

# An Easily Accessible Isoindigo-Based Polymer for High-Performance Polymer Solar Cells

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 Supporting Information

**ABSTRACT:** A new, low-band-gap alternating copolymer consisting of terthiophene and isoindigo has been designed and synthesized. Solar cells based on this polymer and PC<sub>71</sub>BM show a power conversion efficiency of 6.3%, which is a record for polymer solar cells based on a polymer with an optical band gap below 1.5 eV. This work demonstrates the great potential of isoindigo moieties as electron-deficient units for building donor–acceptor-type polymers for high-performance polymer solar cells.

Polymer solar cells (PSCs) have received close attention in both academia and industry in recent years, as they constitute a simple and efficient way to convert solar energy into usable electricity. In comparison with inorganic solar cells, PSCs have the potential advantages of fabrication on flexible and lightweight substrates by roll-to-roll solution processing, providing cost-effective production.<sup>1</sup> The most efficient device structures of PSCs are based on the concept of a bulk heterojunction (BHJ) as the active layer,<sup>2</sup> which consists of an interpenetrating blend of conjugated polymers and fullerene derivatives as electron donors and acceptors, respectively. Intense interdisciplinary research has led to great progress in the power conversion efficiency (PCE) of PSCs.<sup>3</sup> The main contribution to this progress was made by developing new donor polymers for BHJ systems, with [6,6]-phenyl-C61-butyric acid methyl ester (PC<sub>61</sub>BM) or [6,6]-phenyl-C71-butyric acid methyl ester (PC<sub>71</sub>BM) as electron acceptors.<sup>1</sup> Most donor polymers have been designed to meet three important criteria: (i) well-matched energy level offsets between the polymer and PCBM to ensure enough driving force for charge separation while maintaining a high open-circuit voltage ( $V_{oc}$ ), (ii) appropriate solubility and miscibility to achieve an optimal nanostructure in the active layers, and (iii) a narrow optical band gap, broad absorption spectra, and a high extinction coefficient to harvest more solar photons for a thin active layer. To reach the desired properties, the combination of electron-rich (donor) and electron-deficient (acceptor) moieties as repeating units, forming internal donor–acceptor (D–A) structures, has been widely used for developing low-band-gap polymers.<sup>3,4</sup> Although a number of electron-rich and electron-deficient units have been explored, it is still a challenge to develop an

ideal low-band-gap polymer by choosing appropriate donor and acceptor units.

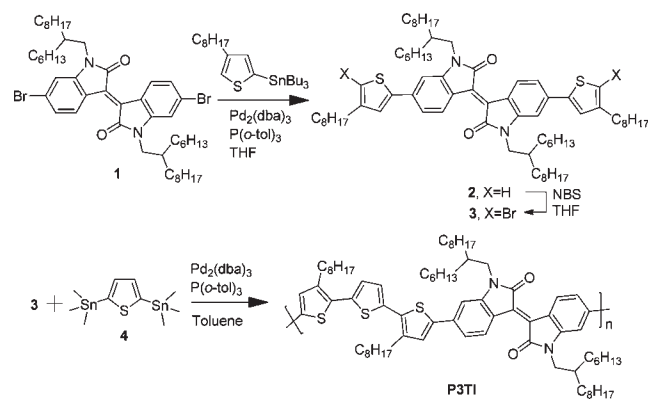
Isoindigo has strong electron-withdrawing character due to its two lactam rings. It has been widely used in the dye industry and can be obtained easily from various natural sources.<sup>5</sup> Hence, applications of isoindigo fall into the scope of renewable and sustainable synthetic sources. Isoindigo-based oligomers were first investigated by Reynolds and co-workers<sup>6</sup> and showed promising absorption spectra and favorable electrochemical and photovoltaic (PV) properties. Although they also disclosed the photophysical and electrochemical properties of isoindigo-based polymers, their PV performance was not investigated.<sup>7</sup> Very recently, the PV performance of isoindigo-based polymers was independently reported by Zhang et al.,<sup>8</sup> Liu et al.,<sup>9</sup> and our group.<sup>10</sup> Only moderate PV performance was registered for the particular class of isoindigo-based polymers. However, their broad absorption spectra, high extinction coefficients, and appropriate energy levels inspired us to modify their chemical structures and explore their PV performance further. It was noted that when different donor units were combined with isoindigo, the resulting polymers exhibited totally different optoelectronic properties.<sup>7–10</sup> PTI-1 with one thiophene as the donor unit exhibited a low absorption intensity in a high-energy band as a result of the strong electron-withdrawing effect of the isoindigo group and showed moderate PV performance.<sup>10</sup> To enhance the absorption intensity in the high-energy band, one possible way would be to increase the conjugation length of the donor unit.<sup>11</sup> Terthiophene is thought to be a promising donor unit since it is quite planar and thus beneficial for  $\pi$ – $\pi$  stacking of polymer backbones.<sup>12</sup> Here, by choosing the appropriate electron-rich unit terthiophene as the donor and isoindigo as the acceptor, we synthesized a new alternating polymer, P3TI, that shows PCEs of up to 6.3% in PSCs with PC<sub>71</sub>BM as the acceptor.

