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Effect of processing additive 1,8-octanedithiol on the lifetime of PCPDTBT based Organic Photovoltaics

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ABSTRACT

The effect that processing additives have upon the lifetime of PCPDTBT-based OPVs has been investigated. The results show conclusively that whilst ODT processing additive enhances the initial performance of PCPDTBT:PCBM OPVs, it is detrimental to their long term performance, when measured under light soaking at 1 Sun of irradiance. Results are shown for both encapsulated and non-encapsulated devices. Topographical and morphological measurements made using AFM and small- and wide-angle X-ray scattering of active layers show that there are greater morphological changes of devices fabricated with the 1,8-octanedithiol upon light soaking, revealing a relatively venerable morphology of the active layer processed with the additive, when subject to light soaking.

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1. Introduction

The behaviour of Organic Photovoltaic (OPVs) devices has been extensively researched over the past decade [1,2], particularly with respect to the way in which the processing steps optimise the performance [3]. Many reports of high performing OPVs emphasise that the active layer requires further processing after coating, normally using thermal treatment [4,5]. In polymer-fullerene blended OPVs, this can result in an increase in crystallinity in the polymer or improvement in the phase separation of the blend morphology, which is assumed to lead to an improved interconnected pathway to the electrodes for charge carriers.

Poly[4,4-bis(2-ethylhexyl)-4*H*-cyclopenta[2,1-*b*;3,4-*b*′] dithiophene-2,6-diyl-*alt*-2,1,3-benzothiadiazole-4,7-diyl] (PCPDTBT) is one of the most commonly reported low band-gap polymers and when blended with fullerene derivatives such as [6,6]-phenyl C_{71}-butyric acid methyl ester (C_{71}-PCBM) gives a Power Conversion Efficiency (PCE) close to 5.5% [6]. This decreases however when the device is subjected to a post-bake or annealing step [2] to stabilise the morphology. An alternative method to control the morphology of the blended film is normally employed, which involves adding a second non-reacting solvent, known as a processing additive, to the donor and acceptor solution prior to coating. The most studied and understood processing additives are alkanethiol based compounds [7]. It has been shown that the incorporation of a few volume percent of alkanethiols into the PCPDTBT:C_{71}-PCBM OPVs improves the PCE by about a factor of two. It does this by modifying the morphology [2,6,7] and controlling the phase separation between the donor and acceptor components [8]. Using a transistor configuration to determine the charge carrier mobility, it was found that the electron mobility increases in the

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PCPDTBT:C_{71}-PCBM active layers, when fabricated with alkanethiols, which was attributed to the enhanced connectivity of C_{71}-PCBM networks [9,10]. Other reports have also indicated that processing using alkanethiols in active layer blends leads to small increases in polymer crystallinity [11], reduced charge recombination and reduced charge carrier loss [12].

Despite the significant research progress in understanding the experimental application and fundamental physics behind using processing additives in PCPDTBT:PCBM OPVs, no reports exist on how the long term stability of devices is affected using such processing additives. This is a critical issue if these additives are to be used in the commercialisation of the technology. In this publication, the results of lifetime studies of PCPDTBT-based OPVs are discussed for devices fabricated with and without alkanethiol processing additives included in the initial active layer blend. The results given below show that while the additive enhances the initial performance of a device, it is detrimental to the long term performance.

2. Experimental

Cell devices were prepared on pre-patterned ITO glass slides with a surface resistivity of 16 Ω/square. The ITO slides were cleaned using deionised water, acetone and isopropyl alcohol (IPA) and blown dry using nitrogen. The slides were then placed in an oxygen plasma for 10 min. A layer of the conductive polymer PEDOT:PSS mixture (HC Starck Clevios PAI 4083) was spin coated (in air) onto the substrate at 5000 rpm for 30 s before a 20 min bake at 120 °C on a hotplate to remove any residual H_2O.

