Two Novel Small Molecule Donors and the Applications in Bulk-Heterojunction Solar Cells

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Two novel small molecules DTRDTQX and DTIDTQX, based on ditolylaminothienyl group as donor moiety and quinoxaline as middle acceptor moiety with different terminal acceptor groups were synthesized and characterized in this work. In order to study the photovoltaic properties of DTRDTQX and DTIDTQX, bulk-heterojunction solar cells with the configuration of FTO/c-TiO$_2$/DTRDTQX(or DTIDTQX):C$_70$/MoO$_3$/Ag were fabricated, in which DTRDTQX and DTIDTQX acted as the donors and neat C$_70$ as the acceptor. When the weight ratio of DTRDTQX:C$_70$ reached 1:2 and the active layer was annealed at 100$^\circ$C, the optimal device was realized with the power conversion efficiency (PCE) of 1.44%. As to DTIDTQX:C$_70$-based devices, the highest PCE of 1.70% was achieved with the optimal blend ratio (DTIDTQX:C$_70$ = 1:2) and 100$^\circ$C thermal annealing treatment. All the experimental data indicated that DTRDTQX and DTIDTQX could be employed as potential donor candidates for organic solar cell applications.

Keywords: bulk-heterojunction, small molecule, donor, solar cell, ditolylaminothienyl, quinoxaline

INTRODUCTION

Recently, organic solar cells (OSCs) based on bulk-heterojunction structure have attracted much attention due to the distinctive characteristics of low cost, easy fabrication, flexibility and light weight, etc. (Gustafsson et al., 1992; Shaheen et al., 2001; Chen and Cao, 2009). Compared with polymers employed in solar cells, small molecule donors have the advantage of less batch-to-batch variation, well-defined molecular structure, easier purification, etc. (You et al., 2013; Chen et al., 2014, 2015; He et al., 2015; Zhou et al., 2015). Therefore, much work focused on small molecule donors and the photovoltaic performance of OSCs was improved accordingly (Sun et al., 2011; Liu et al., 2013; Love et al., 2013; Coughlin et al., 2014). In general, the active layers of the solar cells consisted of small molecule donors and fullerene/fullerene derivative acceptors (Chen et al., 2012; Huang et al., 2016). In order to optimize the photovoltaic characteristics of OSCs, narrow band-gap and deep highest occupied molecular orbital (HOMO) of small molecule donors should be considered, which resulted in broad absorption and high open-circuit voltage ($V_{oc}$) of devices. Then, various small molecules composed of electron rich moieties (donor, “D”) and electron deficient moieties (acceptor, “A”), have been reported with the molecular configuration such as D-A (Roquet et al., 2006), A-D-A (Schulze et al., 2006), D-A-D (Lin et al., 2011) and D-D-A conjugated structures. In this regard, the HOMO and lowest unoccupied molecular orbital (LUMO) of the small molecules were effectively tuned, mainly due to the intramolecular charge transfer (ICT) between donors and acceptors (Zhang et al., 2011).
Herein, the photovoltaic properties of two novel small molecule donors (named DTRDTQX and DTIDTQX, Figure 1) based on D-A-A structure were studied in this work. DTITDQX or DTRDTQX consisted of ditolylaminothiophenyl group as the donor moiety, quinoxaline as middle acceptor moiety with different terminal acceptor groups such as 1,3-indandione or 3-ethylrhodanine, respectively. To investigate the photovoltaic properties of the small molecules, bulk-heterojunction (BHJ) solar cells based on DTRDTQX or DTIDTQX as the donor together with C70 as the acceptor were fabricated and the optimal cells showed PCE of 1.44 and 1.70%, respectively.

EXPERIMENTAL

Materials and Characterization

All materials in this work were purchased commercially, except for the tailor made DTRDTQX and DTIDTQX donors. The commercial materials were used without further purification.

