Supporting Information

Imide-Functionalized Acceptor-Acceptor Copolymers as Efficient Electron Transport

Layers for High-Performance Perovskite Solar Cells

Yongqiang Shi,^{a,b,†} Wei Chen,^{a,c,†} Ziang Wu,^d Yang Wang,^a Weipeng Sun,^a Kun Yang,^a Yumin

Tang,^a Han Young Woo,^d Ming Zhou,^{*,b} Aleksandra B. Djurišić,^{*,c} Zhubing He,^{*,a} and Xugang

Guo*,a

^a Department of Materials Science and Engineering and The Shenzhen Key Laboratory of Printed Organic Electronics, Southern University of Science and Technology (SUSTech), No. 1088, Xueyuan Road, Shenzhen, Guangdong 518055, China. E-mail: hezb@sustech.edu.cn; guoxg@sustech.edu.cn

^b School of New Energy and Materials, and State Key Laboratory of Oil and Gas Reservoir Geology and Exploitation Engineering, Southwest Petroleum University, Chengdu, Sichuan 610500, China. E-mail: mr.zhouming@163.com

^c Department of Physics, The University of Hong Kong, Pokfulam Road, Hong Kong SAR. Email: dalek@hku.hk

^d Department of Chemistry, College of Science, Korea University, 145 Anam-ro, Seongbuk-gu, Seoul 136-713, Republic of Korea

[†]Y. Shi and W. Chen contributed equally to this work.

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1. Materials and Methods.

All commercially available solvents, reagents, and chemicals were used as received without further purification unless otherwise stated. Anhydrous tetrahydrofuran and toluene were distilled from Na/benzophenone under inert atmosphere. The tributyltin chloride was purchased from Adamas-Beta Inc. (Shanghai, China). 4,9-Dibromo-2,7-bis(2ethylhexyl)benzo[lmn][3,8]phenanthroline-1,3,6,8(2H, 7H)-tetraone (NDI-Br) and N,N'-Bis(2-ethylhexyloxyl)-1,7-dibromo-3,4,9,10-perylene diimide (PDI-Br) were purchased from Suna Tech (Suzhou, China). N-(2-hexyldecyl)-2,2'-bithiophene-3,3'-dicarboximide was prepared from according to the published procedure.¹ Unless otherwise stated, all operations and reactions were carried out under argon using standard Schlenk line techniques. Polymerizations were carried out on Initiator+ Microwave Synthesizer (Biotage, Sweden). ¹H and ¹³C NMR spectra were recorded on a Bruker Ascend 400 and 500 MHz spectrometer, and the chemical shifts were referenced to residual protio-solvent signals. C, H, N, and S elemental analyses (EAs) of polymers were conducted at Shenzhen University (Shenzhen, China). Polymer molecular weights were characterized on Polymer Laboratories GPC-PL220 high temperature GPC/SEC system (Agilent Technologies) at 150 °C vs polystyrene standards using trichlorobenzene as the eluent. Differential scanning calorimetry (DSC) curves were recorded on Mettler STARe (TA Instrument) in nitrogen with a heating ramp of 10 °C min⁻¹, and thermogravimetric analysis (TGA) curves were collected on Mettler STARe (TA Instrument). UV-vis absorption spectra were collected on a Shimadzu UV-3600 UV-VIS-NIR spectrophotometer. Cyclic voltammetry (CV) measurements of polymer films were performed under argon atmosphere using a CHI760E voltammetric analyzer with 0.1 M tetra-nbutylammonium hexafluorophosphate in acetonitrile as the supporting electrolyte. A platinum disk working electrode, a platinum wire counter electrode, and a silver wire reference electrode were employed, and the ferrocene/ferrocenium (Fc/Fc⁺) was used as the external reference for all measurements. The scanning rate was 100 mV s⁻¹. Polymer films were drop-casted from chloroform solutions on a Pt working electrode (2 mm in diameter). The supporting electrolyte solution was thoroughly purged with argon before all CV measurements. AFM characterization of polymer films was performed on a Dimension Icon Scanning Probe Microscope (Asylum Research, MFP-3D-Stand Alone) in tapping mode.

