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The introduction of conjugated isolation groups into the common acceptor cyanoacrylic acid: an efficient strategy to suppress the charge recombination in dye sensitized solar cells, and the dramatically improved efficiency from 5.89% to 9.44%

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1. Additional graphs

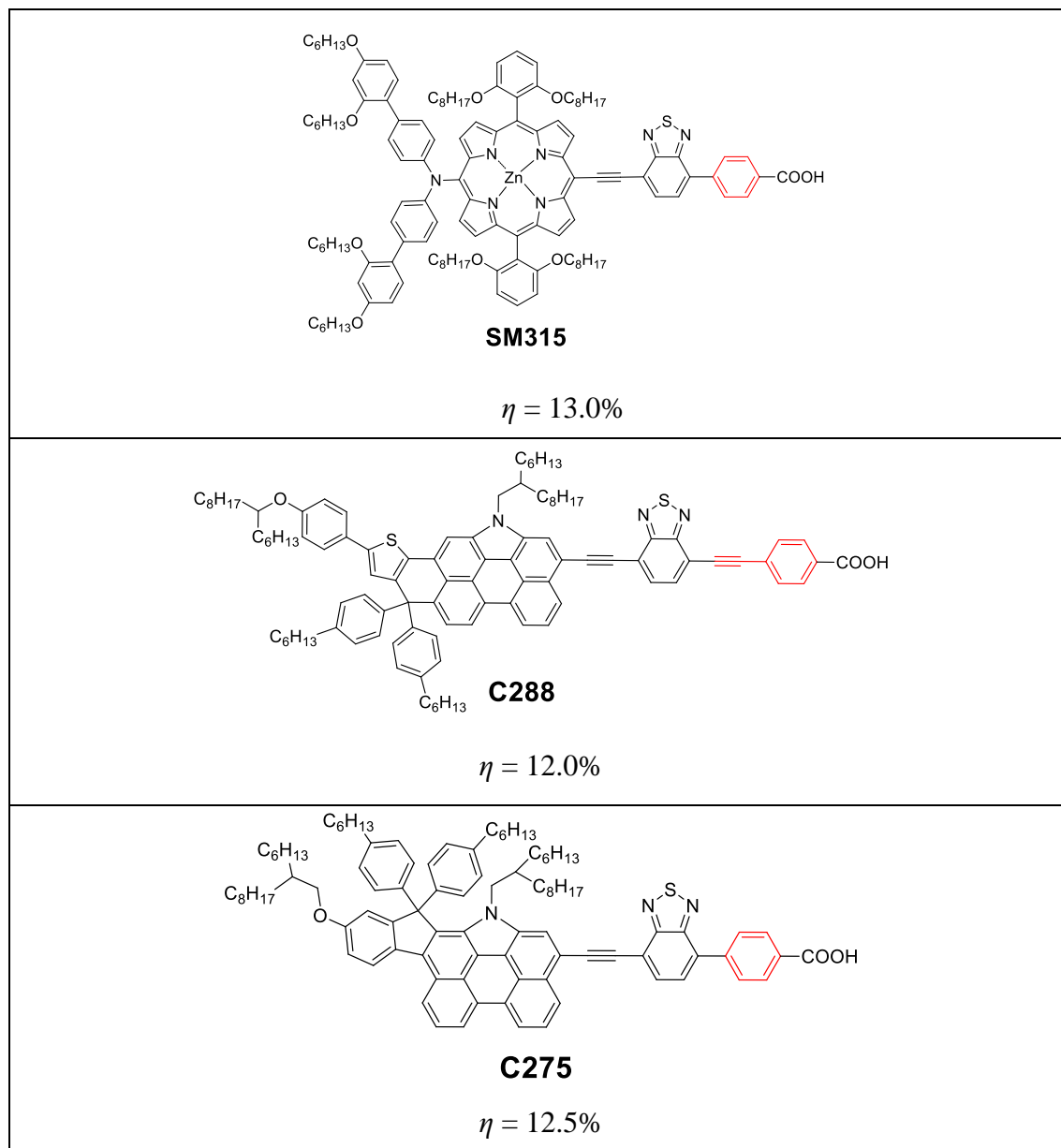


Chart S1. Chemical structures and efficiencies of **SM315**, **C288**, and **C275**.

