Boron Subphthalocyanine Chloride as an Electron Acceptor for High-Voltage Fullerene-Free Organic Photovoltaics

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High-efficiency fullerene-free single-heterojunction (SHJ) organic photovoltaic (OPV) cells consisting of tetracene (Tc) as a typical donor material and boron subphthalocyanine chloride (SubPc) as an acceptor material are reported. Cells containing SubPc as a direct replacement for C_{60} exhibit an ~60% improvement in open circuit voltage (V_{oc}) achieving a maximum V_{oc} of 1.24 V, which is amongst the highest values acheived to date for SHJ devices. This resulted in an overall improvement of ~60% in power conversion efficiency from 1.8%, for Tc/C_{60} cells, to 2.9% for Tc/SubPc. The OPV device results are complemented by soft X–ray photoelectron spectroscopy (PES) measurements of the interfacial energetics of both systems. The results demonstrate that SubPc shows considerable promise as an electron acceptor material for future cell designs.

1. Introduction

Organic photovoltaics (OPVs) have received increasing attention over the past few years in the hopes of utilising them as a renewable, cheap, and economical source of power.[1] In 1986 Tang introduced the first organic donor–acceptor OPV cell demonstrating a power conversion efficiency (η_p) of ~1%.[2] Improvements in cell architecture,[3] choice of organic materials,[4–6] and electrodes[7] have all helped to push efficiencies to around 8% for state-of-the-art cells,[8] and significant further improvements in performance are predicted.

Oligoacenes have been shown to be attractive small molecule materials for OPVs as donor-type semiconductors due to their planar fused ring systems and crystalline structures which allow for high charge carrier mobilities (>1 $cm^2\ V^{-1}\ s^{-1}$),[9] and relatively long exciton diffusion lengths.[10] Pentacene has been the most extensively studied resulting in a relatively high η_p of ~2.0% when used in a single heterojunction (SHJ) cell in combination with C_{60} as the acceptor.[11,12] Although pentacene achieves a relatively high photocurrent in comparison to other small molecule donors, the restriction on the maximum achievable η_p is the relatively small ionisation potential (IP) of 4.9 eV.[10] It has been established that the maximum open circuit voltage in an OPV cell, V_{oc}, is related to the difference in energy between the highest occupied molecular orbital (HOMO) of the electron donor and the lowest unoccopied molecular orbital (LUMO) of the electron acceptor, i.e., the interface gap (I_g).[13] In pentacene/C_{60} cells the small IP limits the I_g at the heterojunction (~0.7 eV) and hence the measured V_{oc} (0.40 V).[11]

By comparison, tetracene (Tc) has received very little attention with regards to OPV applications despite having the potential, when used with C_{60}, to achieve a much greater I_g and therefore V_{oc} due to its larger IP of 5.4 eV.[14] Evaporated films of Tc can exhibit high molecular ordering with organic field effect transistors (OFETs) revealing a high hole mobility (μ_h) of 1.32 $cm^2\ V^{-1}\ s^{-1}$.[15] The exciton diffusion length of Tc is also expected to be similar to other oligoacenes such as pentacene, allowing for thicker films to be utilised in OPV devices. Chu *et al.* incorporated Tc as the donor material in conjunction with C_{60} in SHJ devices and achieved a η_p of 2.2%.[14] The devices were further improved using heat treatments which led to improved crystallinity, increased hole mobility and better overall performance.[16]

The lack of available electron accepting materials remains a significant limitation in the development of efficient OPV cells, with the vast majority of devices using fullerenes such as C_{60}, C_{70} or their soluble derivatives, $PC_{60}BM$ and $PC_{70}BM$. Although C_{60} is extremely efficient as an electron accepting material,[17] successful donor/acceptor heterojunctions depend on well tuned HOMO–LUMO energies and due to the relatively small band gap of C_{60} the V_{oc} is largely limited in these devices.[18] Peisert *et al.* synthesised fluorinated copper phthalocyanine, ($F_{16}CuPc$),[19] which without fluorination exhibits p-type behaviour, but after fluorination demonstrates n-type character with an increased IP, but with a relatively similar HOMO–LUMO gap.[19] Recently, $F_{16}CuPc$ was succesfully incorporated into an OPV device as an acceptor with boron subphthalocyanine chloride (SubPc) as the donor, demonstrating a η_p of 0.56%,[20] much lower than the reference SubPc/C_{60} cell (~3%).[7] In an attempt to create highly successful heterojunctions whilst still efficiently separating charges, perfluorinated SubPc ($F_{13}SubPc$) was recently

