The Influence of polymer purification on photovoltaic device performance of a series of indacenodithiophene donor polymers

Raja Shahid Ashraf,* Bob C. Schroeder, Hugo A. Bronstein, Zhenggang Huang, Stuart Thomas, R. Joseph Kline, Christoph J. Brabec, Patrice Rannou, Thomas D. Anthopoulos, James R. Durrant, and Iain McCulloch*

Dr. R. S. Ashraf, B. C. Schroeder, Dr. H. A. Bronstein, Z. Huang, Prof. J. R. Durrant, Prof. I. McCulloch
Department of Chemistry and Centre for Plastic Electronics
Imperial College London
London, SW7 2AZ (United Kingdom)
E-mail: r.ashraf@imperial.ac.uk, i.mcculloch@imperial.ac.uk

S. Thomas, Dr. T. D. Anthopoulos
Department of Physics and Centre for Plastic Electronics
Imperial College London
London, SW7 2AZ (United Kingdom)

Dr. R. J. Kline
Polymers Division
National Institute of Standards and Technology (NIST)
Gaithersburg, Maryland 20899 (United States)

Prof. C. J. Brabec
Institute of Materials for Electronics and Energy Technology (I-MEET)
Friedrich-Alexander-University, Erlangen-Nuremberg
Martensstraße 7, 91058 Erlangen (Germany)

Dr. P. Rannou
UMR5819-SPrAM (CEA/CNRS, Univ. J. Fourier-Grenoble I)
Institut Nanosciences et Croyogénie (INAC), CEA-Grenoble
17 Rue des Martyrs, 38054 Grenoble Cedex 9 (France)

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Plastic electronics have experienced increasing interest, both in academia and industry, during the last decade. Organic semiconducting materials are considered an inexpensive alternative to replace silicon based semiconducting devices and are of particular interest for light-weight applications. A great effort has been made in (macro)molecular design, synthesis, processing and devices in this field recently to improve the efficiency. Power conversion efficiencies (PCE) have been rapidly increasing both in polymer solar cells
(PSCs) and in small molecule solar cells.\[^2\] One main advantage of small molecules over polymeric counterparts is their ease of characterization and purification. The purification of semiconducting materials has recently been shown to be crucial to achieve high PCEs in “small molecule” organic photovoltaic devices (OPV).\[^3\] Even though, small molecules can be synthesized with excellent purity, their performance is extremely sensitive to processing conditions and small molecule single crystals are often brittle in nature.\[^4\] Polymers on the other hand are more difficult to purify, but beyond a certain molecular weight threshold the electronic properties are less sensitive to processing conditions and the polymers exhibit mechanical properties superior to those of small molecules.\[^5\] Polymers can be more readily formulated into printable inks, which make them viable for large scale solution deposition processing techniques. It is important however to ensure a minimal batch to batch variation, especially for industrial synthetic scale-up and manufacture. Not only is it important to reproduce the same molecular weight polymer with similar weight average dispersities (D\(_w\)) from batch to batch, but one has to ensure that the polymer has no structural defects and contains a minimum of organic and inorganic impurities.\[^6\]

Herein we describe the fractionation of a series of indacenodithiophene (IDT) based donor-acceptor copolymers by preparative-scale recycling size exclusion chromatography (recSEC) and we investigate the effects of this additional purification step on device performances in OPV compared to non-purified polymers.\[^7, 8, 9\] In order to study the effect of purification on the device performance we investigated a series of highly soluble indacenodithiophene polymers (Scheme 1).\[^9\] The additional purification step allowed us to isolate very similar molecular weight fractions of the studied polymers and to investigate the optoelectronic properties by eliminating molecular weight variations as a complicating variable.

The indacenodithiophen-co-benzo thiadiazole (IDT-BT) and germainindacenodithiophen-co-benzo thiadiazole (GeIDT-BT) polymers have been synthesized using published synthetic
pathways.\(^8, 10\) To broaden the scope of the comparison, the silaindacenodithiophene (SiIDT) donor with branched 2-ethylhexyl side chains was synthesized. Contrary to the carbon and germanium bridged IDT polymers, the SiIDT-BT alternated copolymer was not stable under Suzuki coupling conditions and the polymer was synthesized via Stille coupling. The synthetic details and complete characterizations of this polymer can be found in the supporting information.