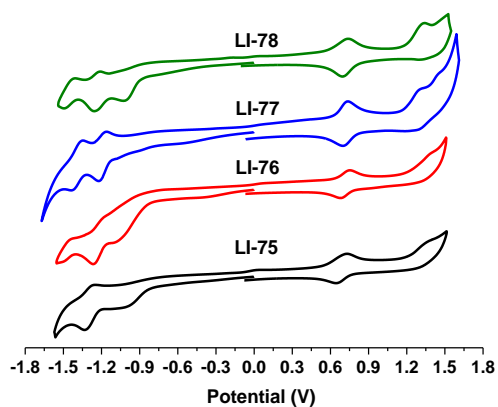


Figure S1. Cyclic voltammograms of **LI-75–LI-78** in CH_2Cl_2 .

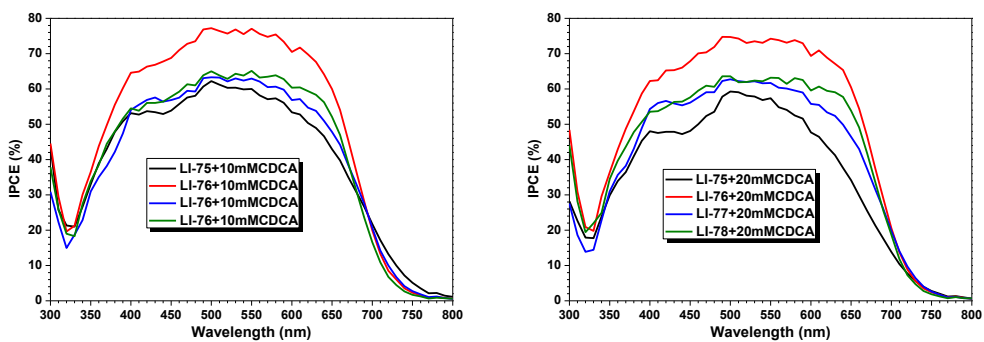


Figure S2. Spectra of monochromatic IPCE for DSCs based on **LI-75–LI-78** with CDCA.

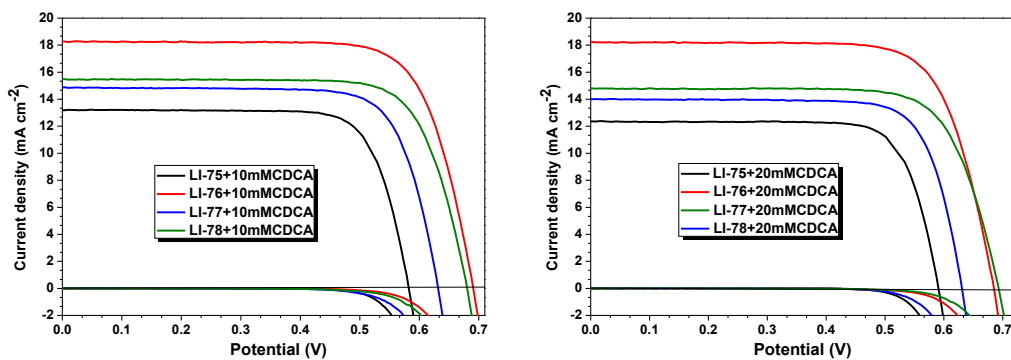


Figure S3. J – V characteristics of DSCs measured at simulated 100 mW cm^{-2} AM 1.5 conditions with CDCA.

