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## Subtle Balance Between Length Scale of Phase Separation and Domain Purification in Small-Molecule Bulk-Heterojunction Blends under Solvent Vapor Treatment

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Organic photovoltaic (OPV) devices are a promising next-generation technology to produce lightweight, low-cost, and flexible solar-cell panels.<sup>[1,2]</sup> Currently, power conversion efficiencies (PCEs) over 10% have been achieved for OPVs using the bulkheterojunction (BHJ) device structure,<sup>[3-9]</sup> owing to the continuous material evolution<sup>[10-16]</sup> and device optimization (interface layer engineering and morphology control).<sup>[17-23]</sup> For donor materials, including polymers and small molecules, several criteria have to be met, i.e., strong absorption, suitable energy levels, high hole mobility, and good solubility and film-forming property. In device optimization, one of the major challenges to further improve device performance is controlling the activelayer morphology. It is suggested that a bicontinuous network of phase separated donor-acceptor morphology with size scale commensurate with the exciton diffusion length will be ideal to harvest sunlight.<sup>[24]</sup> In practice, the vertical segregation and domain connectivity in a device sandwich are also critical. Various morphological control methods have been developed, including appropriated solvent choice, thermal annealing,

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solvent vapor annealing (SVA), additives, etc., showing promising enhancement in device performance.<sup>[19,25]</sup>

SVA in controlling the nanomorphology of block copolymers has been well established in the past decade.<sup>[26]</sup> Most recently, it has been applied to OPVs.<sup>[10,27-36]</sup> In SVA, solvent vapor can penetrate into the blend and drastically lower the glass transition temperature of the materials, and thus polymers/molecules in blends gain high mobility to organize into a lower energy state. It is of high interest to OPVs since it can be used to fine-tune the non-equilibrium morphology to a state that is necessary to deliver a good performance. When applying SVA into small-molecule BHJ blends, phase separation and nucleation and growth will be two processes that dominate the kinetics of morphology evolution. Thus, a systematic study of the morphology change during SVA treatment,[27,29,31,35] and correlating the morphology details with device performance are of high importance. In this study, we performed a systematic SVA study on a BHJ photoactive layer based on the DR3TBDTT molecule<sup>[37]</sup> (Figure 1a) (DR3TBDTT:[6,6]-phenyl-C<sub>71</sub>-butyric acid methyl ester (PC71BM)) and elucidated the morphology reasons for device performance improvement. The well-defined chemical structure, good crystallinity, and suitable length scale of phase separation make this system ideal to study the performance/morphology and SVA relationship. Four annealing solvents, carbon disulfide (CS2), chloroform (CHCl3), tetrahydrofuran (THF), and methylene chloride (CH2Cl2) with different solubilities for donor and acceptor, and different boiling points, were used to systematically investigate the impact of these solvents on the morphology. Our study demonstrates that: i) the presence of solvent molecules inside the BHJ thin film promotes mobility of both DR3TBDTT and PC71BM in mixing regions, which induces molecule crystallization, and ii) the growth of crystalline domains is more of a domain purification process in a phase-separated structure. With these, the best performance was achieved when CS2 was used for the treatment, with a high PCE of 9.58%, a short-circuit current density  $(J_{\rm sc})$  of 14.21 mA cm<sup>-2</sup>, and markedly improved fill factor (FF) of 76.1%.

Devices with normal device structure of ITO/PDEDOT:PSS/ DR3TBDTT:PC<sub>71</sub>BM/PrC<sub>60</sub>MA/Al were fabricated. CS<sub>2</sub>, CHCl<sub>3</sub>, THF and CH<sub>2</sub>Cl<sub>2</sub> were chosen as solvents for SVA. The optimization process for each solvent can be found in Table S3-S6 in the Supporting Information. The solvent properties and optimized photovoltaic performance parameters with SVA are listed

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**Figure 1.** a) Chemical structure of DR3TBDTT, b) current density-voltage (J-V) curves of DR3TBDTT:PC<sub>71</sub>BM-based devices, c) absorption spectra of the blends, d) the EQE curves for the devices, and e) photocurrent density versus effective voltage ( $J_{ph}-V_{eff}$ ) characteristics for the devices under constant incident light intensity (AM 1.5G, 100 mW cm<sup>-2</sup>) without and with SVA.