The commercialization of PSCs necessitates the availability of simple, low-cost synthetic routes toward conjugated polymers.<sup>13</sup> An abundant supply of isoindigo from natural sources and simple synthetic routes to prepare polymers such as P3TI make isoindigo-based polymers attractive for solar cell applications. Scheme 1 depicts the synthesis of P3TI. To ensure enough

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## Scheme 1. Synthesis of the Polymer P3TI



solubility of the resulting polymer, octyl was substituted on the thiophene units. To reduce steric hindrance and increase the planarity of the polymer backbone, octyl was designed to face the middle thiophene unit instead of facing the isoindigo unit.<sup>14</sup> Compound 1 and tributyl(4-octylthiophen-2-yl)stannane, which were previously prepared in our lab,<sup>10,15</sup> were coupled by Stille reaction to yield 2. The bromination of 2 with *N*-bromosuccinimide (NBS) offered 3 in 87.1% yield. The copolymerization of 3 and commercially available 2,5-bis(trimethylstannyl)thiophene (4) via Stille coupling reactions afforded crude P3TI, which was purified by Soxhlet extraction and column chromatography. The column chromatography purification was found to be crucial for the PV performance of the polymer, since the low-molecular-weight portion and residual catalyst impurities could be further removed. By control of the polymerization time, two batches of P3TI with different molecular weights were obtained. The higher-molecular-weight batch showed a number-average molecular weight ( $M_n$ ) of 73 000 with a polydispersity index (PDI) of 2.9 and the lower-molecular-weight batch an  $M_n$  of 43 000 with a PDI of 3.1, as determined by size-exclusion chromatography (SEC) relative to monodisperse polystyrene standards. Both batches of the polymer were soluble in common organic solvents such as chloroform, toluene, and *o*-dichlorobenzene (DCB). Thermogravimetric analysis demonstrated an excellent thermal stability of the polymer, which had a decomposition temperature of 380 °C (1% weight loss).

The UV–vis absorption spectra of P3TI in chloroform solution and in the solid state are shown in Figure 1. The strong low-energy peak at 645 nm in the solid state can be attributed to intramolecular charge transfer (ICT) between the terthiophene and isoindigo segments and indicates the strong electron-withdrawing effect of the isoindigo groups. In comparison with the absorption spectrum in solution, the broader absorption spectrum in the solid state indicates that aggregation or orderly  $\pi$ – $\pi$  stacking occurs in the solid state, which is beneficial for improving the charge mobility of the resulting films. In the solid state, P3TI exhibits a rather low optical band gap ( $E_g$ ) of 1.50 eV [as determined by the onset of the absorption spectrum (826 nm)], which is ideal for PSCs.<sup>16</sup>

