Photostability of benzodithiophene based polymer: effect of PC$_{60}$BM and intermolecular interactions

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Abstract

Organic solar cells working under continuous light irradiation are prone to photo-induced degradation. Photostabilities of benzodithiophene-alt-2-decyltetradecyl substituted isoindigo (PBDTI-DT) copolymer in solution, and as pristine film, and a PBDTI-DT:PC$_{60}$BM bulk heterojunction (BHJ) film were investigated for more than 70 h under simulated AM 1.5 solar irradiation. The photodegradation kinetics studied in standalone polymer chains were found to be fast due to the absence of intermolecular interaction while the inter-chain interaction in the polymer films kept the backbone intact against light-induced degradation. Further addition of PC$_{60}$BM in the polymer made the BHJ film more stable as PC$_{60}$BM serves as photoprotective layer and radical scavenger. This conclusion was supported by the similar XRD traces of PBDTI-DT:PC$_{60}$BM while the traces of the pristine film indicated bleaching and shifted with increasing photon stress. In addition, the absorption of PBDTI-DT:PC$_{60}$BM and pristine PBDTI-DT films were reduced to 80 and 56%, respectively, after 70 h of photo-degradation.

1. Introduction

Organic solar cells (OSCs) have a huge potential for long-term solar energy harvest technology due to their low cost fabrication, light weight and possibility of roll-to-roll manufacturing of large area devices [1]. The research has witnessed an advancement in the last 10 years that milestone power conversion efficiencies (PCE) over 10% are reported regularly [2, 3]. Low band gap π-conjugated polymers that harvest a substantial amount of the solar irradiation are usually used as donor materials in efficient OSCs. Synthesis of low band gap polymers can be achieved through incorporation of units that have a valence band formed with the contributions of quinoid resonance of the units or by coupling electron donor (D) and electron acceptor (A) units alternatively [4–6]. The band gap of D-A copolymers is narrowed through formation of quinoid resonance structure via intramolecular charge transfer (ICT) between the units.

Among the many electron donor units introduced to OSCs research benzo[1,2-b:4,5-b']dithiophene (BDT) has gained a lot of attention and has been successfully used in highly performing donor polymers such as (poly)[4,8-bis(2-ethylhexyl)oxy]benzo[1,2-b:4,5-b']dithiophene-2,6-diyl][3-fluoro-2-[(2-ethylhexyl)carbonyl][thieno[3,4-b]thiophenediyl]] (PTB7) mainly due to its structural rigidity, planarity, extended π-conjugation length and favorable interchain π–π stacking in solid state [7–10]. Recently, PCE exceeding 17% was reported using BDT based copolymers [11, 12]. The band gap of BDT based copolymers can be fine-tuned by using appropriate acceptor units such as isoindigo. Recently, the synthesis and photophysics of two BDT-alt-isoindigo copolymers with band gap below 1.6eV and broad absorption that extends over 750 nm are reported. A good interchain interaction is expected in the copolymers due to their planar backbone structure [7].

The application of polymers in OSCs will be ensured when both high PCE and environmental stability of the devices can be attained. However, the stability of OSCs has been a bottleneck for the commercialization of the
technology even after achieving efficiencies exceeding 18% [13]. Degradation by extrinsic factors that include exposure to light, heat and environmental conditions should be addressed to facilitate the application of OSCs as energy source to meet the global energy demand [14–16]. Most importantly, light-induced degradation in OSCs is a crucial problem that has to be addressed to elongate the working life-time of the devices. This kind of degradations can occur in any of the layers in a multi-stack OSCs devices. Active layer materials usually composed of donor polymers and acceptors such as the fullerene, should be stable while working under irradiation to ensure an uninterrupted charge generation in the device. A stable donor material is a prerequisite for the stability of the active layer. Its stability is determined by many factors that include the stability of the units incorporated on its backbone structure and aggregate formation in the film. The interchain interaction that is responsible for fast charge transport must be maintained under degradation stresses. Light-induced degradation of the pristine donor material and when the same materials is blended with the acceptor in a bulk heterojunction (BHJ) active layer are usually different [6]. A number of researchers have found that oxidation of a polymer through triplet formation can be inhibited when PC_{60}BM is blended with it [6, 17, 18]. Additionally, radical scavenging and UV-screening of PCBM in a BHJ active layer were found to be potential stabilization techniques [19, 20]. This motivated us to investigate the effect of PCBM and intermolecular interaction on the photostability of a BDT-based copolymer that was synthesized by our group [7].