2. Monomer and Polymer Synthesis.



Synthesis of *N*-(2-hexyldecyl)-2,2'-bithiophene-3,3'-dicarboximide.¹ To a suspension of 2,2'-bithiophene-3,3'-dicarboxylic anhydride (1.0 g, 4.23 mmol) in 20 mL dichloromethane was added a solution of 2-hexyldecyl amine (1.02 g, 4.23 mmol) in 10 mL dichloromethane. After the addition, the reaction mixture was stirred at reflux for overnight. Upon removal of solvent, 10 mL thionyl chloride was added in one portion, and the mixture was refluxed for overnight. The excess thionyl chloride was then removed under a reduced pressure, and the residue was purified by column chromatography on silica gel with petroleum ether:dichloromethane (1:1) as the eluent to give the product as a yellow oil (1.7 g, 89%). ¹H NMR (500 MHz, CDCl₃) δ (ppm): 7.72 (d, 2H), 7.23 (d, 2H), 4.24 (d, 2H), 1.93-1.91 (m, 1H), 1.37-1.23 (m, 24H), 0.89-0.84 (t, 6H). ¹³C NMR (125 MHz, CDCl₃) δ 162.28, 137.38, 133.28, 133.06, 124.22, 53.46, 49.40, 31.92, 31.84, 30.09, 29.78, 29.57, 29.33, 26.42, 22.69, 22.68, 14.16, 14.13.



Synthesis of N-(2-hexyldecyl)-5,5'-bis(tributylstannyl)-2,2'-bithiophene-3,3'dicarboximide (BTI-Tin). A solution of LDA (5mL, 4.42 mmol) was added dropwise to a solution of bithiophene imide BTI (925 mg, 2.01 mmol) in dry THF (15 mL) at -78 °C under N₂ and stirred for 1 h. Bu₃SnCl (1.45 mL, 4.42 mmol) was added dropwise, and the reaction mixture was allowed to reach room temperature and stirred overnight. The mixture was quenched with water and extracted with CH₂Cl₂ three times. The combined organic layer was dried over Na₂SO₄ and filtered. After the removal of solvent under a reduced pressure via rotvap, the residue was purified over flash column chromatography on silica gel basified with trimethylamine using hexane as the eluent to give the target compound as a colorless oil (1.2 g, 60%). ¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.74 (s, 2H), 4.25 (d, 2H), 1.94-1.88 (m, 1H), 1.61-1.54 (m, 12H) 1.38-1.14 (m, 48H), 0.92 (t, 18H), 0.86 (t, 6H). ¹³C NMR (100 MHz, CDCl₃) & 162.91, 142.75, 141.05, 138.46, 133.00, 132.87, 49.32, 31.93, 31.85, 29.80, 29.59, 29.33, 28.89, 27.24, 22.70, 22.68, 14.14, 14.12, 13.64, 11.03. HRMS: C₅₀H₈₉NO₂S₂Sn₂ Calcd: 1037.44, Found: 1038.44 (M+H).



P(BTI-NDI): To a 5 mL reaction tube equipped with a stirring bar was added the BTI-Tin monomer (131.7 mg, 0.13 mmol), NDI-Br monomer (82.3 mg, 0.13 mmol), $Pd_2(dba)_3$ (1.7 mg, 0.002 mmol), P(o-tolyl)₃ (4.6 mg, 0.015 mmol) and anhydrous toluene (3 mL). The reaction tube was purged with argon and sealed under argon flow. The sealed reaction mixture was heated to 80 °C for 10 min, 100 °C for 10 min, and then 140 °C for 3h under microwave irradiation, respectively. And 0.1 mL 2-(tributylstanny)thiophene was then added and the reaction mixture was stirred at 140 °C for 0.5 h under microwave irradiation. Finally, 0.2 mL

