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Conjugated Polymers

Diketopyrrolopyrrole-Porphyrin Based Conjugated Polymers for Ambipolar Field-Effect Transistors

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Abstract: Porphyrin-based molecules have been widely used in dye-sensitized solar cells and bulk heterojunction solar cells, but their application in field-effect transistors (FETs) is limited. In this work, two conjugated polymers based on diketopyrrolopyrrole and porphyrin units were developed for FETs. The polymers exhibit extra-low band gap with energy levels close to -4.0 eV and -5.0 eV due to the strong electron-donating and withdrawing ability of porphyrin and diketopyrrolopyrrole. With additionally high crystalline properties, ambipolar charge carrier transports with a hole mobility of $0.1 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$ in FETs were realized in these polymers, representing the highest performance in solution-processed FETs based on porphyrin unit.

Organic field-effect transistors (OFETs) as the components to build organic electric devices, such as electronic skin, electronic paper, sensor and organic RFID tag, have received tremendous attention in recent years. Solution-processed conjugated polymers with high molecular weight and viscosity are able to apply into roll to roll printing technique, providing the advantage to realize large-area flexible OFET devices. Conjugated polymers with "donor-acceptor" structures have been explored toward high charge carrier mobilities. While many conjugated polymers have shown high hole/electron mobility, the study of conjugated polymers with ambipolar charge transport has been lagged behind. Since the transistors that transport hole

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and electron in single-component material have some particular application, such as CMOS-like circuits^[6] and light-emitting transistors,^[7] it is important to explore new conjugated polymers with high ambipolar mobilities.

Frontier energy alignment is the crucial parameter to realize both hole and electron transport in one conjugated polymer, due to charge injection between conjugated polymers and electrodes.[8] In OFET devices with gold as source and drain electrode, the highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) levels close to -5.0 eV and -4.0 eV are beneficial for hole and electron injection. This indicates that conjugated polymers with extra-low band gaps approaching 1.0 eV are the candidates to realize ambipolar FETs. One design strategy to lower the band gap and realize these energy levels is to use "acceptor-acceptor" structure, in which two electron-deficient units are incorporated into conjugated polymers. [5d, e, 9] For example, by using strong electron-deficient diketopyrrolopyrrole (DPP) and benzothiadiazole, the corresponding conjugated polymer exhibited a small band gap of 1.2 eV with HOMO and LUMO levels of -5.2 eV and -4.0 eV, consequently resulting in high hole and electron mobilities of $0.35 \text{ cm}^2 \text{V}^{-1} \text{s}^{-1}$ and $0.40 \text{ cm}^2 \text{V}^{-1} \text{s}^{-1}$. [5d] Another route to realize small band gap is to use strong electron-deficient and electron-rich units to construct conjugated polymers, in which the HOMO levels are usually close to -5.0 eV.[10] However, the LUMO levels of this kind of polymers are positioned far away from -4.0 eV, resulting in hole-only FETs. Therefore, a rational selection of alternating building blocks for "donor-acceptor" polymers is important to realize ambipolar transistors.

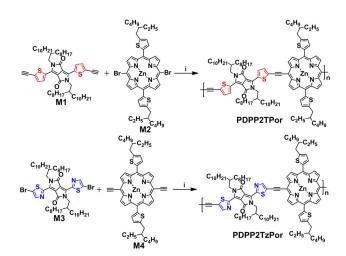
Porphyrin (Por) is a strong electron-rich unit that has been used to build small band gap conjugated materials for application in organic electronics. However, porphyrin-based conjugated polymers have been rarely reported in FETs, which is possibly due to their low hole mobility below $10^{-4} \, \mathrm{cm^2 \, V^{-1} \, s^{-1}}$. Since porphyrin based small molecules as electron-donor to electron acceptor have been successfully applied in organic solar cells with high photon-conversion efficiencies and broad photo-response up to 900 nm, these results indicate that porphyrin can be potentially used as hole electron transporting materials in FETs. In this work, we develop two conjugated polymers containing DPP and porphyrin units connected by thiophene or thiazole, and their charge transport properties in FETs were symmetrically studied. The ambipolar FETs based on the polymers were obtained, in





which the hole mobility of 0.1 cm²V⁻¹s⁻¹ represents the highest mobility in porphyrin-based conjugated polymers.

