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Solution-processed bulk heterojunction solar cells based on porphyrin small molecules with very low energy losses comparable to perovskite solar cells and high quantum efficiencies

Ke Gao‡, Liangang Xiao‡, Yuanyuan Kan†, Binglin Yang‡, Junbiao Peng‡, Yong Cao‡, Feng Liu‡, Thomas Russell‡ and Xiaobin Peng*‡

Two new A-D-A conjugated small molecules Por-Rod and Por-CNRod are developed using a porphyrin core as the donor unit and 3-ethylrhodanine and 2-(1,1-dicyanomethylene)rhodanine as the acceptor units. Por-Rod and Por-CNRod show broad absorptions to ~850 nm with optical energy band gap of 1.47 and 1.45 eV, and their blend films with PC$_{71}$BM show SCLC hole mobilities of 8.5 × 10$^{-5}$ and 7.5 × 10$^{-5}$ cm$^2$ V$^{-1}$ s$^{-1}$, respectively. Though their bulk heterojunction solar cells with PC$_{71}$BM processed without additive, only with thermal annealing or only with pyridine additive show very low power conversion efficiencies (PCEs), the Por-Rod-based solar cells processed with pyridine and then thermal annealing show a PCE up to 4.97% with a remarkable $V_{OC}$ up to 0.94 V, showing a very low energy loss of only 0.53 eV. This is the first report that small molecule-based solar cells show such a low energy loss comparable to perovskite solar cells but with a good PCE of about 5% and a maximum external quantum efficiency up to 61%. To further understand the effect of different processing conditions on the blend films, the morphology of their blend films is studied by grazing incidence X-ray diffraction and resonant soft X-ray scattering.

Compared with perovskite solar cells, the main issue of present OSCs is how to obtain a high open circuit voltage ($V_{OC}$) and a short circuit current ($J_{SC}$) simultaneously. Firstly, it is impossible to convert the whole energy bandgap ($E_g$) of the donor material to $V_{OC}$ because driving forces are needed between the donor and acceptor materials in OSCs. A key parameter of the energy loss ($E_{loss}$), which defines as $E_{loss}=E_g - E_{OC}$, create a link between the $V_{OC}$ and the $E_g$ and the minimum $E_{loss}$ was suggested to be 0.6 eV for efficient BHJ OSCs. Secondly, though the $E_{loss}$ below 0.6 eV sometimes can be achieved for OSCs, the external quantum efficiency (EQE) drops remarkably because a too low $E_{loss}$ enhances the charge recombination significantly even though a lower $E_{loss}$ can also help the charge separation. On the contrary, the $E_{loss}$ values can be low to 0.5-0.55 eV but still with very high EQE for perovskite solar cells. Therefore to lower the $E_{loss}$ values comparable perovskite solar cells but still maintaining high EQE is important to further improve the performance of BHJ OSCs. However, most BHJ OSCs show energy losses above 0.7 eV, and the OSCs with energy losses below 0.6 eV usually show low EQE values as show in Figure 1.7, 24, 28, 29, 32 Although there are many ways to reduce the energy losses and obtain high $V_{OC}$ values such as: (1) tuning the energy levels of donor materials, (2) increasing crystallinity without decreasing D/A interfaces to provide more paths for charge separation, (3) reducing the exciton binding energy and getting a higher $E_{eff}$ by increasing the permittivity, and (4) lowering bimolecular non...
radiative recombination,\textsuperscript{35} it is still not easy to obtain a low energy loss but with high EQE values for BHJ solar cells.