Devices were fabricated and tested in a glovebox, which maintained H_2O and O_2 concentrations to typically less than 0.1 part per million. Active layers of PCPDTBT:C_{71}-PCBM with weight ratios of 1:3 were prepared and dissolved in chlorobenzene solvent with a concentration of 30 mg/ml, using the same manner as previous papers by this group on PCPDTBT [2,13,14], where the optimisation of polymer:fullerene blend ratio, film thickness and annealing conditions have already been rigorously studied. For devices with alkanethiol additive, 1,8-octaneditiol (ODT), Sigma-Aldrich (03605), was mixed with the initial blend with a concentration of 3% by weight. The solutions were then allowed to equilibrate for 24 h on a hot plate stirrer at 60 °C and then filtered using a 0.45 μm PTFE filter. Active layers were applied by spin-casting in a glovebox from a 60 °C heated solution (2200 rpm for 60 s for devices without ODT, 2500 rpm for devices with ODT). It is important to note that ODT possesses a high boiling point of 269 °C and is unlikely to be lost from the active layer.

Other groups [6,10] have reported that blends made with 1,8-diiodooctane (DIO) instead of ODT additive yields better a slightly higher PCE in final devices (approximately 10–20% relative increase). However, through our optimisation, we find that ODT is slightly better performing; this difference is likely to originate in the differing molecular weights of polymer synthesised through Suzuki coupling (M_n = 20 k) and Stille coupling (M_n = 28–50 k).

Finally, a 5 nm layer of Calcium (Ca) and an 80 nm layer of Aluminium (Al) were deposited on each sample by thermal evaporation through a shadow mask to form a composite cathode electrode, which defined six active areas of 0.2 cm^2. For encapsulated devices, UV encapsulant was supplied by Polymetronics ltd., Oxford, UK, which was sandwiched between the OPV device and a rear coverslip.

The initial performance of the devices was determined using a Newport solar simulator with 100 mW cm^{-2} AM1.5 output calibrated using a silicon reference cell verified by (RERA, Netherlands). Subsequent light soaking measurements was provided from a halogen light soaker (GB-Sol ltd, Cardiff, UK). This was conducted in accordance with ISOS-L-2 standards [15]. *I–V* measurements were made every 30 min and devices were biased at the Maximum Power Point (MPP) in between measurements. The device temperature was not controlled but remained at 50 °C ± 5 °C during the measurements. Both encapsulated and non-encapsulated devices were measured for 1000 h, or until the PCE reached 10% of its initial value.

Measurements of the External Quantum Efficiency (EQE) were conducted periodically to follow the trend as light soaking proceeded. They were made with a monochromator with nm resolution (Bentham) and an SRS lock-in amplifier. A Xenon quartz light source (Osram HLX64623) with an output spectrum from 350 nm to 1000 nm was used and measurements were taken every 2 nm over a range of 375–920 nm. A silicon photodiode (Thorlabs SM1PD1A) is used to make the reference measurement. Two order sorting filters were used to ensure that the correct wavelength was being used to illuminate the devices. For EQE measurements, only one non-encapsulated device was used and was light soaked in between measurements. This device was not biased at maximum power point in between measurements.

3. Results

3.1. J–V measurements under light soaking

For the devices without ODT, a reasonable photovoltaic effect was observed with mean values of short-circuit current density (J_{SC}) of −7.37 mA/cm^2 ± 0.87, open-circuit voltage (V_{OC}) of 0.571 V ± 0.07 and fill factor (FF) of 0.393 ± 0.04, resulting in PCE of 1.78% ± 0.18. For the devices with ODT, a significant improvement in the device performance was observed with J_{SC} increases to −13.06 mA/cm^2 ± 1.07, FF to 0.46 ± .01, V_{OC} at 0.630 V ± 0.007 leading to an overall efficiency of 3.78% ± 0.22. Encapsulation of the device did not affect device performance.

Light soaking, as described earlier, was then begun with data collected every 30 min. Fig. 1 shows how the key performance parameters V_{OC}, J_{SC}, FF and PCE for the PCPDTBT:C_{71}-PCBM solar cells fabricated with and without ODT changed relative to their initial values during light soaking. As expected for the overall performance of devices without encapsulation decreased rapidly, emphasising the need for encapsulation if devices are to be put to practical use [15,16]. It is also clear that devices with ODT degraded faster that those without. For non-encapsulated devices

the half life ($t_{1/2}$), defined when the PCE falls to half value, is 46 h without ODT and only 25 h when ODT has been included. For encapsulated devices, the half-life is 156 h for devices with ODT and 2820 h without ODT (fitted from a trend line after 1000 h).