Scheme 1 depicts the synthesis of DTIDTQX and DTRDTQX. By following the protocols established by Krebs et al. (Jørgensen and Krebs, 2005) and Janssen et al. (Bijleveld et al., 2009), we could get 4-bromo-7-methyl-2,1,3-benzothiadiazole (3). Then the heterocyclic 3 was converted to diamine intermediate 4 by treating Fe/HCl, which was then followed by condensation with glyoxal to afford 5-bromo-8-methylquinoxaline (5) without further purification. The 8-bromoquinoxaline-5-carbaldehyde (7) was synthesized by benzylic bromination with N-bromosuccinimide (NBS) initiated by azobisisobutyronitrile (AIBN) and followed by hydrolysis with CaCO3 in H2O/acetonitrile (Lin et al., 2011). Aldehyde 7 was reacted with N,N-di-p-tolyl-5-(tri-n-butylstannyl)-thiophen-2-amine (8) through Stille coupling reaction and gave key intermediate 9. Finally, the condensation of 9 with 1,3-indandione and 3-ethylrhodanine via Knöevenagel reaction afforded DTIDTQX and DTRDTQX, respectively. The absorption spectra were measured with JASCO V-670 spectrophotometer. Thermogravimetric analysis (TGA) was determined on a TA Instruments Model TGA Q500 V20.13 (build 39) with a heating rate of 10°C/min. Differential Scanning Calorimeter (DSC) was carried out at a heating rate of 10°C/min on a TA Instruments Model DSC Q100 V9.9 (build 303). The thickness of the films was evaluated using a surface profilometer. The electrochemical cyclic voltammetry (CV) was recorded by a CHI619B potentiostat with glassy carbon electrode, Pt wire and Ag/AgCl which were used as the working electrode, counter electrode, and reference electrode, respectively, further calibrated with the ferrocene/ferrocenium (Fc/Fc+) redox couple. The oxidation waves were recorded in CH2Cl2 (for 1.0 mM) with 0.1 M tetraethylammonium hexafluorophosphate [(Bu4N)PF6] as supporting electrolyte, while reductive waves were recorded in THF (for 1.0 mM) with 0.1 M tetrabutylammonium perchlorate [(Bu4N)ClO4] as supporting electrolyte.

Solar Cell Fabrication and Characterization

In order to investigate the photovoltaic properties of DTRDTQX and DTIDTQX, the OSCs with the configuration of FTO/c-TiO2/DTRDTQX or DTIDTQX:C70/MoO3/Ag were fabricated as shown in Figure 1. The compact TiO2 layer in OSCs acted as the electron transporting layer (Heo et al., 2015) and MoO3 as the hole buffer layer. As to the photoactive layers, DTRDTQX and DTIDTQX served as the donors and C70 as the acceptor, respectively. The FTO cathode was pre-cleaned in an ultrasonic cleaner with deionized water, acetone and alcohol for 15 min respectively and then treated with oxygen plasma for 15 min. The TiO2 films were fabricated according to the literatures (Kim et al., 2012; Zhang et al., 2016) and sintered at 500°C for 15 min in a muffle furnace. And then, the TiO2 films were naturally cooled to room temperature. Blended solutions (total concentration: 20 mg/ml) of DTRDTQX or DTIDTQX:C70 in ortho-dichlorobenzene (odCB) were spin-coated (700 rpm, 18 s) onto FTO/TiO2 substrates in a glove box and then thermal annealed at 100°C or 150°C. The effect of thermal annealing on the photovoltaic properties of the active layers was also studied in this work. Finally, 7 nm MoO3 buffer layers and 100 nm Ag anodes were thermal evaporated successively below 10−6 Torr. The photovoltaic performance of the OSCs were evaluated by current density-bias voltage (J-V) measurement (using a Keithley 2400 source meter) under AM 1.5G simulated solar illumination (Newport model 94021A, 100 mW cm−2).

RESULTS AND DISCUSSION

Thermal Property

Thermal properties of the two small molecules were investigated by TGA measurement as shown in Figure 2 and the thermal decomposition temperatures (Td, 5% weight loss) were evaluated to be 362°C and 312°C for DTRDTQX and DTIDTQX respectively, indicating the good thermal stability of the small molecules. According to the DSC plots shown in Figure 3, the melting temperatures (Tm) were evaluated to be 187.8°C and 185.8°C for DTRDTQX and DTIDTQX respectively.
263.3°C for DTRDTQX and DTIDTQX, respectively. Moreover, the glass transition temperatures (T_g) were measured to be 94.0°C and 149.7°C for DTRDTQX and DTIDTQX, respectively. Therefore, both DTRDTQX and DTIDTQX were stable donors for OSCs due to their decent thermal stability.

**Absorption Properties**

The UV-Vis absorption of DTIDTQX and DTRDTQX in CH_2Cl_2 were shown in Figure 4 and the corresponding data were summarized in Table 1. The compounds showed broad band absorption from 480 to 750 nm with high extinction coefficient (3.3–3.5 × 10^4 M^{-1} cm^{-1}) in the visible range (450–700 nm). DTIDTQX absorbed longer wavelength than DTRDTQX (631 vs. 588 nm), mainly due to the stronger electron withdrawing ability of 1,3-indanedione group than that of N-ethylrhodanine group.