Porphyrim and their derivatives have been studied as the active materials in initial OPV studies because of the easy modifications of the periphery or by metal insertion into the cavity, extensively \(\pi\)-conjugated systems and high molar absorption coefficients.\textsuperscript{26-38} Very recently, we developed conjugated donor-acceptor-donor (A-D-A) porphyrin small molecules, in which electron-deficient units were linked to the electron-rich porphyrin core through ethynylene, for solution-processed BHJ OSCs, and the PCEs were finally enhanced up to 8%.\textsuperscript{7} Considering the good performance of the electron-deficient units of 3-ethylrhodanine and 2-(1,1-dicyanomethylenepyridine)rhodanine in OPV molecules,\textsuperscript{39-41} herein, we design and prepare two new A-D-A SMs of Por-Rod and Por-CNRod, in which 3-ethylrhodanine and 2-(1,1-dicyanomethylenepyridine)rhodanine units, respectively, are symmetrically conjugated through thiophenylethynyl bridges to a porphyrin core for BHJ OSCs.\textsuperscript{42, 43} We not only explore the photovoltaic properties of the BHJ OSCs based on them as the donor materials but also the morphology of the blend films under different device fabrication conditions.

**Experimental**

**General methods**

\(^1\)H NMR spectra were conducted on a Bruker AVANCE Digital 300 MHz spectrometer in deuterated chloroform using tetramethylsilane as an internal standard. Mass Spectrometry (MS) data were obtained on a Bruker Daltonics BIFLEX III MALDI-TOF Analyzer using MALDI mode. UV-vis-NIR absorption spectra of the films on quartz substrates were measured using a Shimadzu UV-3600 spectrophotometer. Photoluminescence spectra were recorded on a Horiba Fluoromax-4 spectrophotometer. Cyclic voltammetry (CV) was carried out on a CHI660A electrochemical workstation with platinum electrodes at a scan rate of 50 mV s\(^{-1}\) against an Ag/AgCl reference electrode and ferrocene/ferrocenium (Fc/Fc+) as the internal potential standard with nitrogen-saturated solution of 0.1 M tetrabutylammonium hexafluorophosphate (Bu\(_4\)NPF\(_6\)) in chloroform. The \(J-V\) characteristics were measured under AM 1.5 solar simulator (Japan, SAN-EL, XES-40S1) at 100 mW cm\(^{-2}\) calibrated with a standard Si solar cell, and the data were collected using a Keithley 2400 digital source meter.

**Synthesis of Por-Rod and Por-CNRod**

All operations including air-sensitive reagents were conducted under an inert atmosphere of nitrogen or argon. All chemicals and solvents were used as received unless otherwise indicated from commercial sources (TCI, Sigma Aldrich, J&K, Alfa Aesar). Toluene, tetrahydrofuran (THF) and triethylamine were all dried by distillation over sodium prior to use. Rhodanine-(CN)\(_2\) was synthesized according to reported procedures,\textsuperscript{39} and the detailed synthetic procedures of 6 are shown in supporting information. The main synthetic routes of Por-Rod and Por-CNRod are shown in Scheme 1. The chemical structures of Por-Rod and Por-CNRod were confirmed by \(^1\)H NMR, MALDI-TOF mass spectroscopy and elemental analysis.

**Compound 4**

40 mL acetic acid was added to the mixture of 1 (546.7 mg, 2.862 mmol) and 2 (506.9 mg, 3.148 mmol) under argon before ammonium acetate (484.8 mg, 6.297 mmol) was added. After stirred and heated at 110 °C for 36 h, the mixture was extracted with CH\(_2\)Cl\(_2\), washed with water and dried over anhydrous Na\(_2\)SO\(_4\). After the removal of solvent, the crude product was first purified by column chromatography and then recrystallized from CH\(_2\)Cl\(_2\) and CH\(_3\)OH to afford 4 as a yellow solid (915 mg, 96%). \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.75 (s, 1H), 7.16 (d, 2H), 4.18 (q, 2H), 1.29 (t, 3H).

**Scheme 1.** Synthetic routes for Por-Rod and Por-CNRod. Reaction condition: (a) ammonium acetate/acetic acid for aldol condensation reaction reactions; (b) Pd(PPh\(_3\))\(_4\)/CuI, toluene/triethylamine for Sonogashira reaction coupling reactions.
Compound 5
Similar procedures as the synthesis of 4 but with 3 as one of the starting materials to afford 5 as a light yellow solid (yield: 90%). 1H NMR (300 MHz, CDCl3) δ (ppm): 7.96 (s, 1H), 7.24 (d, 1H), 7.21 (d, 1H), 4.34 (q, 2H), 1.39 (t, 3H).

