

Supporting Information

Enhancement of Photovoltaic Performance by Utilizing Readily Accessible Hole Transporting Layer of Vanadium (V) Oxide Hydrate in a Polymer-Fullerene Blend Solar Cell

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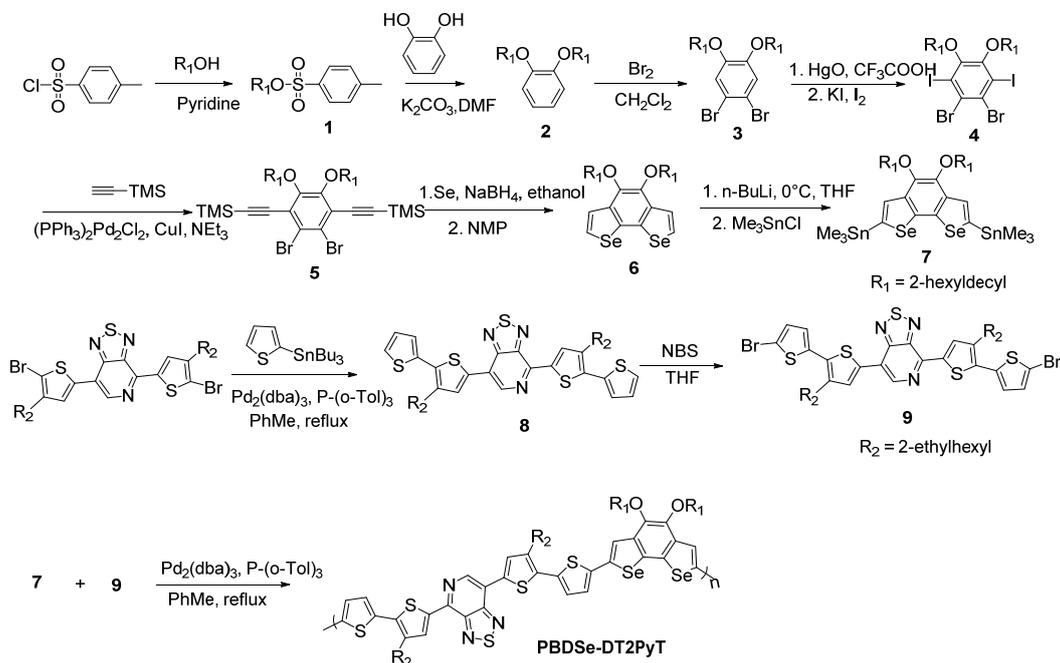
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General Information on Experiments.

^1H NMR and ^{13}C NMR spectra were recorded on a Mercury plus-400 MHz spectrometer at 400 MHz and 100 MHz in CDCl_3 , respectively. The mass spectroscopy of compounds was performed on a Bruker Biflex III MALDI-TOF mass spectrometer. The molecular weights of the polymer were determined by gel permeation chromatography (GPC) with 1,2,4-trichlorobenzene as the eluent at 120 °C and polystyrenes as standards to calibrate on an Agilent PL-GPC 220 system.

UV-Vis absorption spectra were obtained with a Shimadzu UV-1750 spectrophotometer. Cyclic voltammograms (CV) measurements were obtained in a solution of 0.1 M tetrabutylammonium hexafluorophosphate (Bu_4NPF_6) in anhydrous acetonitrile as the supporting electrolyte on a CHI 660D Electrochemical Analyzer using a standard three-electrode configuration, with a glass carbon disk as the working electrode, a platinum wire as the counter electrode and Ag/AgNO_3 (0.01 M AgNO_3 in supporting electrolyte) electrode as the reference electrode. The measurement of the polymer film drop-casted on the working electrode from a 2 mg mL^{-1} solution in CHCl_3 was done at a constant scan rate of 100 mV s^{-1} at room temperature under highly purified N_2 . The HOMO level of PBDS_e-DT2PyT was estimated through the formula of $\text{HOMO} = - (E_{\text{ox, onset}} + 4.8) \text{ eV}$, the LUMO level was determined by the energy difference between the optical band gap and the HOMO value.

Synthesis and characterization of the polymer PBDSe-DT2PyT



Scheme S1. The synthetic route to the monomers and the polymer of **PBDSe-DT2PyT**.