The chemical structure and the synthetic routes of the two polymers are shown in Scheme 1. We select thiophene or thia-



Scheme 1. Chemical structures of the DPP-Por polymers and their synthetic routes. (i) Sonogashira polymerization by using $Pd(PPh_3)_{4r}$ Cul and diisopropylamine in THF at 80 °C.

zole as bridge to construct the polymers, since thiazole is able to effectively lower the energy levels of the polymers.^[13] Thiophene-based polymer PDPP2TPor was synthesized via Sonogashira polymerization by using ethynyl-linked DPP-based monomer M1 and dibromono-Por monomer M2, while thiazole-contained polymer PDPP2TzPor was prepared by using dibromo-DPP monomer M3 and ethynyl-Por monomer M4. The attempt to synthesis ethynyl-thiazole DPP monomer from the monomer M3 was failed. It is worthy to mention that conjugated polymers with the same backbone as PDPP2TPor have been reported for application in organic solar cells.^[14] The polymers show good solubility in CHCl₃ and chlorobenzene. The molecular weight of the polymers was determined by gel permeation chromatography (GPC) measurement with ortho-dichlorobenzene (o-DCB) as eluent under 140 °C as shown in Figure S1 and S2 in the supporting information and the data was summarized at Table 1. PDPP2TPor and PDPP2TzPor have the peak molecular weight (M_p) of 29.3 kg mol⁻¹ and 17.4 kg mol⁻¹, but the polydispersity index (PDI) was relatively high. Therefore, the number average molecular weight (M_n) was relatively low,

Table 1. Molecular weight, optical and electrochemical properties of the DPP-Por polymers.

| Polymer | $M_{\rm p}^{\rm [a]}$ [kg mol ⁻¹] | $E_{ m g}^{ m film}$ [eV] | E _{HOMO} [b] [eV] | E _{LUMO} ^[c] [eV] |
|------------|---|------------------------------|-------------------------------|--|
| PDPP2TPor | 29.3 | 1.29 | -5.28 | -3.99 |
| PDPP2TzPor | 17.4 | 1.26 | -5.34 | -4.08 |

[a] Determined by GPC at 140 °C using o-DCB as the eluent. [b] Estimated from the onset potential of the first oxidation wave using a work function value of -4.8 eV for Fc/Fc $^+$. [c] Determined as $E_{\text{HOMO}} + E_{\text{q}}^{\text{flim}}$.

which was due to the small molecular weight fraction in the polymers (Figure S1 and S2). Since we apply high temperature GPC and low concentration (0.1 mg mL⁻¹) to perform molecular weight measurement, we infer that high molecular weight fraction is not from the aggregation. Therefore, we infer that PDPP1 and PDPP2 have high molecular weight fractions along with some oligomers.

The two polymers perform broad absorption spectra from 300 nm to 1000 nm with three distinct regions, in which the region in 400–500 nm can attribute to the Soret-band of porphyrin (Region I), and the near-infrared absorption between 650 nm and 1000 nm (Region II) was due to the intramolecular interaction of DPP and Por. The region around 550 nm has weak intensity, which may be due to the Q-bands of porphyrin (Figure 1). Although the two polymers perform similar optical band gap (E_g) of 1.29 eV and 1.26 eV (Table 1), the relative intensity between Region I and Region II is different (Figure 1). PDPP2TPor shows high absorption intensity at Region II, indicating that thiophene unit can facilitate the intramolecular charge transfer between DPP and Por unit compared to thiazole.

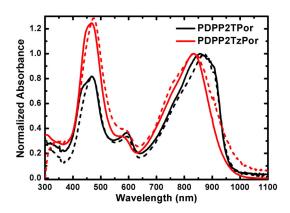


Figure 1. Optical absorption spectra of PDPP2TPor and PDPP2TzPor in CHCl₃ (solid lines) and in thin films (dashed lines).

