

# Spin-Coated Small Molecules for High Performance Solar Cells

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Organic solar cells (OSCs) have attracted significant attention as a clean and competitive renewable energy source due to their attractive features such as low-cost, light weight, solution processability and high mechanical flexibility.<sup>[1–4]</sup> Benchmark power conversion efficiencies (PCEs) of 10% or higher have been predicted if a suitable low bandgap donor material can be designed and implemented.<sup>[5]</sup> More recently, bulk heterojunction (BHJ) OSCs using solution-processed small molecules as the donor have attracted great attention.<sup>[6–8]</sup> This long-time but recently increased interest lies in the fact that solution-processed small molecule based OSCs have numerous advantages, such as relatively simple synthesis and purification methods, monodispersity and well defined structures, high open circuit voltage and charge carrier mobilities, and better batch-to-batch reproducibility.<sup>[6–8]</sup> However, solution-processed small molecule OSCs have not met such high expectations as those of their polymeric counterparts due to their limited PCEs. In most cases, small molecule devices using solution processing always seem to have poorer film quality than that of their polymeric counterparts in BHJ OSCs.<sup>[6,7]</sup> It is thus expected that better PCE could be achieved if the intrinsic poor film quality and morphology in BHJ architecture could be improved. However, in order to achieve this, careful molecule design has to be carried out to address many factors simultaneously, including the material's solar light absorption associated with its highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) positions, mobility, film forming quality, electronic band structure and morphology compatibility with the acceptors, and so on. Indeed, several families of solution processed small molecules, such as oligothiophenes,<sup>[9]</sup>

triarylaminines,<sup>[10,11]</sup> diketopyrrolopyrroles (DPP),<sup>[12]</sup> squaraines,<sup>[13,14]</sup> merocyanine,<sup>[15]</sup> and dipyrromethene boron difluoride (BODIPY)<sup>[16]</sup> derivatives, have offered great promise in this direction. Although still lower than the PCE of their polymeric counterparts (~6–8%),<sup>[17–21]</sup> the highest published PCE of 4.4% for solution-processed small molecule/fullerene derivative based BHJ OSCs was recently achieved by a University of California Santa Barbara group using a DPP-thiophene derivative.<sup>[12]</sup>

Push-pull chromophores involving electron-donating and electron-withdrawing groups have been widely investigated for OSC molecules.<sup>[8]</sup> This type of molecular architecture can lower the material bandgap and extend the absorption spectrum toward longer wavelengths, and at the same time, the HOMO and LUMO levels can be tuned effectively.<sup>[11,22]</sup> Oligothiophenes with well defined structures possess highly delocalized  $\pi$ -electrons along the molecular backbone and are well known as one of the most effective hole-transporting materials. Oligothiophenes with a push-pull structure have been widely investigated in solution processed BHJ OSCs and PCEs of 1.7–3.7% have been achieved.<sup>[8]</sup> In addition, in the field of organic electronic materials, the choice of solubilizing group and the attachment strategy is gaining growing attention.<sup>[23,24]</sup> Carefully designing and controlling the position and density of different alkyl side or end chains can not only increase solubility of materials in organic solvents for spin-casting devices but also improves their packing structure and solid-state miscibility with fullerenes.<sup>[23,24]</sup> Continuing our pursuit of small molecules for high photovoltaic performance, herein, we have designed and synthesized a series of thiophene-based small molecules end-capped with electron-withdrawing alkyl cyanoacetate groups (DCAE7T, DCAO7T and DCAEH7T, Scheme 1) and investigated the correlation between these different end groups and their BHJ device performance. Using the simple solution spin-coating fabrication process, DCAO7T/PC<sub>61</sub>BM ([6,6]-phenyl-C<sub>61</sub>-butyric acid methyl ester) based OSCs exhibit a PCE as high as 5.08% without any special treatment, which is, to our knowledge, the highest efficiency for solution processed small molecule based BHJ OSCs reported so far.