The crude polymers were precipitated into methanol after polymerization, followed by three 24 hours Soxhlet extractions in acetone, \(n\)-hexane and chloroform under argon atmosphere. The chloroform solution was washed with an aqueous sodium diethyldithiocarbamate solution to remove residual palladium impurities and precipitated in methanol.\(^{11}\) Throughout the manuscript, polymers purified by Soxhlet extractions and salt wash only will be referred to as non-purified (non-purif.) polymers. After the salt wash the polymers were further purified by recSEC using chlorobenzene as eluent and an Agilent PLgel 10μm MIXED-D column.\(^{12}\) The column temperature was kept at 80°C to ensure sufficient solubility and to minimize polymer chain aggregation. Fractionating the polymers by recSEC allows isolating narrower dispersity fractions with well defined molecular weights. In addition, we were able to remove low molecular weight components (\(M_n < 10 \text{ kDa}\)), which we attribute to oligomers (i.e. relatively low average degree of polymerization in number of ca. \(\text{DP}_n < 10\)) and to chains with chemical defects, such as miscoupled or cross-linked polymer chains. From the variety of fractions we collected, we chose a low (\(L M_w\)) and a high (\(H M_w\)) molecular weight fraction for each polymer. We excluded very low and very high molecular weight fractions from this study because the processing conditions for these two extreme cases needed to be adapted, thus making a direct comparison with the other fractions invalid.

For all three polymers acceptable molecular weights and polymerization degrees could be obtained after Soxhlet purification, as summarized in Table 1 (entries 1, 4 and 7). After the
polymers had been further purified by recSEC, the molecular weights could be further increased, but most importantly the dispersities of the various fractions could be significantly reduced, especially in the case of IDT-BT and GeIDT-BT, compared to the initial non-purified polymers (Figure 1). The high and low molecular weight fractions of each polymer were chosen that the lower fraction has roughly half the $D_{\text{Pn}}$ value of the higher molecular weight fraction. Both samples have a sufficiently high degree of polymerization to be significantly beyond the polymer effective conjugation length and thus the optoelectronic properties of the polymers will be identical; however we expect that the difference in molecular weight will have a significant influence on the active layer morphology in OPV devices.

Thermal gravimetric analysis (TGA) confirmed the stability of all the polymers and 5% weight loss was not observed at temperatures lower than 400°C (see Figure S1 in the Supporting Information). The choice of bridging atom has a significant effect on the molecular packing and the crystallinity of the polymer. IDT-BT has been shown to be rather amorphous, whereas GeIDT-BT shows clear indications of semi-crystalline behaviour. These observations were further supported by Differential scanning calorimetry (DSC) studies. DSC revealed a broad melt with an onset at about 290°C for $HM_w$ GeIDT-BT and the crystallization was observed at 280°C. For the lower molecular weight $LM_w$ GeIDT-BT both the melt (275°C) and the crystallization (270°C) shifted towards lower temperatures (see Figure S4 in the Supporting Information) compared to $HM_w$ GeIDT-BT. Even though the GeIDT-BT polymer has four 2-ethylhexyl side chains per repeating unit, the observation of obvious phase transitions by DSC suggest that the polymer tends to adopt some sort of macromolecular order. The phase transitions observed for the SiIDT-BT polymers were of much weaker intensity, than the signals obtained for the two GeIDT-BT polymers, suggesting that the interactions between polymer chains are weaker in SiIDT-BT, than in
GeIDT-BT. A weak melt was identified around 260°C for HM<sub>w</sub> SiIDT-BT and the recrystallisation occurred at 260°C during cooling. LM<sub>w</sub> SiIDT-BT and both IDT-BT polymers on the other hand showed no significant thermal transitions in the temperature range of 0°C to 350°C.