Two highly thermally stable BDT-alt-isooindigo copolymers with thermal decomposition temperatures of over 380 °C were synthesized and reported in [7]. The application of these copolymers for stable OSCs can be ensured if they are also stable under light exposure. In this contribution, light-induced degradations of a copolymer composed of BDT and 2-decyltetradecyl (DT) substituted isoindigo, PBDTI-DT is investigated. The effects of both PC_{60}BM loading and intermolecular interactions in the photostability of PBDTI-DT were studied using a light source with intensity that matched the AM 1.5 solar irradiation. A strong inter-chain interaction in a polymer film is beneficial for an enhanced charge transport. However, its effect in the light induced degradation of polymers has not been well explored. Hence, the role of intermolecular interaction in the photostability of PBDTI-DT copolymer is studied in this work. The results confirmed that intermolecular interaction is beneficial not only for charge transport but also to reduce photo-induced decoupling of the donor and acceptor units.

2. Experimental section

2.1. Optical characterization

The absorption spectra of the solution of PBDTI-DT and spin-coated thin films of both the pristine PBDTI-DT and its blend with [6,6]-Phenyl-C_{61}-butyric acid methyl ester (PC_{60}BM) (1:1) were recorded using an Eidenburgh instruments model DS5 UVvis spectrophotometer. The films were prepared from 1,2-dichlorobenzene (o-DCB) solution with a concentration of 25 mg/mL that was stirred for 3 h at 60 °C. The thin films were spin–coated at 1000 rpm and annealed at 80 °C for 10 minutes. Moreover, their photoluminescence (PL) spectra were recorded by exciting at their absorption maxima in the longer wavelength region using JY Horiba, Fluoromax-4 spectrofluorometer.

2.2. Ageing

Photo-induced degradations of the pristine PBDTI-DT in solution and as thin film, and as BHJ PBDTI-DT:PC_{60}BM blended films were monitored using their absorbance losses during light exposure. A tungsten lamp operating at an intensity of 100 mW cm^{-2} was used as a light source and the degradation was recorded for more than 70 h. The samples temperature was maintained by using a fan throughout the ageing process to eliminate any thermally-induced degradation in the measurements. The remaining peak absorbance (A_{\text{max,remaining}}) recorded as a function of the ageing time were used to quantify the photodegradation of the samples studied. The remaining peak absorbance was determined using equation (1):

\[ A_{\text{max,remaining}} = \frac{A_{\text{max}}(t)}{A_{\text{max}}(0)} \times 100\%, \]

where A_{\text{max}}(0) and A_{\text{max}}(t) are absorbances recorded before and after time ‘t’ of degradation, respectively.

Moreover, the evolution PL spectra and x-ray diffractometer (XRD) traces of the films of both the pristine PBDTI-DT and its blend with PC_{60}BM were also recorded for fresh and photodegraded samples at different light irradiation time intervals to further understand light-induced degradation in this work.

2.3. XRD measurement

The XRD traces of the pristine PBDTI-DT and its blend with PC_{60}BM were recorded for 2θ values from 10 to 40° in drop–casted films on glass substrates using Shimadzu Scientific Instruments XRD-7000S with a copper target, Cu-Kα, radiation operating at 40 kV x 40 mA power. The evolution of XRD traces of both the pristine PBDTI-
DT and its blend with PC_{60}BM films were recorded during light irradiation to understand light induced structural changes in the films. Bragg’s equation was employed to determine the interlayer spacing between the planes (d) using the following relation.

\[ n\lambda = 2d \sin \theta \]  

Where \( n, \lambda \) and \( \theta \) are order of diffraction (1), wavelength of the x-ray (0.154 nm) and angle of diffraction, respectively.

### 3. Results and discussion

#### 3.1. Photophysical properties of PBDTI-DT and PBDTI-DT:PC_{60}BM films

The absorption of PBDTI-DT and PBDTI-DT:PC_{60}BM films were recorded using UV-Vis spectrometer as shown in figure 1(a). The absorption of the pristine film exhibited the common two-band profile due to the alternating donor-acceptor bonding in the copolymer as reported in [7]. The two transitions in the short (below 500 nm) and long (above 500 nm) wavelength regions are attributed to \( \pi - \pi^* \) and intramolecular charge transfer (ICT), respectively. The absorption onset of PBDTI-DT in film was found to be red-shifted from its solution by 29 nm. Consequently, the optical band gap of the copolymer calculated using Einstein’s relation \( E_g = \frac{1240}{\lambda_{onset}} \) (where \( \lambda_{onset} \) is the onset of absorption) was found to be 1.54 eV. The ICT band on the other hand is blue-shifted by 5 nm upon solidification indicating the formation of H-aggregates in the films. The cofacial arrangement of the polymer chains improves the interchain interaction in the film due to \( \pi - \pi \) stacking which is beneficial for charge transport [7]. The absorption of PBDTI-DT:PC_{60}BM BHJ film revealed a clear superposed absorption of the donor and the fullerene acceptor. In this regard, PC_{60}BM makes the most contribution to the blend’s absorption below 400 nm while the copolymer makes a considerable contribution above 400 nm. Since the photon flux of solar irradiance below 400 nm is small, PC_{60}BM’s contribution to exciton generation is negligible indicating its main role is as electron transport material in the blend film [6].