2-bromothiophene was added and the reaction mixture was stirred at 140 °C for another 0.5 h. After cooled to room temperature, the reaction mixture was slowly dripped into 100 mL methanol containing 5 mL 12 N hydrochloric acid, under vigorous stirring. After stirring for 1 h, the solid precipitate was transferred to a thimble followed by Soxhlet extraction. The solvent sequence for the extraction was methanol, acetone, hexane and dichloromethane. The final dichloromethane fraction was concentrated by removing most of solvent and precipitated into methanol. The solid was collected by filtration and dried in vacuum to afford the polymer product as a deep colored solid (86 mg; yield: 70%). ¹H NMR (400 MHz, C₂Cl₄D₂, 80 °C) δ (ppm): 8.76 (s, 1H), 7.93 (s, 1H), 4.23 (s, 2H), 4.05 (s, 4H), 1.95 (s, 1H), 1.88 (s, 2H), 1.30 (m, 16H), 1.23 (m, 26H), 0.87 (m, 6H), 0.79 (m, 12H). $M_n = 27$ kDa, PD = 1.6. Anal. Calcd. for C₅₆H₇₃N₃O₆S₂ (%) : C, 70.93; H, 7.76; N, 4.43; S, 6.76. Found (%): C, 70.89; H, 7.80; N, 4.41; S, 6.73.



P(BTI-PDI): To a 5 mL reaction tube with a stirring bar was added the BTI-Tin (149.9 mg, 0.14 mmol), PDI-Br (111.6 mg, 0.14 mmol), $Pd_2(dba)_3$ (1.9 mg, 0.002 mmol), $P(o-tolyl)_3$ (5.2 mg, 0.017 mmol) and anhydrous toluene (3 mL). The reaction tube was purged with argon and sealed under argon flow. The sealed reaction mixture was heated to 80 °C for 10 min, 100 °C for 10 min, and then 140 °C for 3h under microwave irradiation, respectively. And 0.1 mL 2-(tributylstanny)thiophene was then added and the reaction mixture was stirred at 140 °C for 0.5 h under microwave irradiation. Finally, 0.2 mL 2-bromothiophene was added and the reaction mixture was stirred at 140 °C for 0.5 h. After cooled to room temperature, the reaction

mixture was slowly dripped into 100 mL methanol containing 5 mL 12 N hydrochloric acid, under vigorous stirring. After stirring for 1 h, the solid precipitate was transferred to a Soxhlet thimble followed by Soxhlet extraction. The solvent sequence for the extraction was methanol, acetone, hexane and dichloromethane. The final dichloromethane fraction was concentrated by removing most of solvent and precipitated into methanol. The solid was collected by filtration and dried in vacuum to afford the polymer as a deep colored solid (97 mg; yield: 65%). ¹H NMR (400 MHz, C₂Cl₄D₂, 80 °C) δ (ppm): 8.62 (s, 2H), 8.32 (s, 4H), 7.84-7.92 (s, 2H), 4.17 (s, 2H), 4.03 (s, 4H), 1.88 (m, 3H), 1.24-1.18 (m, 40H), 0.88-0.74 (m, 18H). $M_n = 11$ kDa, PD = 1.6. Anal. Calcd. for C₆₆H₇₇N₃O₆S₂ (%) : C, 73.92; H, 7.24; N, 3.92; S, 5.98. Found (%): C, 73.89; H, 7.26; N, 3.89; S, 5.93.

3. NMR and Mass Spectra of Compounds.



Figure S1. ¹H NMR spectrum of BTI (500 MHz, r.t., in CDCl₃).



Figure S2. ¹³C NMR spectrum of BTI (500 MHz, r.t., in CDCl₃).



Figure S3. ¹H NMR spectrum of distannylated BTI monomer (400 MHz, r.t., in CDCl₃).



Figure S4. ¹³C NMR spectrum of distannylated BTI monomer (400 MHz, r.t., in CDCl₃).



Figure S5. ¹H NMR spectrum of polymer P(BTI-NDI) (400 MHz, 80 °C, in C₂Cl₄D₂).



Figure S6. ¹H NMR spectrum of polymer P(BTI-PDI) (400 MHz, 80 °C, in C₂Cl₄D₂).



Figure S7. HRMS spectrum of distannylated BTI monomer.

4. Polymer Thermal Properties.



Figure S8. (a) Thermogravimetric analysis of polymers P(BTI-NDI) and P(BTI-PDI) at a heating ramp of 10 °C min⁻¹. (b) DSC thermograms of polymers P(BTI-NDI) and P(BTI-PDI). The DSC curves are from the second heating and first cooling scans with a ramp of 10 °C min⁻¹. N₂ was used as the purge gas for both TGA and DSC measurements.