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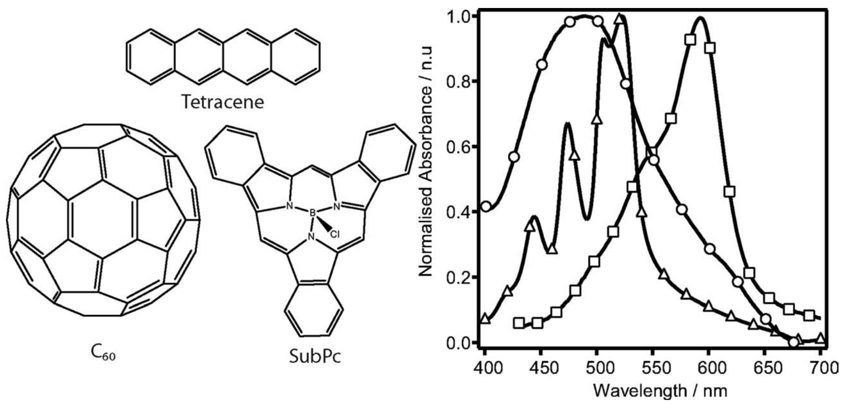

Figure 1. Molecules used within this study and their normalized UV-vis electronic absorption spectra: Tetracene (triangles), C_{60} (circles) and SubPc (squares).

developed as an acceptor by Gommans *et al.*[6] When used in combination with oligoacenes and other phthalocyanines, a marginal improvement in V_{oc} was achieved but a large decrease in both short circuit current density (J_{sc}) and fill factor (FF) in comparison to using C_{60} was found, resulting in a much lower η_p.[6] Recently, Sullivan *et al.*, through functionalization of SubPc via selective halogenation, tuned the interfacial energy level offset sufficiently to use it as an electron acceptor. A significant improvement in the V_{oc}, FF, and overall stability of the device was seen when using Cl_6-SubPc in conjuction with a SubPc donor.[5]

In this paper we demonstrate how a typical electron donor material, SubPc, can be used as a good electron acceptor in vacuum deposited SHJ OPVs using Tc as the donor. The electronic structure and energy level alignments of Tc/C_{60} and Tc/SubPc heterojunctions are studied using soft X–ray photoelectron spectroscopy (PES) and the performance of OPV devices based on these two donor/acceptor combinations optimised with regards to Tc and SubPc thickness. Through the replacement of C_{60} with SubPc we show significant improvements in V_{oc}, J_{sc}, and η_p.

2. Results and Discussion

The molecules used within this study and their normalized UV–vis electronic absorption spectra are shown in **Figure 1**. The overlap in absorption between Tc and C_{60} is quite large (Figure 1b) indicating competition for absorption from the layers in this region and therefore a limit to the J_{sc} obtained in the more conventional Tc/C_{60} cell. SubPc has the potential to replace C_{60} as the acceptor material due to the reported energy levels of the material, and its strong absorption between 500–600 nm allows for a greater percentage of the solar spectrum to be harvested.[21]

Various thicknesses of Tc films were grown on indium tin oxide (ITO) substrates and the morphology studied using atomic force microscopy (AFM). **Figure 2**a, which was taken from a 10 nm Tc film, reveals large voids between the micrometer-sized crystals, which still partially exist in the thicker 40 nm films (Figure 2b). This is consistent with previous reports by Chu *et al.*[14] The growth of Tc films on various substrates has been thoroughly documented and Stranski–Krastanov type growth is evident in the 60 nm film[22] (Figure 2c), with the steps of the multilayers most clearly apparent in the corresponding phase image (Figure 2d). Complete films were found to form at measured thicknesses (d_T) >40 nm, putting a lower limit on the thickness required for use as a donor layer in OPVs. The root mean square roughness, R_q, of the 60 nm films was found to be 8.5 nm, which is higher than other more commonly used donor materials, e.g., chloroaluminium phthalocyanine (ClAlPc), which has an R_q ~ 4 nm.[23]