2-hexyldecyl 4-methylbenzenesulfonate (1)

To a flame-dried 250 mL round-bottom flask was added *p*-toluenesulfonylchloride (38.12 g, 200 mmol). After treated through three vacuum-nitrogen refilling cycles, 2-hexyl-1-decanol (48.48 g, 200 mmol) and pyridine (200 mL) were added under nitrogen. The mixture was stirred for 1 hour at $0^\circ C$, and then stirred for another 2 hours at room temperature. Afterwards, the resulted mixture was diluted with 220 mL of aqueous 2 mol L^{-1} HCl solution and extracted with ethyl acetate. The obtained organic layer was dried over magnesium sulfate ($MgSO_4$) and concentrated under reduced pressure. The crude product was further purified by gel column chromatography using petroleum ether/ethyl acetate (100/1, v/v) as the eluent to afford 70.42 g of oil product (yield, 88.8%). 1H NMR (400 MHz, $CDCl_3$) δ : 7.78 (d, $J = 8.4$ Hz, 2H), 7.34 (d, $J = 8.8$ Hz, 2H), 3.90 (d, $J = 5.2$ Hz, 2H), 2.44 (s, 3H), 1.60-1.54 (m, 1H), 1.23 (m, 24H), 0.93-0.80 (m, 6H). ^{13}C NMR(100 MHz, $CDCl_3$) δ : 144.38, 132.64, 129.58, 127.70, 72.54, 37.40, 31.70, 31.55, 30.42, 29.60, 29.31, 29.26, 29.09, 26.26, 26.19, 22.49, 22.42, 21.34, 13.88. MALDI-TOF MS for $C_{23}H_{40}O_3S$: calcd. 396.3; found, 419.1 ($M + Na^+$).

1,2-Bis(2-hexyldecyloxy)benzene (2)

Catechol (1.10g, 10 mmol), 2-hexyldecyl 4-Methylbenzenesulfonate (9.12 g, 23 mmol), potassium carbonate (5.8 g, 42 mmol) and N, N- dimethylformamide (DMF) (20 mL) were added to the flask, the reaction mixture was heated to reflux under N₂. After 32 hours, the reactant was cooled to room temperature and poured into 100 mL of water solution, and then extracted with petroleum ether (50 mL × 2). The extracted organic phase was dried over MgSO₄ and concentrated in vacuum. Further purification by gel column chromatography using petroleum ether as the eluent afforded 3.23 g of the target product (yield, 57.8%). ¹H NMR (400 MHz, CDCl₃) δ: 6.86 (s, 4H), 3.84 (d, *J* = 5.6 Hz, 4H), 1.80 (m, 2H), 1.45-1.26 (m, 48H), 0.879 (t, *J* = 6.6 Hz, 12H). ¹³C NMR(100 MHz, CDCl₃) δ: 149.64, 120.77, 113.82, 71.80, 38.24, 31.95, 31.40, 30.14, 29.80, 29.69, 29.42, 26.91, 14.08. MALDI-TOF MS for C₃₈H₇₀O₂: calcd. 558.5; found, 581.5 (M + Na⁺).

1, 2-Dibromo-bis(2-hexyldecyloxy)benzene (3)

To a solution of 1, 2-bis(2-hexyldecyloxy)benzene (1.66 g, 2.89 mmol) in 5 mL of CH₂Cl₂ solution was added dropwise bromine (0.93 g, 5.8 mmol), the mixture was stirred at room temperature. After 6 hours, the resulted reaction solution was diluted with aqueous Na₂S₂O₃ solution, the organic phase was then washed with water solution, dried over MgSO₄ and concentrated in vacuum. Further purification by a short silica gel column using petroleum ether as the eluent afforded 2.05 g of the target product (yield, 98%). ¹H NMR (400 MHz, CDCl₃) δ: 7.04(s, 2H), 3.79 (d, *J* = 5.6 Hz, 4H), 1.78 (m, 2H), 1.36-1.20 (m, 48H), 0.879 (t, *J* = 6.8 Hz, 12H). ¹³C NMR(100 MHz, CDCl₃) δ: 149.37, 117.61, 114.39, 72.05, 38.01, 31.92, 31.88, 31.28, 30.06, 29.72, 29.63, 29.38, 26.83, 22.69, 14.09. MALDI-TOF MS for C₃₈H₆₈Br₂O₂: calcd. 714.4; found, 720.4 (M + Li⁺).

1, 2-Dibromo-3,6-diiodo-4,5-bis(2-hexyldecyloxy)benzene (4)

1, 2-Dibromo-bis(2-hexyldecyloxy)benzene (2.05 g, 2.86 mmol) and HgO (1.86 g, 8.58 mmol) were dissolved in 15 mL of CF₃COOH. The mixture was heated to reflux for 6 hours. After cooled to room temperature, KI (2.17 g, 13 mmol), I₂ (3.32 g, 13 mmol) and 60 mL of water solution were directly added to the reaction mixture of. The mixture was stirred vigorously and heated at 80 °C. After 24 hours, the mixture was cooled to room temperature, diluted with 10% NaHSO₃ solution (100 mL) and then extracted with petroleum ether (50 mL × 3). The organic phase was separated, washed with water (50 mL × 3) and dried over MgSO₄. Further purification by a short silica gel column using petroleum ether as the eluent afforded 2.29 g of the target product (yield, 83%). ¹H NMR (400 MHz, CDCl₃) δ: 3.82 (d, *J* = 6.4 Hz, 4H), 1.89 (m, 2H), 1.4-1.2 (m, 48H), 0.95-0.84 (m, 12H). ¹³C NMR(100 MHz, CDCl₃) δ: 152.35, 126.04, 102.78, 77.53, 38.97, 31.91,

