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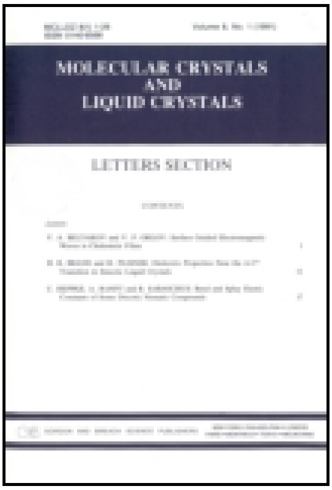

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Synthesis and Photovoltaic Properties of Moderate Band Gap Diketopyrrolopyrrole Based Small Molecules for Solution Processed Organic Solar Cells

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Synthesis and Photovoltaic Properties of Moderate Band Gap Diketopyrrolopyrrole Based Small Molecules for Solution Processed Organic Solar Cells

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Two new small molecules, based on diketopyrrolopyrrole core flanked by cyanothiophene units (CN-DPP-CN, CN-TH-DPP-TH-CN) were synthesized using CuCN and palladium catalyzed Suzuki coupling and explored in organic solar cells (OSCs). The HOMO/LUMO energy levels of CN-DPP-CN, CN-TH-DPP-TH-CN having moderate band gap of 1.83 eV and 1.44 eV were estimated to be −5.63/−3.84 eV, −5.20/−3.75 eV respectively. The device efficiency was found to be 0.013, 0.21% for CN-DPP-CN, CN-TH-DPP-TH-CN respectively as donors for BHJ solar cells. When CN-DPP-CN (0.05%) was added in P3HT:$PC_{60}BM$ *device, its PCE was enhanced to 2.45% from 2.08% signifying its ability to be used as potential n-type additive.*

Keywords Diketopyrrolopyrrole; cynothiophene; organic solar cells; solution process; small molecules

1. Introduction

Along with the development of organic solar cells (OSCs) with desired power conversion efficiency (PCE) of 10% to be attained, it is important to develop organic materials with appropriate design and understand the mechanism of charge generation, recombination and mobility in OPV devices [1]. Thus far, with the continuous efforts in novel molecular designs, device optimization and device structure improvements, PCE upto 6∼8 % has been reported for polymer materials [2]. Compared with well explored polymeric counterparts, however, small molecules are less investigated. Though a 7% highest PCE has been reported for few small molecules [3], their efficiency is still inferior than that polymer solar cells. Considering the ease of synthesis and purification along with monodisperity of small molecules, they are undoubtedly the best alternatives for the polymer based solar cells. Among various small molecules, diketopyrrolopyrrole (DPP) was firstly observed by Farnum et al. in 1974 and largely explored by Iqbal's groups [4]. Since it is known to have remarkable fluorescence and good stability, it was exclusively used in paints, inks,

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plastics, lacquers and printing. The aryl group attached to DPP chromophore unit endows it with the ability to possess colors with different shades [5]. Moreover, DPP based materials show strong light absorption capability and are known to have high charge carrier mobilities in organic field effect transistors [6]. Recently, DPP has gained immense interest in OSCs [7]. Hence in an attempt to explore the DPP based small molecules, we report here synthesis, characterization and application in OPVs of two rational designs of DPP small molecules with A-D-A architechture, each having a different number of thiophene spacer groups in molecules incorporated with cyano group at the peripheral position. PCEs of 0.013 and 0.21% were achieved when CN-DPP-CN, CN-TH-DPP-TH-CN molecules were used as donors with $PC_{70}BM$. CN-DPP-CN delivered very poor PCE of 0.00025%, as an acceptor with P3HT. However, a more significant improvement in PCE to 2.45 % was achieved, when CN-DPP-CN was used as n-type additive in P3HT: $PC_{60}BM$ based bulk heterojunction (BHJ) devices.