The PV performance of P3TI was investigated in PSCs in the standard configuration glass/ITO/PEDOT:PSS/P3TI:PC<sub>71</sub>BM/LiF/Al. The P3TI:PC<sub>71</sub>BM weight ratio was optimized from 1:1 to 1:1.5 to 1:2, and the PV characteristics are summarized in Table 1. PSCs were first fabricated with DCB as the processing solvent for the active layer and showed a PCE of 4.8% for the

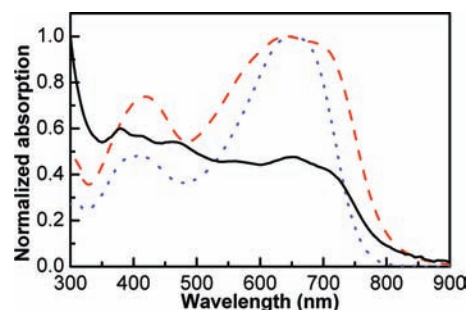


Figure 1. UV–vis absorption spectra of P3TI in chloroform solution (blue dotted line) and the solid state (red dashed line) and of the P3TI:PC<sub>71</sub>BM (1:1.5) blend film (black solid line).

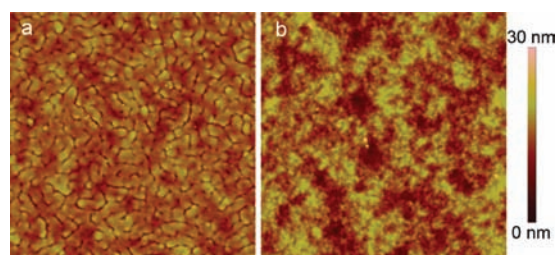
Table 1. PV Parameters of the Solar Cells

solvent	ratio <sup>b</sup>	<i>d</i> (nm) <sup>c</sup>	$V_{oc}$ (V)	$J_{sc}$ (mA cm <sup>-2</sup> )	FF	PCE (%)
DIO <sup>a</sup>	1:1	90	0.70	12.4	0.59	5.2
	1:1.5	90	0.70	13.1	0.69	6.3
	1:2	95	0.70	12.4	0.69	6.0
DCB	1:1.5	85	0.72	10.5	0.63	4.8

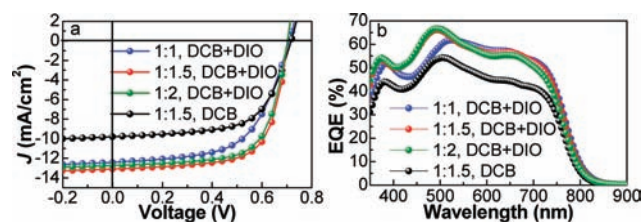
<sup>a</sup> 2.5% (v/v) DIO in DCB. <sup>b</sup> P3TI:PC<sub>71</sub>BM weight ratio. <sup>c</sup> Thickness of the active layer.