31.13, 30.10, 29.77, 29.65, 29.37, 26.86, 22.69, 14.13. MALDI-TOF MS for $C_{38}H_{66}Br_2I_2O_2$: calcd. 966.2; found, 965.7 (M^+)

1, 2-Dibromo-4,5- bis(2-hexyldecyloxy)-3, 6-bis(trimethylsilylethynnyl)benzene (5)

1, 2-Dibromo-3, 6-diiodo-4, 5-bis(2-hexyldecyloxy)benzene (0.96 g, 1 mmol), CuI (0.006 g, 0.03 mmol), $Pd(PPh_3)_2Cl_2$ (0.021 g, 0.03 mmol), trimethylsilylacetylene (0.25 g, 2.5 mmol) and 5 mL Et_3N were sequentially added to the flask under nitrogen. The mixture was heated at 60 °C for 24 hours. Then, 5% aq. HCl (50 mL) solution was poured into the mixture and then extracted with petroleum ether (20 mL \times 3). The combined organic layer was washed with water (20 mL \times 3) and dried over anhydrous magnesium sulfate. The crude product was further purified by gel column chromatography using petroleum ether as the eluent to afford pure 0.63 g of product (yield 70%). 1H NMR (400 MHz, $CDCl_3$) δ : 3.93 (d, J = 6.0 Hz, 4H), 1.80 (m, 2H), 1.28 (s, 48H), 0.88 (m, 12H), 0.27 (s, 18H). ^{13}C NMR (100 MHz, $CDCl_3$) δ : 153.94, 132.20, 122.35, 106.20, 99.85, 77.92, 39.12, 31.92, 31.12, 30.13, 29.80, 29.67, 29.37, 27.00, 22.69, 14.08, -0.28. MALDI-TOF MS for $C_{48}H_{84}Br_2O_2Si_2$: calcd. 906.4; found, 930.3 ($M + Na^+$).

4, 5-bis(2-hexyldecyloxy)benzo[2,1-b:3,4-b']diselenophene (6)

To a suspension of selenium powder (0.63 g, 8 mmol) in 15 mL of ethanol solution was added sodium borohydride (0.30 g, 8 mmol) under N_2 . The mixture was stirred vigorously at 0 °C for 2 hours. 1, 2-Dibromo-4, 5-bis(2-hexyldecyloxy)-3, 6-bis(trimethylsilylethynnyl)benzene (1.82 g, 2 mmol) in 200 mL of N-methyl-2-pyrrolidone (NMP) solution was added into the mixture, and then ethanol was removed by distillation. The mixture was refluxed for another 2 hours. The reaction mixture was diluted with 200 mL of saturated aqueous NH_4Cl solution and extracted with CH_2Cl_2 (50 mL \times 3). The separated organic phase was washed with water (100 mL \times 2), dried over $MgSO_4$ and concentrated under reduced pressure. The crude product was further purified by gel column chromatography using petroleum ether as the eluent to afford 1.07 g of product (yield 70%). 1H NMR (400 MHz, $CDCl_3$) δ : 7.87 (d, J = 5.6 Hz, 2H), 7.76 (d, J = 5.6 Hz, 2H), 4.02 (d, J = 5.6 Hz, 4H), 1.85 (m, 2H), 1.29 (s, 48H), 0.89 (m, 12H). ^{13}C NMR (100MHz, $CDCl_3$) δ : 145.49, 136, 133.39, 126.92, 125.46, 77.52, 39.20, 31.92, 31.32, 30.15, 29.82, 29.66, 28.38, 26.94, 26.91, 22.70, 14.12. MALDI-TOF MS for $C_{42}H_{70}O_2Se_2$: calcd. 766.4; found, 767.4($M + H^+$).

2, 7-Bis(trimethyltin)-4, 5-bis(2-hexyldecyloxy)benzo [2, 1-b:3, 4-b']diselenophene (7)

A solution of 2.4M n-BuLi(2.1 mL, 5.5 mol) in pentane was added dropwise to the solution of 4, 5-bis(2-hexyldecyloxy)benzo[2, 1-b:3, 4-b']diselenophene (1.53 g, 2 mmol)

in 10 mL of anhydrous THF solution at 0 °C under nitrogen. After stirring for 1 hour, a solution of 1M Me₃SnCl (3.0 mL, 3 mmol) in hexane was added and reacted for another 2 hours. The reaction was quenched by adding 50 mL of the distilled water and the organic phase was extracted with 20 mL of ether solution and dried over MgSO₄. Removal of the solvent under reduced pressure afforded 1.96 g of oil product (yield, 90%). ¹H NMR (400 MHz, CDCl₃) δ: 7.90 (s, 2H), 4.02 (d, *J* = 6 Hz, 4H), 1.83 (m, 2H), 1.29 (m, 48H), 0.89 (m, 12H), 0.42 (m, 18H). ¹³C NMR (100MHz, CDCl₃) δ: 144.79, 143.67, 137.42, 136.81, 133.39, 77.42, 39.23, 31.93, 31.41, 30.24, 29.89, 29.70, 29.41, 27.03, 26.98, 22.73, 22.69, 14.11, -8.02. MALDI-TOF MS for C₄₈H₈₆O₂Se₂Sn₂: calcd. 1094.3; found, 1094.5(M⁺).