in **Table 1**. The current density–voltage (*J*–*V*) curves of OPV devices at optimized SVA conditions are shown in Figure 1b. The device without SVA exhibited a PCE of 7.92%, with opencircuit voltage ( $V_{oc}$ ) of 0.918 V,  $J_{sc}$  of 12.95 mA cm<sup>-2</sup>, and FF of 0.667. Compared with the untreated device, the devices after SVA showed obvious improvement in  $J_{sc}$  and FF, and a slight drop in  $V_{oc}$ . SVA with CS<sub>2</sub> gave the best device performance with PCE of 9.58%,  $V_{oc}$  of 0.886 V,  $J_{sc}$  of 14.21 mA cm<sup>-2</sup>, and FF of 0.761. The device treated by CHCl<sub>3</sub> gave a PCE of 9.12%. The devices with THF and CH<sub>2</sub>Cl<sub>2</sub> SVA showed PCEs of 8.28% and 8.66%, respectively. It can be seen that the devices with SVA based on the good solvent (CS<sub>2</sub> and CHCl<sub>3</sub>) for donor and

Table 1.	Solvent properties	used in SVA and OF	V performance	e parameters withou	t and with SVA
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Treatment	Boiling point [°C]	Donor solubility [mg mL <sup>-1</sup> ]	Acceptor solubility [mg mL <sup>-1</sup> ]	V <sub>oc</sub> [V]	∫ <sub>sc</sub> [mA cm <sup>−2</sup> ]	FF	PCE [%]	
							Best	Average <sup>b)</sup>
No SVA	-	-	-	0.918	12.95	0.667	7.92	7.66
$CH_2Cl_2$	40	<0.5	<10	0.889	13.55	0.717	8.66	8.30
THF	66	2.3	3.45 <sup>a)</sup>	0.893	13.38	0.693	8.28	8.14
CHCl <sub>3</sub>	61	29	61.1 <sup>a)</sup>	0.881	13.84	0.748	9.12	9.00
CS <sub>2</sub>	46.5	65	142 <sup>a)</sup>	0.886	14.21	0.761	9.58	9.36

<sup>a)</sup>Solubility parameters for all solvents were obtained from ref.;<sup>[38] b)</sup>The average values were obtained from over 50 devices.

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acceptor display better improvement in photovoltaic performance, indicating that the solubility of the donor and acceptor materials in the annealing solvents is critical. Moreover, the decreased  $V_{\rm oc}$  of the devices with SVA could be due to the increasing intermolecular interaction (the strength of intermolecular interactions could be represented by  $J_{\rm so}$ ) between the donor and acceptor,<sup>[39,40]</sup> which was observed in our previous work.<sup>[33]</sup> As shown in Figure S3 and Table S1 (Supporting Information), compared with the untreated device, the devices with SVA treatments exhibit increased  $J_{\rm so}$ , which is consistent with the decreased  $V_{\rm oc}$ .

The effect of SVA on the photovoltaic performance was investigated by absorption studies and external quantum efficiency (EQE) measurements. Figure 1c shows the absorption spectra of DR3TBDTT:PC71BM films before and after SVA under optimized conditions. The pristine blend film showed absorption maximum at 571 nm, with a small shoulder at 636 nm. After SVA, the film maximum absorption peaks were redshifted by 15, 13, 9 and 12 nm for CS<sub>2</sub>, CHCl<sub>3</sub>, THF and CH<sub>2</sub>Cl<sub>2</sub> SVA, respectively, with noticeable absorption coefficient enhancement. In addition, strong shoulder peaks showed up. This demonstrates that better ordered packing is formed for the DR3TBDTT molecules after SVA process. The EQEs of the devices are shown in Figure 1d. Broad wavelength improvement in EQE was recorded for SVA treated samples. The calculated Jsc values obtained from the integration of the EQE data for the devices using different treatments are close to the  $J_{sc}$  values from the J-V measurement with ca. 4% mismatch.

