44}\text{H}_{60}\text{F}_2\text{N}_2\text{S}_4]$: C, 70.73%; H, 8.36%; N, 3.75%; S, 17.6%. Found: C, 69.85%; H, 7.90%; N, 3.57%; S, 15.56%. Molecular weight evaluated by GPC: $M_n = 20.6$ kDa, $M_w = 27.2$ kDa, PDI = 1.32.

S.3.2 Synthesis of PC16BTH

Monomer 4,7-bis(5-bromo-4-(2-hexyldecyl)thiophen-2-yl)benzo[c][1,2,5]thiadiazole (108.84 mg, 0.12 mmol) and monomer 2,5-bis(trimethylstannyl)thiophene (49.17 mg, 0.12 mmol) were combined with catalyst Pd(PPh₃)₄ (11.55 mg, 0.01 mmol) in a 10 ml microwave reaction vial. The vial contained 4.7 ml of anhydrous toluene and 0.47 ml of DMF as the solvent, resulting in a concentration of 0.025 M. The reaction mixture was sealed with a PTFE cap and placed in a microwave reactor, set at 150°C for 3.5 hours. Upon completion of the reaction, the conjugated polymer was slowly dripped into methanol for reprecipitation, and the solid product was collected through filtration using a fritted funnel. Subsequently, the conjugated polymer PC16BTH was subjected to purification via a Soxhlet extractor, employing methanol, acetone, hexane, DCM, and CHCl₃ sequentially. The final yield of the dark blue solid PC16BTH amounted to 98.7 mg, corresponding to a yield of 62.5%. ¹H NMR (400 MHz, CDCl₃): δ 8.03 (s, 2H), 7.87 (s, 2H), 2.87 (s, 4H), 1.87 (s, 2H), 1.39-1.29 (m, 48H), 0.89-0.86 (m, 12H). Elemental analyses calculated for [C₅₂H₇₈F₂N₂S₄]: C, 72.67%; H, 9.15%; N, 3.26%; S, 14.92%. Found: C, 72.55%; H, 8.94%; N, 3.23%; S, 14.14%. Molecular weight evaluated by GPC: *M_n* = 26.6 kDa, *M_w* = 37.4 kDa, PDI = 1.41.

S.3.3 Synthesis of PC20BTH

In a 10 ml microwave reaction vial, monomer 4,7-bis(5-bromo-4-(2-octyldodecyl)thiophen-2-yl)benzo[c][1,2,5]thiadiazole (122.31 mg, 0.12 mmol) and monomer 2,5-bis(trimethylstannyl)thiophene (49.17 mg, 0.12 mmol) were combined with catalyst Pd(PPh₃)₄ (11.55 mg, 0.01 mmol). The vial contained 4.7 ml of anhydrous toluene and 0.47 ml of DMF as the solvent, resulting in a concentration of 0.025 M. The reaction mixture was sealed with a PTFE cap and placed inside a microwave reactor, set at 150°C for 3.5 hours. Upon the completion of the reaction, the conjugated polymer was gently dripped into methanol for reprecipitation, and the solid product was collected via filtration using a fritted funnel. Subsequently, the conjugated polymer PC20BTH underwent purification using a Soxhlet extractor, with methanol, acetone, hexane, DCM, and CHCl₃ employed sequentially. The final yield of the dark blue solid PC20BTH was 105.1 mg, corresponding to a yield of 61.3%. ¹H NMR (400 MHz, CDCl₃): δ 8.02 (s, 2H), 7.86 (s, 2H), 2.87 (s, 4H), 1.88 (s, 2H), 1.39-1.25 (m, 64H), 0.89-0.86 (m, 12H). Elemental analyses calculated for [C₆₀H₉₂F₂N₂S₄]: C, 74.17%; H, 9.75%; N, 2.88%; S, 13.20%. Found: C, 73.97%; H, 9.57%; N, 2.95%; S, 13.27%. Molecular

weight evaluated by GPC: $M_n = 12.5$ kDa, $M_w = 21.2$ kDa, PDI = 1.69.