optimized P3TI:PC<sub>71</sub>BM weight ratio of 1:1.5. The atomic force microscopy (AFM) image (Figure 2a) of the 1:1.5 blend processed from DCB showed large domains. The formation of PC<sub>71</sub>BM-rich regions in polymer:PC<sub>71</sub>BM blends is often observed and has been found to be detrimental to solar cell performance.<sup>17</sup> Processing solvent additives such as 1,8-dioctadecane (DIO) are known to improve the morphology of the active layer and generate much finer nanostructures, removing grain boundaries and sometimes enhancing polymer crystallization.<sup>17b,18</sup> Also in this case, the active layer from the 1:1.5 blend processed with 2.5% (v/v) DIO in DCB resulted in a much finer phase separation, as shown in the AFM image (Figure 2b). The improved morphology can explain the enhanced photocurrent (10.5 vs 13.1 mA cm<sup>-2</sup>) and fill factor (FF) (0.63 vs 0.69) of the devices processed with DIO as an additive. The PV performance of solar cells fabricated from P3TI:PC<sub>71</sub>BM blends by processing with DCB and 2.5% (v/v) DIO in DCB is summarized in Table 1, and current density–voltage ( $J$ – $V$ ) curves for the solar cells are plotted in Figure 3a. A PCE of 6.3% with  $V_{oc}$  of 0.70 V, a short-circuit current density ( $J_{sc}$ ) of 13.1 mA cm<sup>-2</sup>, and a decent FF of 0.69 was obtained for devices containing the 1:1.5 blend as the photoactive layer (thickness of 90 nm) and processed with the additive, under AM 1.5G simulated solar light illumination (100 mW cm<sup>-2</sup>). To our knowledge, this is the highest PCE reported to date for a PSC fabricated using a polymer with an optical band gap below 1.5 eV.<sup>1,19</sup> This work highlights the great potential of low-band-gap polymers to realize high-performance PSCs by taking advantage of their broad absorption spectra, which yield high  $J_{sc}$  values, while maintaining a reasonably high  $V_{oc}$ .

To confirm the accuracy of the  $J_{sc}$  measurements conducted on the devices, the corresponding external quantum efficiency (EQE) values of the solar cells were measured under illumination of monochromatic light. As shown in Figure 3b, the solar cells fabricated from the 1:1.5 blends processed with the additive exhibited a broad photoresponse extending from 350 to 720 nm



**Figure 2.** AFM images ( $5\ \mu\text{m} \times 5\ \mu\text{m}$ ) of the active layers of P3TI:PC<sub>71</sub>BM (1:1.5) blends processed with (a) DCB and (b) 2.5% (v/v) DIO in DCB.

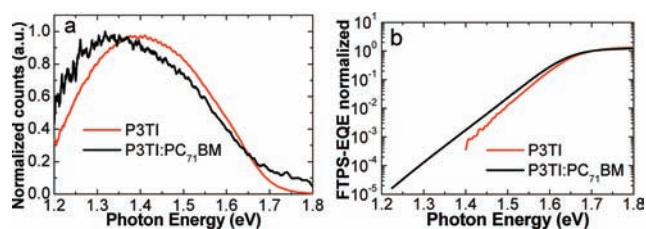


**Figure 3.** (a)  $J$ - $V$  curves and (b) EQEs for solar cells based on P3TI:PC<sub>71</sub>BM with different stoichiometries processed with DCB or 2.5% (v/v) DIO in DCB.

with EQE values over 50% and a maximum EQE of 66% at 491 nm, which is consistent with the UV-vis absorption spectrum of the blend film (Figure 1). The  $J_{sc}$  calculated from integration of the EQE with an AM 1.5G reference spectrum agreed well with the  $J_{sc}$  obtained from the  $J$ - $V$  measurements.

To achieve a high-efficiency PV device, a high photocurrent must be combined with a high photovoltage. Therefore, the energy level offset of the polymer and fullerene must be optimized.<sup>20</sup> To ensure efficient electron transfer from the polymer to the fullerene, the energy of the charge transfer (CT) state ( $E_{CT}$ ) must be lower than the optical band gap of the polymer.<sup>20,21</sup> However,  $V_{oc}$  is maximized when  $E_{CT}$  is as high as possible. A first approximation of the relevant energies can be deduced from electrochemical measurements. The HOMO and LUMO positions of P3TI were estimated to be  $-5.82$  and  $-3.83$  eV, respectively, from the oxidation and reduction potentials as determined by square-wave voltammetry measurements.<sup>22</sup> The LUMO level of  $-3.83$  eV is positioned 0.3 eV above that of PC<sub>71</sub>BM ( $-4.13$  eV, measured under the same conditions<sup>12</sup>), which is often considered to be an empirical lower limit for efficient electron transfer from the polymer to the fullerene.<sup>16</sup>