When considering the relative changes of each parameter for non-encapsulated devices, the main cause of the rapid decrease in PCE for devices with ODT is a significant reduction in J_{SC} and FF. The fall in FF could be attributed to changes in morphology influencing charge extraction from the active layer. However the temperature of the device was always in the range 50 °C ± 5 °C and previous Differential Scanning Calorimetry (DSC) measurements of PCPDTBT has indicated that the material does not morphologically change significantly upon heating, even up to 220 °C [17]. In addition, no obvious changes in the colour of the devices were observed after light soaking.

Whilst the degradation of devices with ODT was more rapid, it is important to note that this device is initially much higher performing than those without. Such is the rapid degradation of the device with ODT that after just 47 h of light soaking, the output of devices without ODT overtook those with the additive. Over the lifetime of those fabricated without ODT generated 171 mW h/cm^2, whilst the device with ODT generated the lesser amount of 168 mW h/cm^2.

For encapsulated devices (see Fig. 1), the effect of ODT on long-term performance is even more striking. Whilst the degradation of J_{SC} is actually up 200 h, this is offset by a much larger reduction in FF. At 200 h, the FF drops to 65% of its initial value (i.e. FF = 31.85%) and J_{SC} is influenced by parasitic resistances. For the encapsulated devices the time at which the device without ODT starts exceeding the output of the device with ODT is at 312 h. Likewise, if we consider the power generated over the lifetime of each cell; we see that the device fabricated without ODT is still better performing; over the 1000 h of operation, generating 1457 mW h/cm^2, compared to 840 mW h/cm^2 for the device with ODT.

3.2. EQE measurements under light soaking

In addition to *J–V* characteristics, a series of External Quantum Efficiency (EQE) measurements were made as part of the lifetime study on the non-encapsulated devices. These allow the spectral response and degradation of the cells to be measured. The EQE of a device without ODT is compared to that of a device with ODT and Fig. 2(a and b) shows how the EQE of both devices evolves with light soaking time. The general trend of Fig. 2(a and b) is similar to previous reports of EQE for this material system [3,6]. For these measurements, the devices were not biased at MPP and *J–V* sweeps were not conducted every 30 min. Because these devices were not electrically stressed, we found the lifetime was significantly longer than measured in Section 3.1. This gives an interesting insight into another failure mechanism behind OPVs; device lifetime is dependent on bias conditions in between measurements, which will be reported in greater detail in a later publication.

The EQE curves of PCPDTBT:C_{71}-PCBM OPVs with and without ODT (Fig. 2(a and b)) are similar to those reported elsewhere for this material system. Both show the effect of the low band gap polymer causing absorption up to 850 nm, however it is shown that the photoconductivity