Compound 8

4, 7-Bis(5-bromo-4-(2-ethylhexyl)thiophen-2-yl)-[1, 2, 5]thiadiazolo[3, 4-c]pyridine was synthesized according to the reported literature (Angew. Chem. Int. Ed. 2010, 49, 7992-7995).

To a flame-dried flask with a condenser were added 4, 7-bis(5-bromo-4-(2-ethylhexyl)thiophen-2-yl)-[1, 2, 5]thiadiazolo[3,4-c]pyridine (1.36 g, 2 mmol), Pd₂(dba)₃ (20 mg, 0.02 mmol), P(o-Tol)₃ (40 mg, 0.13 mmol) and 80 mL of anhydrous THF solution under N₂. 2-(Tributylstannyl) thiophene (1.87 g, 5 mmol) was then added to the above mixture. The resulted mixture was heated under reflux overnight under N₂. After removal of the solvent under reduced pressure, the crude product was further purified by gel column chromatography using CH₂Cl₂ as the eluent to afford 1.13 g of product (yield 82%). ¹H NMR (400 MHz, CDCl₃) δ: 8.78 (s, 1H), 8.48 (s, 1H), 7.93 (s, 1H), 7.42-7.16 (m, 4H), 7.15-7.05 (m, 2H), 2.8(m, 4H), 1.76(m, 2H), 1.40-1.11 (m, 16H), 0.87 (m, 12H). ¹³C NMR (100MHz, CDCl₃) δ: 154.35, 147.78, 145.28, 140.06, 139.51, 138.52, 136.64, 135.66, 135.13, 133.92, 133.08, 131.22, 127.41, 127.30, 126.50, 126.33, 126.19, 126.09, 125.92, 125.61, 119.59, 39.95, 39.84, 33.62, 33.36, 32.36, 29.65, 28.01, 26.75, 25.59, 23.06, 17.28, 14.14, 13.58, 10.68. MALDI-TOF MS for C₃₇H₄₃N₃S₅: calcd.689.2; found, 690.0 (M⁺).

Compound 9

To a solution of compound **8** (0.828 g, 1.2 mmol) in 20 mL of THF solution was added NBS (0.448 g, 2.5 mmol) in small portions. The reaction mixture was stirred in the dark for 2 h at room temperature. After removal of the solvent under reduced pressure, the crude product was purified by gel column chromatography using toluene as the eluent to afford 0.912 g of product (yield 90%). ¹H NMR (400 MHz, CDCl₃) δ: 8.75 (s, 1H), 8.44 (s, 1H), 7.90 (s, 1H), 7.07-6.95 (m, 4H), 2.79-2.69 (m, 4H), 1.73 (m, 2H), 1.43–1.19 (m,

16H), 0.90-0.82 (m, 12H). ^{13}C NMR (100MHz, CDCl_3) δ : 154.22, 147.66, 145.12, 140.45, 140.11, 139.93, 138.86, 137.40, 137.18, 135.58, 135.02, 134.27, 132.09, 131.10, 130.24, 130.13, 126.57, 126.40, 119.46, 112.72, 112.25, 39.97, 39.87, 33.67, 32.36, 28.51, 25.62, 23.07, 14.16, 10.72. MALDI-TOF MS for $\text{C}_{37}\text{H}_{41}\text{Br}_2\text{N}_3\text{S}_5$: calcd. 845.03; found, 846.7 ($\text{M} + \text{H}^+$).

PBDSe-DT2PyT

To a two-necked round bottom flask were added 2, 7-Bis(trimethyltin)-4, 5-bis(2-hexyldecyloxy)benzo [2, 1-b:3, 4-b']diselenophene (338 mg, 0.31mmol), monomer 9 (262 mg, 0.31mmol), $\text{Pd}_2(\text{dba})_3$ (8 mg, 0.009mmol), $\text{P}(\text{o-Tol})_3$ (16 mg, 0.054mmol) and 10 mL of anhydrous toluene under nitrogen, then the mixture was degassed and filled with nitrogen three times. After refluxed for 48 hours, the mixture was cooled to room temperature and added dropwise into 100 mL of anhydrous methanol solution. The crude solid was filtered and washed with acetone. The polymer was further purified by Soxhlet extraction with acetone, hexane, CH_2Cl_2 , CHCl_3 . The solution of polymer in CHCl_3 was concentrated and reprecipitated in 10 mL of methanol solution, and then the resulting solid was filtered and dried under vacuum to get the polymer 280 mg (yield 64 %). GPC: $M_n = 20.37\text{kDa}$; $M_w = 43.67\text{kDa}$; PDI = 2.8.