Por-Rod
To a 50 mL two necked round-bottom flask were added compound 6 (103.3 mg, 0.1 mmol), 4 (99.9 mg, 0.3 mmol), anhydrous toluene (12 mL) and triethylamine (6 mL), and the mixture was deoxygenated with Ar for 30 min before Pd(PPh3)4 (11.55 mg, 0.01 mmol) and CuI (1.9 mg, 0.01 mmol) were added. Then the mixture was stirred at 80 °C for 72 h under the protection of Ar. After cooled to room temperature, the mixture was washed with water and dried over anhydrous Na2SO4. Then the solvent was removed, and the residue was purified by column chromatography to give a black solid Por-Rod (yield: 75%). Mass (MALDI-TOF): Obs. 1539.6; Calcd. for C77 H6 N2 O6 Zn: C, 65.45; H, 6.15; N, 5.45. Found: C, 65.29; H, 6.28; N, 5.39.

Por-CNRod
Similar procedures as the synthesis of Por-Rod but with 5 as one of the starting materials to afford Por-CNRod as a black solid (yield: 75%). Mass (MALDI-TOF): Obs. 1604.1; Calcd. for C84 H6 N2 O6 Zn: 1538.4. Anal. Calcd. for C84 H6 N2 O6 Zn: C, 65.45; H, 6.15; N, 5.45. Found: C, 65.29; H, 6.28; N, 5.39.

Results and discussion
Synthesis of Por-Rod and Por-CNRod
Two new A-D-A porphyrin organic semiconductor materials are synthesized. The aldol condensation reaction of commercially available 1 with 2 or 3 produced compound 4 or 5. The bis-Sonogashira reaction of 4 or 5 with 6 affords the target product Por-Rod or Por-CNRod in good yield about 75%. More important, no toxic tin compound or dangerous lithium reagent was used in the whole synthetic routes. Optical and electrochemical properties

Optical and electrochemical properties

Table 1 Optical and electrochemical data for Por-Rod and Por-CNRod.

<table>
<thead>
<tr>
<th>Donor</th>
<th>λmax [nm] (solution)</th>
<th>λmax [nm] (film)</th>
<th>Eon [V]</th>
<th>Eon0 [eV]</th>
<th>ELumo [eV]</th>
<th>Eg [eV]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Por-Rod</td>
<td>500</td>
<td>843</td>
<td>0.44</td>
<td>-5.24</td>
<td>-3.77</td>
<td>1.47</td>
</tr>
<tr>
<td>Por-CNRod</td>
<td>488</td>
<td>854</td>
<td>0.52</td>
<td>-5.32</td>
<td>-3.87</td>
<td>1.45</td>
</tr>
</tbody>
</table>

* Optical band gap estimated from the formula of 1240/λ_{onset}, and λ_{onset} is the onset of the absorption spectrum in film.
The electrochemical properties of the two molecules were investigated by CV in order to obtain the frontier orbit energy levels. HOMO energy levels (E_{HOMO}) are estimated according to the empirical equation of E_{HOMO} = -(4.8 + E_{ox}), where E_{ox} is the onset oxidation potential of molecules relative to that of Fe/Fe'. As shown in Figure 2, E_{ox}(Por-Rod) and E_{ox}(Por-CNRod) after the deduction of E_{ox}(Fe/Fe') were measured to be 0.44 and 0.48 V, thus the HOMO energy levels are calculated to be -5.24 and -5.32 eV, and the LUMO energy levels (E_{LUMO}) are calculated to be -3.77 and -3.87 eV for Por-Rod and Por-CNRod, respectively, according to E_{LUMO} = E_{HOMO} + E_g. Their electrochemical data and the HOMO and LUMO energy levels are summarized in Table 1.

