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Rational design of a difluorobenzo[c]cinnoline-based low-bandgap copolymer for high-performance polymer solar cells†

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A copolymer with A–A structure is rationally designed and synthesized by using difluorobenzo[c]cinnoline (DFBC) as weak electron-withdrawing (A) moieties and 1,4-diketo-pyrrolo[3,4-c]pyrrole (DPP) as strong A moieties. Polymer solar cells based on **PDFBC-DPP** and PC₇₁BM showed a promising power conversion efficiency of 7.92%, which is one of the highest values in polymer solar cells based on A–A structure.

Polymer solar cells (PSCs) have attracted considerable interest because of their facile solution processability, low cost, and flexibility.^{1–5} Although power conversion efficiencies (PCEs) of fullerene-based and fullerene-free singlet junction PSCs both have exceeded 10% recently,^{6–8} it is necessary that new high-performance photovoltaic materials should be continually developed to realize the commercial purpose in the future. To exploit high-performance polymer photovoltaic materials, several design strategies have been carried out, such as exploiting new building blocks,^{9–19} designing the structure of polymer backbones,^{20–28} and optimizing side chains.^{29–37} Many efforts have been devoted to designing polymer backbones. Copolymers based on weak electron-donating (D) moieties and strong electron-withdrawing (A) moieties have been widely studied because of their low bandgap and appropriate energy levels. Moreover, terpolymers with A–D1–A–D2^{38,39} or D–A1–D1–A2^{40,41} structures have been proposed in order to broaden the absorption spectra of D–A polymers. Recently, a few conjugated polymers with A–A structure were reported and used as

the donor materials of PSCs.^{42,43} The A–A type polymer has not been paid enough attention because it currently shows a poor photovoltaic performance (PCE < 5%). The polymer PDPP2TPCL with pentacyclic lactam (PCL) and diketopyrrolopyrrole (DPP) as the A units showed a PCE value of 4.7%.⁴³

Benzo[c]cinnoline (BC) and its derivatives possess rigid and planar conjugated structure, and it has been used as weak A units in materials for n-channel organic field-effect transistors and photoluminescence.^{44,45} In the manuscript, 2,9-difluorobenzo[c]cinnoline (DFBC) was designed as a new weak A unit in A–A copolymers for the first time (Fig. 1) in order to lower the low-lying highest occupied molecular orbital (HOMO) energy level and enhance the molecular aggregation because of fluorination.^{46–49} Simultaneously, the DPP units were introduced as strong A units to reduce the bandgap and broaden the absorption spectra of the polymer. Additionally, two long alkyl-substituted thiophene derivatives were introduced between DFBC and DPP units in order to improve the solubility of the polymer. As a result, a novel copolymer **PDFBC-DPP** with DFBC as weak A units and DPP as strong A units was synthesized as a polymer photovoltaic material. As expected, the synthesized copolymer shows a low bandgap of 1.53 eV and a low HOMO energy level (−5.35 eV), which is lower than most DDP-based polymers with typical value above −5.25 eV. Consequently, the PSCs fabricated with **PDFBC-DPP** and fullerene derivatives could achieve high open circuit voltages (V_{oc}) (0.78–0.80 V), which are higher than those of most DDP-based polymer PSCs (<0.70 V).^{50,51} Moreover, the PSC device fabricated with **PDFBC-**

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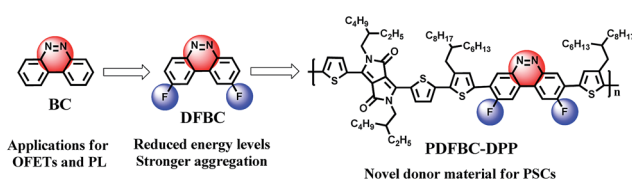


Fig. 1 The design strategy of DFBC and the structure of the conjugated polymer **PDFBC-DPP**.

DPP and (6,6)-phenyl- C_{71} -butyric acid methyl ester (**PC₇₁BM**) as the photoactive layer shows a promising PCE of 7.92%, which is one of the highest PCE values for copolymers with A-A structure.