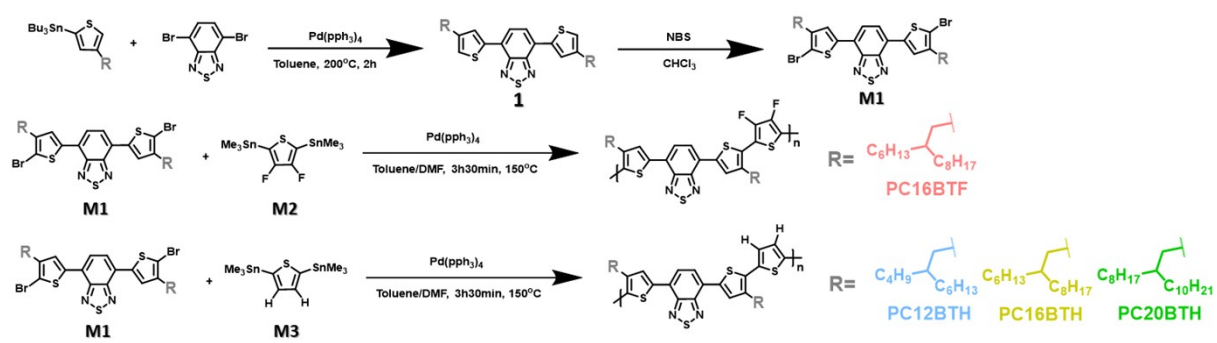
S.3.4 Synthesis of PC16BTF

Monomer 4,7-bis(5-bromo-4-(2-hexyldecyl)thiophen-2-yl)benzo[c][1,2,5]thiadiazole (108.84 mg, 0.12 mmol) and monomer (3,4-difluorothiophene-2,5-diyl)bis(trimethylstannane) (53.48 mg, 0.12 mmol) were combined with catalyst Pd(PPh₃)₄ (11.55 mg, 0.01 mmol) in a 10 ml microwave reaction vial. The vial contained 4.7 ml of anhydrous toluene and 0.47 ml of DMF as the solvent, resulting in a concentration of 0.025 M. The reaction mixture was sealed with a PTFE cap and placed in a microwave reactor, set at 150°C for 3.5 hours. Following the completion of the reaction, the conjugated polymer was gradually dripped into methanol for reprecipitation, and the solid product was collected through filtration using a fritted funnel. The conjugated polymer PC16BTF was purified through a Soxhlet extractor, sequentially using methanol, acetone, hexane, DCM, and CHCl₃. The final yield of the dark blue solid PC16BTF was 105.6 mg, representing a yield of 65.1%. ¹H NMR (400 MHz, CDCl₃): δ 8.05 (s, 2H), 7.92 (s, 2H), 2.80 (s, 4H), 1.83 (s, 2H), 1.34-1.28 (m, 48H), 0.88-0.86 (m, 12H). Elemental analyses calculated for [C₅₂H₇₈F₂N₂S₄]: C, 69.75%; H, 8.56%; N, 3.13%; S, 14.32%. Found: C, 69.09%; H, 8.12%; N, 3.10%; S, 14.00%. Molecular weight evaluated by GPC: $M_n = 33.1$ kDa, $M_w = 45.3$ kDa, PDI = 1.36.

S.4 Instrumentation

The thermal characteristics of the specimens were assessed using a PerkinElmer DSC 8000 apparatus, involving heating and cooling cycles spanning from 50 to 250 °C, with a heating rate of 10 °C min⁻¹, all conducted in an environment of nitrogen. Thermogravimetric analysis (TGA) was carried out utilizing a HITACHI STA 7200 thermogravimetric analyzer under a continuous flow of nitrogen. All samples underwent heating from 50 to 700 °C at a rate of 10 °C min⁻¹. Electrochemical cyclic voltammetry (CV) was performed employing a BioLogic SP-150 electrochemical analyzer. Glassy carbon, Pt wire, and Ag/AgCl served as the working, counter, and reference electrodes, respectively. The measurements were conducted in a solution of 0.10 M ferrocene in acetonitrile, with a scan rate of 50 mV s⁻¹. UV-vis-NIR spectra were recorded on a Hitachi U-4100 spectrophotometer. AFM studies of thin films were performed with Hitachi AFM5100N at tapping mode under ambient conditions using Hitachi SI-DF3PS tips with a resonant frequency = 70 kHz and force constant = 2.0 N m⁻¹). Grazing-incidence wide-angle X-ray