2. Experimental Section

Materials and Synthesis

3,6-bis(5-bromothiophen-2-yl)-2,5-bis(2-ethylhexyl)pyrrolo[3,4-c]pyrrole-1,4(2H,5H)-dione (Br-DPP-Br) was synthesized by the procedure reported by our group previously [8]. 5-cyanothiophen-2-ylboronic acid, CuCN and tetrakis (triphenylphosphine)-palladium(0) [$Pd(PPh_3)_4$] were purchased from sigma Aldrich. All other chemicals were reagent grade and were obtained from commercial sources and used as-received.

5,5′-(2,5-bis(2-ethylhexyl)-3,6-dioxo-2,3,5,6-tetrahydropyrrolo[3,4-c]pyrrole-1,4-diyl) dithiophene-2-carbonitrile (CN-DPP-CN). To the suspension of Compound 1 (0.1 gm, 0.146 mmol) and Dry DMF (10 mL) in N_2 atmospere, CuCN (1.18 gm, 13.18 mmol) was added, resulting mixture was heated at 140°C for 8 hr. After water was added to quench the reaction and organic layer was extracted with ethyl acetate. Organic layer was washed with water and dried over $MgSO_4$. Removal of solvent afforded the crude which was further purified by column chromatography eluting with hexane/CH_2Cl_2 (3:2) to produce CN-DPP-CN as red solid (80 mg, 95%). ^1H NMR (400 MHz, $CDCl_3$): δ 8.80 (d, 2H), 7.66 (d, 2H), 3.85 (d, 4H), 1.71 (m, 2H), 1.39–1.17 (m, 18 H), 0.91 (t, 12H). ^{13}C NMR ($CDCl_3$, 100MHz): δ 161.0, 139.4, 137.7, 134.9, 134.6, 113.6, 113.2, 110.3, 46.1, 39.3, 30.0, 28.1, 23.4, 22.9, 13.9, 10.3. IR: ν (cm^{-1}), 3441, 3085, 2926, 2343, 2211, 1674, 1552, 1454, 1398, 1258, 1095.

5′,5″-(2,5-bis(2-ethylhexyl)-3,6-dioxo-2,3,5,6-tetrahydropyrrolo[3,4-c]pyrrole-1,4-diyl) di-2,2′-bithiophene-5-carbonitrile (CN-TH-DPP-TH-CN). A degassed 5 mL H_2O solution of K_2CO_3 (2.5M) was added to a degassed 15 mL THF solution of 5-cyanothiophen-2-ylboronic acid (0.138 gm, 0.879 mmol), $Pd(PPh_3)_4$ (34 mg, 0.029 mmol) and compound 1 (0.2 gm, 0.29 mmol) under argon atmophere. After the mixture was heated to 80°C for 12 hr, solvent was removed under vaccum. Column chromatography eluting with hexane/CH_2Cl_2 (2:3) affords CN-TH-DPP-TH-CN as blue solid (100 mg,46%). ^1H NMR (400 MHz, $CDCl_3$): δ 8.83 (d, 2H,), 7.51 (d, 2H), 7.34 (d, 2H), 7.21 (d, 2H), 3.96 (d, 4H), 1.75 (m, 2H), 1.48-1.18 (m, 18 H), 0.8 (t, 12H). ^{13}C NMR ($CDCl_3$, 100MHz): δ 161.15, 142.96, 139.45, 138.42, 136.04, 130.33, 126.91, 124.68,109.10,46.07, 39.31, 30.27, 28.46, 23.68, 23.05, 14.03, 10.54. IR: v (cm^{-1}), 3441, 2955, 2352, 2216, 1655, 1552, 1440, 1262, 1095, 1024.