The synthesis process of the monomer and the copolymer **PDFBC-DPP** is shown in Scheme 1, and the detailed experimental procedures are described in the ESI† (S1, Experimental section). The polymer **PDFBC-DPP** was synthesized by a typical Suzuki coupling reaction with monomers **M1** and **M2** in toluene with $Pd_2(dba)_3$ as the catalyst. The polymer was purified through sequential Soxhlet extractions with methanol, acetone, hexane, dichloromethane and chloroform. The polymer exhibits good solubility in chloroform, chlorobenzene (CB) and *o*-dichlorobenzene (*o*-DCB) at room temperature, which provides convenience for device processing. The average molecular weight and polydispersity index (PDI) of the copolymer, determined by using Waters 1515 gel permeation chromatography (GPC) analysis with $CHCl_3$ as the eluent and polystyrene as the standard, are 73.2 kDa and 1.7, respectively. The polymer **PDFBC-DPP** exhibits a decomposition temperature (T_d , 5% weight loss) of 415 °C (Fig. S1†) and no glass transition (Fig. 3), which indicates that its thermal stability is sufficiently good for application in photovoltaic devices.

The optimized molecular geometry of **PDFBC-DPP** was calculated using the Gaussian 09 program suite at the B3LYP/6-31G(d) level. As shown in Fig. S2,† the dihedral angle between DFBC and alkylthiophene is 8.2°, and the dihedral angle between alkylthiophene and DPP is 7.7°. These results indicate that **PDFBC-DPP** possesses good molecular coplanarity, which would be beneficial to molecular aggregation and hole transport.

Temperature-dependent absorption spectra of **PDFBC-DPP** in *o*-DCB solution are shown in Fig. 2a, indicating an obvious temperature-dependent aggregation property.²² At 95 °C, the polymer is almost disaggregated, showing an absorption peak at 619 nm. With the decrease of the temperature, a new peak around 713 nm emerges, which reveals a strong aggregation in solution. At 25 °C, the polymer shows two distinct absorption

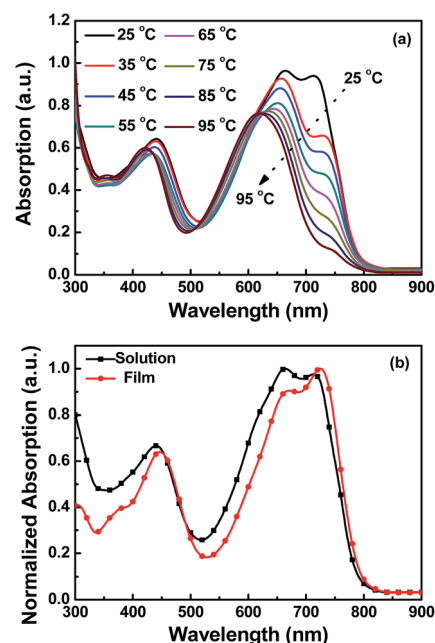
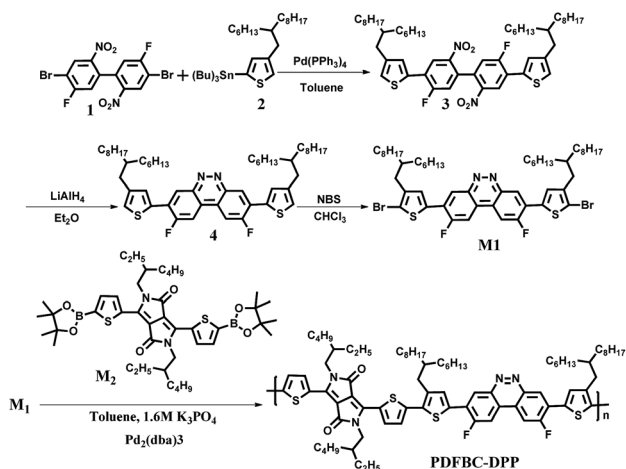