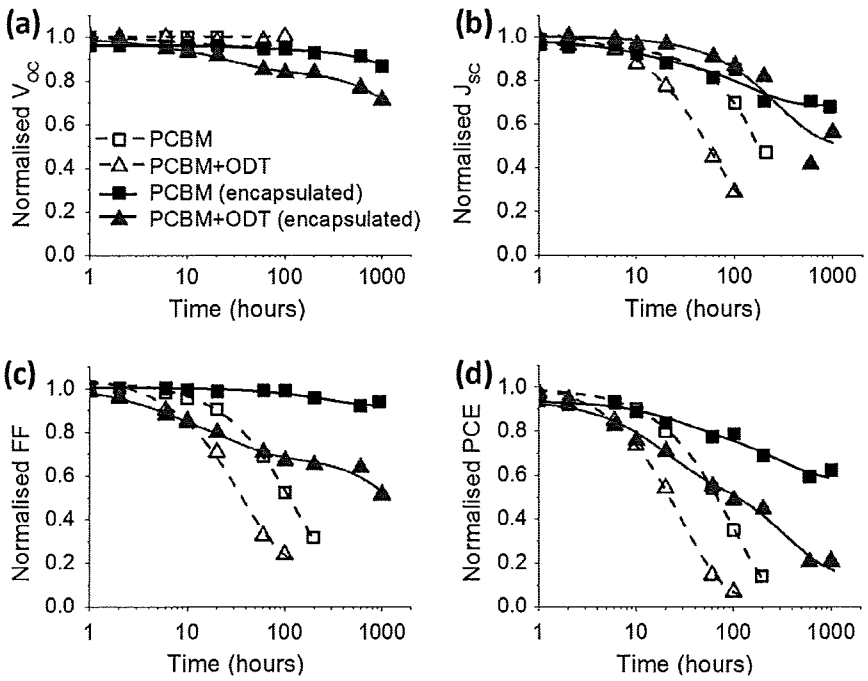

Fig. 1. Device characteristics for PCPDTBT:PCBM solar cells without ODT (black) and with ODT (red) during continuous light soaking for 1000 h under 1 sun of irradiance at 50 °C ± 5 °C. *J–V* sweeps were conducted every 30 min and devices were biased at maximum power point in between measurements. Bold lines indicate encapsulated devices and 'broken' lines non-encapsulated devices. The plots show the variation in (a) V_{OC}, (b) J_{SC}, (c) fill factor and (d) power conversion efficiency and are each normalised to the initial value at the start of the aging process. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

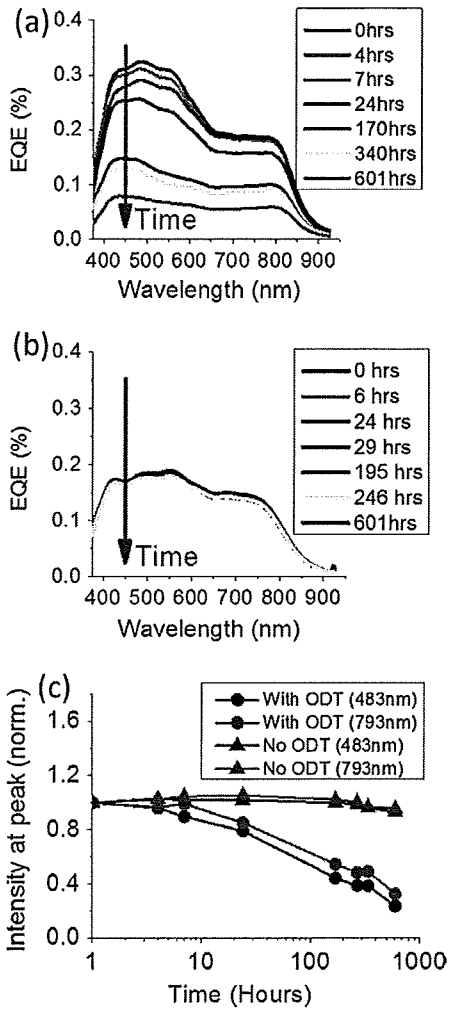

Fig. 2. External Quantum Efficiency (EQE) characteristics for non-encapsulated PCPDTBT:PCBM solar cells (a) with ODT and (b) without ODT during continuous light soaking for 800 h under 1 sun of irradiance at 50 °C. The time dependence of the EQE peaks at 483 nm and 793 nm (corresponding to peak C_{71}-PCBM and PCPDTBT absorption) for devices with ODT and without ODT are shown in (c).

response increases strongly by incorporating ODT to the solution [18–20]. As the devices are light soaked, the general shape of the curves only change slightly, indicating that PCPDTBT and C_{71}-PCBM in the active layer are degrading at only slightly different rates. The peak polymer absorption (793 nm) and the wavelength where C_{71}-PCBM absorption dominates (483 nm) are plotted for devices made with and without ODT, shown in Fig. 2(c). The overall degradation rates are much slower than in Section 3.1 as the devices are not being electrically stressed by biasing in between measurements or undergoing *J–V* measurements at regular intervals. It is shown that devices with ODT degrade possess a much lower intrinsic stability and do so more rapidly than devices without ODT. In addition, both PCPDTBT:C_{71}-PCBM devices showed slower degradation at the $\lambda = 793$ nm, corresponding to the PCPDTBT absorption, than at $\lambda = 483$ nm, indicating that C_{71}-PCBM is the less stable component of the active layer (see Fig. 3).