The nature of the bridging atom not only influences the molecular packing, but also has a significant effect on the optoelectronic properties of π-conjugated polymers. The UV-vis. absorption spectra of the three polymer families (Figure 2) in solution and solid state exhibit similar features, a strong internal charge transfer (ICT) absorption peak at higher wavelength and a smaller absorption peak at lower wavelengths originating from the π-π* transitions. Of all three polymers, IDT-BT is the most bathochromically shifted, whereas the absorption features of SiIDT-BT and GeIDT-BT are very similar, with SiIDT-BT hypsochromically shifted by 10 nm, both in solution and solid state (Table 2).

The nature of the bridging atom is responsible for the overall shape and wavelength of the absorption peaks, whereas the molecular weight and purification seems to have an influence on the vibronic structures of the peaks. In solution all three IDT-BT polymers behave similarly, in the solid state however the LM<sub>w</sub> IDT-BT differs slightly from the two other IDT polymers. The shoulder appearing around 600 nm is more defined in the case of LM<sub>w</sub> IDT-BT, leading to the assumption that the lower molecular weight polymer aggregates stronger in solid state than the higher molecular weight fraction or the non-purified polymer. All three GeIDT-BT polymers show the same absorption features in solution, but behave differently in the solid state. The non-purified GeIDT-BT polymer has a well defined shoulder around 600 nm, which decreases in both purified fractions. As the dispersity values of the purified fractions are significantly lower than for the non-purified fraction, we assume that highly aggregating, low molecular weight material responsible for the shoulder at 600 nm has been removed by the recSEC purification. The effect of the additional purification is even more
pronounced in the case of SiIDT-BT. In solution, the ICT absorption peak of SiIDT-BT (non-purif.) is broadened by 12 nm hypsochromically compared to the two purified fractions. In the solid state both purified fractions of the SiIDT-BT exhibit shoulders towards shorter wavelengths, but they are less pronounced than for the non-purified polymer. In this case the observed differences in UV-vis. absorption spectra are not related to the narrower dispersity value of LM\textsubscript{w} and HM\textsubscript{w} SiIDT-BT because the non-purified polymer had already a rather small D\textsubscript{w}, but to the increase in molecular weight. From initially 14 kg/mol, the number average molecular weight (M\textsubscript{n}) was increased by recSEC to 17 kg/mol, respectively 28 kg/mol in the case of HM\textsubscript{w} SiIDT-BT, which induced a bathochromic shift of the absorption spectra in solution and causes the polymers to aggregate less in the solid state, thus reducing the intensity of the shoulder at 590 nm compared to SiIDT-BT (non-purif.).

It has previously been demonstrated in the case of poly(3-hexylthiophene) that molecular weight has a significant impact on OPV performance.\textsuperscript{[13]} Other critical and related parameters essential for optimal OPV device performance include both the weight average dispersity (D\textsubscript{w}), the shape of the molecular weight profile, particularly at the low molecular weight tail, and the bulk purity.\textsuperscript{[14]} The device data of these purified fractions were compared to non-purified polymer fractions.

To evaluate the photovoltaic performances of the fractionated and non-purified polymers, bulk hetero-junction (BHJ) solar cells with conventional device structure consisting of ITO/PEDOT:PSS/Polymer:PC\textsubscript{71}BM/Ca/Al were prepared and tested under simulated 100 mW cm\textsuperscript{-2} AM1.5G illumination. The J-V curves and external quantum efficiencies (EQE) of the organic photovoltaic cells are presented in Figure 3. The corresponding open-circuit voltages (V\textsubscript{OC}), short-circuit currents (J\textsubscript{SC}), fill factors (FF) and power conversion efficiencies (PCE) are summarized in Table 3.
A significant improvement in the OPV performance was observed in all fractionated polymers compared to non-purified parent polymers (Table 3), even for the low molecular weight fractions. In case of IDT-BT, the $V_{OC}$ was almost unchanged in all three polymer fractions, $J_{SC}$ however was significantly improved in the fractionated polymers, leading to a better performance compared to the non-purified one. Overall the higher fill factor and $J_{SC}$ of HM$_w$ IDT-BT lead to a higher PCE of 6.5 %. A similar trend was observed in both SiIDT-BT and GeIDT-BT polymers, with improved performances for the fractionated polymers. Here it is important to note that replacement of C with both Si and Ge as bridging atoms led to an improvement in $V_{OC}$, SiIDT-BT polymer showing the highest $V_{OC}$ in the series, followed by the GeIDT-BT polymer. There was however a trade-off in this higher $V_{OC}$, with the absorption spectrum of SiIDT-BT polymer blue shifted compared to IDT-BT polymer leading to lower $J_{SC}$ and thus to a lower performance than IDT-BT. The absorption spectrum of GeIDT-BT sits between SiIDT-BT and IDT-BT, showing higher $V_{OC}$ than IDT-BT in OPV devices and as the loss in $J_{SC}$ was minimal, a high PCE of 6.5% was obtained.