Photovoltaic properties and exciton dissociation rate

The solution-processed BHJ OSCs were fabricated with PC_{71}BM as the electron acceptor and Por-Rod or Por-CNRod as the electron donor in a conventional device structure of ITO/PEDOT:PSS/BHJ blends/PFN/Al (ITO: indium tin oxide; PEDOT:PSS: poly(styrene sulfonate)-doped poly(ethylene-dioxythiophene); PFN: poly[(9,9-bis(N,N-dimethylamino)propyl)-2,7-fluorene]-alt-2,7-(9,9-dioctyfluorene)]) under different conditions, and then measured under AM 1.5 illumination. The ratio of Por-Rod or Por-CNRod to PC_{71}BM was optimized to be 1:1 (w/w). Device results are summarized in Table 2, and their corresponding J-V curves are shown in Figure 3 and Figure S1. All the OSCs fabricated just using CB as the solvent without any post treatment show very poor performance. And the addition of pyridine (Py) additive (1 v%), which was often used to improve the film morphology of zinc(II) porphyrins, only improves the device performance slightly. However, it is noted that Py additive can enhance the Voc. Especially, the Por-CNRod-based OSCs show a high Voc up to 0.94 V. Since the E_g of Por-CNRod is 1.45 eV, the E_{loss} for Por-CNRod-based OSCs fabricated with Py additive is just 0.51 eV, which is smaller than the 0.55 eV value for perovskite solar cells when PEDOT:PSS is used. Therefore, the Voc is very high for such a small bandgap molecule-based OSCs. However, the small J_{sc} and FF lead to a poor PCE of only 0.6%.

Thermal annealing does not take effects for these OSCs either, inducing similar or inferior performance to those fabricated without any post treatments. However, when the devices were further optimized with Py additive and then thermal annealing, Por-Rod-based devices exhibit a significant PCE enhancement up to 4.97% with a J_{sc} of 12.39 mA/cm^2, a fill factor of 42.71% and a Voc of 0.94 V while Por-CNRod-based ones do not show an improved performance. The Voc values of the OSCs changes obviously under different device fabrication conditions, showing that film morphology impacts significantly on the open-circuit voltages. As show in Table 2, the Voc values for the devices fabricated with pyridine additive are increased compared with those without pyridine, which can be ascribed to the more D/A interfaces induced by pyridine. This is another case for the high Voc of Por-Rod-based OSCs fabricated with pyridine and then thermal-annealed. It is also worthy to note that Por-Rod-based devices show not only an extremely low energy loss of 0.53 eV but also a good J_{sc} up to 12.39 mA/cm^2, which can be confirmed by the EQE as shown in Figure 3b. The EQE of Por-Rod-based solar cells processed with pyridine additive and then thermal annealing exhibits a broad photo response from 300 to 850 nm with the maximum EQE value up to 61% at 520 nm and other two peak values over 50%, indicating it is possible to further enhance the efficiency by reducing the E_{loss} while maintaining high EQE.
values. To the best of our knowledge, the EQE up to 61% is the highest for the BHJ OSCs with an EQE less than 0.55 eV. And the high EQE can be partially ascribed to the efficient exciton dissociation, which is demonstrated by photoluminescence (PL) quenching experiments. As seen in Figure S3, Por-Rod pristine film exhibits PL emission peak at 805 nm, and it is quenched almost completely by PC$_7$BM in the blend films, which suggests the efficient exciton separation. And the very weak PL of the blends is further quenched when processed with Py or Py+TA, indicating that more efficient exciton dissociation occurs in the active layers.