Besides photon harvest by the active layer of an OSCs which is usually a BHJ of donor and acceptor organic semiconductors, the exciton dynamics both in the pristine films and blend plays a crucial role in the performance of device. The PL spectra of the pristine PBDTI-DT and PBDTI-DT:PC_{60}BM BHJ films were recorded by exciting the polymer to study charge transfer dynamics in the BHJ layer (figure 1(B)). A large Stokes shift of 182 nm was recorded in the pristine film confirming a significant electronic structural shift from the ground to excited state of PBDTI-DT has occurred. This shift is from D - A to D^+ - A^- due to its ICT characteristics [7, 21]. The electron transfer from the polymer to the fullerene acceptor is found to quench the PL from the donor polymer confirming efficient charge transfer in the BHJ film with exciton quenching efficiency over 75%. This further shows that the copolymer is miscible with the PC_{60}BM acceptor material, creating better nanomorphology for the excitons created to diffuse to the donor-acceptor interface before recombination [22].

#### 3.2. Photoinduced degradation of pristine PBDTI-DT and BHJ PBDTI-DT:PC_{60}BM films

The photochemical stability of both the pristine PBDTI-DT and BHJ PBDTI-DT:PC_{60}BM films were investigated by recording their absorption recorded during irradiation as shown in figure 2(A). The absorbance...
of fresh PBDTI-DT exhibits a broad band confirming its extended π-conjugation. In the first few hours of irradiation, the absorption of the pristine film was bleached while keeping its profile. This could be due to the peeling off of the polymer making it thinner [21, 23, 24]. Upon further increase of the light stress on the film, the main absorption started to blue-shift by more than 16 nm as shown in figure 2(D). Evidently the blue-shifted and narrowed absorbance after the long light stress is due to the breaking of the π-conjugation on the backbone of PBDTI-DT. The scission of the chains can be initiated by the formation of radicals, R, from the long side chains [25]. This scission can lead to photooxidation that will finally produce a product with a different chemical property. However, PBDTI-DT did not reveal a change in the absorption profile after 70 h of irradiation that would indicate the change in its chemical structural which confirmed it stability against photo-oxidation [21, 25, 26]. The absorption of a similar copolymer, a thiophene-alt-isoindigo, was found to be completely bleached in less than 40 h [6]. Comparing this result with PBDTI-DT, which maintained more than 50% of its absorbance after 70 h of irradiation, it can be concluded that BDT is a better donor unit for photostable copolymers. One reason can be its deeper HOMO (−5.5 eV) energy level [7] compared to the thiophene and isoindigo copolymer (−5.2 eV) reported in [6]. Martynov et al recently reported the effect of oxidation potential on the photoinduced degradation of 16 polymers using a sensitive electron spin resonance measurement [27]. In their report, they elucidated the importance of deep-lying HOMO level to inhibit photooxidation. Likewise, it could be clearly seen that PBDTI-DT, which has a donor with more delocalized electrons than the thiophenes, is more stable against photooxidation due to its deeper-lying HOMO level. Interestingly, the photobleaching of PBDTI-DT was suppressed upon blending it with PC60BM as shown in figure 2(B).

The remaining absorbance of both the pristine and the blended films were used to quantify the role of acceptor blending in the light induced degradation of PBDTI-DT as shown in figure 2(C). In the first 20 h, an absorbance loss of 15 and 4% were recorded in the pristine and PBDTI-DT:PC60BM BHJ films, respectively. After 70 h of light stress the absorbance loss increased to 44 and 20% in the pristine and PBDTI-DT:PC60BM films, respectively, further confirming the stabilizing effect of PC60BM against photodegradation. However, in the first three hours, both the pristine and the BHJ films exhibited similar degradation kinetics. The superior stability of the BHJ film is attributed to three possible mechanisms i) exciton quenching by the acceptor reducing the exited polymer species in the film which is highly reactive to oxygen [20] ii) UV-screening effect of PC60BM. This could
be confirmed from the less than 5% absorbance loss after 70 h irradiation in the BHJ film in the region where PC_{60}BM absorbs (see figure 2(C) (blue)) and iii) the less rapid penetration of oxygen to the film as PC_{60}BM has higher density than the polymer. This argument is supported by the similar degradation of both the pristine and BHJ film in the first three hours of exposure to light. During these hours the film must be going thorough rearrangement of its morphology before PC_{60}BM could be on top of the active layer.