In order to investigate further the role of the CT state, highly sensitive measurements of the gap region in the EQE spectrum and the electroluminescence (EL) spectrum were performed. Such measurements allow the CT state to be probed directly and its energy to be compared to the polymer optical band gap.<sup>23</sup> The low-energy region of the EQE spectra, as measured by Fourier transform photocurrent spectroscopy (FTPS), and the EL emission spectra (at 2 V forward bias) of both pure P3TI processed with DCB and P3TI:PC<sub>71</sub>BM (1:1.5 w/w) processed with 2.5% (v/v) DIO in DCB are shown in Figure 4. Only a modest red shift of  $\sim 0.1$  eV was observed for the EL spectrum of the blend relative to the pure polymer. The FTPS-EQE measurement showed that some extra absorption was present in the optical gap region extending down to 1.2 eV (Figure 4b). Previously the EL



**Figure 4.** (a) EL spectra of P3TI processed with DCB and P3TI:PC<sub>71</sub>BM (1:1.5) processed with 2.5% (v/v) DIO in DCB. (b) Low-energy part of the EQE spectra (normalized at 1.7 eV) measured by FTPS for pure P3TI processed with DCB and P3TI:PC<sub>71</sub>BM (1:1.5) processed with 2.5% (v/v) DIO in DCB.

spectrum and extra subgap absorption of the blend were ascribed to CT emission and absorption.<sup>23</sup> The energy of the CT state can be deduced from the onset of CT absorption or the peak of CT emission and was estimated to be 1.2–1.3 eV for P3TI.<sup>20,23,24</sup> Radiative and nonradiative recombination losses lead to a typical offset of 0.5–0.6 V between  $V_{oc}$  and  $E_{CT}$ ,<sup>23b,24a</sup> in this case resulting in a  $V_{oc}$  of 0.7 V. The electrochemical, EL, and FTPS measurements all demonstrate only a small driving force for electron transfer, indicating that the  $V_{oc}$  is nearly optimized for P3TI. However, this small offset is still sufficient for efficient electron transfer between P3TI and PC<sub>71</sub>BM, as evidenced by the high EQE and almost completely quenched photoluminescence of P3TI in P3TI:PC<sub>71</sub>BM blends processed from 2.5% (v/v) DIO in DCB (see the Supporting Information).

Polymers are prone to suffer batch-to-batch variations in quality and molecular weight, which lead to low reproducibility of the PV performance of solar cells fabricated from different batches of polymers.<sup>18a,25</sup> It was therefore interesting to look into the influence of molecular weight on the photophysical properties and PV performance of P3TI, since we had two batches with different molecular weights. It was striking to note that the two batches showed almost the same photophysical properties and very similar PV performance. The fact that the PV performance of P3TI is independent of molecular weight (in a certain region) gives this easily accessible polymer great potential for synthesis and wide investigation. To avoid confusion, all the data shown in this communication are based on the low-molecular-weight batch.

In summary, an easily accessible alternating copolymer of terthiophene and isoindigo, P3TI, was designed and synthesized. The polymer presents a promising absorption spectrum and appropriate HOMO/LUMO positions, leading to PCEs of up to 6.3% in the resulting PSCs. This is the highest efficiency reported for isoindigo-based polymers and also a record for a PSC based on a polymer with an optical band gap as low as 1.5 eV. The air stability<sup>26</sup> of isoindigo and the fact that it is easily accessible from renewable and sustainable synthetic sources make it a highly promising electron-deficient unit for building D-A-type polymers. This work opens a door to the applications of isoindigo-based polymers in high-performance PSCs.

## ■ ASSOCIATED CONTENT

Supporting Information. Detailed synthetic procedures, electrochemical data, and details of solar cell fabrication. This material is available free of charge via the Internet at <http://pubs.acs.org>.

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