Studying the effect of purification and fractioning on the device performance of the highly soluble indacenodithiophene polymers actually yielded surprising results. Typically, one would expect that impurities, defects, chain ends, etc. act as recombination centers, affecting all performance parameters, i.e. $V_{OC}$, $J_{SC}$ and FF. However, in our case, we dominantly observe that purification leads to an increase in the short circuit current, while $V_{OC}$ and FF remain more or less constant (with the exception of IDT-BT, where the FF increases from slightly under 50 % to over 50%). It would be difficult to explain such a trend exclusively by reduced recombination. Rather, J-V and EQE data suggest that a combination of enhanced generation and reduced recombination may simultaneously be responsible for the enhanced photocurrent generation upon purification. Atomic force microscopy (AFM) images on IDT-
BT suggest a better intermixed phase morphology upon purification, but more extensive
device investigations are necessary to verify this hypothesis (Figure 4).

Grazing-incidence X-ray diffraction (GIXD) studies of the polymer:PC$_{71}$BM blends was
dominated by scattering from the PC$_{71}$BM. Diffraction patterns for the GeIDT-BT and
SiIDT-BT polymers show weak scattering peaks in the region associated with the lamellar
repeat between molecules (Figure 5). The scattering in the GeIDT-BT films was stronger
than in the SiIDT-BT films, consistent with the relative strength of the crystallization
transformation peaks revealed by the DSC studies. Additionally the scattering for LM$_w$
SiIDT-BT was noticeably higher than HM$_w$ SiIDT-BT, also consistent with the DSC
measurements. This scattering reveals the presence of some regions of aggregation in the
films, but the weak intensity of the peaks is suggestive of a low crystallinity. No scattering
was identified for the IDT-BT polymers, which is consistent with the DSC measurements
and suggests a rather amorphous nature for the IDT-BT polymer when blended with
PC$_{71}$BM.

In summary, we have synthesized three highly soluble indacenodithiophene polymers and
studied the effect of purification and molecular weight control on their optoelectronic
properties. All three classes of polymers required high molecular weight and purification by
recycling SEC to obtain optimal performance, with improvements of up to 30% in power
conversion efficiency observed.

Experimental

Device Fabrication and Testing: All organic photovoltaic devices have a conventional device
architecture, ITO/PEDOT:PSS/Polymer: PC$_{71}$BM/Ca/Al. The precoated ITO glass substrates
were cleaned with acetone and isopropyl alcohol under sonication, followed by drying and
oxygen plasma treatment. A 30 nm layer of PEDOT:PSS was spin-coated onto the plasma-
treated ITO substrate and baked at 150°C for 20 minutes. An 80 nm active layer consisting of
a 1:3.5 blend of polymer and PC_{71}BM dissolved in o-dichlorobenzene (ODCB) was spin-coated on the PEDOT:PSS layer and then Ca (30 nm)/Al (100 nm) cathode was finally deposited by thermal evaporation under high vacuum (10^{-6} mbar) through a shadow mask. The pixel size, defined by the spatial overlap of the ITO anode and Ca/Al cathode, was 0.045 cm^2. The device characteristics were obtained using a xenon lamp with AM1.5G filters and 100 mW/cm² illumination (Oriel Instruments). Short circuit currents under AM1.5G conditions were obtained from the spectral response and convolution with the solar spectrum, measured with a Keithley source meter. Spectral response was measured under operation conditions using bias light from a 532 nm solid state laser (Edmund Optics). Monochromatic light from a 100 W tungsten halogen lamp in combination with monochromator (Oriel, Cornerstone 130) was modulated with a mechanical chopper. The response was recorded as the voltage over a 50 Ω resistance, using a lock-in amplifier (Stanford research Systems SR830). A calibrated Si cell was used as reference. All the device measurements were carried out behind a quartz window in a nitrogen filled container.