To support the argument on the stabilization mechanism of the BHJ film by PC_{60}BM, the PL of the blend was recorded as shown in figure 3. The PL of PBDTI-DT:PC_{60}BM film decreased in the first 3 h of irradiation followed by a slight increase at 20 h. The PL of the BHJ film started to decrease after longer light irradiation which is mainly due to the degradation of the absorbance of the film. The rearrangement of the PBDTT-DT:PC_{60}BM morphology in the first three hours increased its exciton quenching efficiency as confirmed by the lower PL intensity. However, further increase in photon stress must have segregated the PC_{60}BM from the BHJ which could benefit its stability but not the charge dynamics as revealed by the decrease in the exciton quenching efficiency at 20 h. In summary, blending PBDTT-DT with PC_{60}BM could stabilize the absorbance of the BHJ active layer but not the charge dynamics indicating that further modifications are required to maintain both the photon absorption and charge dynamics in the active layer to ensure a photostable OSC.

3.3. X-ray diffraction traces with photon stress
The XRD traces of pristine PBDTI-DT and BHJ PBDTI-DT:PC_{60}BM films were recorded before and after exposure to light to further understand light-induced degradation in the films and the results are shown in figure 4. As seen from figure 4, the XRD traces of the pristine and BHJ films revealed a broad halo (010) centred around 2\(\theta\) = 23.62° and 20.76°, respectively. This is due to the \(\pi-\pi\) interchain stacking in the polymer that are separated by 0.38 and 0.43 nm in pristine PBDTI-DT and PBDTI-DT:PC_{60}BM BHJ films, respectively, as calculated by Bragg’s law (equation (2)). The packing confirms the presence of a strong intermolecular interaction in the copolymer backbones [28].

To investigate photoinduced structural ordering changes, the XRD traces of pristine PBDTI-DT and PBDTI-DT:PC_{60}BM BHJ films were recorded for 24 hours. The XRD-traces of PBDTI-DT revealed a decrease in intensity accompanied by a peak shift to lower diffraction angle during light exposure while the BHJ film kept its profile. The decrease in the diffraction intensity of PBDTI-DT upon exposure of light was due the bleaching of polymer film. However, this decrease in diffraction peak intensity due to photoinduced degradation was suppressed in the BHJ film because PC_{60}BM serves as a photoprotective layer. In our recently published work, the XRD traces of the push-pull terthiophene-isooindigo copolymer was found to remain intact due to the suppressed photodegradation by the addition of PC_{71}BM [6].

3.4. Intermolecular interaction and photostability of PBDTI-DT
Intermolecular interaction is the key to efficient charge transport in OSCs. The properties of non-interacting polymer chains can be usually extrapolated to films [29, 30]. Hence, the role of intermolecular interactions in the photostability of PBDTI-DT is investigated. Thus, a thin film of PBDTI-DT, where there is a strong
intermolecular interaction, and solutions of PBDTI-DT in oDCB with concentrations of 0.02 and 0.1 mg/mL, with almost no and small intermolecular interactions, respectively, were prepared. The absorption spectra of the the samples were recorded at intervals of 5 minutes as shown in figure S1 available online at stacks.iop.org/MRX/9/055502/mmedia.

The absorptions of the three samples taken before and after 2 h degradation (figure 5(A)) reveal a decrease in absorbance of the long wavelength absorption maximum in all measurements. This decrease corresponds to values of 8.42 and 7.95% for solutions with concentrations 0.02 and 0.1 mg/mL, respectively, and 5.62% in the thin film indicating that an intermolecular interaction can benefit the photostability of PBDTI-DT. To further elaborate on the role of intermolecular interaction, the remaining absorbance of PBDTI-DT during irradiation both in solutions and film are plotted as shown in figure 5(B). The degradation kinetics in the solutions clearly follows two steps which are a decrease in absorbance both at the ICT and $\pi - \pi^*$ peaks until 20 minutes followed by a stable ICT and an increase in absorbance on the $\pi - \pi^*$ transition. On the contrary, the absorbance of the film decreases in the ICT transition while its $\pi - \pi^*$ remained almost unaffected by the photon stress. The $\pi - \pi^*$ transition peak of PBDTI-DT has a main contribution from the donor BDT unit. The growing of the $\pi - \pi^*$ absorption in the three samples clearly show the decoupling of the donor BDT unit from the polymer.