Frontier energy levels of the polymers determined by cyclic voltammetry measurement were shown in Figure S3 in the Supporting Information and the data was summarized at Table 1. HOMO levels calculated from oxidation potential are $-5.28~\rm eV$ and $-5.34~\rm eV$ for PDPP2TPor and PDPP2TzPor, and LUMO levels were $-3.99~\rm eV$ and $-4.08~\rm eV$ as determined by HOMO levels and $E_{\rm g}$. PDPP2TzPor shows low-lying energy levels compared to those of PDPP2TPor, which is due to the electron-negative thiazole units. Both of the polymers desire HOMO levels close to $-5.0~\rm eV$ and LUMO levels around $-4.0~\rm eV$, which would be beneficial for ambipolar charge transport.

The charge transport properties of the polymers were studied by fabricating FET devices with bottom-gate bottom-contact (BGBC) configuration, and the detailed experimental procedure was shown in the Supporting Information. The polymer thin films were solution-processed from CHCl₃ with 10% *o*-DCB onto the OTS-modified Si/SiO₂ substrate. The thin films were

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thermal-annealed at different temperature in order to achieve optimized mobility (Figure S4, Table S1 and S2). Both of the polymers perform the best hole and electron mobilities under thermal annealing at 200 °C (Table 2, Figure 2). The improved mobility at this annealing temperature may be due to better crystallization of the polymers with ordered structures as shown in atomic force microscopy (AFM) images in Figure S5 in the Supporting Information.

A high hole mobility of $0.1~\text{cm}^2\text{V}^{-1}\text{s}^{-1}$ and electron mobility of $0.004~\text{cm}^2\text{V}^{-1}\text{s}^{-1}$ could be obtained for the polymer PDPP2TPor, while PDPP2TzPor exhibits relatively low hole mobility of $0.017~\text{cm}^2\text{V}^{-1}\text{s}^{-1}$ and electron mobility of $0.002~\text{cm}^2\text{V}^{-1}\text{s}^{-1}$ in FET devices. To the best of our knowledge, this is the first time for porphyrin-based polymer to achieve ambipolar transistors and also $0.1~\text{cm}^2\text{V}^{-1}\text{s}^{-1}$ in PDPP2TPor

Table 2. Hole and electron mobility of the polymers based on field-effect transistors and their crystalline properties in thin films.

| Polymer | $\mu_{\rm h}^{{ m [a]}}$ [cm 2 V $^{-1}$ s $^{-1}$] | $\mu_{\rm e}$ [cm 2 V $^{-1}$ s $^{-1}$] | d(100) ^[b] [Å] | <i>d</i> (010) ^[c] [Å] |
|------------|---|--|------------------------------|--------------------------------------|
| PDPP2TPor | 0.1 | 0.004 | 22.4 | 4.1 |
| PDPP2TzPor | 0.017 | 0.002 | 25.1 | 3.9 |

[a] The average hole mobilities from eight devices for PDPP2TPor and PDPP2TzPor were $0.084~\rm cm^2V^{-1}~s^{-1}$ and $0.015~\rm cm^2V^{-1}~s^{-1}$. [b] Calculated from q values as $0.28~\rm \AA^{-1}$ and $0.25~\rm \AA^{-1}$ for PDPP2TPor and PDPP2TzPor. [c] Calculated from q values as $1.55~\rm \AA^{-1}$ and $1.62~\rm \AA^{-1}$ for PDPP2TPor and PDPP2TzPor. The thin films were spin coated from CHCl $_3$ with 10% o-DCB and thermal annealed at $200~\rm ^{\circ}C$ for $10~\rm min$ before measurement.

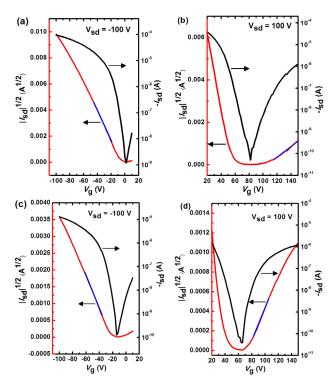


Figure 2. Transfer curves obtained from BGBC FET devices. (a, b) PDPP2TPor, (c, d) PDPP2TzPor, (a, c) for hole mobility calculation, (b, d) for electron mobility calculation. The blue lines are used for mobility calculations.

represents the highest hole mobility based on porphyrin-contained polymers.^[12] The low hole mobility and electron mobility in PDPP2TzPor is possibly due to the low molecular weight,^[15] deep HOMO levels^[16] or the preferentially "face-on" orientation of the polymers in thin films as discussed below.^[17]