To further understand the mechanism responsible for the enhanced performance in the devices with SVA, the relationship between photocurrent  $(J_{\rm ph})$  and effective voltage  $(V_{\rm eff})$ has been carried out (Figure 1e). The  $J_{ph}$  in devices without or with SVA increase sharply with effective voltage, and reached gradually a saturated value (J<sub>ph,sat</sub>) in higher effective voltage. The J<sub>ph,sat</sub> was correlated to the maximum exciton generation rate, which is a measure of the maximum number of photons absorbed.<sup>[41,42]</sup> The devices with SVA show higher J<sub>ph,sat</sub> compared to the device without SVA (Table S7, Supporting Information), indicating that more photons could be absorbed. In addition, the ratio of  $J_{\rm ph}/J_{\rm ph,sat}$  can be used to judge the overall exciton dissociation efficiency and charge collection efficiency.<sup>[43]</sup> At the maximal power output condition, the ratio of the device without SVA is 79.8%, while the devices with CS<sub>2</sub>, CHCl<sub>3</sub>, THF and CH<sub>2</sub>Cl<sub>2</sub> annealing obtained higher J<sub>ph</sub>/J<sub>ph,sat</sub> of 89.3%, 90.0%, 83.1% and 82.5%, respectively, showing dramatically improved charge collection efficiency. Figure S4 (Supporting Information) shows transient photovoltage curves under one sunlight using the same pulsed light intensity. The untreated device showed relatively short carrier lifetime of 0.85 µs. The CH<sub>2</sub>Cl<sub>2</sub> and THF treated devices had slightly increased lifetimes of 1.54 and 1.21 µs, respectively. For the devices with good solvents (CS2 and CHCl3), the lifetimes improved to 2.38 and 2.30 µs, respectively. Besides, higher and more balanced hole and electron mobilities were observed in the devices with SVA (Figure S5 and Table S7, Supporting Information). All of these demonstrate that SVA improves the device exciton dissociation efficiency, and charge transport and collection efficiency, and reduces carrier recombination.



In order to fully understand the effect of SVA on the photovoltaic performance, the structure order of the pure DR3T-BDTT and its BHJ blends with different treatments was accessed by grazing-incidence X-ray diffraction (GIXD). As shown in Figure 2a, the as-cast pure material thin film showed an edge-on crystal orientation with four orders of (100) diffraction showing up in the out-of-plane (OOP) direction. A complimentary (010) diffraction in in-plane (IP) direction appeared. The (100) peak in pure film located at 0.31 Å<sup>-1</sup>, giving an alkyl-to-alkyl spacing of 20.4 Å. (010) peak located at 1.72 Å<sup>-1</sup>, giving a  $\pi$ - $\pi$  stacking distance of 3.65 Å. The (100) peak in pristine and annealed samples was consisted of two peaks, which could be split and fitted by two Gaussians (with results shown in Figure S7, Supporting Information). The smaller peak in a slightly higher q region ( $\approx 0.32-0.33$  Å<sup>-1</sup>) and the difference in (100) spacing comparing to the major peak is about 1 Å, indicating DR3TBDTT could form two crystalline structures, which most probably come from different tilting angles of alkyl chains. The crystalline information of DR3TBDTT in BHJ blends was of more interest since it was directly related to the device performance. BHJ thin-film samples were prepared under the same conditions with device fabrication. The diffractogram for BHJ thin films was slightly different from the pure DR3TBDTT, with a new diffraction ring in 1.32  $Å^{-1}$ coming from PC71BM. Although DR3TBDTT still adopted an edge-on orientation in BHJ thin films, the azimuthal spreading of crystal conformations were broadened comparing to pure DR3TBDTT films. Both (100) and (010) peaks spread in 0-180° chi-angle space. Line-cut profiles in the OOP direction and IP direction were summarized in Figure 2b and 2c. These overlaid traces showed that DR3TBDTT in BHJ films under different solvent annealing remained the same crystal form and no polymorph was seen. Detailed crystal information was accessed by fitting the OOP (100) peak and IP (010) peak, and the results were shown in Figure 3. For the four solvents used in vapor annealing experiment, increase in relative crystallinity in both (100) and (010) directions was seen. In both directions, CS<sub>2</sub> solvent showed the most pronounced effect in increasing relative crystallinity. (100) d-spacing changed under different solvent vapor treatment yet without a clear trend. (100) crystal size increased under vapor annealing, with CS<sub>2</sub> case showing a maximum value of 177 Å. Vapor treatment more profoundly affected the (010) directions where charge transport took place. (010) *d*-spacing reduced by about 0.03 Å, and (010) crystal size increased under vapor treatment. CHCl<sub>3</sub> and CS<sub>2</sub> vapor treated films showed more obvious increase in (010) crystal size, relative to the films with THF and CH<sub>2</sub>Cl<sub>2</sub> treatment. CS<sub>2</sub> vapor annealed samples showed a 7.5 Å increase in (010) crystal size, corresponding to two more  $\pi$ - $\pi$  stacks. The enhanced crystalline content, elevated crystal size and reduced  $\pi$ - $\pi$  stacking are favorable for the charge transport in BHJ thin film, which can lead to enhanced device performance. It is also important to note that different choice of solvents will be quite different in controlling the thin film morphology and crystalline structure. In general both vapor pressure and solvent-molecular interaction need to be considered. The solvents with low boiling point and high solubility for donor and acceptor will be more effective in morphology control, as the CS2 and CHCl3 cases in current experiment.