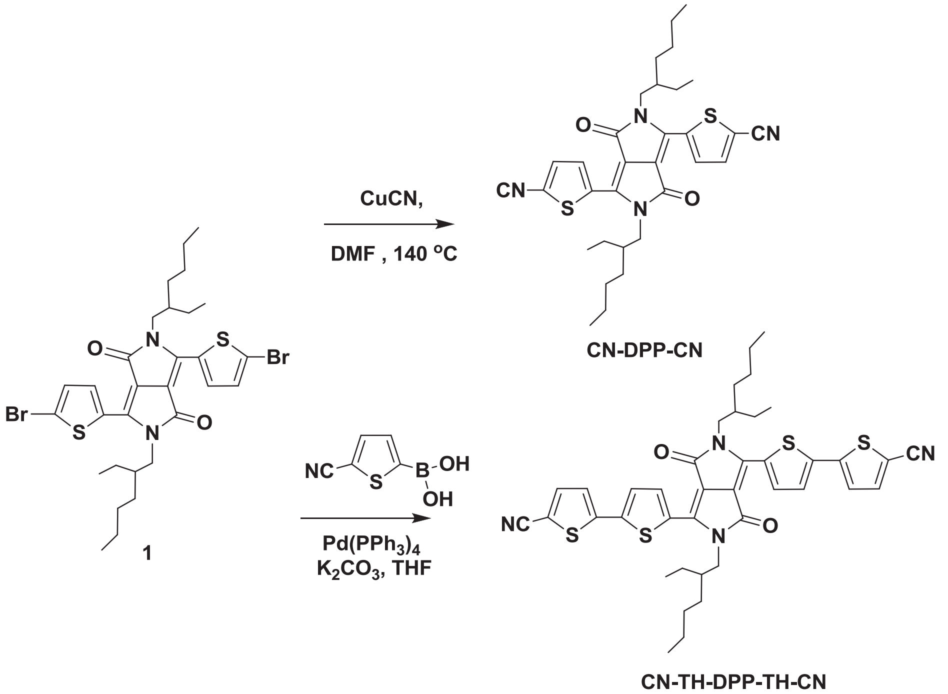
Characterization and Device Fabrication

^{1}H spectra was recorded on JEOL FT-NMR (400 MHz) spectrometer using $CDCl_3$ as the solvent. Chemical shift were reported as δ values (ppm) relative to the internal standard tetramethylsilane. The UV-vis absorption spectral films or solutions were obtained using a shimadzu UV-2550 spectrometer. Photoluminescene (PL) spectra of the thin films were obtained using a Jasco FP- 6500 spectrometer. Cyclic Voltametry (CV) measurements were performed on a VersaSTAT3 (METEK) under argon at a scan rate of 50 mV s^{-1} at room temperature, wherein a platinum wire and Ag/AgCl were used as the counter and reference electrodes respectively. The reference electrode was calibrated with Fc/Fc$^+$ as an external standard. The samples were prepared in a chloroform solution with 0.10 M tetrabutylammonium hexafluorophosphate (n-Bu_4NPF_6) as the electrolyte. The chemical bonding characteristics of the samples were characterized by Fourier transform infrared spectroscopy (FT-IR, Shimadzu FT-IR-8700) using a standard potassium bromide pellet technique. Each FT-IR spectrum was collected after 32 scans at a resolution of 2 cm^{-1} from 4000 to 600 cm^{-1}.

All of the OPV cells were prepared on a commercial indium-tin oxide (ITO)-coated glass substrates with a structure of glass/ ITO/PEDOT:PSS(small molecule): $PC_{70}BM$/LiF/Al, according to the general procedure reported previously [9] in brief, the patterened ITO-covered glass substrates were cleaned with deionized water, acetone, and isopropyl alcohol using ultrasonication, followed by treatment with UV and O_3. PEDOT:PSS (AI 4083, H.C.Starck) was spin-coated (2600 rpm, 40 s) onto the cleaned ITO glass and dried (with and without annealing) and transferred into glovebox filled with Nitrogen. Blends of small molecule and PCBM with different weight ratios were solubilized overnight in a chloroform (concentration: 10 mg mL^{-1}) solution, filtered through a 0.45- μm poly(tetrafluroethylene) filter, and spin coated on PEDOT: PSS layer of the ITO. The resulting films were dried at room temperature for 20 mins and then under vaccum for 12 hr. The devices were completed by the deposition of a 0.5-nm layer of LIF and a 120-nm layer of aluminum.These layers were thermally evaporated at a pressure of 1×10^{-6} Torr at room temperature. The active area of every device was 9 mm^2. The current-voltage (J-V) characteristic of the photovoltaic devices were measured in dark and under white-light illumination at AM 1.5G using a solar simulator (Newport) at 100 mW cm^{-2}, adjusted with a standard PV reference (2×2 cm) and monocrystalline silicone solar cell (calibrated at NREL, USA) with Keithley 2400 source-measure unit. The external quantum efficiency (EQE) was determine using Polaronix K 3100 spectrometer.