### Table 2. Summary of device performances for Por-Rod and Por-CNRod

<table>
<thead>
<tr>
<th>Molecule</th>
<th>Conditions</th>
<th>$J_{sc}$ (mA/cm$^2$)</th>
<th>$V_{OC}$ (V)</th>
<th>FF (%)</th>
<th>PCE (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Por-Rod</td>
<td>w/o</td>
<td>0.81</td>
<td>0.73</td>
<td>25.19</td>
<td>0.15</td>
</tr>
<tr>
<td></td>
<td>Py</td>
<td>1.44</td>
<td>0.86</td>
<td>22.35</td>
<td>0.28</td>
</tr>
<tr>
<td></td>
<td>TA$^a$</td>
<td>0.94</td>
<td>0.74</td>
<td>25.84</td>
<td>0.18</td>
</tr>
<tr>
<td></td>
<td>Py+TA</td>
<td>12.39</td>
<td>0.94</td>
<td>42.71</td>
<td>4.97</td>
</tr>
<tr>
<td>Por-CNRod</td>
<td>w/o</td>
<td>0.72</td>
<td>0.87</td>
<td>19.68</td>
<td>0.12</td>
</tr>
<tr>
<td></td>
<td>Py</td>
<td>2.43</td>
<td>0.94</td>
<td>26.39</td>
<td>0.60</td>
</tr>
<tr>
<td></td>
<td>TA</td>
<td>0.37</td>
<td>0.81</td>
<td>21.09</td>
<td>0.06</td>
</tr>
<tr>
<td></td>
<td>Py+TA</td>
<td>1.37</td>
<td>0.89</td>
<td>26.79</td>
<td>0.33</td>
</tr>
</tbody>
</table>

$^a$1 v% pyridine, thermal annealing at 100 °C.

### Hole-Mobility Measurement

The hole and electron mobilities of the blend films under different conditions shown in Figure 5, Figure S1 and Table S1 were estimated according to the electric-field dependent space charge limited current (SCLC) model in the configuration of ITO/PEDOT:PSS (40 nm)/active layer (85 nm)/MoO$_3$ (10 nm)/Ag (~60 nm) using the following equation:

$$J = \frac{9}{8} \varepsilon_r \varepsilon_0 \frac{E^2}{d} \mu_0 \exp(\beta E)$$

Where $J$ is the current, $\varepsilon_r$ is the relative permittivity of the material, $\varepsilon_0$ is the permittivity of free space, $E$ is the effective electric field, $d$ is the thickness of the active layer, $\mu_0$ is the zero-field mobility and $\beta$ is the field activation factor.

![Figure 5. I-V characteristics of the hole-only devices in the configuration of ITO/PEDOT:PSS/active layer/MoO$_3$/Ag.](image)

Compared with those of pure chlorobenzene processed thin films, the electron mobility decreased to the half and hole mobility increased by five times for the bends fabricated with pyridine additive. However, thermal annealing and thermal annealing after adding pyridine lead to obvious improvements in hole and electron mobilities compared with processing with pyridine, which can be an important reason for the increase of FF and $J_{sc}$. The other reason is the smaller $\mu_h/\mu_e$ ratio than that of the devices fabricated with Py and TA. Also, The SCLC hole mobilities of Por-Rod and Por-CNRod blend films with PC$_7$BM under Py+TA condition are calculated to be $8.5 \times 10^{-5}$ and $7.5 \times 10^{-5}$ cm$^2$ V$^{-1}$ s$^{-1}$, respectively. The higher hole mobility of Por-Rod can be one of the reasons why Por-CNRod-based devices show the higher $J_{sc}$ because a higher mobility usually lead to a smaller photocurrent loss caused by charge recombination.

### Morphology Characterizations

![Figure 6. GIXD of Por-Rod and Por-CNRod in BHJ blends. (a, e) as spun from CB; (b, f) thermal annealed film casted from CB; (c, g) thin film casted from CB+Py mixture; (d, h) thermal annealed films from CB+Py processing.](image)
lamellae distance. It should be noted that in our previous studies that porphyrin and DPP based molecules are quite weak in molecule packing, even without obvious π-π stacking signal. The modification in chemical structure, replacing the DPP moiety with thiophene and rhodanine, leads to stronger intermolecular packing. The crystal size is estimated using Scherr equation. The (100) crystal size is estimated to be 15.64 nm, and the π-π stacking peak is estimated to be 5.97 nm. For Por-CNRod, (100) distance is estimated to be 22.14 Å (located at 0.28 Å⁻¹), π-π stacking distance is estimated to be 3.46 Å (located at 1.82 Å⁻¹). Thermal annealing of as casted BHJ thin film leads to shifting of (100) peaks, which is more server in Por-CNRod blends. The (100) peak shifted to 0.24 Å⁻¹, a ~2 Å distance shifting. The π-π stacking peak shifted to 1.86 Å⁻¹; the size is also increased about 9.67, corresponding to ~3 layer of π stacks. When pyridine is used in thin film processing, the nanostructure of BHJ thin film changed completely. Both (100) and π-π stacking peak became much less intensive and crystal size is largely reduced because pyridine is quite effective in reducing the crystallinity of porphyrin based materials due to the coordination with the metal center, which will strongly retard the molecular assembly. The subsequent thermal annealing does not help to improve the structure order of the BHJ thin film. Quite similar GIXD results are obtained.