2. Experimental details

2.1 Instrumentation

^1H and ^{13}C NMR spectroscopy study was conducted with a Varian Mercury 300 or 400 spectrometer or using tetramethylsilane (TMS; $\delta = 0$ ppm) as internal standard. HRMS (ESI) spectra were measured on a Finnigan LCQ advantage mass spectrometer. UV-visible spectra were obtained using a Shimadzu UV-2550 spectrometer. Electrochemical cyclic voltammetry was performed with a CHI 660 voltammetric analyzer with Pt disk, Pt plate, and Ag/Ag^+ electrode as working electrode, counter electrode, and reference electrode, respectively, in nitrogen-purged anhydrous CH_2Cl_2 with tetrabutylammonium hexafluorophosphate (TBAPF_6) as the supporting electrolyte (scanning rate: 100 mV/s). The ferrocene/ferrocenium redox couple was used for potential calibration. Elemental analyses were performed with a 73 CARLOERBA-1106 microelemental analyzer.

2.2 Materials

Tetrahydrofuran (THF) was dried over and distilled from K-Na alloy under an atmosphere of dry Argon. Compound **1**^[1] and 2-(5-bromothiophen-2-yl)acetonitrile^[2] were prepared according to literature methods. Commercially available reagents were used as received.

Synthesis of compound 2

Under an atmosphere of nitrogen, to a mixture of 4,7-dibromobenzo[*c*][1,2,5]thiadiazole (882 mg, 3.0 mmol), compound **1** (1.14 g, 2.0 mmol) and $\text{Pd}(\text{PPh}_3)_4$ (0.05 eq) in THF (30 mL) was added a solution of K_2CO_3 (2.0 M, 5 mL) in water, the reaction solution was refluxed overnight.

Then the reaction mixture was poured into water, extracted with CH_2Cl_2 and dried over anhydrous Na_2SO_4 . The crude product was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1) to give compound **2** as a red oil (910 mg, 69%). ^1H NMR (300 MHz, CDCl_3) δ (ppm): 7.88 (d, $J = 7.5$ Hz, 1H, ArH), 7.74 (d, $J = 9.0$ Hz, 2H, ArH), 7.51 (d, $J = 7.5$ Hz, 1H, ArH), 7.13 (d, $J = 8.4$ Hz, 4H, ArH), 7.03 (d, $J = 8.7$ Hz, 2H, ArH), 6.86 (d, $J = 8.4$ Hz, 4H, ArH), 3.95 (t, $J = 6.6$ Hz, 4H, $-\text{OCH}_2-$), 1.81-1.74 (m, 4H, $-\text{CH}_2-$), 1.47 (s, br, 4H, $-\text{CH}_2-$), 1.37-1.35 (m, 8H, $-\text{CH}_2-$), 0.92 (t, $J = 6.6$ Hz, 6H, $-\text{CH}_3$).

Synthesis of compound **3**

Under an atmosphere of nitrogen, to a mixture of compound **2** (882 mg, 3.0 mmol) and $\text{Pd}(\text{PPh}_3)_4$ (0.05 eq) in THF (20 mL) was added a solution of K_2CO_3 (2.0 M, 5 mL) in water. Then the reaction solution was heated at 45 °C for 0.5 h, followed by the addition of a solution of 5-formylthiophene-2-boronic acid (248 mg, 1.59 mmol) in THF (10 mL). The resulting reaction solution was refluxed overnight. Then the reaction mixture was poured into water, extracted with CH_2Cl_2 and dried over anhydrous Na_2SO_4 . The crude product was purified by column chromatography on silica gel (petroleum ether/ ethyl acetate/ $\text{CHCl}_3 = 15/1/ 5$) to give compound **3** as a red oil (360 mg, 49%). ^1H NMR (300 MHz, CDCl_3) δ (ppm): 9.97 (s, 1H, $-\text{CHO}$), 8.21 (d, $J = 3.9$ Hz, 1H, ArH), 8.04 (d, $J = 7.8$ Hz, 1H, ArH), 7.85-7.82 (m, 3H, ArH), 7.71 (d, $J = 7.8$ Hz, 1H, ArH), 7.13 (d, $J = 8.7$ Hz, 4H, ArH), 7.04 (d, $J = 8.7$ Hz, 2H, ArH), 6.86 (d, $J = 8.4$ Hz, 4H, ArH), 3.95 (t, $J = 6.6$ Hz, 4H, $-\text{OCH}_2-$), 1.81-1.74 (m, 4H, $-\text{CH}_2-$), 1.47 (s, br, 4H, $-\text{CH}_2-$), 1.35 (s, br, 8H, $-\text{CH}_2-$), 0.91 (m, 6H, $-\text{CH}_3$).