Fig. 2 Temperature-dependent absorption spectra of **PDFBC-DPP** in *o*-DCB solution (a) and absorption spectra of **PDFBC-DPP** in chloroform solution and thin film (b).

peaks in the longer wavelength region. The maximum absorption peak at 664 nm can be identified with the intramolecular charge transfer (ICT), and the shoulder peak at 713 nm is attributed to the intermolecular charge transfer. The absorption spectra of **PDFBC-DPP** in chloroform solution and thin film on a quartz plate are shown in Fig. 2b, and the corresponding data are collected in Table S1.† Obviously, the maximum absorption peak of the polymer film is 725 nm, which is red-shifted compared with that in *o*-DCB solution (664 nm) and chloroform solution (664 nm). In other words, the polymer film possesses stronger molecular aggregation than that in solution. According to the absorption edge (793 nm), the optical bandgap is calculated to be 1.56 eV.

The electrochemical properties of **PDFBC-DPP** were investigated using cyclic voltammetry. As shown in Fig. S3,† the onset oxidation and reduction potentials of the polymer were measured to be about 0.58 V and −0.95 V (vs. Ag/Ag^+), respectively. Since the redox potential of Fc/Fc^+ was measured and



Scheme 1 Synthetic route of the polymer **PDFBC-DPP**.

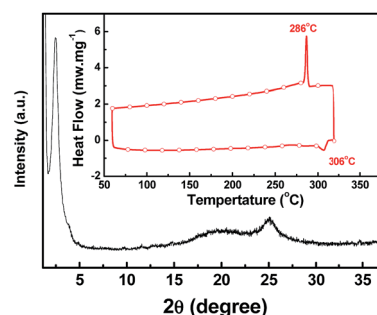


Fig. 3 DSC curve and WAXRD pattern of the polymer film.

calculated to be 0.03 V, the HOMO and the lowest unoccupied molecular orbital (LUMO) energy levels of **PDFBC-DPP** can be calculated to be -5.35 eV and -3.82 eV, respectively. It is well accepted that the HOMO and LUMO energy levels of a D-A copolymer depend on the HOMO energy level of the D units and the LUMO energy level of the A units.⁵² Therefore, **PDFBC-DPP** possesses a low LUMO energy level of -3.82 eV because of the strong electron-deficient property of DPP units. The LUMO energy level (-3.82 eV) is higher 0.48 eV than that of PC₇₁BM (-4.3 eV),⁵¹ which ensures a downhill driving force for efficient exciton dissociation. Simultaneously, the HOMO energy level of **PDFBC-DPP** is mainly decided by weak electron-deficient DFBC units, since the polymer does not contain any typical D units. Therefore, **PDFBC-DPP** presents a low HOMO energy level of -5.35 eV. It is obviously lower than most DDP-based polymers with typical value above -5.20 eV,⁵⁰ which would lead to better air stability⁵³ and higher V_{oc} in PSCs.

The molecular aggregation and crystallinity property of the polymer **PDFBT-DPP** are investigated by differential scanning calorimetry (DSC) and wide angle X-ray diffraction (WAXRD). As shown in Fig. 3, there is a sharp exothermic peak at 286 °C during cooling, which indicates that **PDFBT-DPP** is a crystalline polymer. The WAXRD of the polymer film gives a similar result (Fig. 3). It presents a sharp 100 peak at 2.46° and a 010 peak at 25.01° , corresponding to a lamellar distance of 35.88 Å and a π - π stacking distance of 3.56 Å, respectively. All these results indicate that **PDFBT-DPP** possesses a strong molecular aggregation and crystallinity property.