3. Results and Discussion

Synthetic routes for the small molecule are depicted in Scheme 1. Starting from the commercially available thiophene-2-carbonitrile, intermediate (1) was synthesized with four step reactions. CN-DPP-CN was obtained in 95% yield as red solid by reacting compound (1) with CuCN at 140 °C in dry DMF. CN-TH-DPP-TH-CN was obtained by $Pd(PPh_3)_4$ assisted Suzuki coupling reaction between (1) and 5-cyanothiophen-2-ylboronic acid in 46% yield as blue solid. UV- visible absorption spectra for CN-DPP-CN and CN-TH-DPP-TH-CN in chloroform and as thin films are shown in Fig. 1. Chloroform solutions of both the molecules exhibit broad optical absorption band covering the region around



Scheme 1. Synthetic routes for CN-DPP-CN and CN-TH-DPP-TH-CN.

300- 700 nm, with the longest wavelength absorption maximum at 584 nm and 586 nm respectively. Compared with the solution absorption spectra, the thin film absorption spectra were broadened and red shifted by 45 nm in CN-DPP-CN and 116 nm in CN-TH-DPP-TH-CN. It also shows two well define peaks, consistent with more rigid and ordered structure in the solid state. The red shift and broader absorption could be attributed to well ordered π—π^* stacking of molecules [10]. Additional red shift around 71 nm in case of CN-TH-DPP-TH-CN could be attributed with increased conjugation length afforded by two additional thiophene moieties. CN-TH-DPP-TH-CN possesses a narrow optical bandgap of 1.44 eV due to its largest π – conjugation system, while bandgap of CN-DPP-CN is 1.83 eV.

From the CV measurement, the HOMO energy levels of CN-DPP-CN and CN-TH-DPP-TH-CN are estimated to be −5.63 and −5.20 eV respectively. Conjugated polymers, especially DPP-containing molecules, are known to possess higher-lying HOMOs (∼5.00 eV), which is expected for structures having more effective conjugation [11]. Comparing with polymer, CN-DPP-CN has a low-lying HOMO of −5.63 eV, which is common for small conjugated molecule [12]. The additional thiophene unit in CN-TH-DPP-TH-CN which is extending the molecular conjugation is responsible for raising the HOMO to −5.20eV, as compared with CN-DPP-CN. Note that low-lying HOMOs are generally correlated with large open-circuit voltage (V_{OC}) in bulk heterojunction (BHJ) OPV system. LUMO is predominantly localized at cyano group and found to be −3.84 and −3.75 eV. There is difference of −0.09eV in LUMO values of two molecules. This difference is attributed to the additional thiophene in CN-TH-DPP-TH-CN, which diminishing the electron withdrawing ability of cyano group from central core, rising the LUMO. The electrochemical bandgap of two molecules are 1.79 and 1.45 eV respectively, which are comparable to their respective optical bandgap.