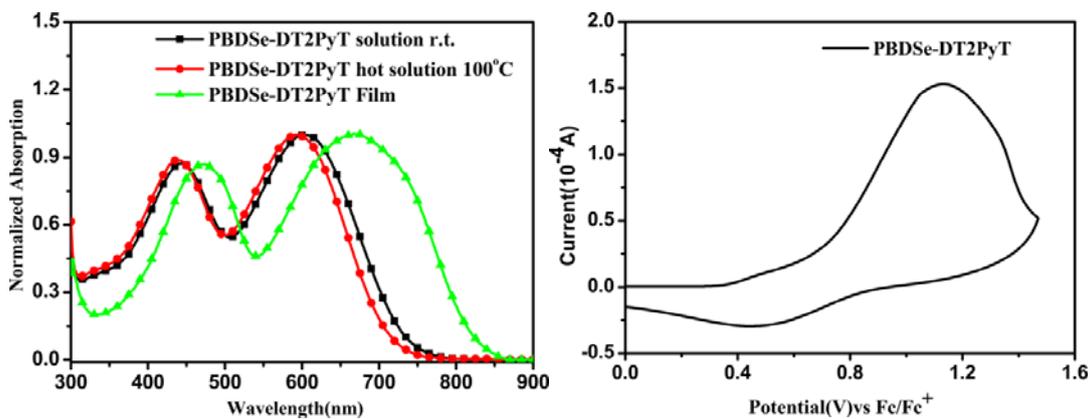


Figure S1. UV-Vis absorption spectra of **PBDSe-DT2PyT** in toluene solution at room temperature (r.t.) and 100 °C and in thin film (left) and Cyclic voltammogram curve of **PBDSe-DT2PyT** in 0.1 M Bu_4NPF_6 solution in anhydrous methylene dichloride at a scan rate of 0.1 V s^{-1} under N_2 (right).

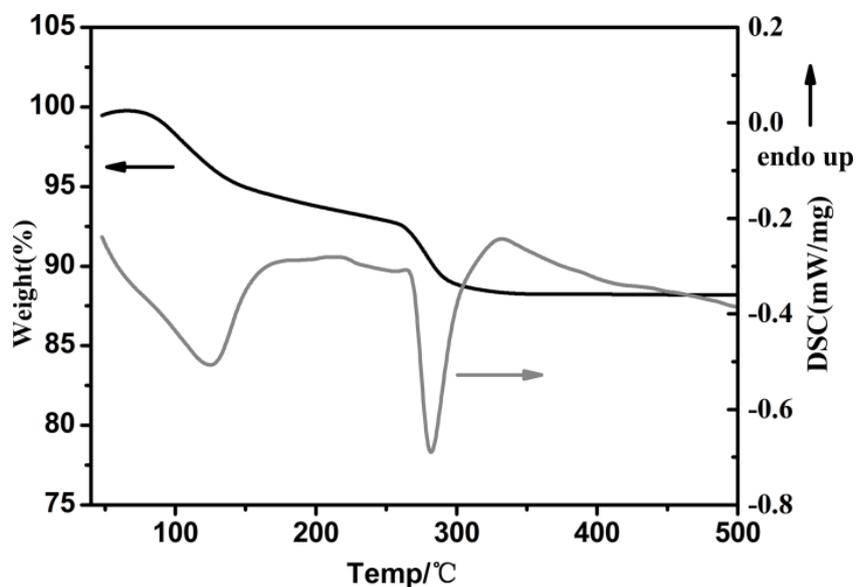


Figure S2. TGA and DSC curves of $\text{V}_2\text{O}_5 \cdot n\text{H}_2\text{O}$ at a heating rate of $10 \text{ }^\circ\text{C min}^{-1}$ under nitrogen.

Table S1. The average electrical parameters of PBdSe-DT2PyT:PC₇₁BM solar cells with various D:A ratio and corresponding optimized thicknesses of active layer processed with and/or without DIO additive when using PEDOT:PSS as HTL.

PBdSe-DT2 PyT:PC ₇₁ BM	DIO (% v/v)	<i>d</i> (nm)	<i>V</i> _{oc} (V)	<i>J</i> _{sc} (mA cm ⁻²)	<i>FF</i>	PCE(%)
1:0.8	0	98	0.71±0.01	9.75±0.14	0.50±0.01	3.47±0.16
	0	90	0.67±0.01	12.53±0.12	0.54±0.01	4.55±0.11
	0.1	90	0.61±0.01	11.45±0.20	0.58±0.01	4.04±0.15
1:1	0.25	85	0.57±0.01	11.92±0.25	0.54±0.02	3.68±0.24
	0.5	91	0.61±0.01	11.25±0.16	0.52±0.02	3.56±0.26
	1	83	0.56±0.01	10.39±0.18	0.57±0.02	3.28±0.15
1:2	0	85	0.66±0.01	8.34±0.30	0.53±0.01	2.92±0.16