The photovoltaic properties of **PDFBC-DPP** were investigated in PSCs with the conventional configuration ITO/PEDOT:PSS (30 nm)/**PDFBC-DPP**:PCBM/LiF (0.5 nm)/Al (100 nm). The active layers were spin-coated from chlorobenzene solution. At the beginning, the preliminary device optimization was carried out by optimizing the weight ratios of **PDFBC-DPP** and PC₆₁BM from 1 : 1 to 1 : 3 (w/w), and the J - V curves are shown in Fig. S4.† As shown in Table S2,† the optimized **PDFBC-DPP** : PC₆₁BM devices, prepared with a blend weight ratio of 1 : 1 (w/w), exhibited the best PCE value of 7.33%, with a short circuit photocurrent density (J_{sc}) of 13.15 mA cm⁻², a V_{oc} value of 0.80 V, and a fill factor (FF) of 0.68. It is noted that the PSC device shows a higher V_{oc} value than most DPP-based PSC devices.⁵⁰ Subsequently, PC₇₁BM was used as the acceptor material to substitute PC₆₁BM. Obviously, the PC₇₁BM-based

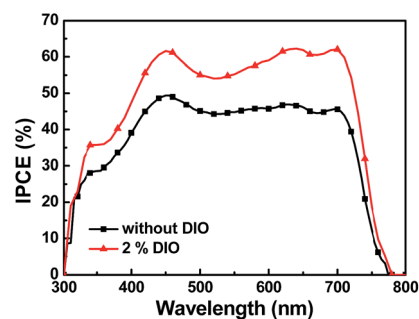


Fig. 5 IPCE curves of the PSC devices (**PDFBC-DPP** : PC₇₁BM = 1 : 1).

PSC device shows higher J_{sc} , FF and PCE values than those of the PC₆₁BM-based PSC devices (Table S2†). A similar phenomenon has been reported because PC₇₁BM possesses stronger absorption than PC₆₁BM in the visible region.⁵⁴ Finally, the optimized additive content of the blend solution was investigated in the PSC devices with **PDFBC-DPP** and PC₇₁BM. As shown in Fig. S5,† the PC₇₁BM-based PSC device prepared without 1,8-diiodooctane (DIO) shows inferior PCE values, it only shows the J_{sc} value of 11.87 mA cm⁻² and the PCE value of 6.11%. With the increase of the DIO content (vol%), the PSC devices exhibit better photovoltaic properties. The PSC device shows the best photovoltaic parameters when the DIO content is 2%. It presents the highest J_{sc} value of 14.58 mA cm⁻² and the best PCE value of 7.92% (Table S2† and Fig. 4). The incident photon-to-current conversion efficiency (IPCE) curves are shown in Fig. 5. The PSC device prepared with 2% DIO exhibits a high IPCE response over 300–780 nm with a maximum IPCE value 61.62% at 450 nm. The corresponding J_{sc} value obtained from the IPCE curve is 13.88 mA cm⁻² for the PSC device (2% DIO), which agrees within experimental error with the measured J_{sc} . All the PC₇₁BM-based PSC devices prepared with

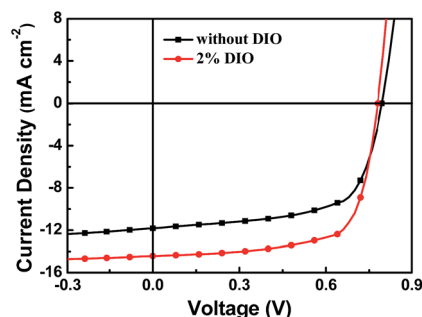


Fig. 4 J - V curves of the PSC devices (**PDFBC-DPP** : PC₇₁BM = 1 : 1).

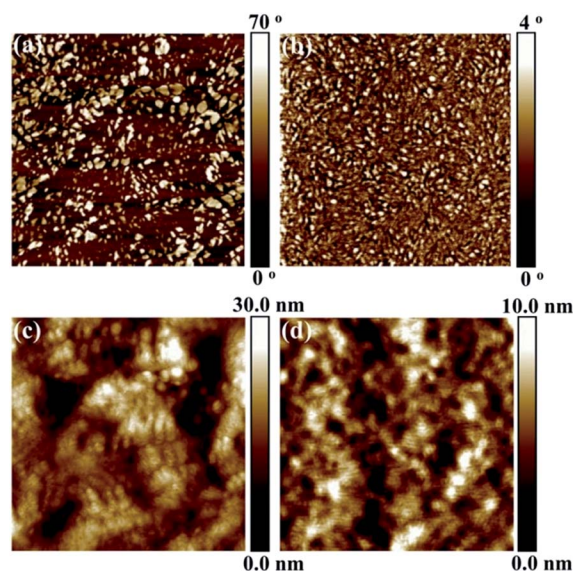


Fig. 6 AFM phase images and height images of the **PDFBC-DPP**/PC₇₁BM blend film. (a and c) Without DIO and (b and d) with 2% DIO.