3.3. Morphological characterisation

To judge whether the ODT facilitates an additional degradation route, AFM images of the surfaces of non-encapsulated devices with and without ODT and before and after light soaking for 250 h were undertaken and are shown in Fig. 4. For typical devices with ODT, the initial surface roughness (R_A) was measured at 1.11 nm and the peak-to-peak roughness measured (R_{MAX}) at 9.35 nm. After light soaking, this reduced to $R_A = 0.63$ nm and $R_{MAX} = 7.08$ m, a relative decrease of 44% and 25%, respectively. By comparison a device without ODT prior to light soaking had a surface roughness of 0.55 nm and peak to peak value of 7.83 nm, which reduced to $R_A = 0.47$ nm and $R_{MAX} = 7.62$ nm, relative changes of 16% and 3%, respectively.

For further understanding of the structural changes that occur as a result of ODT inclusion in the active layer, we investigated the effect of light soaking on morphology using small- and wide-angle X-ray scattering (GISAXS/GIWAXS) at the National Synchrotron Radiation Research Center, Taiwan. Experimental setup is discussed elsewhere [20,21]. These layers were prepared in the same manner as the layers used for devices as reported in Sections 3.1 and 3.2, so were prepared onto PEDOT:PSS-coated glass substrates. Fig. 4(a) shows in-plane GISAXS profiles (20) measured for the active layers of PCPDTBT:C_{71}-PCBM with/without ODT and before and after light soaking for 300 h. Prior to light soaking, the GISAXS profiles of active layers with/without ODT very closely overlap. From Fig. 4(a), it was seen that light soaking resulted in increased scattering intensity at 0.004–0.002 $Å^{-1}$ (150–300 nm) for both layers with/without ODT, revealing a relatively large scale PCBM agglomeration [22,23]. However for the layers processed with ODT, scattering intensity in the higher-q region (>0.01 $Å^{-1}$) is enhanced substantially (10–20 nm), which corresponds to smaller and compact

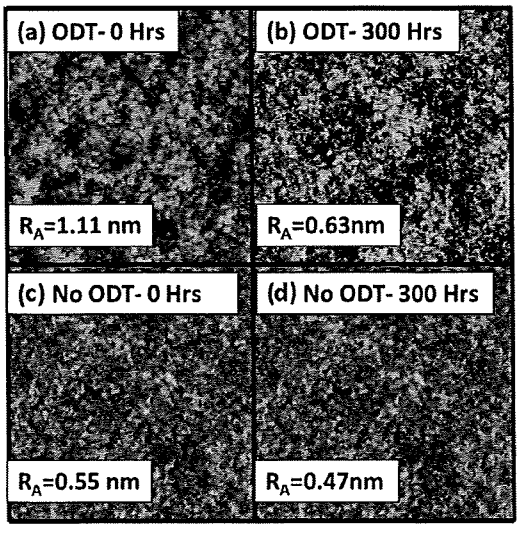

Fig. 3. Atomic Force Microscope (AFM) topography of PCPDTBT:C_{71}-PCBM OPVs with ODT (a) before light soaking and (b) after light soaking and PCPDTBT:C_{71}-PCBM OPVs without ODT (c) before light soaking and (d) after light soaking. From measurements of surface roughness, a larger relative change in morphology of devices fabricated with ODT is observed.

C_{71}-PCBM aggregates of an average size of 14 nm, as revealed from the sphere model fitting shown in Fig. 4(a) [21,22,24]. It is evident that morphology of the active layer processed with ODT is relatively unstable to light soaking.

Fig. 4(b) shows the corresponding normal-to-plane GIWAXS profiles for the same samples. The halo peak centers at 1.30 $Å^{-1}$ for the film processed without ODT moves to 1.15 $Å^{-1}$ for the film with ODT, revealing a substantial increase of an intermolecular distance of PCBM (22) from 4.8 Å to 5.5 Å. This effect may be attributed to less aggregated (or more disperse) C_{71}-PCBM in the film when spin coated with ODT (22). After light soaking, the intermolecular distance for the sample with ODT decreased from 5.5 Å to 5.3 Å (the halo peak moved from 1.15 to 1.20 $Å^{-1}$ in Fig. 4b), together with a substantially enhanced intensity, suggesting a stronger PCBM aggregation. In contrast, there is less changes in the halo peak for the film processed without ODT. Overall, the film processed with ODT exhibited larger local and global morphological changes associated closely to PCBM.