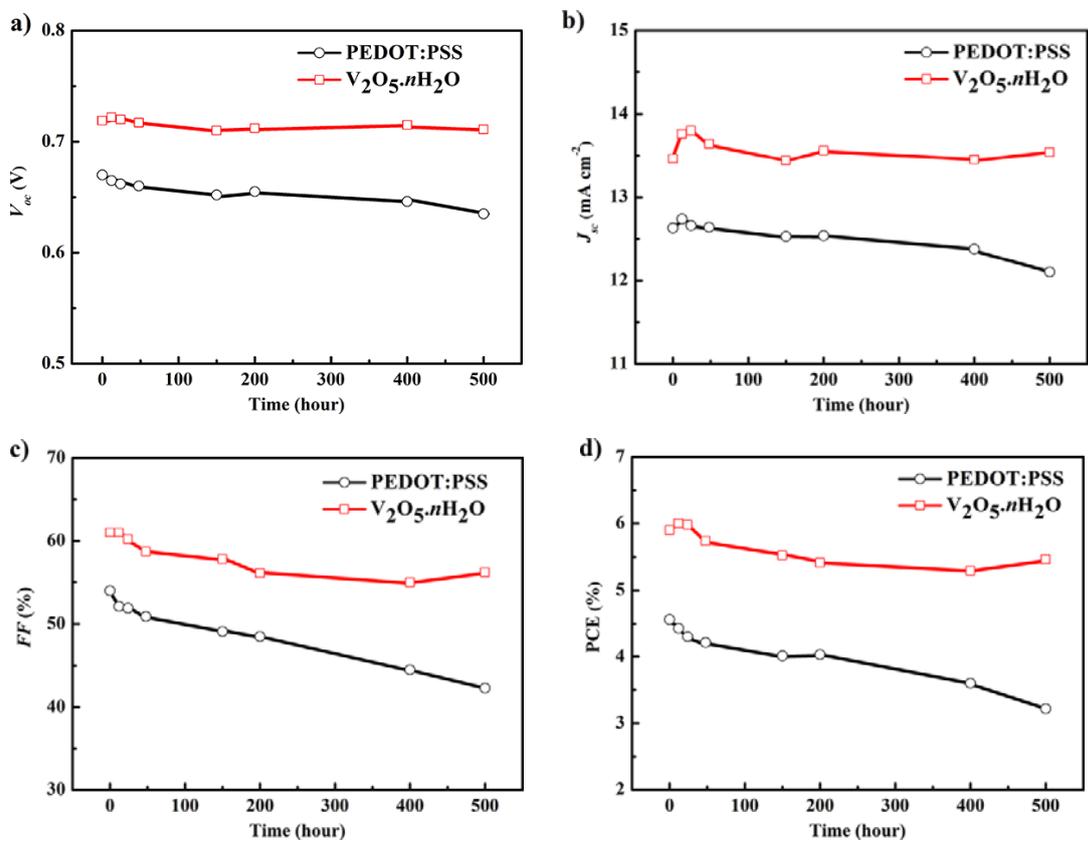


Figure S3. Variation of the solar cell parameters of the devices with different HTLs of PEDOT:PSS and $V_2O_5 \cdot nH_2O$ respectively from fabrication day to 500 hours stored in a glovebox; (a) J_{sc} , (b) V_{oc} , (c) FF, and (d) Power conversion efficiency (PCE).

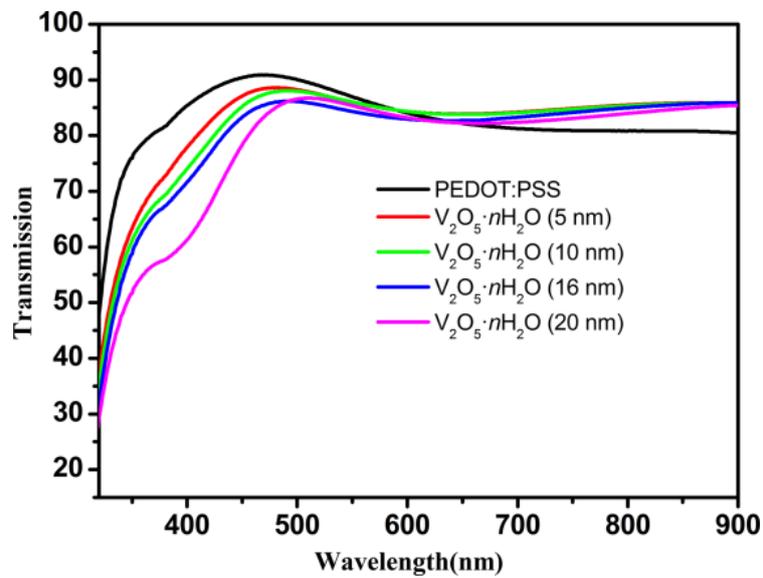


Figure S4. Optical transmittance spectra of PEDOT:PSS and V₂O₅·nH₂O.