**Electrochemical Properties**

The electrochemical properties of DTRDTQX and DTIDTQX were studied with cyclic voltammetry (CV) as shown in
Qi et al. Bulk-Heterojunction Solar Cells

FIGURE 2 | TGA diagrams of DTRDTQX (up) and DTIDTQX (down).

FIGURE 3 | Differential scanning calorimetry measurements of DTRDTQX and DTIDTQX.

FIGURE 4 | UV-Vis absorption spectra of DTIDTQX and DTRDTQX in CH$_2$Cl$_2$.

Figure 5. In addition, the energy levels as well as the band gaps of DTRDTQX and DTIDTQX were summarized in Table 1. With the oxidation and reduction potentials recorded, the HOMO and LUMO levels of the two materials could be calculated (HOMO = $-5.1$ eV – $E_{\text{onset}}^{\text{ox}}$, LUMO = $-5.1$ eV – $E_{\text{onset}}^{\text{red}}$), which were $-5.33$ eV, $-3.96$ eV for DTIDTQX and $-5.29$ eV, $-3.59$ eV for DTRDTQX respectively. Interestingly, the HOMO and LUMO levels of DTIDTQX were both deeper than those of DTRDTQX. The phenomenon implied that the electron withdrawing ability of 1,3-indanedione group was stronger than that of N-ethylrhodanine group, which was consistent with the observation of UV-Vis absorption. The energy levels of the materials used in the OSCs were depicted in Figure 6. The large gap between the low-lying HOMO level ($-5.33$ eV) of DTIDTQX and LUMO ($-4.20$ eV) of C$_{70}$ was evaluated to be $1.13$ eV, which resulted in the large $V_{\text{oc}}$ (0.71 V) of the optimal DTIDTQX-based OSCs in this work. Furthermore, the electrochemical energy band gap ($\Delta E_{\text{CV}}^\text{opt}$) of DTIDTQX was $0.33$ eV lower than that of DTRDTQX and strong absorption of DTIDTQX active layer in red region could be realized, which was matched well with the UV-Vis absorption spectrum shown in Figure 4. Therefore, the light-harvesting capability as well as the photovoltaic performance of DTIDTQX-based devices could be superior to that of DTRDTQX-based counterparts, which will be discussed further in following.

Photovoltaic Properties

To study the photovoltaic properties of the small molecules, OSCs with the structure of FTO/c-TiO$_2$/donor:C$_{70}$/MoO$_3$/Ag were fabricated. The weight ratios of DTRDTQX:C$_{70}$ and DTIDTQX:C$_{70}$ varied from 1:1 to 1:3 and the corresponding J-V curves of the OSCs were shown in Figures 7, 8. All the photovoltaic data of OSCs were summarized in Table 2. When the weight ratio of DTRDTQX:C$_{70}$ reached 1:2 and the photoactive layer was thermal annealed at 100°C, the
TABLE 1 | Physical properties of DTIDTQX and DTRDTQX.

| Compounds | λ_{abs} Solution (nm)^a (ε, M^{-1} cm^{-1}) | ΔE_{opt} sol. (eV)^a | E_{onset}^{ox} (V)^b | E_{onset}^{red} (V)^b | ΔE^{CV} (eV) | HOMO (eV)^b | LUMO (eV)^b | Td (°C) |
|-----------|--------------------------------|-----------------|------------------|------------------|-------------|-------------|-------------|--------|
| DTIDTQX   | 631 (34,900)                | 1.97            | 0.23             | -1.14            | 1.37        | -5.33       | -3.96      | 312    |
| DTRDTQX   | 588 (32,800)                | 2.11            | 0.19             | -1.32            | 1.70        | -5.29       | -3.59      | 361    |

^a Measured in CH_{2}Cl_{2} solution (10^{-5} M) and the value was estimated from the onset. ^b Estimated from the HOMO (~5.1 eV) (Cardona et al., 2011) of Fc^+/Fc as reference. ^c Temperature corresponding to 5% weight loss obtained from TGA analysis.