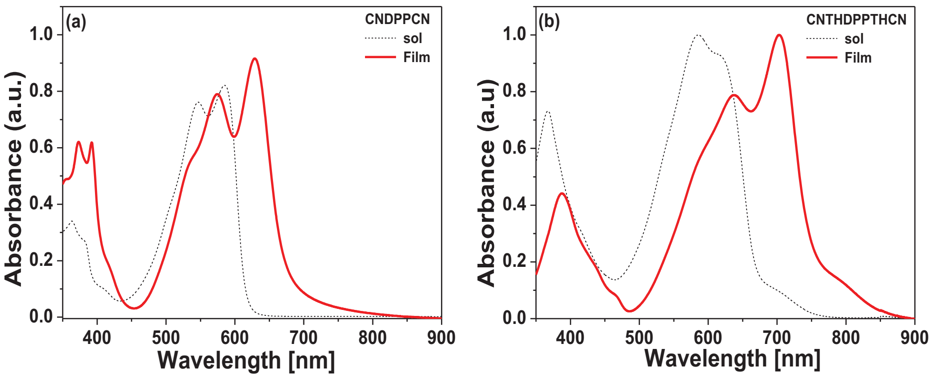


Figure 1. UV-vis absorption spectra of (a) CN-DPP-CN and (b) CN-TH-DPP-TH-CN in chloroform and solid state.

Devices containing these small molecules, CN-DPP-CN and CN-TH-DPP-TH-CN, as donors along with $PC_{70}BM$ as an acceptor in BHJ active having ratios of 1:1 and 1:2 (w/w) in chloroform were fabricated. The photovoltaic characteristics of all devices are summarized in Table 1. It is observed that OSCs containing CN-TH-DPP-TH-CN as a donor exhibited better device performance with maximum PCE of 0.21 with J_{sc} of 1.03 mA cm^{-2}, V_{oc} of 0.8 V, and FF of 0.25. This result reveals that J_{sc} of OSCs with CN-TH-DPP-TH-CN is many orders higher in comparison to OSCs with CN- DPP-CN. This is very obvious that the bandgap of CN-TH-DPP-TH-CN is comparatively lower and it can harvest more solar photons in the longer wavelength than CN-DPP-CN molecule [9]. We also fabricated OSCs with annealing their BHJ active layers at 90°C in order to further improve device performance. Although J_{sc} was slightly enhanced in CN-DPP-CN based devices, unfortunately, V_{oc} was drastically reduced after annealing for all devices. To obtain a better understanding of the different photovoltaic performance, AFM measurements

Table 1. Performance summary of photovoltaic devices of CN-DPP-CN and CN-TH-DPPTH-CN as donors with $PC_{70}BM$

Active layer	J_{sc} (mA cm^{-2})	V_{oc} (V)	FF	η (%)
CN-DPP-CN:PC_{70}BM (1:1) No annealing	0.053	0.69	0.23	0.0086
CN-DPP-CN:PC_{70}BM (1:1) 90°C annealing	0.060	0.37	0.27	0.0060
CN-DPP-CN:PC_{70}BM (1:2) No annealing	0.081	0.61	0.27	0.013
CN-DPP-CN:PC_{70}BM (1:2) 90°C annealing	0.133	0.23	0.32	0.0098
CN-TH-DPP-TH-CN:PC_{70}BM (1:1) No annealing	0.75	0.77	0.28	0.16
CN-TH-DPP-TH-CN:PC_{70}BM (1:1) 90°C annealing	0.40	0.22	0.29	0.027
CN-TH-DPP-TH-CN:PC_{70}BM (1:2) No annealing	1.03	0.80	0.25	0.21
CN-TH-DPP-TH-CN:PC_{70}BM (1:2) 90°C annealing	0.78	0.48	0.27	0.10