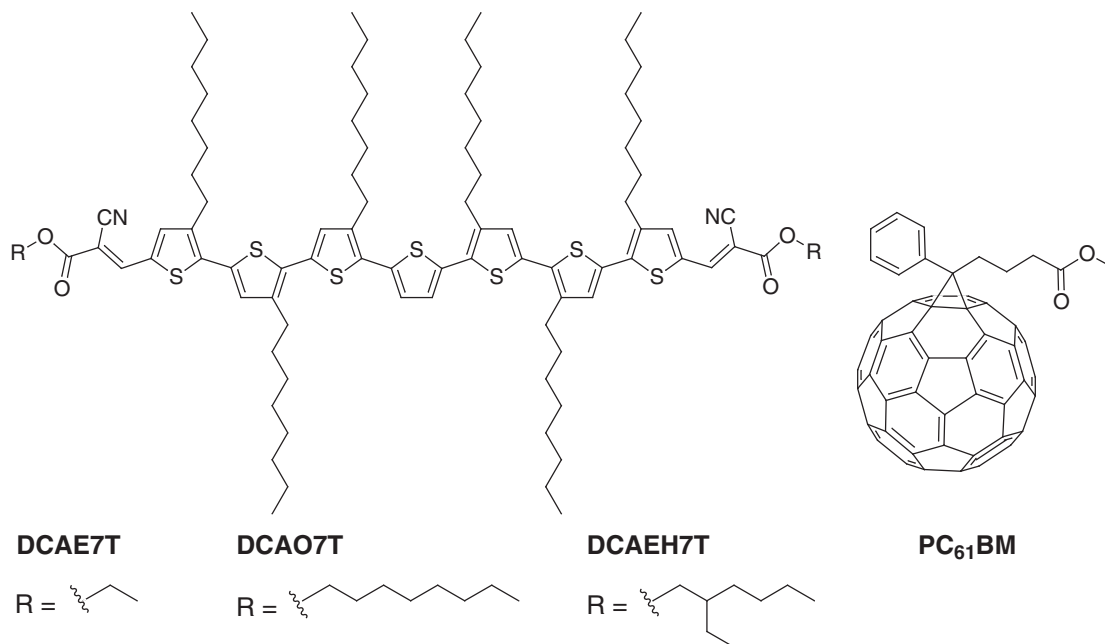
The synthesis of these compounds was carried out using a Knoevenagel condensation, and the detailed procedure is shown in the Supporting Information. As shown in Figure 1a, DCAE7T, DCAO7T and DCAEH7T have similar absorption maxima (ca. 492 nm) in CHCl<sub>3</sub> and the corresponding absorption coefficients are  $7.6 \times 10^4$ ,  $6.3 \times 10^4$ ,  $6.0 \times 10^4$  m<sup>-1</sup> cm<sup>-1</sup>, respectively. Absorption spectra of thin films (Figure 1b) show an obvious broadening and bathochromic shift (98 nm for DCAE7T, 88 nm for DCAO7T and 77 nm for DCAEH7T) of the bands compared to the solution spectra. The absorption

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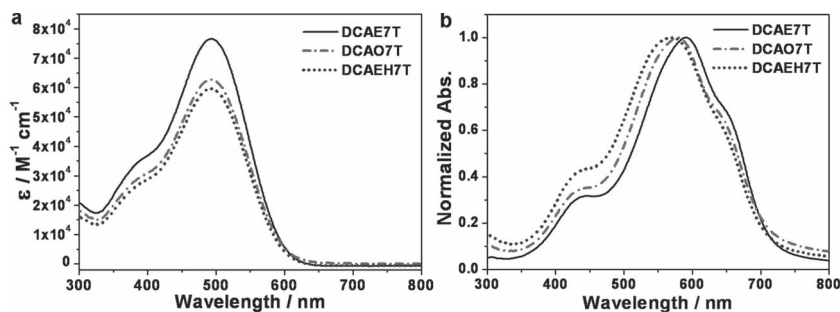