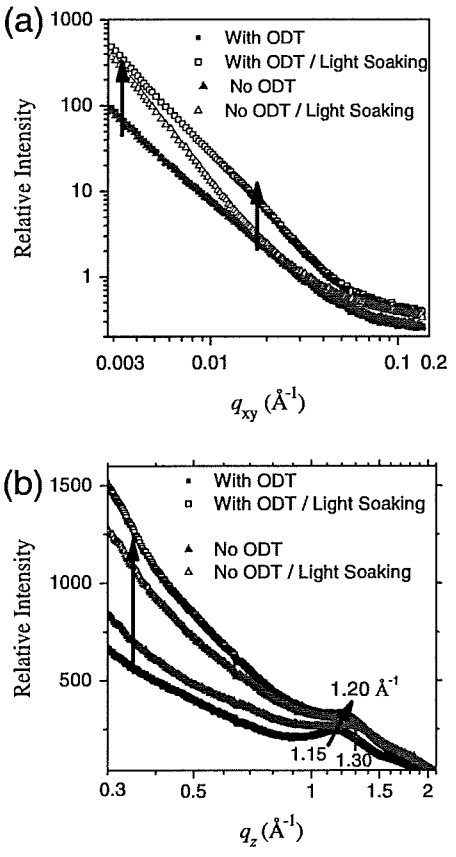

Fig. 4. (a) GISAX and (b) GIWAX profiles of PCPDTBT:C_{71}-PCBM active layers with /without ODT and before/after light soaking for 300 h. The arrows in (a) and (b) depict the characteristic intensity growths in different in-plane q_{xy}-region and normal-to-plane q_z resulted from light soaking, which indicates that films prepared with ODT undergo more substantial morphological changes. The GISAXS data for the film with ODT and subject to light soaking in (a) are fitted (solid curve) with a model of spherical PCBM aggregates of an average diameter 14 ± 2 nm, with 5% polydispersity in the higher-q regime (>0.01 $Å^{-1}$), together with a fractal agglomeration of a correlation length of 134 nm and a fractal dimension of 2.4 in the low-q region.

It is clear that from AFM data and the GISAXS/GIWAXS measurements that the morphological change induced by light soaking is greater when ODT has been included. ODT possesses a high boiling point of 269 °C and is unlikely to be evaporated at the low processing temperatures used in device fabrication, or even under the high vacuum for Ca/Al contact evaporation. It is therefore likely that some ODT remains in the active layer when devices are removed from the evaporation chamber and lifetime tests start. This conclusion is supported by Cho et al. [9], who showed that the ODT was not completely removed from the active layer films by placing them in high vacuum ($\sim 10^{-6}$ Torr). Residual ODT left in the active layer will selectively dissolve the C_{71}-PCBM component, as the PCPDTBT is less soluble in ODT [6] and will lead to increased aggregation and a reduction in the connectivity of C_{71}-PCBM percolation networks, thus limiting the amount of charge that can be extracted from the active layer. Therefore, in order to have stable PCPDTBT:C_{71}-PCBM devices made using ODT, it might be necessary to remove the ODT after processing. This could pose difficulties as ODT has a high boiling point and PCPDTBT:C_{71}-PCBM OPVs tend to decrease in performance with higher temperature annealing/baking steps [2]. An alternative is to use a crosslinkable PCPDTBT analogue to stabilize the film morphology optimised by ODT during spincasting, thereby, a sustainable high PCE performance upon light soaking.

4. Conclusions

It has been demonstrated that the inclusion of ODT in the active layer during the fabrication of PCPDTBT-based OPV devices led to a reduced overall performance under light soaking. Measurements of the FF as well as AFM and GISAXS-GIWAXS studies indicate that, while the incorporation of ODT during processing enhances the initial performance with an optimised morphology, it leads to greater and apparently detrimental changes in the morphology, when subject to long-term light soaking. Both the additive effects on morphology optimisation and long-term stability subject to light soaking, of the active layers, require balanced considerations.

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