As previously discussed, although the I_g is extremely important when achieving a larger V_{oc} in OPV cells, the magnitude of the $HOMO_D$–$HOMO_A$ and $LUMO_D$–$LUMO_A$ offsets are vital to achieving efficient dissociation at the heterojunction interface since too small an offset can have a detrimental effect on device performance. PES studies of the Tc/C_{60} and Tc/SubPc systems were therefore used to obtain quantitative information on the energy level alignment of the two organic heterojunctions. The

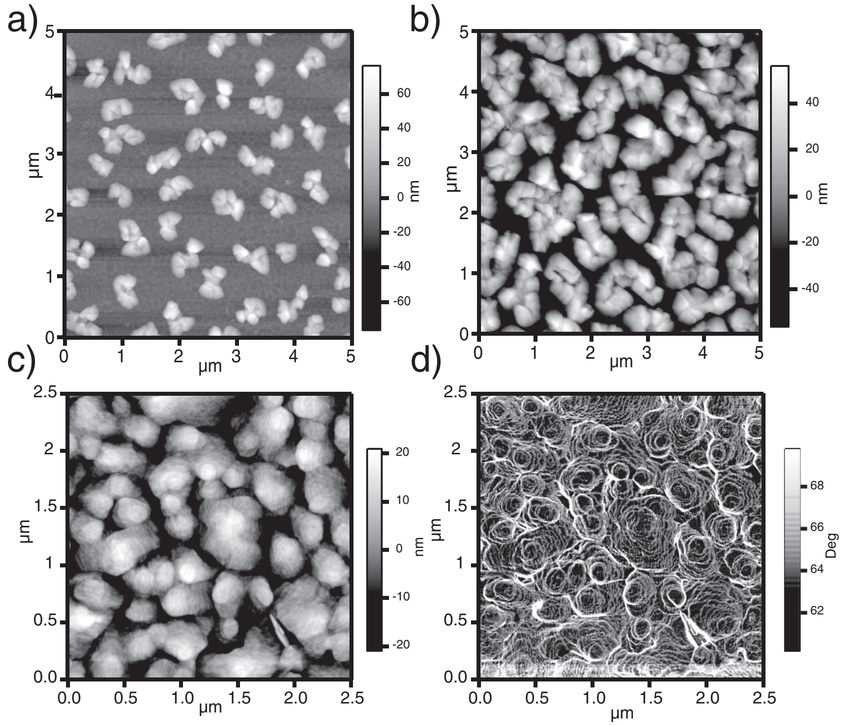

Figure 2. AFM images of: a) 10 nm, b) 40 nm, and c) 60 nm films of Tc grown on ITO; d) the corresponding phase image of the 60 nm Tc film.