The length scale of phase separation was studied by using resonant soft X-ray scattering methods (RSoXS) by taking advantage of high scattering contrast at the carbon k-edges. For both Por-Rod and Por-CNRod blends, the as-cast samples show a quick decay of scattering signals in the very low q region, indicating a large scale phase separation outside of the probing length scales. Thermal annealing does not generate new length scale of phase separation and a nearly identical morphology is seen in this system. When pyridine additive is used, a small length scale of phase separation at ~20 nm (0.032 Å⁻¹) is seen for Por-Rod blends. Further thermal annealing leads to enhancement of this scattering peak at an even larger q positions, corresponding to a length scale of ~16 nm (0.039 Å⁻¹). This new scattering feature can be the critical of enhancing the device performance, giving a much larger short circuit current and power conversion efficiency. For Por-CNRod blends, using pyridine as the additive do lead to a smaller length scale of phase separation showing a peak at 0.025 Å⁻¹ with low scattering intensities. Further thermal annealing does not improve this length scale of phase separation. A quite similar scattering profile is recorded. From morphological aspects, it should be concluded that both Por-Rod and Por-CNRod blends processed from pyridine additive and the subsequent thermal annealing should lead to improved device performances. The low lying LUMO energy level of Por-CNRod, which cannot align well with PCBM, can be a major issue to efficiently split excitons and generate current.

Figure 7. Line-cut profiles of GIXD results. (a) out-of-plane line cut of Por-Rod blends; (b) in-plane line cut of Por-Rod blends; (c) out-of-plane line cut of Por-CNRod blends; (d) in-plane line cut of Por-CNRod blends.

Figure 8. RSoXS of (a) Por-Rod blends; (b) Por-CNRod blends.

Conclusions
In summary, two new conjugated A-D-A porphyrin small molecules of Por-Rod and Por-CNRod are designed and synthesized through Sonogashira reactions. The BHJ OSCs based on the two porphyrins and PC₇BMB as the active materials in a conventional device structure under different conditions were investigated, and the Por-Rod-based devices processed with pyridine additive and then thermal annealing exhibit a PCE up to 4.97% with a V₉₄ of 0.94 V and a maximum EQE up to 61%. The energy loss for Por-Rod-based devices is only 0.53 eV which can compare to perovskite solar cells, and this is the first report that such low energy loss BHJ OSCs can exhibit EQE values more than 60%. However, Por-CNRod-based OSCs show very poor performance under the investigated condition even though a very low energy loss of only 0.51 eV has been recorded. Morphology studies using GIXD and RSoXS show that while pyridine additive reduces the crystallinity of porphyrin and the length scale of phase...
separation, the subsequent thermal annealing leads to the enhancement of the scattering peak corresponding to an even smaller length scale, which can be critical for the enhanced performance of Por-CN-based devices. The morphology of Por-CNRod blends is quite similar with that of Por-CN blends under the same fabrication conditions, and the low lying LUMO energy level of Por-CNRod, which cannot align well with PCBM to efficiently split excitons and generate current, can be a major issue for the poor performance of all the Por-CNRod-based OSCs. The results provide a further understanding of the energy loss and help to design new donors with high $V_{oc}$ values for more efficient organic solar cells.

Acknowledgements

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Notes and references

TOC:

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The BHJ OSCs show an energy loss of 0.53 eV but a maximum external quantum efficiency up to 61%.