**Scheme 1.** Structure of DCAE7T, DCAO7T, DCAEH7T and PC<sub>61</sub>BM.

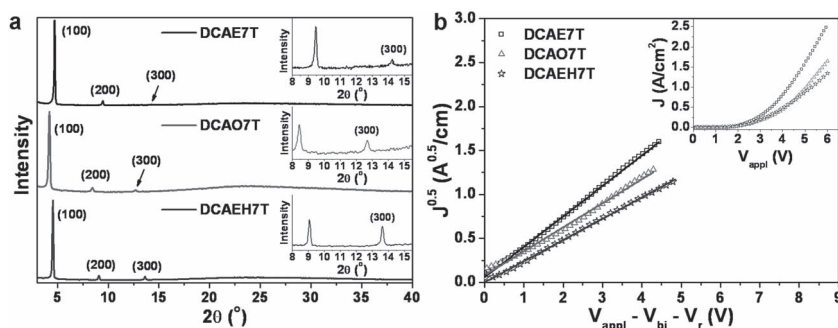
maxima of these materials in thin films are gradually blue-shifted to shorter wavelengths by changing the ethyl end group to longer octyl and 2-ethylhexyl end group. Absorption maxima were observed at 591 nm for DCAE7T, 580 nm for DCAO7T and 569 nm for DCAEH7T, respectively. This means that DCAE7T and DCAO7T have better conjugated backbones and more efficient packing than DCAEH7T. The optical bandgap of DCAE7T, DCAO7T and DCAEH7T in thin films was estimated from the onset of the absorption spectra and found to be 1.73, 1.74 and 1.75 eV, respectively. Cyclic voltammograms of all these compounds (Figure S3, Supporting Information) show two reversible oxidation waves and one irreversible reduction wave. The energy levels of the HOMO and LUMO, which are  $-5.09$  and  $-3.33$  eV for DCAE7T,  $-5.13$  and  $-3.29$  eV for DCAO7T, and  $-5.10$  and  $-3.26$  eV for DCAEH7T, were calculated from the onset oxidation potential and the onset reduction potential.<sup>[25]</sup> The electrochemical bandgap of these materials is very similar ( $\sim 1.8$  eV) and in agreement with their optical bandgap.

To investigate the structural ordering of DCAE7T, DCAO7T and DCAEH7T in the solid state, we performed X-ray diffraction (XRD) analysis for thin films spin-coated from CHCl<sub>3</sub> solution onto glass substrates and detailed *d*-spacing values are summarized in Supporting Information (Table S3). As shown in Figure 2a, all the three  $\pi$ -conjugated molecules exhibited strong (100) reflection peaks at  $2\theta = 4.7^\circ$  for DCAE7T,  $4.2^\circ$  for DCAO7T and  $4.5^\circ$  for DCAEH7T, corresponding to *d*<sub>100</sub>-spacing values of 18.7, 21.0 and 19.4 Å, respectively. These *d*<sub>100</sub>-spacing values are the distance between the planes of the main conjugation chains of these molecules separated by alkyl side chains, which are similar to that observed for P3OT (20.1 Å).<sup>[26]</sup> The second-order diffraction peaks (200) and the third-order diffraction peaks (300) for DCAE7T, DCAO7T and DCAEH7T were also clearly observed, implying a highly organized assembly of these  $\pi$ -conjugated molecules at solid state. The difference of the *d*-spacing values and the peak intensity indicate that the organization of these films can be tuned by using different end groups of these molecules, which could have impact on their morphology and thus device performance discussed below.