scattering (GIWAXS) patterns were obtained at the TPS 25A beamlines at National Synchrotron Radiation Research Center (NSRRC, Taiwan). Ultraviolet photoelectron spectroscopy (UPS) and X-ray photoelectron spectra (XPS) measurements were conducted by a PHI 5000 VersaProbe III (ULVAC-PHI, Inc) with an ultra-high vacuum 6.7×10^{-8} Pa. Excitation sources of the He I radiation (21.22 eV) and a monochromatic Al K α X-ray (1486.6 eV) were taken as the excitation source. The film thicknesses were measured using an Alpha-Step[®] D-300 surface profiler (KLA-Tencor).



Scheme S1. Synthesis of monomers and studied polymers.

Table S1. Hall effect measurement results.

Polymer	Doping	μ [cm ² V ⁻¹ s ⁻¹] ¹	n [cm ⁻³]	σ [S cm ⁻¹]
	concentratio n [mM]			
PC12BTH	Pristine	4.68×10^{-2}	3.5×10^{15}	10^{-4}
	3	3.56×10^{-2}	2.67×10^{20}	5.73
	6	3.71×10^{-2}	8.68×10^{20}	19.4
	9	2.95×10^{-2}	9.46×10^{20}	16.8
PC16BTH	Pristine	4.49×10^{-2}	3.6×10^{15}	10^{-4}
	3	3.72×10^{-2}	2.95×10^{20}	6.62
	6	3.56×10^{-2}	1.25×10^{21}	26.8
	9	3.78×10^{-2}	9.70×10^{20}	22.1
PC20BTH	Pristine	3.44×10^{-2}	4.8×10^{15}	10^{-4}
	3	3.15×10^{-2}	2.16×10^{20}	4.1
	6	3.04×10^{-2}	1.14×10^{21}	21.0
	9	1.54×10^{-2}	1.73×10^{21}	16.1
PC16BTF	Pristine	6.69×10^{-2}	2.4×10^{15}	10^{-4}
	3	4.48×10^{-2}	1.54×10^{20}	4.18
	6	4.75×10^{-2}	3.53×10^{20}	10.1
	9	3.18×10^{-2}	3.38×10^{20}	6.48

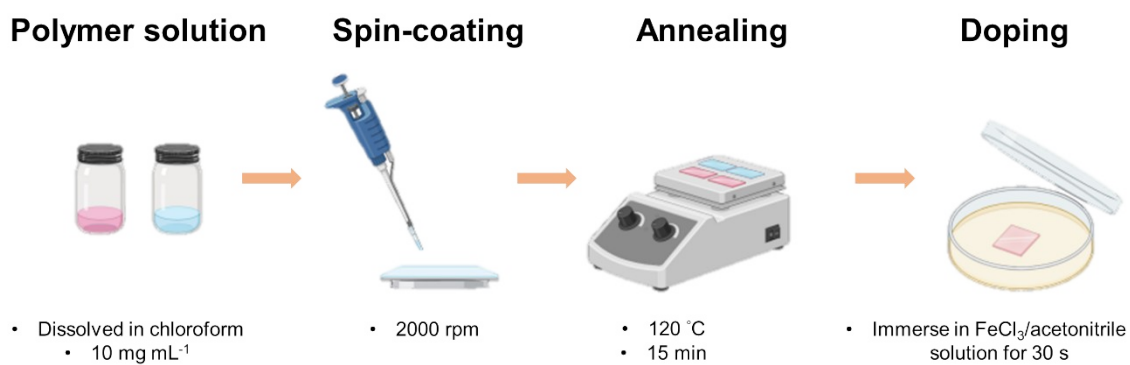


Fig. S1 Schematic illustration of fabrication of polymer thin film and doping process.