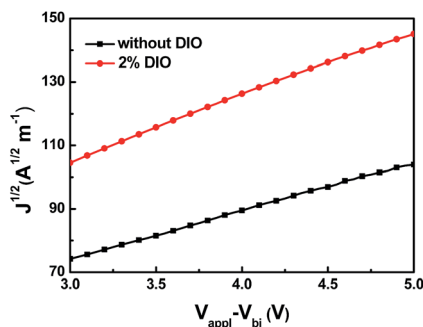


Fig. 7 The $J^{1/2}$ - V curves of the hole-only devices based on the PDFBC-DPP:PC₇₁BM blend film without or with 2% DIO. The thicknesses for the blend films are 110 nm.

DIO show the same V_{oc} value of 0.78 V, which indicates that these PSC devices present a low photon energy loss of 0.75 eV (E_{loss})⁵⁵ due to the appropriate energy levels and optical bandgap.

To better understand the effect of the morphology on the device performance of PDFBC-DPP : PC₇₁BM (1 : 1, w/w) based PSCs, the morphologies of PSCs prepared without DIO or with 2% DIO are characterized by atomic force microscopy (AFM). According to the AFM phase and height images (Fig. 6), the blend film obtained without DIO shows a large domain size and RMS (surface root mean square roughness) value of 4.70 nm. However, the blend film obtained with 2% DIO exhibits a small domain size and RMS (surface root mean square roughness) value of 1.88 nm. Smaller domain size and RMS value would lead to more effective charge separation and higher charge transport capacity.

The hole mobility was measured by the SCLC method with the configuration of ITO/PEDOT:PSS (30 nm)/PDFBC-DPP:PC₇₁BM/MoO₃ (10 nm)/Al (100 nm). The mobility was determined by fitting the dark current, which is described by:¹⁰

$$J = \frac{9}{8} \epsilon_0 \epsilon_r \mu \frac{V^2}{d^3}$$

where J is the current density, μ is the zero-field mobility of holes (μ_h), ϵ_0 is the permittivity of the vacuum, ϵ_r is the relative permittivity of the material, d is the thickness of the blend film, and V is the effective voltage, $V = V_{appl} - V_{bi}$, where V_{appl} is the applied voltage and V_{bi} is the built-in potential determined by the work function difference of the electrodes. Fig. 7 shows J - V curves for the hole-only devices. The mobilities are calculated from the slope of the curves. As mentioned before, the blend film with 2% DIO possesses smaller domain size and smaller RMS value than those without DIO, so the former exhibits higher hole mobility ($1.85 \times 10^{-4} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$) than the latter ($1.03 \times 10^{-4} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$) (Table S2†). As a result, the corresponding PSC device prepared with 2% DIO shows a higher J_{sc} value (14.58 mA cm^{-2}) and PCE value (7.92%) compared with that fabricated without DIO (Table S2†).

In summary, a novel alternate copolymer PDFBC-DPP with DFBC as weak A units and DPP as strong A units was rationally designed and synthesized. The copolymer shows a low bandgap

of 1.53 eV, a low HOMO energy level of -5.35 eV , and a wide absorption spectrum. The PSCs fabricated with PDFBC-DPP and PC₇₁BM achieve a promising PCE of 7.92%, which is one of the highest values in PSCs based on the polymer with A-A structure. This work not only brings a novel electron-withdrawing building block for PSCs, but also provides a novel strategy for designing effective polymer photovoltaic materials by combining weak A units and strong A units.

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