The hole mobility of the pristine DCAE7T, DCAO7T and DCAEH7T was measured by the space charge limited current (SCLC) method,<sup>[27,28]</sup> giving mobility values of  $4.51 \times 10^{-4}$ ,  $3.26 \times 10^{-4}$  and  $1.94 \times 10^{-4}$  cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>, respectively, as plotted in Figure 2b. The hole mobility values of these materials are comparable to that of the widely used donor polymer poly(3-hexylthiophene) (P3HT) ( $(1.4\text{--}3.0) \times 10^{-4}$  cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>)<sup>[28]</sup> and that of the very promising new donor polymers PTB4 ( $7.7 \times 10^{-4}$  cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>)<sup>[27]</sup> and



**Figure 1.** Absorption spectra of DCAE7T, DCAO7T and DCAEH7T in CHCl<sub>3</sub> and in film.



**Figure 2.** (a) XRD patterns of DCAE7T, DCAO7T and DCAEH7T films spin-coated from  $\text{CHCl}_3$ . Insets are enlarged graphs of the corresponding patterns at the range of 8–15.5°. (b)  $J^{0.5}$ - $V$  plots for the DCAE7T, DCAO7T and DCAEH7T based device. The solid lines are fits of the data points. The inset depicts the  $J$ - $V_{\text{app}}$  plot of the devices from these materials before correction for the applied voltage.

**Table 1.** Photovoltaic performance of DCAE7T/ $\text{PC}_{61}\text{BM}$ , DCAO7T/ $\text{PC}_{61}\text{BM}$  and DCAEH7T/ $\text{PC}_{61}\text{BM}$  based devices with blend ratio of 1:0.5 (w:w)

active layer	$J_{\text{sc}}$ ( $\text{mA cm}^{-2}$ )	$V_{\text{oc}}$ (V)	$FF$ (%)	$PCE$ (%)
DCAE7T/ $\text{PC}_{61}\text{BM}$	9.94	0.88	51.0	4.46
DCAO7T/ $\text{PC}_{61}\text{BM}$	10.74	0.86	55.0	5.08
DCAEH7T/ $\text{PC}_{61}\text{BM}$	9.91	0.93	49.1	4.52

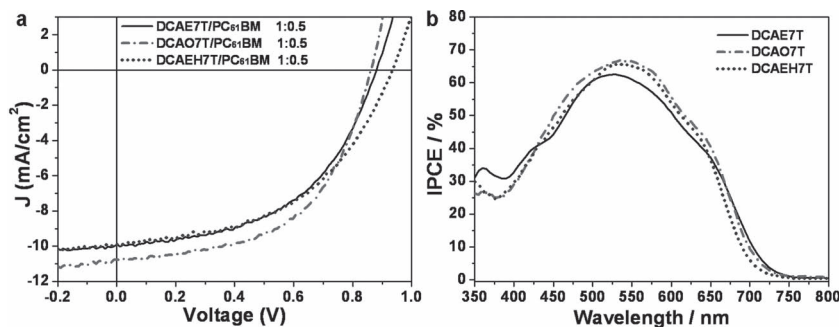
PBDTTT-C ( $2 \times 10^{-4} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ )<sup>[29]</sup> measured using the same SCLC method. The slightly different hole mobility of these compounds demonstrates that introduction of longer or branched end groups such as octyl and 2-ethylhexyl on molecule backbone has an impact on their hole mobility. This is probably due to their different packing and/or crystalline structure, as observed from XRD data above.