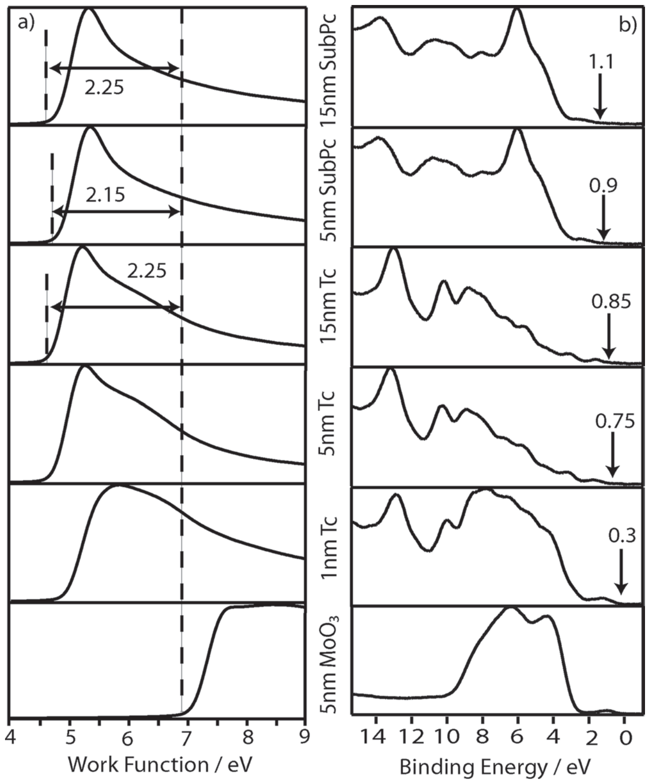

Figure 3. a) Change in onset of secondary electron PES spectra after sequential deposition of Tc and C_{60} on ITO/MoO_x. b) Valence band PES spectra recorded near E_f after the deposition of each layer.

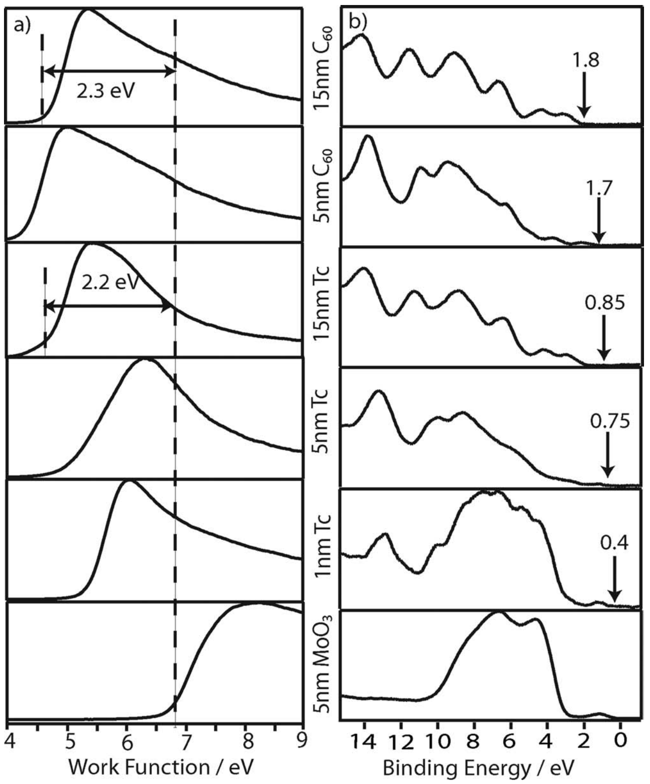

Figure 4. a) Change in onset of secondary electron PES spectra after sequential deposition of Tc and SubPc on ITO/MoO_x. b) Valence band PES spectra recorded near E_f after the deposition of each layer.

HOMO energies and shifts in vacuum level and work function were determined using the standard method given by Schlaf *et al.*[24] **Figure 3** shows the work function and the valence band spectra after deposition of a MoO_x (5 nm) film on ITO and subsequent growth of both Tc (1–15 nm) and C_{60} (5–15 nm).

The work function (measured from the E_{cutoff} in PES) changes significantly by ~2 eV when Tc is deposited onto the MoO_x layer due to the large interface dipole created. The large dipole at the MoO_x/Tc interface is clearly seen by the band bending observed in the Tc films in the range 1–15 nm, whereas at the interface between Tc and C_{60} there is only a small change in work function of 0.1 eV. The Tc HOMO onset saturates at around 0.85 eV below E_f giving an IP of 5.4 eV, which compares well to previous literature.[14] C_{60} was found to have an IP of 6.3 eV with the measured HOMO onset at 1.8 eV below the E_f.[21]