5. PVSC Fabrication and Characterization.

Materials: Solvents including anhydrous N,N-dimethylformamide (DMF, 99.8%), anhydrous dimethyl sulfoxide (DMSO, 99.8%), anhydrous isopropanol (IPA, 99.8%), anhydrous chlorobenzene (CB, 99.8%) and all other solvents were purchased from Across Organics. Cesium iodide (CsI, 99.999%) was purchased from Sigma-Aldrich. Lead (II) iodide (PbI₂) and lead (II) bromide (PbBr₂) were purchased from TCI. Methylammonium bromide (MABr) and formamidinium iodide (FAI) were purchased from Great Cell Solar Ltd (Australia). Bathocuproine (BCP) and [6,6]-Phenyl-C₆₁-butyric acid methyl ester (PC₆₁BM) (99.5%) were purchased from Daeyeon Chemicals Co., Ltd. Poly[bis(4-phenyl)(2,4,6-trimethylphenyl)amine (PTAA) HTL was obtained from Xi'an Polymer Light Technology in China. All materials above were used directly as received.

Device Fabrication: Inverted planar perovskite solar cells were fabricated with a structure of ITO/PTAA/perovskites/PC₆₁BM, P(BTI-NDI) or P(BTI-PDI)/BCP/Ag. ITO glass was cleaned by sequentially washing with detergent, deionized water, acetone, and isopropanol. The

substrates were dried with N₂ and cleaned by UV ozone for 15 min. PTAA HTLs (1.5 mg mL⁻¹ in toluene) were spin-coated onto the clean ITO substrates with 6000 r.p.m and annealed at 100 °C for 10 mins. The CsFAMA mixed perovskite layers were fabricated according to our previously reported one-step antisolvent recipe.^[2-4] Briefly, perovskite precursor solution was made by mixing PbI₂, PbBr₂, FAI and MAI in DMF/DMSO (v/v 4/1). The mole concentration was kept at 1.3 M with 0.1 M PbI₂ excess. The I/Br and FA/MA mole ratios were maintained at 0.85/0.15. After stirring for 1 hour, 35 µL of CsI (2 M in DMSO) was added into the precursor solution and stirred another 1 h at 65 °C. The CsFAMA perovskite films were prepared by spin coating the precursor solution (4000 rpm, 35s) followed by 300 µL CB drop-casting in the last 25 s. After the perovskite deposition, the PC₆₁BM (2 wt% in CB) (1000 r.p.m, 30s), P(BTI-NDI) and P(BTI-PDI) (10 mg mL⁻¹ in CB) (1500 r.p.m, 30s) were spin-coated on top and the films were annealed at different temperatures (100 °C for PC₆₁BM, 110 °C for P(BTI-NDI) and P(BTI-PDI)) for 30 min. To complete the device fabrication, BCP (8 nm) and Ag (100 nm) top electrode were deposited by thermal evaporation.

Characterization: *J*-*V* measurements were carried out using a Keithley 2400 source meter in ambient environment of 25-27 $^{\circ}$ C and 60-65% RH. The devices were measured both in reverse scan (1.2 V \rightarrow -0.2 V, step 0.01 V) and forward scan (-0.2 V \rightarrow 1.2 V, step 0.01 V) with 10 ms delay time. Illumination was provided by an Oriel Sol3A solar simulator with AM1.5G spectrum and light intensity of 100 mW cm⁻² was calibrated by means of a KG-5 Si diode. The active area (7.5 mm²) of the devices was calibrated with shadow mask during the measurements. EQE measurements for devices were conducted with an Enli-Tech EQE measurement system. Top-view morphology was analyzed by TESCAN MIRA3, cross-section SEM, STEM and EDX mapping images were characterized by Helios Nanolab 600i FIB and FEI Talos transmission electron microscope (TEM) with Super-X EDS was employed to acquire the STEM-EDX data with high-angle annular dark field (HAADF) mode. Time resolved photoluminescence spectroscopy was measured using a Spectrofluorometer (FS5, Edinburgh instruments) and 405 nm pulsed laser was used as excitation source for the measurement. Contact angle measurements were performed with VCA Optima (AST Products, Inc.). 2D Grazing-Incidence Wide-Angle X-ray Scattering (GIWAXS) measurements of polymer ETLs were carried out at the PLS-II 9A U-SAXS beam line of Pohang Accelerator Laboratory, Korea.