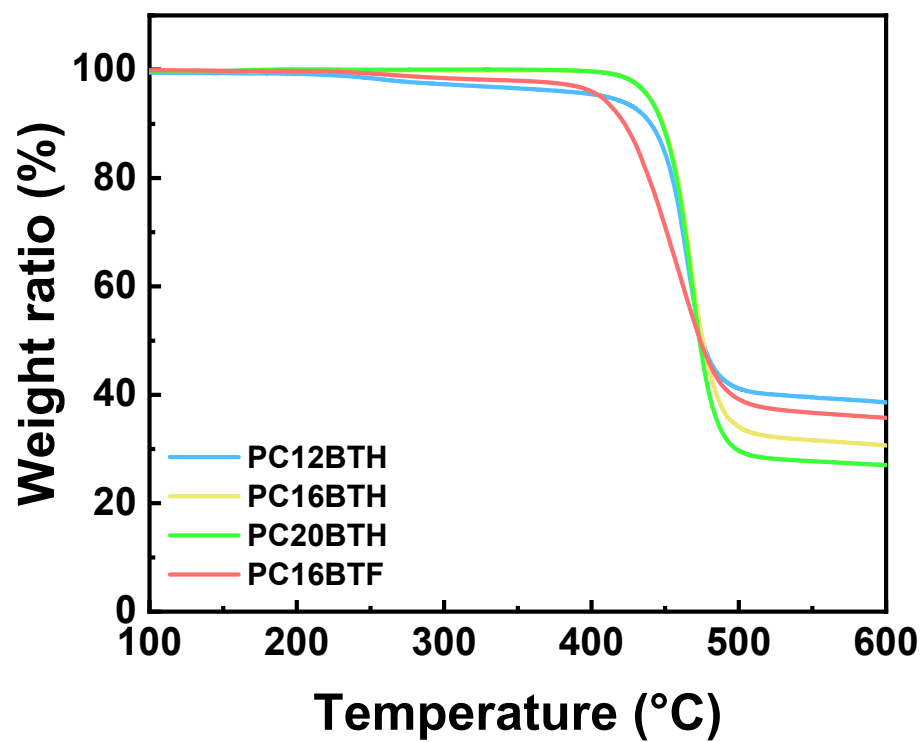


Fig. S2 TGA thermograms of studied polymers.

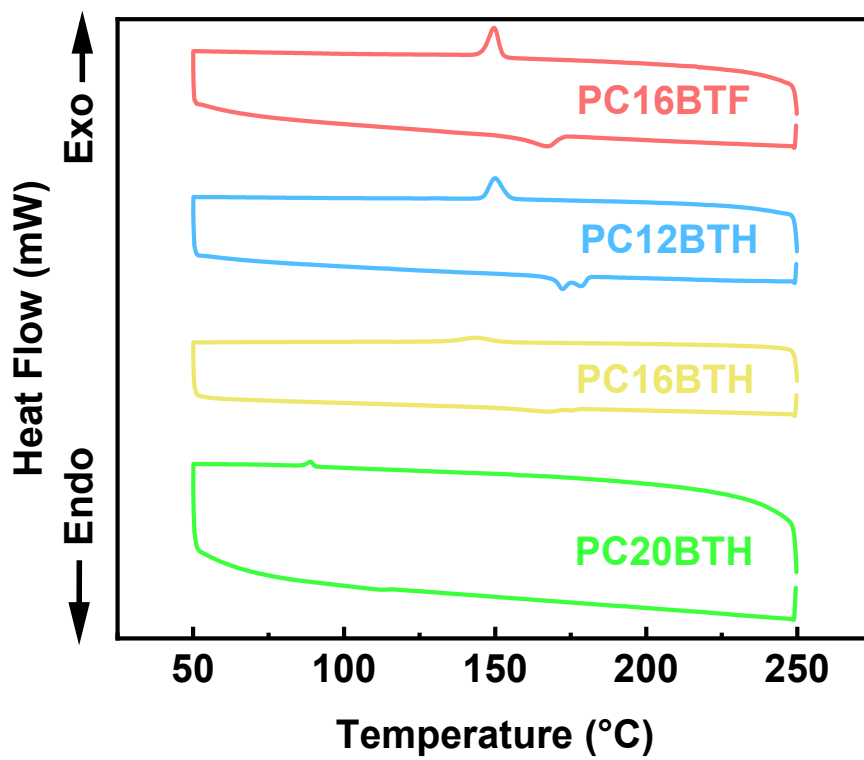


Fig. S3 DSC thermograms of studied polymers.