The best DTRDTQX-based OSC was realized with the short-circuit current density (J_{sc}) and PCE of 5.66 mA/cm² and 1.44%, respectively. The champion DTRDTQX-based OSC exhibited almost the same open-circuit voltage (V_{oc}) of ~0.65 V as other OSCs with different weight ratios (1:1 and 1:3) of DTRDTQX:C_{70}. Moreover, for the devices based on DTRDTQX:C_{70} with the weight ratios of 1:1 and 1:3, the decreased J_{sc} was mainly ascribed to the imbalanced electron and hole diffusion in the OSCs (Kim et al., 2009). The photovoltaic data in Table 2 implied that the weight ratio (DTRDTQX:C_{70}) of 1:2 was advantageous to the photovoltaic performance of DTRDTQX:C_{70}-based OSCs. The photovoltaic properties of DTRDTQX:C_{70}(1:2)-based OSCs with 150°C thermal annealing and without thermal annealing were also studied and compared. The V_{oc} and PCE of the OSC with 150°C thermal annealing were decreased to 0.51 V and 1.19%, respectively. As to the OSC without thermal annealing, the PCE was decreased to 1.14% and V_{oc} (~0.66V) was almost unchanged compared with the champion DTRDTQX-based OSC. Therefore, 100°C
thermal annealing treatment was necessary for the reasonable photovoltaic performance of DTRDTQX:C\textsubscript{70}(1:2)-based OSCs according to the experimental data.

As to DTIDTQX-based OSCs, the photovoltaic performance was modulated by the weight ratios of DTIDTQX:C\textsubscript{70} from 1:1 to 1:3. When the blend ratio of DTIDTQX:C\textsubscript{70} reached 1:2, the best DTIDTQX-based OSC was realized as shown in Table 2. The $V_{oc}$, $J_{sc}$, FF, and PCE of the champion device were 0.71V, 6.24 mA/cm$^2$, 0.38 and 1.70%, respectively. It was worthy to note that the $V_{oc}$ of DTIDTQX:C\textsubscript{70}(1:2)-OSC was 0.06 V higher than that of DTRDTQX:C\textsubscript{70}(1:2)-OSC, mainly due to the low-lying HOMO ($−5.33$ eV) of DTIDTQX as shown in Figure 6. Moreover, the $J_{sc}$ and PCE of DTIDTQX:C\textsubscript{70}(1:2)-OSC were both higher than those of DTRDTQX:C\textsubscript{70}(1:2)-OSC. Therefore, the photovoltaic properties of DTIDTQX-based devices were superior to those of DTRDTQX-based counterparts, which was mainly ascribed to the narrow band gap ($\sim1.37$ eV) of DTIDTQX and the consequent effective absorption in solar spectrum. The photovoltaic performance of DTIDTQX:C\textsubscript{70}(1:2)-OSC was deteriorated when the active layer was treated with 150$^\circ$C thermal annealing as shown in Table 2. And when DTIDTQX:C\textsubscript{70}(1:2)-OSC was fabricated without thermal annealing, the PCE decreased to 1.26%. Therefore, 100$^\circ$C thermal annealing was favorable to DTIDTQX:C\textsubscript{70}(1:2)-OSC and a decent PCE of 1.70% was obtained accordingly. However, the FF values of the OSCs were relatively low in this work and much work should be required to further increase FF as well as PCE of the OSCs, such as inserting buffer layers (Ji et al., 2016; Li et al., 2016; Mbuyise et al., 2016), introducing optical spacers (Ben Dkhil et al., 2014), employing solvent annealing (Sun et al., 2014; Li et al., 2015), chemical treatments (Bai et al., 2015), etc.

The morphology of DTRDTQX:C\textsubscript{70}(1:2) and DTIDTQX:C\textsubscript{70}(1:2) films was studied by atomic force microscopy (AFM) (Agilent Series 5500) as shown in Figure 9. The root-mean-square roughness (RMS) of DTIDTQX:C\textsubscript{70}
CONCLUSIONS

Two small molecules DTRDTQX and DTIDTQX with the D-A-A structure were studied in this work. DTRDTQX and DTIDTQX were used as the donors in bulk-heterojunction solar cells. The optimal OSCs based on DTRDTQX:C70(1:2) and DTIDTQX:C70(1:2) were achieved with the PCE of 1.44% and 1.70%, respectively. The photovoltaic properties of DTIDTQX were superior to those of DTRDTQX, which was attributed to the narrow band gap (1.37 eV) and the high hole mobility ($2.27 \times 10^{-5}$ cm$^2$ V$^{-1}$ s$^{-1}$) of DTIDTQX. Therefore, DTRDTQX and DTIDTQX would be promising donor materials for organic solar cells in future.

AUTHOR CONTRIBUTIONS

All authors listed have made a substantial, direct and intellectual contribution to the work, and approved it for publication.

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SUPPLEMENTARY MATERIAL

The Supplementary Material for this article can be found online at: https://www.frontiersin.org/articles/10.3389/fchem.2018.00260/full#supplementary-material

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