Synthesis of dye LI-75

Under an atmosphere of nitrogen, a solution of compound **3** (230 mg, 0.33 mmol), cyanoacrylic acid (85 mg, 1.0 mmol) and piperidine (0.1 mL) in the mixture of THF (4 mL) and CH₃CN (8 mL) was refluxed overnight. Then the mixture was poured into a solution of HCl (1.0 M, 20 mL) and stirred for 0.5 h. The resulting solution was extracted with CH₂Cl₂ and dried over anhydrous Na₂SO₄. The crude product was purified by column chromatography on silica gel (CHCl₃/ methanol = 20/1) to give dye **LI-75** as a black solid (190 mg, 75%). ¹H NMR (300 MHz, DMSO-*d*₆) δ (ppm): 8.33 (s, 1H, -CH=), 8.24-8.22 (m, 2H, ArH), 7.94 (d, *J* = 3.6 Hz, 1H, ArH), 7.90-7.83 (m, 3H, ArH), 7.08 (d, *J* = 8.7 Hz, 4H, ArH), 6.93 (d, *J* = 8.7 Hz, 4H, ArH), 6.86 (d, *J* = 8.4 Hz, 2H, ArH), 3.94 (t, *J* = 6.3 Hz, 4H, -OCH₂-), 1.72-1.68 (m, 4H, -CH₂-), 1.41 (s, br, 4H, -CH₂-), 1.30 (s, br, 8H, -CH₂-), 0.88 (t, *J* = 6.6 Hz, 6H, -CH₃). ¹³C NMR (100 MHz, DMSO-*d*₆) δ (ppm): 164.82, 155.87, 153.22, 152.16, 149.04, 146.40, 144.30, 139.74, 137.59, 137.07, 133.28, 130.04, 127.72, 127.54, 127.28, 126.36, 123.14, 118.66, 117.93, 115.62, 103.33, 68.01, 31.50, 29.20, 25.68, 22.53, 14.25. HRMS (*m/z*): [M-H]⁻ calcd for C₄₄H₄₃N₄O₄S₂, 755.2726; Found, 755.2731. Anal. Calcd for C₄₄H₄₄N₄O₄S₂: C, 69.81; H, 5.86; N, 7.40. Found: C, 69.51; H, 6.28; N, 7.43.

Synthesis of compound **4**

Under an atmosphere of nitrogen, a solution of compound **3** (207 mg, 0.30 mmol), methyl-4-(cyanomethyl)benzoate (279 mg, 1.59 mmol) and piperidine (0.1 mL) in the mixture of THF (4 mL) and CH₃CN (8 mL) was refluxed overnight. Then the solution was diluted with CH₂Cl₂ (50 mL), washed with water and dried over anhydrous Na₂SO₄. The crude product was purified by column chromatography on silica gel (petroleum ether/DCM = 1/2) to give