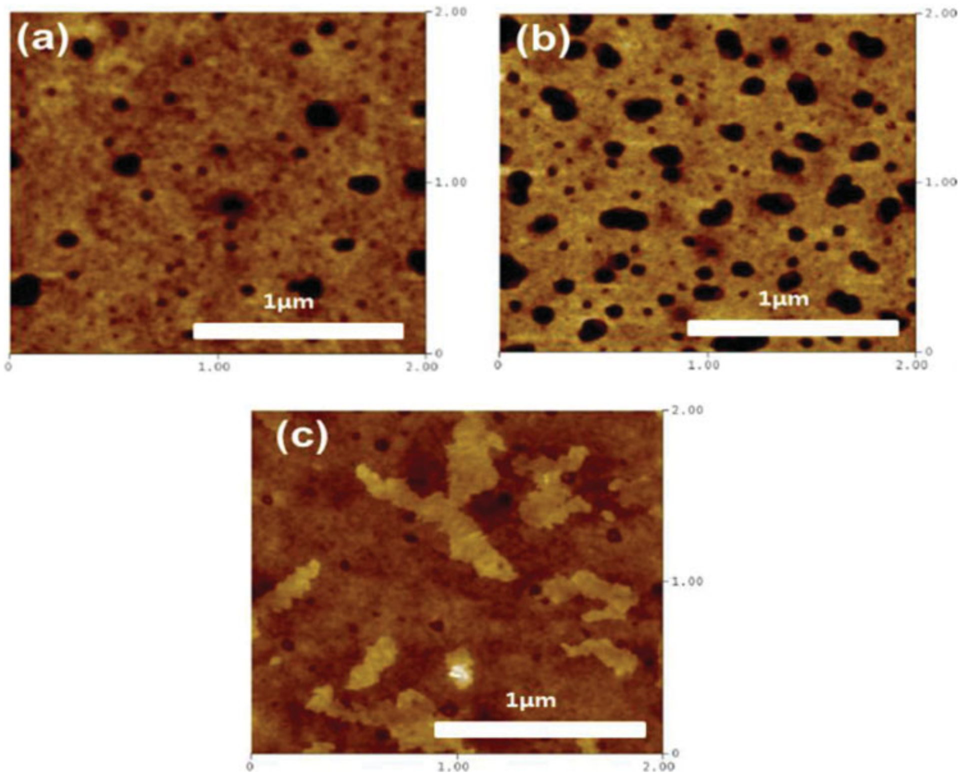


Figure 2. AFM images (2 μm × 2 μm) of (a) CN-TH-DPP-TH-CN: $PC_{70}BM$ (1:2) without annealing (b) with 90° annealing; and (c) P3HT: CN-DPP-CN (1:1).

were performed to examine the surface morphology of the devices. Figures 2a—b show AFM topography of CN-TH-DPP-TH-CN: $PC_{70}BM$ (1:2). Both sample show very poor miscibility with $PC_{70}BM$, more severely in the annealed sample, which is resulting in large phase aggregations throughout the surfaces. The increased phase aggregation induced a decrease J_{sc} and FF due to decrease in the D/A interface and high surface roughness.

Since the LUMO of CN-DPP-CN molecule is near to that of PCBM, CN-DPP-CN can also be used as an acceptor material. In order to explore this possibility, we carried out a PL quenching study of P3HT with CN-DPP-CN. It was observed that the PL emission of P3HT in the chloroform solution was almost quenched when the CN-DPP-CN was added (1:1), which suggest that the photo-excited electrons of the P3HT are effectively transferred to CN-DPP-CN; hence CN-DPP-CN is expected to be a good acceptor in P3HT based solar cell devices. However, the devices containing CN-DPP-CN as an acceptor with P3HT were not successful and photovoltaic performance of these devices were much inferior to those containing CN-DPP-CN as a donor (see result of P3HT:CN-DPP-CN (1:1) device in Table 2). AFM study revealed that low device performance may be due to very severe phase aggregations occurred in the P3HT:CN-DPP-CN film (Fig. 2c).

The potential use of CN-DPP-CN as an n-type additive was also tested in P3HT:$PC_{60}BM$ BHJ devices. Photovoltaic devices containing ternary mixture of P3HT:$P_{60}BM$:CN-DPP-CN for active layer with chlorobenzene are fabricated to investigate the role of CN-DPP-CN as an n-type additive. The J-V curves of devices containing

Table 2. Performance summary of photovoltaic devices of P3HT: CN-DPP-CN and P3HT:PC$_{60}$BM: CN-DPP-CN