_Grazing-incidence X-ray Diffraction:_ The grazing-incidence X-ray diffraction (GIXD) measurements were done at beam line 11-3 at the Stanford Synchrotron Radiation Laboratory. The measurements were done with a beam energy of 12.735 keV and an incidence angle of 0.12°. The samples were enclosed in a He environment to limit beam damage and reduce air scattering. The data was collected using a MAR345 image plate. The measurement was calibrated using a LaB_{6} crystal standard.

**Supporting Information**

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

**Acknowledgements**
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[12] Certain commercial equipment, instruments, or materials are identified in this paper in order to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.


Figures and captions:

Scheme 1. Chemical structures of the synthesized IDT-BT, SiIDT-BT and GeIDT-BT alternated copolymers.

Table 1. Macromolecular parameters ($M_n$, $M_w$, $D_w$) of the non-purified and purified IDT polymers.

<table>
<thead>
<tr>
<th>Polymer</th>
<th>$M_n$ [kg/mol] [a]</th>
<th>$M_w$ [kg/mol] [a]</th>
<th>$D_w$ [a]</th>
<th>$D_P$ n [b]</th>
</tr>
</thead>
<tbody>
<tr>
<td>IDT-BT (non-purif.)</td>
<td>40</td>
<td>87</td>
<td>2.2</td>
<td>47</td>
</tr>
<tr>
<td>LM$_n$ IDT-BT</td>
<td>20</td>
<td>30</td>
<td>1.5</td>
<td>24</td>
</tr>
<tr>
<td>HM$_n$ IDT-BT</td>
<td>50</td>
<td>70</td>
<td>1.4</td>
<td>59</td>
</tr>
<tr>
<td>SiIDT-BT (non-purif.)</td>
<td>14</td>
<td>23</td>
<td>1.6</td>
<td>16</td>
</tr>
<tr>
<td>LM$_n$ SiIDT-BT</td>
<td>17</td>
<td>23</td>
<td>1.4</td>
<td>19</td>
</tr>
<tr>
<td>HM$_n$ SiIDT-BT</td>
<td>28</td>
<td>36</td>
<td>1.3</td>
<td>32</td>
</tr>
<tr>
<td>GeIDT-BT (non-purif.)</td>
<td>32</td>
<td>75</td>
<td>2.3</td>
<td>33</td>
</tr>
<tr>
<td>LM$_n$ GeIDT-BT</td>
<td>19</td>
<td>29</td>
<td>1.5</td>
<td>20</td>
</tr>
<tr>
<td>HM$_n$ GeIDT-BT</td>
<td>37</td>
<td>48</td>
<td>1.3</td>
<td>38</td>
</tr>
</tbody>
</table>

[a] Average molecular weight in number ($M_n$), in weight ($M_w$) and weight average dispersity $D_w$ ($M_w/M_n$) as determined by SEC using narrow weight average dispersity ($D_w < 1.10$) polystyrene (PS) standards and chlorobenzene as eluent. [b] The degree of polymerization ($D_P$) is defined in this case as the number of repeating units.
Figure 1. Normalized SEC traces of the various purified and non-purified **IDT-BT** polymers. Similar molecular weight fractions were isolated for each polymer to eliminate molecular weight as an unfavorable variable.

Figure 2. Normalized UV-vis. absorption spectra of the various **IDT-BT** based polymers in dilute chlorobenzene solution (**top row**) and thin-film (**bottom row**), spin-cast from chlorobenzene.
Table 2. Optical properties and energy levels of IDT-BT, SiIDT-BT and GeIDT-BT polymers.