Figure 4 shows the PES spectra after deposition of Tc and SubPc on ITO/MoO_x. As seen in the Tc/C_{60} system there is a large change in the work function after deposition of the Tc layer onto MoO_x with a small change in work function at the Tc/SubPc interface. The SubPc HOMO saturation occurs around 1.1 eV below E_f and, similar to previous work, has an IP of 5.6 eV.[21] The energy level diagrams of the two different heterojunctions are summarised in **Figure 5**. This diagram was derived using the data shown in Figures 3 and 4 and the optical gaps of Tc, C_{60} and SubPc which are 2.2 eV, 2.0 eV and 2.1 eV respectively. The main difference between the two systems is the interface gap (I_g) which in the Tc/SubPc heterojunction was found to be 0.7 eV larger than the Tc/C_{60} system. This should allow for a much larger V_{oc} to be achieved in OPV cells containing SubPc as the acceptor, provided efficient exciton dissociation occurs. The $\mathrm{HOMO_D}$–$\mathrm{HOMO_A}$ and $\mathrm{LUMO_D}$–$\mathrm{LUMO_A}$ offsets are 0.2 eV and 0.3 eV respectively for Tc/SubPc, considerably smaller than the 1.1 eV and 0.9 eV found in Tc/C_{60}.

To optimise the Tc/C_{60} system a set of planar heterojunction OPV cells were grown with varying Tc thickness (d_T). The thickness of C_{60} was kept contant at 40 nm as this is believed to be optimal due to a reported L_D of ~40 nm.[17] *J–V* characteristics in the dark and under 1 sun illumination for these cells are shown in **Figure 6** with key device parameters listed in **Table 1** for the device structure ITO/MoO_x (5 nm)/Tc (d_T nm)/C_{60} (40 nm)/BCP (8 nm)/Al. The insertion of the 5 nm MoO_x interlayer allows for improved energy level alignment at the electrode/Tc interface.[23]

It is clear that the optimal Tc thickness is 60 nm, with a J_{sc} of 3.57 mA cm^{-2}, a V_{oc} of 0.76 V, and a FF and n_p of 0.68 and 1.83% respectively. Below this thickness ($d_T < 60$ nm) there is a decrease in both J_{sc} and V_{oc}. The reduction in J_{sc} is probably due to reduced exciton formation from the thinner Tc layer. The J_{sc} decreases with $d_T > 60$ nm, consistent with the creation of an increased number of non-dissociated excitons, and a reduction in charge collection efficiency. There is little change in the V_{oc} past the optimum thickness indicating that the maximum V_{oc} achievable has been reached.

To optimise the Tc/SubPc planar heterojunction OPV devices a set of cells were grown with varying SubPc thickness (d_S). The

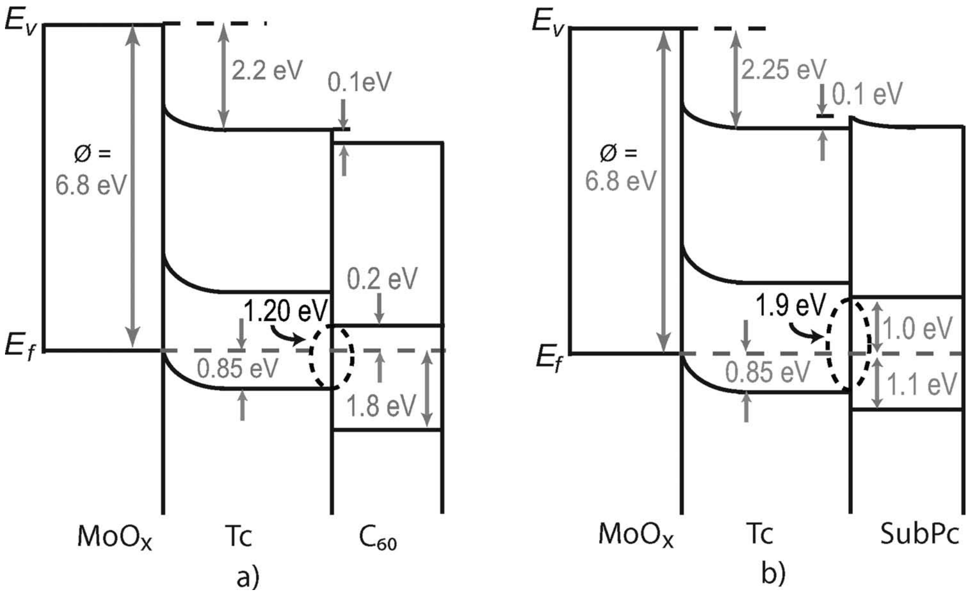

Figure 5. Energy level diagrams of a) ITO/MoO$_x$/Tc/C$_{60}$ and b) ITO/MoO$_x$/Tc/SubPc.