Active layer	J_{sc} (mA cm^{-2})	V_{oc} (v)	FF	η (%)
P3HT:CN-DPP-CN (1:1) No annealing	0.0031	0.52	0.19	0.00025
P3HT: PC$_{60}$BM (1:1) 90°C annealing	5.08	0.64	0.64	2.08
P3HT:PC$_{60}$BM:CN-DPP-CN (1:1:0.05) No annealing	2.04	0.68	0.43	0.60
P3HT:PC$_{60}$BM:CN-DPP-CN (1:1:0.05) 90°C annealing	7.50	0.61	0.54	2.45
P3HT:PC$_{60}$BM:CN-DPP-CN (1:1:0.2) No annealing	1.52	0.65	0.13	0.13
P3HT:PC$_{60}$BM:CN-DPP-CN (1:1:0.2) 90°C annealing	3.97	0.60	0.22	0.52

P3HT:PC$_{60}$BM with different ratios of CN-DPP-CN (0.05 and 0.2) are shown in Fig. 3a and their performance is summarized in Table 2. Considering the high CN-DPP-CN additive loading of 0.2 weight ratio showed detrimental effect on the performance of P3HT/PC$_{60}$BM devices, which showed PCE of 0.13%, while device fabricated with 0.05 weight ratio delivered the PCE of 0.60% with higher J_{sc} of 2.04 mA cm^{-2}. In order to further improve the PCE, devices were annealed at 90°C. J_{sc} dramatically increased to 7.50 mA cm^{-2}, resulting in PCE of 2.45%, which is better than that of device without CN-DPP-CN additive (2.08%). The EQE of the devices are shown in Fig. 3b. P3HT:PC$_{60}$BM:CN-DPP-CN(1:1:0.05) blended device shows photoconversion efficiencies from 300 to 700 nm, with a maximum EQE of more than 50% at 550 nm, suggesting that CN-DPP-CN additive contributed to photocurrent generation by an effective electron transfer from P3HT through CN-DPP-CN to PCBM, considering LUMOs of CN-DPP-CN and PC$_{60}$BM in P3HT-PC$_{60}$BM device.

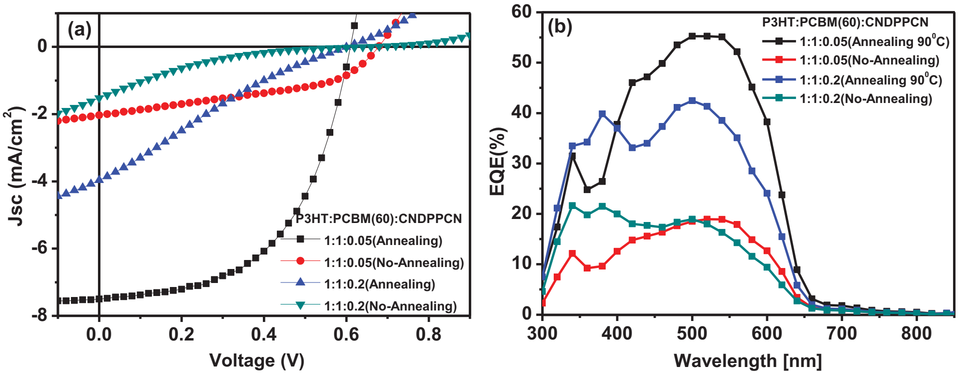


Figure 3. (a) The *J-V* curves and (b) External quantum efficiency of P3HT: CN-DPP-CN and P3HT:PC$_{60}$BM: CN-DPP-CN photovoltaic devices.

4. Conclusions

In summary, we synthesized two new cyano peripheral DPP based small molecules, CN-DPP-CN and CN-TH-DPP-TH-CN with A-D-A architecture. These molecules have been employed in BHJ devices to investigate their photovoltaic properties as donors, an acceptor and n-type additive. As donor materials, CN-DPP-CN and CN-TH-DPP-TH-CN delivered about 0.013% and 0.21% PCE respectively. Further CN-DPP-CN was used as an acceptor due to its similar LUMO value with PCBM. However, it delivered very low PCE of 0.00025 % due to the bad film morphology. It was found that, when CN-DPP-CN was used as an n-type additive with 0.05 weight ratio with P3HT:PC_{60}BM BHJ device, it increase the PCE of devices from 2.08 % to 2.45 %, indicating CN-DPP-CN could be a promising n-type additive in polymer based solar devices.

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