Crystalline properties of the polymer thin films have the significant impact on the FET mobility, so we use atomic force microscopy (AFM) and grazing-incidence wide-angle X-ray scattering (GIWAXS) techniques to study the PDPP2TPor and PDPP2TzPor based thin films, as shown in Figure 3. AFM shows

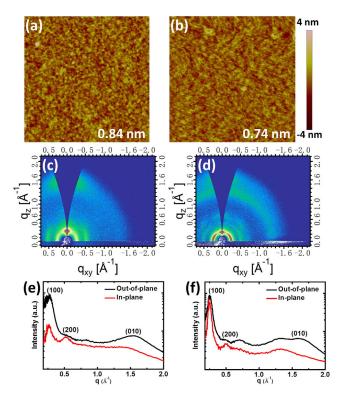


Figure 3. Characteristics of the polymer thin films spin coated from CHCl $_3$ with 10% o-DCB and thermally annealed at 200 °C for 10 min. (a, b) AFM height image (3×3 μ m 2). (c, d) GIWAXS patterns, and (e, f) the out-of-plane and in-plane cuts of the corresponding GIWAXS patterns.

the phase separated information on the surface of thin films, and both of the polymers were found to have small domain regions, indicating the crystalline polymers in thin films. This can be further confirmed by GIWAXS measurement in Figure 3 c-f. The polymer PDPP2TPor performs high diffraction peaks (100) both at out-of-plane and in-plane direction, corresponding to the lamellar stacking of alkyl side chains with d-spacing of 22.4 Å (Table 2). High ordered diffraction peak (200) can be observed at in-plane direction, indicating the good crystalline property of PDPP2TPor. A broad diffraction peak (010) is presented at out-of-plane direction, corresponding to the π - π stacking of conjugated backbone with d-spacing of 4.1 Å. The results reveal that PDPP2TPor performs mixed orientation of "edge-on" and "face-on" stacking. PDPP2TzPor has similar (100) and (010) diffraction peaks at out-of-plane direction, and (100) and (200) at in-plane direction as PDPP2TPor. In addition, the





intensity of (100) peak at in-plane direction is much higher than that at out-of-plane direction, indicating that PDPP2TzPor has the tendency to form "face-on" orientation in thin films. The d-spacings of the lamellar stacking and π - π stacking is 25.1 Å and 3.9 Å for PDPP2TzPor, revealing its condensed π – π stacking compared with PDPP2TPor. We also observed that PDPP2TPor showed clear diffraction peak (010) compared to that of PDPP2TzPor, corresponding to the large crystal correlation length (0.9 nm vs. 0.5 nm) calculated from the full width at half maximum values. This may facilitate the charge transport via the π - π stacking direction, partially responsible for the high mobility in PDPP2TPor. GIWAXS results demonstrate that porphyrin with large π -conjugated cycles can provide good crystalline property for their conjugated polymers, and hence are able to realize high charge transport in FETs.

In conclusion, two conjugated polymers containing electrondeficient DPP unit and electron-rich porphyrin unit were developed for application in FETs. The two polymers exhibit HOMO and LUMO levels around -5.3 eV and -4.0 eV, and good crystallinity as evidenced by GIWAXS measurement. These properties enable the two polymers exhibit ambipolar charge transport in FET devices, including the highest hole mobility of 0.1 cm²V⁻¹s⁻¹ among porphyrin-based conjugated polymers. The results show the potential application of porphyrin unit into high performance FETs.

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Conflict of interest

The authors declare no conflict of interest.

Keywords: ambipolar mobility · conjugation · field-effect transistors · polymers · porphyrinoids

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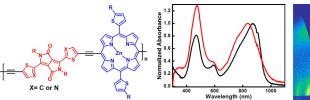
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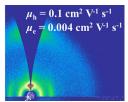




COMMUNICATION



Mind the gap: Small-band-gap diketopyrrolopyrrole-porphyrin based conjugated polymers were developed for application in ambipolar organic field-



effect transistors, in which the hole and electron mobility of 0.1 and 0.004 cm²V⁻¹s⁻¹ was achieved.

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