BHJ OSCs were fabricated using DCAE7T, DCAO7T and DCAEH7T as the electron donor materials and  $\text{PC}_{61}\text{BM}$  as the electron acceptor material with a general device structure of ITO/PEDOT:PSS/donor:acceptor/cathode using the conventional solution spin-coating process. Detailed studies for BHJ device performance of these materials for optimization were presented in Supporting Information, and the optimized results are summarized in **Table 1**. Note that the best result was observed for a donor/acceptor weight ratio of 1:0.5 from chloroform solutions using  $\text{PC}_{61}\text{BM}$  as acceptor and Ca/Al as cathode. The current density vs voltage ( $J$ - $V$ ) curves are shown in **Figure 3a**. The optimized device based on DCAE7T/ $\text{PC}_{61}\text{BM}$  exhibits a short circuit current density ( $J_{\text{sc}}$ ) of  $9.94 \text{ mA cm}^{-2}$ , an open circuit voltage ( $V_{\text{oc}}$ ) of 0.88 V and a fill factor ( $FF$ ) of 51.0%, which yields a PCE of 4.46%. When changing the ethyl end group to octyl group, DCAO7T/ $\text{PC}_{61}\text{BM}$  based device exhibits a PCE as high as 5.08%, with a  $J_{\text{sc}}$  of  $10.74 \text{ mA cm}^{-2}$ , a  $V_{\text{oc}}$  of 0.86 V and a  $FF$  of 55.0%, which is higher than the published record of PCE (4.4%),<sup>[12]</sup> thus become the highest efficiency so far for solution processed small molecule based

BHJ OSCs. When using DCAEH7T as the donor, which possesses a branched 2-ethylhexyl end group, a slightly lower PCE of 4.52% was achieved, with a  $J_{\text{sc}}$  of  $9.91 \text{ mA cm}^{-2}$ , a  $V_{\text{oc}}$  of 0.93 V and a  $FF$  of 49.1%. We note that the device performance is generally lower when other solvents, such as dichlorobenzene or chlorobenzene, were used, where poorer film quality was observed. So, compared with our earlier compound DCN7T,<sup>[9,30]</sup> which has a dicyanovinyl end group, these compounds with one CN end group replaced by ester groups gave a significantly higher PCEs. While systematic investigation is needed, the much better PCEs are believed to be at least partially due to the improved  $FF$  (over 50% vs <40%), which we suspect comes from their better film quality

and morphology as discussed below from AFM and XRD data. It is important to note that the optimized devices all have a high donor:acceptor ratio (1:0.5) in the active layer, compared with a generally lower donor content (20–50% donor content corresponding to donor:acceptor ratios of 1:4 to 1:1) for most polymer based devices.<sup>[4]</sup> The high donor concentration is expected to absorb solar light more efficiently and give more balanced charge carrier transport for donor and acceptor,<sup>[12]</sup> which could improve  $FF$  and PCE. Note that the  $FF$  values of our devices are among the highest reported so far for small molecule based BHJ OSCs.<sup>[8]</sup> During our optimization, the  $FF$  indeed varied in a wide range of 30–61% depending on the film morphology and device structure including blend ratio, solvent, buffer layer, and so on, as detailed in Supporting Information. For example, when using LiF/Al cathode, the device of DCAO7T/ $\text{PC}_{61}\text{BM}$  with the same ratio of 1:0.5 gave a significantly lower PCE of 3.87% with a  $FF$  of 49.1%. Similar results were observed for other compounds (Figure S10 and Table S4, Supporting Information). The lower  $FF$  for devices with LiF/Al cathode could be ascribed to the interfacial contact issue between active layer and electrode.<sup>[31,32]</sup>

BHJ OSCs using [6,6]-phenyl-C<sub>71</sub>-butyric acid methyl ester ( $\text{PC}_{71}\text{BM}$ ) as the acceptor were also fabricated and tested. Though intensive optimization has been performed, lower PCEs (for example, 3.98% for DCAEH7T/ $\text{PC}_{71}\text{BM}$ , w:w, 1:1.2; 3.80% for DCAO7T/ $\text{PC}_{71}\text{BM}$ , w:w, 1:1.5) (Figure S9, Supporting



**Figure 3.** (a)  $J$ - $V$  curves of BHJ OSCs prepared from DCAE7T, DCAO7T and DCAEH7T blend with  $\text{PC}_{61}\text{BM}$  (w:w, 1:0.5). (b) IPCE curves of the corresponding BHJ OSCs.



Information) were achieved for these molecules so far. Also, no clear improvement was observed by thermal annealing.