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**Figure 2.** GIXD data of the films with different treatments. a) The GIXD profiles of the pure DR3TBDTT films and DR3TBDTT:PC<sub>71</sub>BM blend films, b) fitting of OOP line cut profiles of GIXD of the blend films, and c) fitting of in-plane line-cut profiles of GIXD of blend films.

The morphology of pristine and solvent vapor annealed BHJ thin films were also investigated by atomic force microscopy (AFM) and transmission electron microscopy (TEM), wellestablished methods to investigate the phase image of the BHJ blend. From the AFM images (Figure S8, Supporting Information), the blend films without and with CS<sub>2</sub>, CHCl<sub>3</sub>, THF and CH<sub>2</sub>Cl<sub>2</sub> SVA are smooth and uniform with low root-meansquare (rms) surface roughness of 1.07, 1.34, 1.07, 0.979 and 1.01 nm, respectively. Figure 4a shows the TEM images of the blend films. As shown in Figure 4a, all the samples displayed nanoscale phase separation. The bright domains were DR3T-BDTT rich and dark domains were PC71BM rich. The DR3T-BDTT domain size was about 30-40 nm, which was well defined and had good connection. Color inversion analysis was performed on CS2 annealed sample (Figure S9, Supporting Information). The processed TEM image showed a good interconnected light-colored network, consisting of PC71BM rich regions. Thus dual networks of tens of nanometers phase separation existed in BHJ thin films, which would be efficient in exciton diffusion and dissociation, and charge transport. Different annealing times on the morphology of BHJ thin film had also been investigated (see Figure S10 in the Supporting Information). For THF and CH2Cl2 with low solubility, whose

vapor were less effective in driving DR3TBDTT crystallization, did not strongly change the length scale of phase separation over time. For CS<sub>2</sub>, drastic changes were observed during anneal period. The 60 s annealed film showed a well-percolated bicontinuous network, but on extending solvent annealing to 90 s, the phase image changed tremendously to the shape of leaf assemblies, which reduced the PCE from 9.58% to 7.98% (Table S3, Supporting Information). Resonant soft X-ray scattering (RSoXS) was employed to further study the thin film morphology, which provided a better statistics of the size of phase separation. Well-defined scattering peaks were observed in scattering profiles (Figure 4b), which combined with the phase image from TEM, indicating that this BHJ system could be treated as a two-phase system. It was seen that the as-cast BHJ thin film had a length scale of phase separation of 31 nm. THF annealing slightly increased the size of phase separation to 34 nm. CH<sub>2</sub>Cl<sub>2</sub> annealing reduced phase separation to 23 nm with reduced intensity. CS<sub>2</sub> annealing strongly increased the phase separation to 51 nm with much enhanced intensity. CHCl<sub>3</sub> annealing increased the phase separation to 36 nm with enhanced intensity.

The correlation between morphology and device performance was analyzed by taking account of the major parameters **4DVANCED** 

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(a) 1.2 (b) - (100) spacing **Relative Crystallinity** -(100) size 20.0 200 1.0 0 spacing (A) 0.8 Size 19.8 180 0.6 A 0.4 19.6 160 0.2 CHCI As-cast CH<sub>2</sub>Cl<sub>2</sub> THE As-cast CH<sub>2</sub>Cl<sub>2</sub> THF CHCI, CS<sub>2</sub> CS, (c) 1.2 (d) 70 - (010) spacing **Relative Crystallinity** - (010) size 3.66 1.0 ٥ 68 spacing (A) Size 0.8 66 3.64 A 0.6 64 0.4 62 3.62 0.2 60 As-cast CH<sub>2</sub>Cl<sub>2</sub> THF CHCI, CS, As-cast CH<sub>2</sub>Cl<sub>2</sub> THF CHCI, CS,

**Figure 3.** Detailed crystal information of DR3TBDTT:PC<sub>71</sub>BM blend films with different treatments accessed by fitting the OOP (100) peak and IP (010) peak. a) The (100) relative crystallinity of DR3TBDTT in the blend films. b) (100) *d*-spacing and the estimated (100) crystal size of DR3TBDTT in the blend films. c) The (010) relative crystallinity of DR3TBDTT in the blend films. d) (010) *d*-spacing and the estimated (010) crystal size of DR3TBDTT in the blend films. d) (010) *d*-spacing and the estimated (010) crystal size of DR3TBDTT in the blend films.