<table>
<thead>
<tr>
<th>Polymer</th>
<th>$\lambda_{\text{max}}$ soln. [nm] [a]</th>
<th>$\lambda_{\text{max}}$ film [nm] [b]</th>
<th>HOMO / LUMO [eV] [c]</th>
<th>$E_{\text{g opt}}$ [eV]</th>
</tr>
</thead>
<tbody>
<tr>
<td>IDT-BT</td>
<td>657</td>
<td>662</td>
<td>-5.3 / -3.6</td>
<td>1.7</td>
</tr>
<tr>
<td>SiIDT-BT</td>
<td>614</td>
<td>636</td>
<td>-5.3 / -3.5</td>
<td>1.8</td>
</tr>
<tr>
<td>GeIDT-BT</td>
<td>630</td>
<td>645</td>
<td>-5.2 / -3.5</td>
<td>1.7</td>
</tr>
</tbody>
</table>

[a] Measured in dilute chlorobenzene solution. [b] Spin-coated from 5 mg/mL chlorobenzene solution. [c] The LUMO energy is estimated by adding the absorption onset to the HOMO.

Figure 3. (Top) J-V characteristics of IDT-BT, SiIDT-BT and GeIDT-BT polymer:PC$_{71}$BM solar cell under AM1.5 solar illumination and (Bottom) external quantum efficiencies of the cells.
Table 3. Photovoltaic Device Characteristics of IDT polymers.

<table>
<thead>
<tr>
<th>Polymer</th>
<th>J_sc [mA/cm²]</th>
<th>V_oc [V]</th>
<th>FF</th>
<th>PCE [%] [a]</th>
</tr>
</thead>
<tbody>
<tr>
<td>IDT-BT (non-purif.)</td>
<td>11.45</td>
<td>0.79</td>
<td>0.49</td>
<td>4.4</td>
</tr>
<tr>
<td>LMₜ IDT-BT</td>
<td>13.32</td>
<td>0.81</td>
<td>0.51</td>
<td>5.5</td>
</tr>
<tr>
<td>HMₜ IDT-BT</td>
<td>14.48</td>
<td>0.80</td>
<td>0.56</td>
<td>6.5</td>
</tr>
<tr>
<td>SiIDT-BT (non-purif.)</td>
<td>9.60</td>
<td>0.92</td>
<td>0.52</td>
<td>4.6</td>
</tr>
<tr>
<td>LMₜ SiIDT-BT</td>
<td>12.18</td>
<td>0.91</td>
<td>0.52</td>
<td>5.8</td>
</tr>
<tr>
<td>HMₜ SiIDT-BT</td>
<td>12.68</td>
<td>0.88</td>
<td>0.50</td>
<td>5.6</td>
</tr>
<tr>
<td>GeIDT-BT (non-purif.)</td>
<td>10.08</td>
<td>0.86</td>
<td>0.58</td>
<td>5.0</td>
</tr>
<tr>
<td>LMₜ GelIDT-BT</td>
<td>13.06</td>
<td>0.86</td>
<td>0.55</td>
<td>6.2</td>
</tr>
<tr>
<td>HMₜ GelIDT-BT</td>
<td>13.95</td>
<td>0.85</td>
<td>0.55</td>
<td>6.5</td>
</tr>
</tbody>
</table>

[a] 3-4 cells PCE values, corrected by EQE. Standard deviation (0.1-0.3)

Figure 4. AFM topography images of IDT-BT (non-purif.) (left), HMₜ IDT-BT (middle) and LMₜ IDT-BT (right) (tapping-mode, 2 × 2 μm) blended with PC₂₇₁BM (1:3.5).
Figure 5. Out of plane scattering of polymer:PC$_{71}$BM blends (1:3.5) spin-coated on ITO with PEDOT:PSS. The circled areas highlight polymer scattering and the PC$_{71}$BM scattering is marked by arrows.
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A series of low bandgap indacenodithiophene polymers is purified by recycling SEC in order to isolate narrow polydisperse fractions. This additional purification step is found to have a significant beneficial influence on the solar cell performance and the reasons for this performance increase are investigated.

Keywords: Indacenodithiophene, purification, fractionation, organic photovoltaics

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