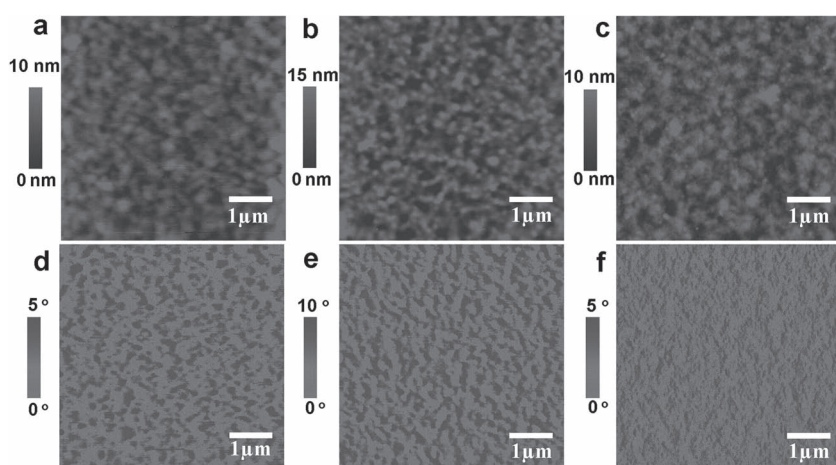
The incident photon to current efficiency (IPCE) curves of the BHJ devices based on these compounds under monochromatic light are shown in Figure 3b. All the IPCE curves exhibit a broad response covering 350–750 nm, with monochromatic IPCE maximum of 63% for DCAE7T, 67% for DCAO7T and 66% for DCAEH7T based devices. These values indicate that the photoresponse is very efficient for these small molecule based BHJ devices. The  $J_{sc}$  calculated from the integral of IPCE curves based on DCAE7T, DCAO7T and DCAEH7T with an AM1.5G reference spectrum were 9.45, 9.98, and 9.43 mA cm<sup>-2</sup>, respectively, consistent with the  $J_{sc}$  values obtained from  $J$ - $V$  measurement (within 8% error).

The atomic force microscopy (AFM) of pristine films of these molecules is shown in Supporting Information (Figure S14). The pristine donor films have similar crystalline domain size and various root mean square (*rms*) roughness, which is 4.41 nm for DCAE7T, 7.76 nm for DCAO7T and 6.63 nm for DCAEH7T. As shown in Figure 4, the blend film morphology is quite different from that of pristine donor films. The *rms* roughness of blend films is 2.51 nm, 4.05 nm and 2.51 nm for DCAE7T/PC<sub>61</sub>BM, DCAO7T/PC<sub>61</sub>BM and DCAEH7T/PC<sub>61</sub>BM films, respectively. The phase image can give good information about the donor and acceptor distribution and interpenetrating network structure.<sup>[12]</sup> As shown in Figure 4, the phase images reveal two distinct feature types, which we assign to the acceptor PC<sub>61</sub>BM-rich and donor-rich domains. These AFM results show that the donor materials have a good miscibility with PC<sub>61</sub>BM molecules in the blend films, and an optimized donor/acceptor interpenetrating network has formed. The interpenetrating network of the blend films indicates that efficient percolation channels are formed, thus improving the carrier collection efficiency and leading to a high short circuit current.

Generally, small molecules for OSCs have higher charge mobility and open circuit voltages, but poorer film quality and fill factor values compared with polymer based devices fabricated using solution process.<sup>[6,8]</sup> Thus, when designing