in our investigations. Device parameters, such as  $J_{\rm sc}$ , PCE and FF showed the same trend and we listed  $J_{sc}$  and PCE in Figure 4c. The crystal-size information is summarized in Figure 4d. The length scale of phase separation is provided in Figure 4e. In terms of the length scale of phase separation, the diminishing of phase size did enhance performance, as shown in CH<sub>2</sub>Cl<sub>2</sub> case. In CS<sub>2</sub> and CHCl<sub>3</sub> cases, the general argument that smaller-sized phase separation would enhance performance did not apply, persuading us searching for new structure-performance relationship. Small-molecule-based OPVs were of high crystalline nature and thus the crystal information was carefully compared. It was seen that the crystal size of DR3TBDTT in both (100) and (010) direction could not fully agree with the trend of device performance, in which (100) peak was off in CHCl<sub>3</sub> case and (010) peak was off in THF case. The general trend of PC<sub>71</sub>BM crystal size agreed with the device results, which grew during SVA, indicating the importance of PC<sub>71</sub>BM aggregation in elevating the device performances. The relative crystallinity (Figure 3c) of the (010) peak agreed well with the PCE trend. The combination of this information together with PC71BM shows essence of SVA: the presence of solvent molecules inside the BHJ thin film promotes molecule mobility of both DR3TBDTT and PC71BM in the mixing regions, which induces molecular crystallization. The growth of crystalline domains is more of a domain purification process in a phase-separated structure, which would veritably change the length scale of phase separation. The increase in crystallinity and crystal size and decrease of phase separation size in CH<sub>2</sub>Cl<sub>2</sub> case suggested that more crystallites formed in BHJ thin film, indicating nucleation and growth process that dominate morphology evolution. CH2Cl2 annealing led to different behaviors of molecular crystallization, which is the main reason of the difference in morphology. The composition variation

(Figure 4e), which was calculated by integrate the area of  $Iq^2$  versus q plot, defined the extend of phase separation, or phase purity in a two-phase system, showed a trend quite similar to  $J_{\rm sc}$  and PCE trends for all the solvents used in this study, pointing out an important factor to be considered in small-molecule-based BHJ OPV. The larger crystallinity of both DR3TBDTT and PC<sub>71</sub>BM and higher phase purity induced by SVA benefit the charge transport and collection, and reduce charge recombination, thus leading to improved device performance of the blends with SVA.

In this contribution, we systematically studied the SVA treatment in small-molecule-based BHJ thin films to control morphology and improve device performance. Various solvents are employed and thus the solvent-quality-morphology-performance relationship can be established. The presence of solvent molecules inside the BHJ thin film promotes the mobility of both donor and acceptor molecules, leading to crystallization and aggregation, which are important in modulating the thin film morphology. Good solvents with high vapor pressures show the most promising results, by increasing both  $I_{sc}$  and FF, with an overall 20+% enhancement in PCEs. Good solvent quality readily crystallizes donor molecules, leading to increased length scale of phase separation and improved domain purity, contributing to enhanced device performance. It should be noted that the length scale of phase separation remained ≈50 nm, which is not far away from the exciton diffusion length. A poor solvent (THF) leads to less pronounced improvements, due to its low ability to drive molecules, and thus a less obvious change in crystalline content and phase separation. In the poor but lowest boiling point CH<sub>2</sub>Cl<sub>2</sub> case, its permeation into BHJ thin films leads to nucleation and growth, leading to higher numerical crystalline density, and thus a reduced length scale of phase separation at an improved relative crystallinity, falling into the

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**Figure 4.** a) TEM images of DR3TBDTT:PC<sub>71</sub>BM blend films without and with SVA. b) RSoXS profiles of DR3TBDTT:PC<sub>71</sub>BM blend films. c) Photovoltaic performance parameters  $J_{sc}$  and PCE of the devices with different treatments. d) Crystal size of (100) direction and (010) direction of DR3TBDTT and PC<sub>71</sub>BM in the blend films with different treatments. e) The composition variation and the length scale of phase separation of the DR3TBDTT:PC<sub>71</sub>BM blend films with different treatments.

paradigm of length scale and performance correlations. However,  $CH_2Cl_2$  annealing led to a larger crystal of both DR3T-BDTT and  $PC_{71}BM$  and a higher phase purity, which benefits both charge transport and collection, and also reduces charge recombination, thus leading to improved device performance. The physical nature of SVA treatment is quite straightforward, and its application is ideal in small-molecule-based BHJ solar cells. It is also an easy process that can be used in future industrial fabrication, adding new handles in controlling morphology and obtaining better device performances.

## **Supporting Information**

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

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