compound **4** as a red solid (220 mg, 87%). ^1H NMR (300 MHz, CDCl_3) δ (ppm): 8.23 (d, $J = 3.6$ Hz, 1H, ArH), 8.10 (d, $J = 8.7$ Hz, 2H, ArH), 8.03 (d, $J = 7.5$ Hz, 1H, ArH), 7.84 (d, $J = 8.7$ Hz, 2H, ArH), 7.79-7.68 (m, 5H, ArH and $-\text{CH}=\text{}$), 7.13 (d, $J = 9.0$ Hz, 4H, ArH), 7.05 (d, $J = 8.4$ Hz, 2H, ArH), 6.86 (d, $J = 9.0$ Hz, 4H, ArH), 3.97-3.93 (m, 7H, $-\text{OCH}_2-$ and $-\text{COOCH}_3$), 1.81-1.74 (m, 4H, $-\text{CH}_2-$), 1.47 (s, br, 4H, $-\text{CH}_2-$), 1.37-1.35 (m, 8H, $-\text{CH}_2-$), 0.92 (t, $J = 6.6$ Hz, 6H, $-\text{CH}_3$).

Synthesis of dye **LI-76**

Under an atmosphere of nitrogen, to a solution of compound **4** (127 mg, 0.15 mmol) in THF (15 mL) was added a solution of NaOH (60 mg, 1.5 mmol) in ethanol (3 mL). The resulting solution was refluxed overnight. Then the solvent was removed under reduced pressure and the solid was dissolved in CHCl_3 (50 mL). The solution was poured into a solution of HCl (1.0 M, 20 mL) and stirred for 0.5 h. The mixture was extracted with CHCl_3 and dried over anhydrous Na_2SO_4 . The crude product was purified by column chromatography on silica gel ($\text{CHCl}_3/\text{methanol} = 20/1$) to give dye **LI-76** as a black solid (115 mg, 92%). ^1H NMR (300 MHz, $\text{DMSO}-d_6$) δ (ppm): 8.42 (s, 1H, $-\text{CH}=\text{}$), 8.20-8.18 (m, 2H, ArH), 8.02 (d, $J = 8.4$ Hz, 2H, ArH), 7.88-7.82 (m, 6H, ArH), 7.06 (d, $J = 8.7$ Hz, 4H, ArH), 6.92 (d, $J = 9.0$ Hz, 4H, ArH), 6.84 (d, $J = 8.7$ Hz, 2H, ArH), 3.93 (t, $J = 6.3$ Hz, 4H, $-\text{OCH}_2-$), 1.72-1.67 (m, 4H, $-\text{CH}_2-$), 1.41 (s, br, 4H, $-\text{CH}_2-$), 1.30 (s, br, 8H, $-\text{CH}_2-$), 0.88 (s, br, 6H, $-\text{CH}_3$). ^{13}C NMR (100 MHz, $\text{DMSO}-d_6$) δ (ppm): 167.18, 155.78, 153.44, 152.32, 149.00, 144.64, 139.73, 137.77, 137.62, 135.66, 135.08, 133.31, 130.88, 130.20, 129.73, 127.58, 127.49, 127.08, 126.88, 126.12, 125.19, 123.39, 118.73, 117.79, 115.33, 105.86, 68.02, 31.43, 29.15, 25.60, 22.45, 14.06. HRMS (m/z): $[\text{M}]^+$ calcd for $\text{C}_{50}\text{H}_{48}\text{N}_4\text{O}_4\text{S}_2$,

832.3117; Found, 832.3166. Anal. Calcd for C₅₀H₄₈N₄O₄S₂: C, 72.09; H, 5.81; N, 6.73. Found: C, 71.92; H, 6.21; N, 7.00.