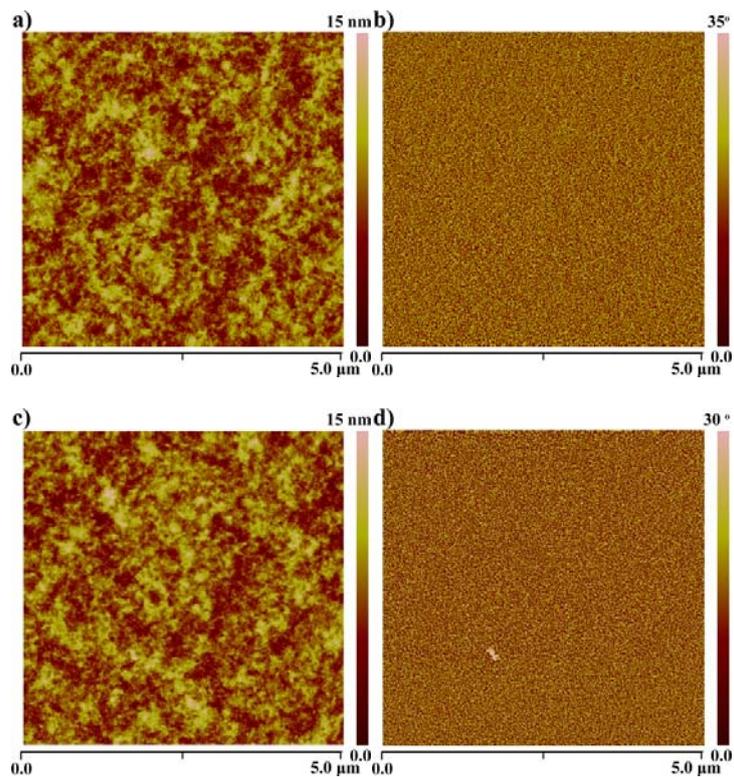


Figure S5. AFM height and phase images of blend films with PEDOT:PSS (a-b) and V₂O₅·nH₂O (c-d).

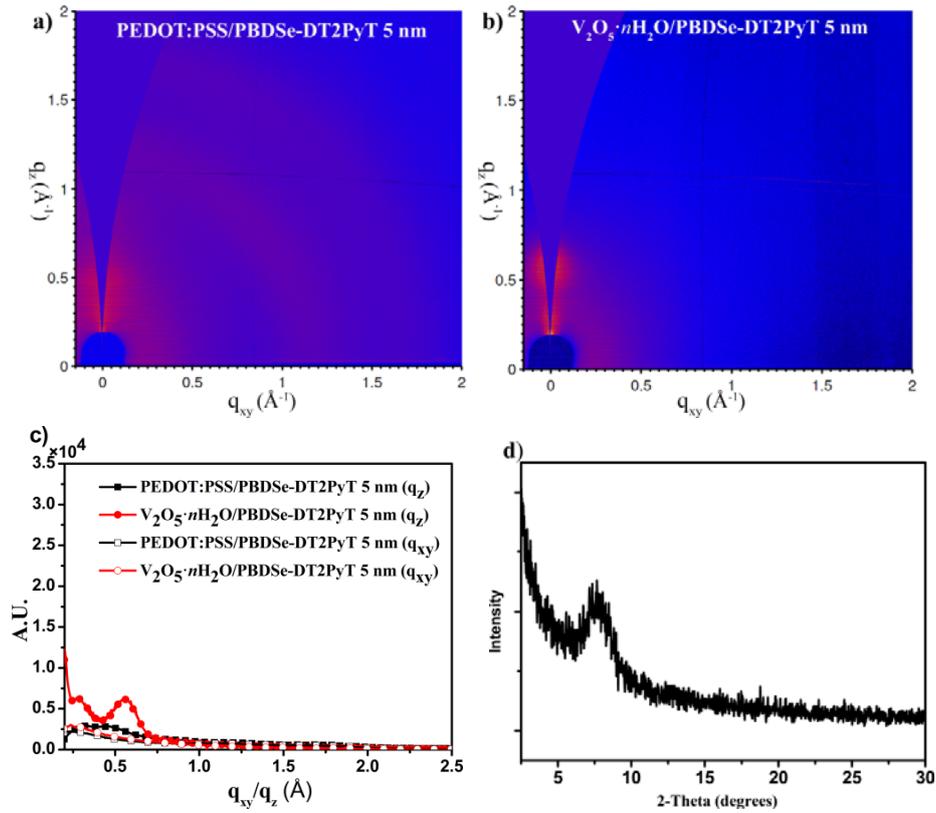


Figure S6. 2D GIWAXS pattern (a-b), out-of-plane and in-plane (c) data of pristine PBDSe-DT2PyT films based on PEDOT:PSS and $V_2O_5 \cdot nH_2O$, and (d) XRD spectra of $V_2O_5 \cdot nH_2O$ annealed at 120 °C.