Tc thickness was maintained at its optimal value of 60 nm. *J–V* characteristics in the dark and under 1 sun illumination for the optimised Tc/SubPc device with structure ITO/MoO$_x$ (5 nm)/Tc (60 nm)/SubPc (35 nm) are shown in **Figure 7** with key device parameters for the series of thicknesses listed in **Table 2**. Data for the optimized Tc/C$_{60}$ OPV device is also shown for direct comparison.

Through the replacement of C$_{60}$ with a 35 nm layer of SubPc, the J_{sc} is improved to 4.01 mA cm^{-2} and the V_{oc} and η_p both increase significantly to 1.23 V and 2.89% respectively. There is only a very small corresponding drop in FF. The devices incorporating SubPc as the acceptor material exhibit a much higher

Table 1. Device parameters obtained from OPV devices. Devices A-D have the structure: ITO/MoO$_x$ (5 nm)/tetracene (d_T nm)/C$_{60}$ (40 nm)/BCP (8 nm)/Al.

d_T [nm]	J_{sc} [mA cm^{-2}]	V_{oc} [V]	FF	η_p [%]
A: 50	3.06	0.65	0.58	1.15
B: 60	3.57	0.76	0.68	1.83
C: 70	3.26	0.74	0.64	1.52
D: 100	2.67	0.72	0.60	1.12

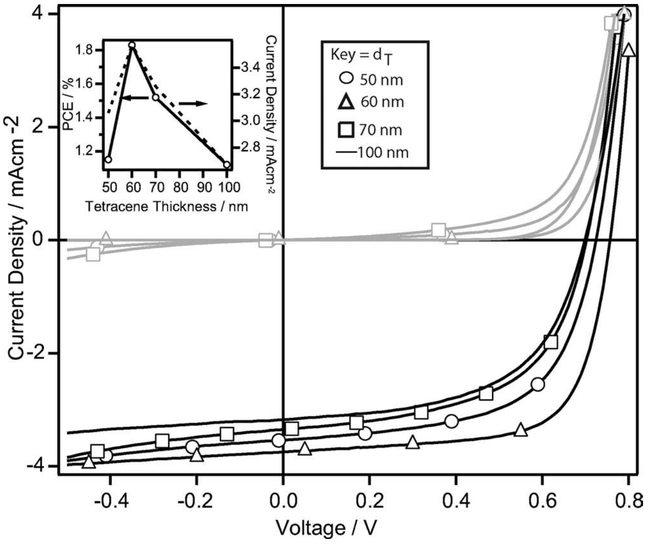

Figure 6. *J–V* data under 1 sun illumination for planar Tc/C$_{60}$ heterojunction OPV devices with the illuminated currents (black) and dark currents (grey). Inset: Shows the change in power conversion efficiency (η_p) and current density with respect to Tc thickness, d_T.

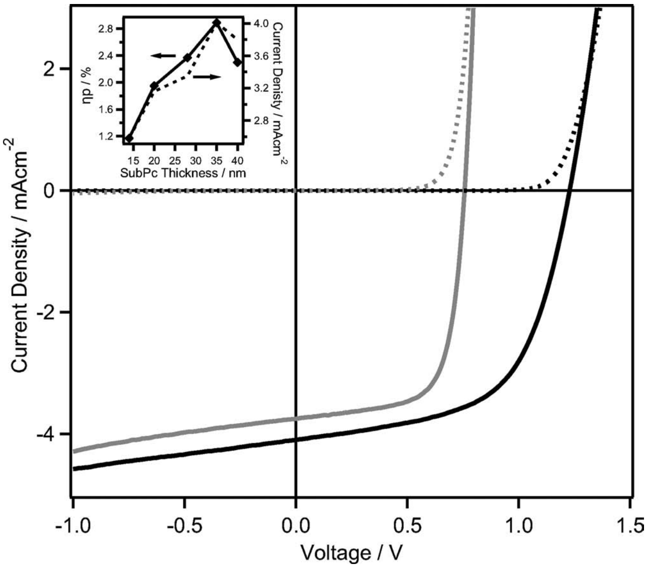

Figure 7. *J–V* data for planar Tc/SubPc heterojunction device (H) (black) with the illuminated currents (solid) and dark currents (dotted) with results from a Tc/C$_{60}$ device (B) included as a reference (grey). Inset: Change in power conversion efficiency and current density with respect to SubPc thickness, d_S.