Synthesis of compound 5

To a solution of compound **3** (340 mg, 0.49 mmol) and 2-(5-bromothiophen-2-yl)acetonitrile (149 mg, 0.74 mmol) in THF (6 mL) was added a solution of NaOH (40 mg, 1.0 mmol) in ethanol (2 mL). The resulting solution was stirred at room temperature overnight. The solvent was removed under reduced pressure. The residue was dissolved in CHCl₃ (50 mL), washed with water and dried over anhydrous Na₂SO₄. The crude product was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 15/1) to give compound **5** as a black solid (260 mg, 60%). ¹H NMR (300 MHz, CDCl₃) δ (ppm): 8.20 (d, *J* = 3.9 Hz, 1H, ArH), 8.01 (d, *J* = 7.8 Hz, 1H, ArH), 7.83 (d, *J* = 8.7 Hz, 2H, ArH), 7.70-7.68 (m, 2H, ArH), 7.37 (s, 1H, -CH=), 7.14-7.12 (m, 5H, ArH), 7.06-7.03 (m, 3H, ArH), 6.86 (d, *J* = 8.4 Hz, 4H, ArH), 3.95 (t, *J* = 6.6 Hz, 4H, -OCH₂-), 1.81-1.74 (m, 4H, -CH₂-), 1.47 (s, br, 4H, -CH₂-), 1.35 (s, br, 8H, -CH₂-), 0.92 (t, *J* = 6.3 Hz, 6H, -CH₃).

Synthesis of compound 6

Compound **6** was prepared according to the similar procedure as described for compound **2** (150 mg, 77%). ¹H NMR (300 MHz, CDCl₃) δ (ppm): 8.22 (d, *J* = 3.9 Hz, 1H, ArH), 8.02-8.00 (m, 3H, ArH), 7.83 (d, *J* = 7.8 Hz, 2H, ArH), 7.70-7.62 (m, 4H, ArH), 7.52 (s, 1H, -CH=), 7.36-7.35 (m, 2H, ArH), 7.12 (s, br, 4H, ArH), 7.06 (s, br, 2H, ArH), 6.86 (d, *J* = 8.7 Hz, 4H, ArH), 3.95

(s, br, 4H, -OCH₂-), 1.81-1.77 (m, 4H, -CH₂-), 1.62 (s, 9H, -CH₃), 1.47 (s, br, 4H, -CH₂-), 1.35 (s, br, 8H, -CH₂-), 0.92 (s, br, 6H, -CH₃).

Synthesis of dye **LI-77**

A solution of compound **6** (75 mg, 0.08 mmol) in the mixture of CH₂Cl₂ (10 mL) and CF₃COOH (1 mL) was stirred at room temperature overnight. Then the solvent was removed under reduced pressure. The solid was dissolved in CH₂Cl₂ (50 mL), washed with water and dried over anhydrous Na₂SO₄. The crude product was purified by column chromatography on silica gel (CHCl₃/ methanol = 20/1) to give dye **LI-77** as a black solid (66 mg, 93%). ¹H NMR (300 MHz, DMSO-*d*₆) δ (ppm): 8.14-8.11 (m, 2H, ArH), 7.99 (s, 1H, ArH), 7.94 (d, *J* = 7.2 Hz, 2H, ArH), 7.83-7.73 (m, 6H, ArH), 7.65 (s, 1H, ArH), 7.38 (s, 1H, -CH=), 7.04 (d, *J* = 8.1 Hz, 4H, ArH), 6.91 (d, *J* = 8.1 Hz, 4H, ArH), 6.82 (d, *J* = 7.8 Hz, 2H, ArH), 3.92 (s, br, 4H, -OCH₂-), 1.70 (s, br, 4H, -CH₂-), 1.40 (s, br, 4H, -CH₂-), 1.30 (s, br, 8H, -CH₂-), 0.88 (s, br, 6H, -CH₃). ¹³C NMR (100 MHz, THF-*d*₈) δ (ppm): 167.11, 157.08, 154.71, 153.61, 150.23, 145.35, 144.29, 141.14, 140.65, 139.07, 138.21, 134.90, 134.40, 132.58, 131.28, 131.12, 130.73, 129.07, 128.70, 128.53, 127.90, 127.70, 127.04, 126.50, 125.89, 124.72, 119.99, 117.26, 116.05, 103.04, 68.76, 32.60, 30.32, 26.76, 23.55, 14.42. HRMS (*m/z*): [M+H]⁺ calcd for C₅₄H₅₁N₄O₄S₃, 915.3072; Found, 915.3076. Anal. Calcd for C₅₄H₅₀N₄O₄S₃: C, 70.87; H, 5.51; N, 6.12. Found: C, 71.25; H, 5.72; N, 6.19.