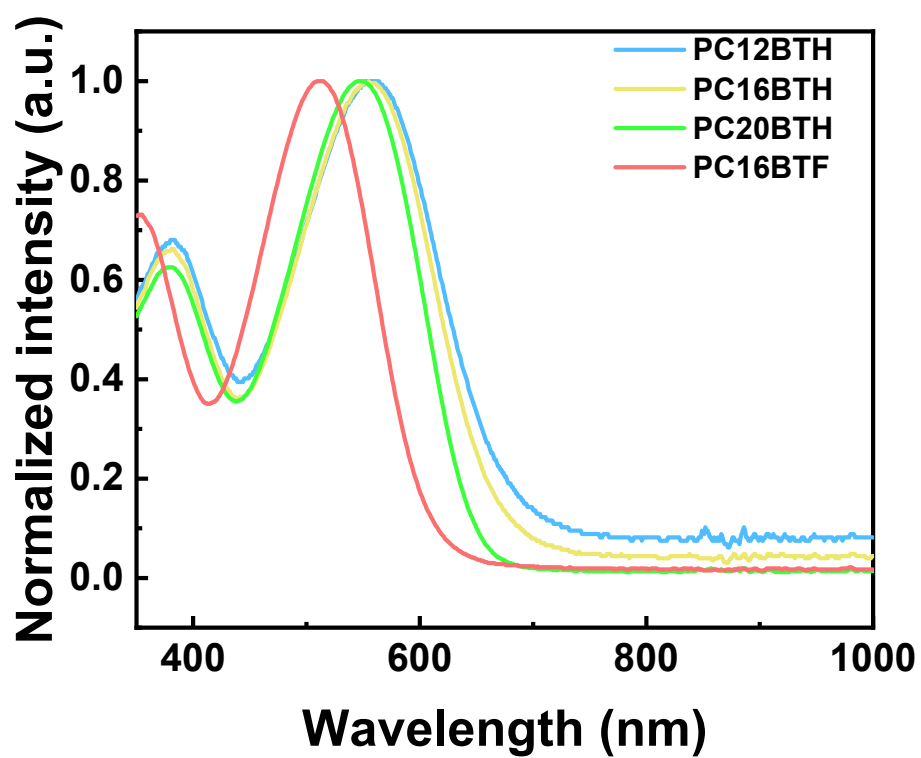


Fig. S4 UV-vis-NIR absorption spectra of PC12BTH, PC16BTH, P20BTH, and PC16BTF in the solution state.