small molecules for high PCEs using solution processes, in addition to realizing their high charge mobility and open circuit voltage, other factors such as maximizing their solar absorption, achieving better film quality and appropriate HOMO and LUMO positions, easy synthesis, and so on, all need to be taken into consideration. Regarding the molecules in this work, their backbone has 7 thiophene units. The effective conjugation length is estimated at approximately 9–10 thiophene units for P3HT.<sup>[33]</sup> So, it should be expected that septithiophene end-capped with electron-withdrawing alkyl cyanoacetate groups should have similar  $\pi$ -conjugation to that of P3HT but better solar absorption. This can be seen from the absorption spectra, the absorption maxima (569–591 nm) of which have a large bathochromic shift compared with that (~520 nm) of P3HT.<sup>[34]</sup> Furthermore, as generally understood, the long side and end alkyl groups could significantly improve film quality required for BHJ OSCs as we observed for our molecules. Probably, the most important issue in addition to all factors mentioned above is the distribution of donor and acceptor in the films, which must meet the desired film morphology and interpenetrating network required for optimized morphology and limited exciton diffusion length. While more studies are needed, we suspect that relatively more comparable size of our molecules (conjugated backbone length of ~34 Å and molar mass of 1490–1700 g mol<sup>-1</sup>) with that of PC<sub>61</sub>BM (~11 Å in diameter and molar mass of 911 g mol<sup>-1</sup>)<sup>[35]</sup> could impart to these small molecules better miscibility with PC<sub>61</sub>BM and the capacity to form a more uniform interpenetrating network at generally lower acceptor PC<sub>61</sub>BM concentrations. Furthermore, in addition to have better light absorption, the observed higher donor/acceptor ratio (1:0.5) for the optimized devices should further improve the balance of the generally unbalanced charge carrier transport of donor and acceptor in most cases. This is because generally, the hole mobility (measured by SCLC method) of polymers used as donors in highly efficient OSCs is lower and on the order of 10<sup>-6</sup> to 10<sup>-4</sup> cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>.<sup>[20,21,36]</sup> while the electron mobility of the acceptor PC<sub>61</sub>BM is much higher, around 2 × 10<sup>-3</sup> cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>.<sup>[37]</sup> On the other hand, small molecules generally have higher hole mobility at around ~10<sup>-5</sup> to 10<sup>-3</sup> cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>.<sup>[8]</sup> Thus, better balanced mobility of acceptor (PC<sub>61</sub>BM) and these small molecule donors could reduce the unbalanced charge transport and improve the FF. Obviously, intensive and systematic design and investigation are needed to fully understand the reasons behind the high PCEs for these molecules. Overall, we believe that better interpenetrating network morphology with optimized bandgap and HOMO/LUMO positions, more balanced charge transport, high donor concentrations and efficient interfacial contact of active layer using Ca/Al cathode, all together, contribute to the high PCEs for these small molecules using simple solution spinning processes.

In conclusion, we have designed and synthesized a new class of solution processable small molecules for application in



**Figure 4.** Tapping-mode AFM height (top) and phase (down) images (5 × 5 μm) of blend films (w:w: 1:0.5) spin-coated from chloroform solutions. (a, d) Blend film of DCAE7T/PC<sub>61</sub>BM. (b, e) Blend film of DCAO7T/PC<sub>61</sub>BM. (c, f) Blend film of DCAEH7T/PC<sub>61</sub>BM.

BHJ OSCs. These small molecules all demonstrate high PCEs (4.46–5.08%) for solution processed BHJ OSCs, and a PCE of 5.08% was achieved based on DCAO7T and PC<sub>61</sub>BM without any special treatment. This is the highest efficiency reported to date for solution processed small molecule based BHJ OSCs. In addition, the high IPCE (67%) for DCAO7T based device indicates that their photoresponse is very efficient for BHJ devices. We believe the fact that all the molecules in this class gave high PCEs is significant, which may indicate further potential for molecules in this class. These results clearly suggest that solution processed small molecules can form high-quality BHJ films and efficient OSCs can be obtained by using simple small molecules and conventional solution process fabrication. We strongly believe that solution processed BHJ OSCs using small molecules can give comparable PCEs, if not higher, to those for the corresponding polymers, which we believe might be achieved in a short time. This should offer an exciting opportunity for the commercialization of low-cost, solution-processed and flexible OSCs to meet the global energy challenge.

## Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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