Figure S9. Different magnification SEM images of the perovskite films and the ETLs coated perovskite films.



Figure S10. (a) Cross-sectional SEM image of a PSCs with PTAA HTL and P(BTI-PDI) ETL;

(b) The corresponding energy dispersive X-ray (EDX) elemental maps.



Figure S11. *J*-*V* hysteresis of the best-performing PSCs with $PC_{61}BM$ ETLs under reverse and forward scans.



Figure S12. Statistics of device performance parameters (J_{sc} , V_{oc} , and FF) of total 20 cells with PC₆₁BM, P(BTI-NDI), and P(BTI-PDI) ETLs.

Samples	A1(%)	$\tau_1[ns]$	A2(%)	$\tau_2[ns]$	Weighted average τ [ns]
Perovskite (PVK)	2.4	44.7	97.6	1045	1043.9
PVK/PC ₆₁ BM	12.2	1.3	87.8	401.2	401.0
PVK/P(BTI-NDI)	27.8	2.4	62.2	281.7	280.6
PVK/P(BTI-PDI)	23.8	0.6	76.2	431.1	430.9

Table S1. Summary of carrier decay time by fitting the PL decay curves.



Figure S13. The photovoltaic performance of PVSCs as a function of the thickness of P(BTI-NDI) electron transport layer.



Figure S14. The photovoltaic performance of PVSCs as a function of the thickness of P(BTI-PDI) electron transport layer.



Figure S15. The corresponding $J^{1/2}$ -V characteristics for the electron-only devices based on the neat films of P(BTI-NDI), P(BTI-PDI), and PC₆₁BM.



Figure S16. Water contact angle measurements of the $PC_{61}BM$, P(BTI-NDI) and P(BTI-PDI) films.

ETLs	V _{oc} (V)	$J_{\rm sc}$ (mA cm ⁻²)	FF (%)	PCE (%)	Ref.
ITIC	0.88	20.67	61.1	11.11	[5]
DS2	0.8	22.65	63	11.4	[6]
t-BPTI	0.97	19.80	60.55	11.63	[7]
SFX-PDI4	1.08	19.9	71.4	15.3	[8]
TPE-PDI4	1.01	21.68	74	16.29	[9]
hPDI3-Pyr-hPDI3	0.93	22.68	78	16.5	[10]
HATNASOC7-Cs	1.08	20.73	78.6	17.62	[11]
HATNT	1.07	21.83	77.8	18.1	[12]
TDTP	1.05	22.4	77.7	18.2	[13]
NDI-PM	1.1	21.1	79.1	18.4	[14]
PDI-C60	1.061	22.1	79.2	18.6	[15]
NDI-ID	1.1	23.0	80	20.2	[16]
NDI-PhE	1.1	23.1	80.8	20.5	[17]

Table S2. Summary of recent representative non-fullerene small molecular electron transporting layers utilized in inverted perovskite solar cells.

 Table S3. Summary of recent presentative polymer electron transporting layers utilized in inverted perovskite solar cells.

ETLs	$V_{\rm oc}$ (V)	$J_{\rm sc}$ (mA cm ⁻²)	FF (%)	PCE (%)	Ref.
pBTTz	0.91	21.95	72.3	14.4	[18]
N2200	1.06	19.3	73.4	15.02	[19]
NDP-V	1.043	21.41	74	16.54	[20]
PFN-2TNDI	0.98	21.9	78	16.7	[21]
P(NDI2DT-TTCN)	1.00	22.0	77.4	17.0	[22]
P(BTI-NDI)	1.10	22.7	78.5	19.5	This work
P(BTI-PDI)	1.12	23.3	79.4	20.8	This work

6. GIWAXS Measurements.



Figure S17. (a, b) 2D-GIWAXS characteristics for the neat P(BTI-NDI) and P(BTI-PDI) ETLs. All samples were prepared on silicon substrate following the optimal device fabrication condition; The in-plane (c) and out-of-plane (d) scattering profiles for the corresponding samples.

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