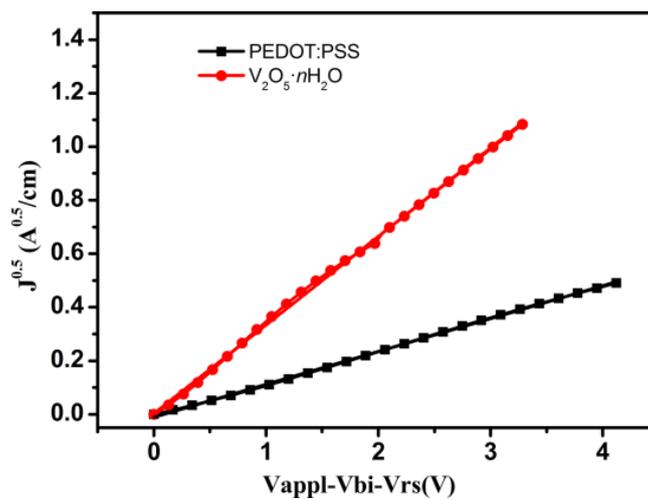


Figure S7. $J^{0.5}$ vs $V_{appl} - V_{bi} - V_{rs}$ plots for hole-only devices with PEDOT:PSS and $V_2O_5 \cdot nH_2O$.

The dependence of related parameters calculated from the J - V curve on FF . (refer to Qi, B.; Wang, J., Fill factor in organic solar cells. *Physical Chemistry Chemical Physics* **2013**, *15* (23), 8972-8982)

For a practical BHJ solar cell under illumination, the equivalent circuit equation can be approximately expressed by the Shockley equation:

$$J = \frac{R_{sh}}{R_s + R_{sh}} \left\{ J_s \left[\exp\left(\frac{q(V - JR_s)}{nkT}\right) - 1 \right] + \frac{V}{R_p} \right\} - J_{ph} \quad (S1)$$

Where J is the measured current, R_{sh} is the shunt resistance, R_s is the series resistance, J_s is dark reverse saturation current, q is the elementary charge, V is the applied bias, n is the diode ideality factor, k is the Boltzmann constant, T is the ambient temperature and J_{ph} is the photocurrent.

Based on such equivalent circuit model, the FF can be approximately expressed as:

$$FF = FF_s \left(1 - \frac{v_{oc} + 0.7}{v_{oc}} \frac{FF_s}{r_{sh}} \right) \quad (S2)$$

While

$$FF_0 = \frac{v_{oc} - \ln(v_{oc} + 0.72)}{v_{oc} + 1} \quad FF_s = FF_0 (1 - 1.1r_s) + \frac{r_s^2}{5.4} \quad (S3)$$

Where FF_0 is the fill factor of ideal solar cells, FF_s is the fill factor of solar cells that only have R_s , r_s ($R_s J_{sc} / V_{oc}$) and r_{sh} ($R_{sh} J_{sc} / V_{oc}$) are the normalized resistances, respectively. The deduced values of FF based on equations are summarized in Table S2.

Table S2. Related parameters in equation S2 and S3 deduced from the equivalent circuit model.

HTL layer	v_{oc}	r_s	r_{sh}	FF_0	FF_s	<i>Predicated FF</i>
PEDOT:PSS	11.76	0.18	6.0	0.72	0.58	0.52
$V_2O_5 \cdot nH_2O$	15.12	0.17	10.6	0.77	0.63	0.59

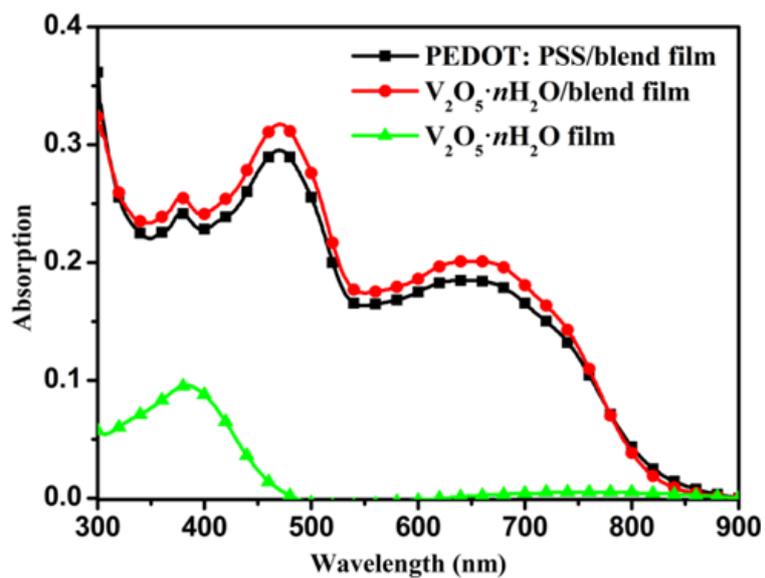


Figure S8. UV-vis absorption spectra of V₂O₅·nH₂O film and PBDS_e-DT2PyT:PC₇₁BM blend films based on PEDOT:PSS and V₂O₅·nH₂O.