Synthesis of compound **7**

Compound **7** was prepared according to the similar procedure as described for compound **5** (520 mg, 96%). ¹H NMR (300 MHz, CDCl₃) δ (ppm): 8.22 (d, *J* = 3.9 Hz, 1H, ArH), 8.02 (d, *J* = 7.8 Hz, 1H, ArH), 7.84 (d, *J* = 8.7 Hz, 2H, ArH), 7.71-7.68 (m, 2H, ArH and -CH=), 7.60-7.52 (m, 4H, ArH), 7.13 (d, *J* = 9.0 Hz, 4H, ArH), 7.05 (d, *J* = 9.0 Hz, 2H, ArH), 6.86 (d, *J* = 9.0 Hz, 4H, ArH), 3.95 (t, *J* = 6.3 Hz, 4H, -OCH₂-), 1.81-1.74 (m, 4H, -CH₂-), 1.48 (s, br, 4H, -CH₂-), 1.37-1.35 (m, 8H, -CH₂-), 0.92 (t, *J* = 6.6 Hz, 6H, -CH₃).

Synthesis of compound **8**

Compound **8** was prepared according to the similar procedure as described for compound **6** (220 mg, 98%). ¹H NMR (300 MHz, CDCl₃) δ (ppm): 8.22 (d, *J* = 3.9 Hz, 1H, ArH), 8.08 (d, *J* = 8.1 Hz, 2H, ArH), 8.02 (d, *J* = 7.2 Hz, 1H, ArH), 7.83 (d, *J* = 8.4 Hz, 2H, ArH), 7.77-7.74 (m, 4H, ArH), 7.71-7.65 (m, 5H, ArH and -CH=), 7.13 (d, *J* = 8.4 Hz, 4H, ArH), 7.05 (d, *J* = 8.4 Hz, 2H, ArH), 6.86 (d, *J* = 8.4 Hz, 4H, ArH), 3.95 (t, *J* = 6.6 Hz, 4H, -OCH₂-), 1.81-1.76 (m, 4H, -CH₂-), 1.62 (s, 9H, -CH₃), 1.47 (s, br, 4H, -CH₂-), 1.37-1.35 (m, 8H, -CH₂-), 0.92 (s, br, 6H, -CH₃).

Synthesis of dye **LI-78**

Dye **LI-78** was prepared according to the similar procedure as described for dye **LI-77** (130 mg, 92%). ¹H NMR (300 MHz, DMSO-*d*₆) δ (ppm): 8.33 (s, 1H, ArH), 8.18-8.14 (m, 2H, ArH), 8.02 (d, *J* = 8.1 Hz, 2H, ArH), 7.82 (s, br, 10H, ArH and -CH=), 7.04 (d, *J* = 8.1 Hz, 4H, ArH), 6.91 (d, *J* = 8.1 Hz, 4H, ArH), 6.83 (d, *J* = 7.8 Hz, 2H, ArH), 3.93 (t, *J* = 6.3 Hz, 4H, -OCH₂-), 1.72-1.68 (m, 4H, -CH₂-), 1.40 (s, br, 4H, -CH₂-), 1.30 (s, br, 8H, -CH₂-), 0.88 (s, br, 6H, -CH₃).
¹³C NMR (100 MHz, DMSO-*d*₆ / THF-*d*₈ = 2/1) δ (ppm): 168.16, 157.03, 154.59, 153.54,