Table 2. Device parameters obtained from OPV devices. Devices E–I have the structure: ITO/MoO$_x$ (5nm)/tetracene (60 nm)/SubPc (d_S nm) BCP (8 nm)/Al. Tc/C$_{60}$ device B is also included for direct comparison.

d_S [nm]	J_{sc} [mA cm^{-2}]	V_{oc} [V]	FF	η_p [%]
E = 14	2.58	1.06	0.43	1.17
F = 20	3.16	1.19	0.52	1.95
G = 28	3.35	1.23	0.58	2.37
H= 35	4.01	1.24	0.58	2.89
I = 40	3.80	1.24	0.49	2.30
B = 40 (C$_{60}$)	3.57	0.76	0.68	1.83

V_{oc} over the range of thicknesses studied with a maximum of 1.24 V compared to the 0.76 V achieved using C$_{60}$. This is due to the increase in the I_g. The reduction in FF in the SubPc cells is thought be due to the amorphous character of the film.[6] When using C$_{60}$ the close packed hexagonal/cubic crystal phases improve the π–π stacking resulting in a higher electron mobility and allow for higher FFs.[6] Although the FF has decreased it is still comparable to other small molecule OPVs. Below and above the optimal thickness there is a decrease in J_{sc} for reasons similar to the Tc/C$_{60}$ system. Interestingly, the optimum thickness, d_s, of 35 nm is much thicker than when using SubPc as a conventional donor material, where the best η_p is typically achieved using films as thin as 14 nm due to the short exciton diffusion length.[7,25] The acceptor/Al interface largely determines the distribution of the optical field within the device and previously, Halls *et al*. demonstrated that an acceptor layer, in their case C$_{60}$, could also be used as a spacer layer to enhance the optical electric field within the device.[26] Through varying the thickness of the acceptor layer the optical electric field at the heterojunction increases, allowing for greater photovoltaic efficiency due to a larger percentage of excitons created close to the interface where they can be easily dissociated.[27,28] This has also been seen by others for various other spacing layers. For example, Kim *et al*. reported both an increase in photocurrent and V_{oc} in their polymer devices when incorporating a TiO$_2$ layer in between the active polymer and the Al cathode.[29]

The external quantum efficiency (EQE) of the Tc/C$_{60}$ and Tc/SubPc OPV cells are shown in **Figure 8**. Although there is a distinct overlap in absorbance, the Tc and C$_{60}$ both show a contribution to the photocurrent as can be seen by the distinct Tc peaks between ~450 and 500 nm and the overall broadness consistent with a C$_{60}$ contribution. Previous studies have indicated that a minimum separation energy at the heterojunction for efficient dissociation is 0.3–0.4 eV[21,30] whereas in the Tc/SubPc case the HOMO$_D$–HOMO$_A$ separation is only 0.2 eV and therefore a marginal contribution to the photocurrent would be expected from the SubPc. Although only a small contribution is expected, it does in fact contribute quite significantly compared to the Tc layer as can be seen by the strong peak at ~590 nm. This indicates that although the HOMO$_D$–HOMO$_A$ gap is small, there is still enough of an energy offset to efficiently separate the exciton and allow sufficient contribution from this layer.