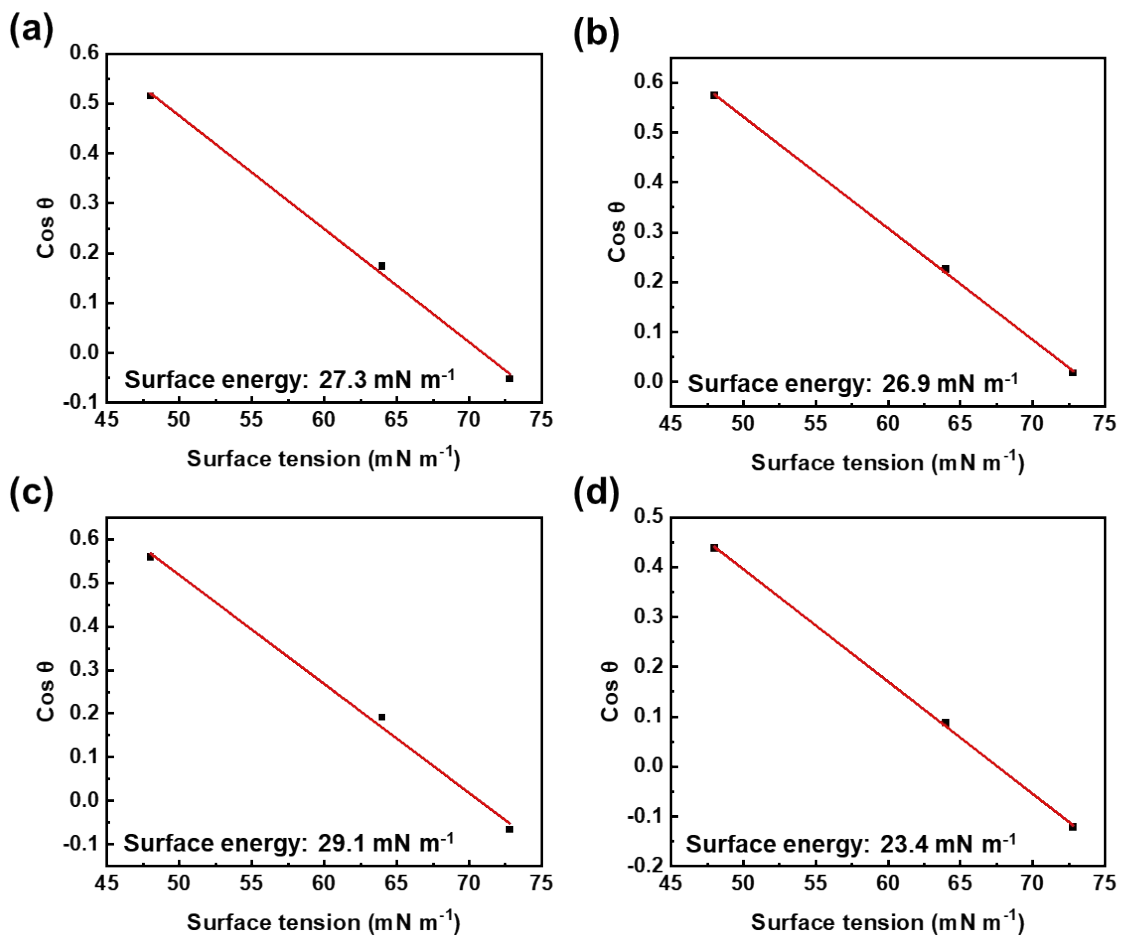


Fig. S5 The relationship of contact angle and surface tension of solution surface tension for (a) PC12BTH, (b) PC16BTH, (c) P20BTH, and (d) PC16BTF. The surface energy is calculated by Zisman method, and the adopted solution are water, glycerol, and ethylene glycol.

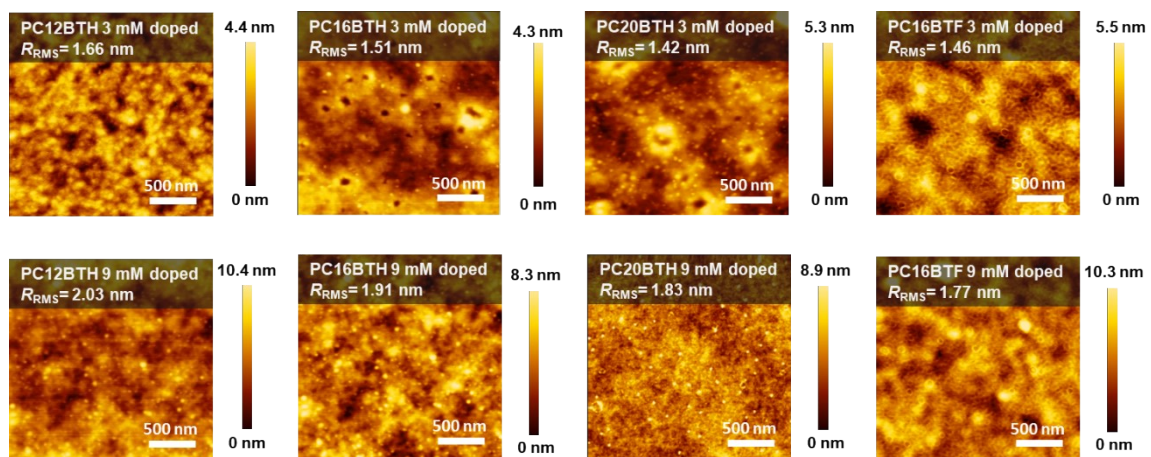


Fig. S6 AFM height image and the root-mean-square surface roughness (R_{rms}) of 3 mM and 9 mM doped polymers.