Figure 4. Evolution of XRD traces of pristine PBDTI-DT (solid) and BHJ PBDTI-DT:PC_{60}BM blend (dash) with photon stress.

Figure 5. Time evolutions of (A) absorption spectra and (B) the remaining absorptions at the ICT and $\pi - \pi^*$ transitions of PBDTI-DT in solutions of concentrations 0.02 and 0.1 mg/mL and film.
In this regard, the solution with lower concentration manifested a larger breaking of intramolecular bonding between the donor BDT and the acceptor-isoindigo units. Interestingly, the breaking of intramolecular bonding is suppressed with increasing intermolecular interactions. This can be due to the stabilized LUMO of the polymer due to intrachain interaction in the film that imparts photostability [29, 31]. The selected PL spectra of the less concentrated solution which was recorded during the 2 h degradation were shown in figure 6(A). The full measured PL spectra are shown in figure S2. Similar to its absorption, the light induced PL kinetics of PBDTI-DT in solution of low concentration depicts two steps, a sharp decrease to about 40 minutes followed by stable PL intensity. This leads to the conclusion that the mechanism that is responsible for the absorbance loss is the cause for PL intensity decrease. Since the emission recorded is from ICT de-excitation, the two possible reasons can be the lower population of the state and/or conformational changes in the backbone of the polymer. The ICT state population is confirmed to get reduced due to the decoupling of the donor and acceptor units as indicated by the growing $\pi-\pi^*$-transition (see figure 5(B)). Therefore, the reduced ICT emission further confirms the role of inter-molecular interaction in keeping the intra-molecular bonding of the copolymer. To elucidate further on the ICT emission quenching, the evolution of Huang-Rhys factor, \( S = I_{0-1}/I_{0-0} \) with photon stress was calculated as shown in figure 6(B), where \( I_{0-1} \) and \( I_{0-0} \) are emission at the 0-1 and 0-0 transitions of the ICT state, respectively. Interestingly, the PL intensity of both the \( I_{0-1} \) and \( I_{0-0} \) decreased at the same rate keeping the Huang-Rhys factor around 0.5. The Huang-Rhys factor can be used as a measure of conformational disorder in a polymer. In this regard, it can be concluded that the role of conformational disorder in the photobleaching kinetics of the copolymer chains is minimal [30]. Note here that the two transitions in the PL spectra were extracted by fitting each spectrum with two-peak Gaussian functions. Hence, the main reason for the quenching of ICT emission in the dilute PBDTI-DT solution is decoupling of the donor and acceptor units. This finding suggests that polymers with strong interchain interaction not only exhibit high charge transport properties but also superior photostability.

4. Conclusion

The photostability of a benzodithiophene-isoindigo (PBDTI-DT) copolymer was studied in solution, as pristine film, and BHJ blend. The photostability investigation of the copolymer in dilute solution revealed that the main degradation mechanism is $\pi$-conjugation breakage in the backbone as indicated by the blue shift of the main PL peak. In addition, its absorption exhibited a growing $\pi-\pi^*$ transition confirming the decoupling of the donors and acceptors in the copolymer chains. This argument was also complimented by the stable conformation of the copolymer during degradation as confirmed by its stable Huang-Rhys factor which was around 0.5. However, when in thin films of the polymer, the main degradation was found to be chain scission while D-A decoupling is suppressed, which led to the conclusion that a stronger interchain interaction made the polymer stable. The photostability of PBDTI-DT was further improved when it was blended with PC_{60}BM. This was due to the UV-screening effect of PC_{60}BM in addition to quenching of the excited polymer, which is highly reactive with oxygen and radical scavenging by the acceptor. However, the charge dynamics in the BHJ film was impaired due to photodegradation as confirmed by the lower excitation quenching efficiency in the first 20 h of irradiation. In summary, the photostability of thin films of the polymer was mainly due to its high intermolecular interaction in the solid state. The active layers of OSCs with PBDTI-DT:PC_{60}BM BHJ blend will be stable if there is way of...
controlling their morphology. Hence, a further work on morphology control, to get both stable absorption and high charge transfer dynamics is needed.

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Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

Conflict of interest

The authors declare that there is no known conflict of interest in this work.

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