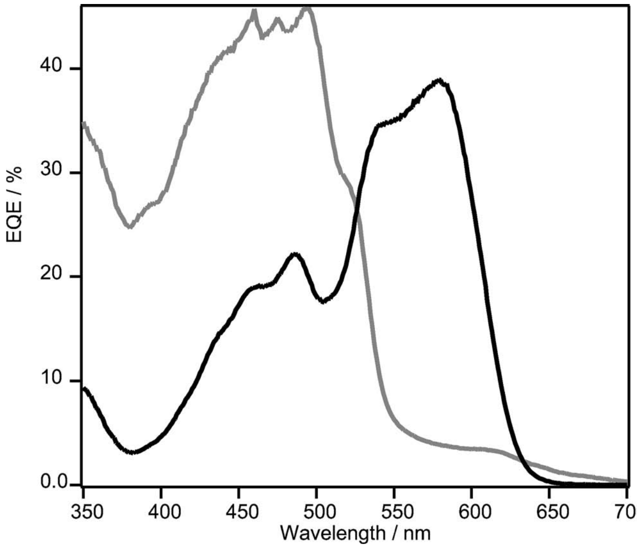

Figure 8. EQE of Tc/C$_{60}$ (grey) and Tc/SubPc (black) devices.

3. Conclusions

We have demonstrated the use of SubPc as an efficient electron accepting material when used in single junction OPV cells in conjunction with tetracene as the donor material. By investigating the replacement of C$_{60}$ with SubPc, significant improvements in V_{oc} and η_p have been demonstrated due to the increase in interface gap energy, I_g. Although the diffusion length of SubPc is thought to be ≤15 nm, thicker layers can be utilised due to an optical spacing effect, which allows for a large contribution to the photocurrent from the SubPc acceptor. Although organic semiconductors show a preference for having donor or acceptor character, we have shown that provided the offsets at the heterojunction are sufficient to dissociate charges efficiently, a typical donor material can be utilised as an acceptor. This leads the way for interesting donor–acceptor material combinations allowing for better overlap of the solar spectrum and improved device performance.

4. Experimental Section

OPV devices were fabricated on commercially available indium tin oxide (ITO) - coated glass substrates (Thin Film Devices, 145 nm thick, $R_s < 15\ \Omega$ square^{-1}) after cleaning by sonication in acetone, detergent, water and isopropanol, and an ultraviolet/ozone treatment decontamination system to remove carbon residues (Novascan PSD-UVT). The OPVs were fabricated using a Kurt. J. Lesker Spectros vacuum evaporation system. The organic materials, Tc (Acros, 98%), SubPc (Aldrich, 85%), C$_{60}$ (Nano-C Inc., 99.5%), were purified using thermal gradient sublimation before deposition and bathocuproine (BCP, Aldrich, 96%) and molybdenum oxide (MoO$_x$, Aldrich) were used as received. The aluminium electrodes were deposited in situ by evaporation through a shadow mask to a thickness of 100 nm to give an active pixel area of 0.16 cm^2. Atomic force microscopy (AFM) images were obtained using an Asylum research MFP-3D in AC mode. Current density–voltage (*J–V*) characteristics were recorded using a Keithley 2400 sourcemeter with simulated AM 1.5 G solar illumination at 100 mW cm^{-2} (1 sun) from a Newport Oriel solar simulator. External quantum efficiency (EQE) measurements were obtained using a Sciencetech SF150 xenon arc lamp and a PTI Monochromator. The monochromatic light intensity was calibrated with a Si photodiode (Newport 818-UV) and chopped at 500 Hz. Signal detection was performed with a current–voltage amplifier (Femto DHPCA-100) and lock-in amplifier (Stanford Research SR 830 DSP).

XPS measurements were carried out at the soft X–ray undulator beamline X1B at the National Synchrotron Light Source (NSLS), Brookhaven National Laboratory. Thin films of Tc/C_{60} were grown in situ in an ultrahigh vacuum (UHV) organic molecular beam deposition (OMBD) custom chamber, attached to a multi-technique soft X–ray spectroscopy system. Clean ITO surfaces were obtained via Ar^+ ion sputtering and annealing in UHV, and the film deposition rate was monitored using a quartz crystal microbalance (QCM). The secondary electron cut–off and valence band spectra were recorded using an incident photon energy of 260 eV and the sample work function was measured with an applied bias voltage of 9 V. Spectra obtained are referenced to the Fermi level (E_f) of atomically clean gold foil in contact with the sample.

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