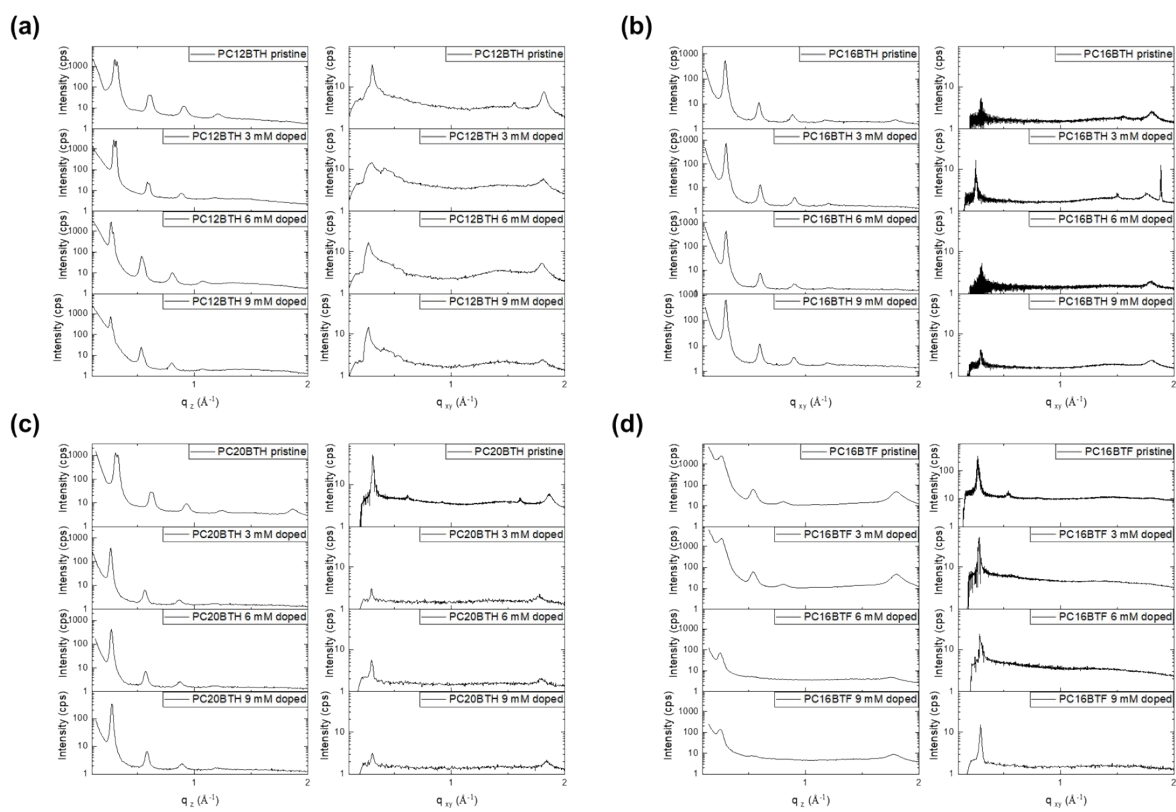


Fig. S7 GIWAXS in-plane and out-of-plane line cut pattern of pristine and doped (a) PC12BTH, (b) PC16BTH, (c) PC20BTH, and (d) PC16BTF.