150.18, 144.99, 144.38, 140.96, 140.71, 139.74, 135.69, 135.52, 134.74, 134.14, 131.60, 131.05, 130.82, 128.62, 128.52, 128.01, 127.51, 127.40, 127.02, 114.76, 119.79, 118.84, 116.25, 107.36, 68.82, 32.51, 30.22, 26.68, 23.48, 14.57. HRMS (m/z): $[M]^+$ calcd for $C_{56}H_{52}N_4O_4S_2$, 908.3430; Found, 908.3438. Anal. Calcd for $C_{56}H_{52}N_4O_4S_2$: C, 73.98; H, 5.76; N, 6.16. Found: C, 73.52; H, 6.08; N, 6.00.

2.3 Device fabrication

The double layer TiO_2 films were prepared according to the reported procedures.^[3] Before be immersed into the dye solutions, the films were soaked in the 0.04 M aqueous $TiCl_4$ solution at 70 °C for 30 min. After being washed with water and ethanol, they were heated at 500 °C for 30 min. Subsequently, the films were dipped into a 3×10^{-4} M $CH_3CN/t\text{-BuOH}/CHCl_3$ (V/V/V = 2/2/1) solution of the sensitizers for 24 h at room temperature. For the coadsorption, chenodeoxycholic acid (CDCA) was added. The counter electrode was made by a conducting glass substrate (FTO, 2.2 mm thickness, 9 ohms/sq, 1.25 cm \times 2.0 cm) with dropping a solution of H_2PtCl_6 in isopropyl alcohol (10 mM, 10 μ L), which was drilled two small holes to allow the injection of the liquid electrolyte, followed by heating at 400 °C for 120 min. The dye-loaded TiO_2 electrode and Pt counter electrode were assembled into a sealed sandwich-type cell with a Surlyn sheet. The redox electrolyte, which was composed of 0.05 M I_2 , 0.2 M LiI, 0.6 M DMPII, 0.1 M GuNCS and 0.5 M 4-TBP in acetonitrile, was injected into the cell through a pre-drilled hole at the counter electrode. Finally, the hole was sealed using a Surlyn sheet and a thin glass.

2.4 Photovoltaic properties measurements

Photovoltaic performances were tested under AM 1.5 G simulated sunlight (100 mW cm^{-2}) provided by a solar simulator ((Model 94023A equipped with a 450W xenon arc lamp, Newport Co.) with a 450 W xenon light source. The light intensity was determined using a Si solar cell (Model 91150, Newport Co.) as a reference. The current-voltage (J - V) curves of the DSCs were obtained by using a Keithley digital source meter (Model 2400). The electrochemical impedance spectra (EIS) measurement was measured by a Modulab XM PhotoEchem system under dark or 0.5 SUN conditions with a series of applied voltages and recorded over a frequency range from 0.1 Hz to 100 KHz (amplitude: 25 mV). Intensity-modulated photovoltage spectroscopy (IMVS) and charge extraction (CE) method measurement were performed using the same electrochemical workstation. Incident photon-current conversion efficiency (IPCE) was recorded in a solar cell QE/IPCE measurement system equipped (Model 2931-C equipped with a 300W xenon arc lamp, Newport Co.) under irradiation with a motorized monochromator (Oriel). All the measurements mentioned above were taken under ambient conditions.

2.5 IMVS and CE measurements

Intensity-modulated photovoltage spectroscopy (IMVS) was measured by a Modulab XM PhotoEchem system at an open-circuit condition. A white light, controlled by a Labview system, was utilized to obtain the photovoltaic response induced by the modulated light. The modulated light was driven with a 10% AC perturbation current superimposed on a DC current, in the frequency ranging from 10^{-1} to 10^5 Hz. The CE was performed with the same monochromatic light source. First, the solar cell was illuminated at an open-circuit condition for 10 s to attain a

steady state; then the light source was switched off, and simultaneously, the device was switched to a short-circuit condition for the extraction of the charges generated at that light intensity.

3. Reference

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