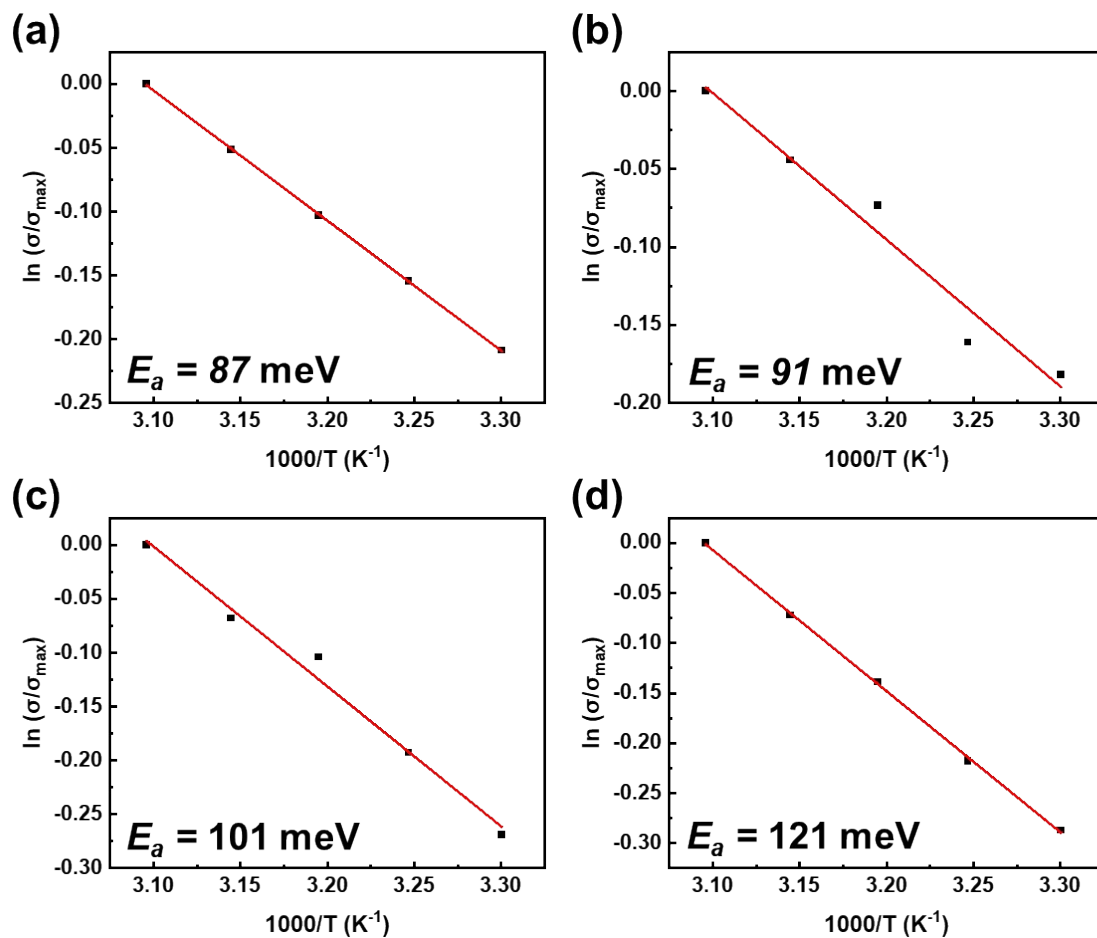


Fig. S8 Temperature-dependent of FeCl₃-doped (a) PC12BTH, (b) PC16BTH, (c) PC20BTH, and (d) PC16BTF films. All polymer films were doped with 6 mM FeCl₃ solution.

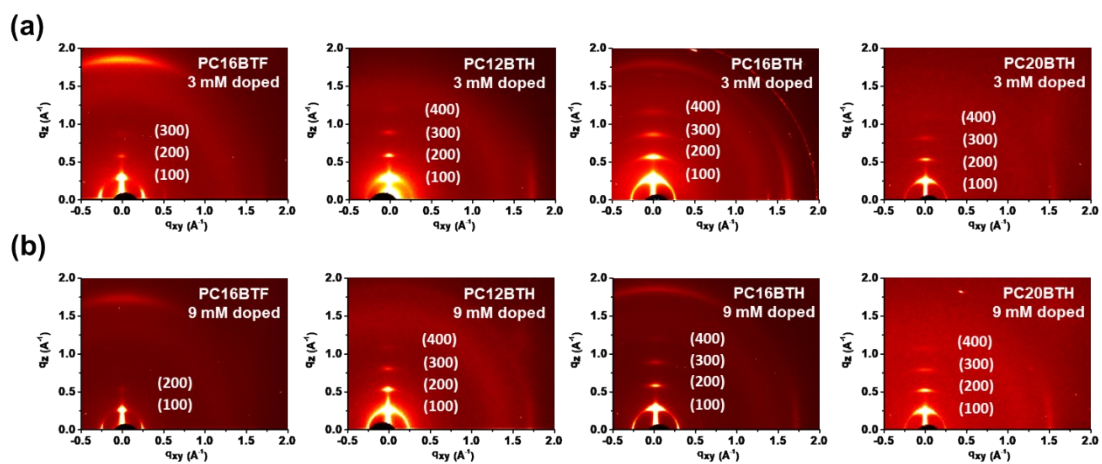


Fig. S9. 2D-GIWAXS patterns of polymer films: (a) 3 mM doped and (b) 9 mM FeCl_3 doped polymers.

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