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The Relationship between the Electric Field-Induced Dissociation of Charge Transfer Excitons and the Photocurrent in Small Molecular/Polymeric Solar Cells

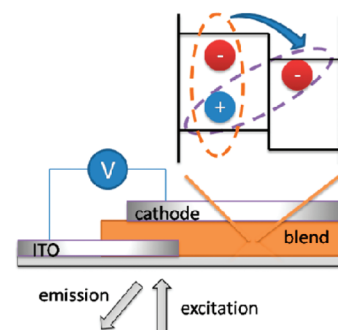
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ABSTRACT A bulk heterojunction system consisting of a soluble poly(*p*-phenylenevinylene) donor and a small molecular electron acceptor based on Vinazene (2-vinyl-4,5-dicyanoimidazole) has been investigated with respect to the correlation between field-induced emission quenching and free charge carrier formation. Transient and steady-state emission properties measured at different voltages indicate that emissive charge transfer (CT) excitons formed at the heterojunction are prone to direct field-induced dissociation. However, the split-up of these CT excitons cannot fully account for the photocurrents observed on the very same devices. It is proposed that the formation of free carriers involves a nonemissive precursor and that the recombination of this precursor does not proceed via the formation of CT excitons.

SECTION Macromolecules, Soft Matter



Organic solar cells comprising a heterojunction between hole and electron transporting materials exhibit far better performances than single component devices.^{1,2} Provided that charge transport to and extraction at the electrodes is optimized, the device performance is entirely determined by processes occurring at the donor/acceptor (D/A) heterojunction. As a result, the excited state and charge carrier dynamics at the heterojunction have been the subject of multiple studies.^{3–6} It is believed that the dissociation of photogenerated excitons at the D/A heterojunction yields Coulombically bound polaron pairs (also denoted as geminate pairs, GPs), which may either separate into free charge carriers or recombine.⁷ Various organic D/A systems exhibit an emission feature at low energies of the solar spectrum, which is attributed to the radiative decay of such interfacial polaron pairs (also called charge transfer (CT) excitons or exciplexes).^{5–5,7–14} An intense debate has developed on the importance of such radiative interfacial recombination processes with respect to the efficiency of free charge carrier formation. Steady-state and time-resolved photoluminescence (PL) studies on a blend of two polyfluorene copolymers suggested that radiative CT excitons are formed via the recombination of more weakly bound and nonemissive GPs.⁷ The latter was identified as the main precursor state to free carriers. As a consequence, application of an electric field reduced the emission from the CT exciton (via field-assisted dissociation of dark GPs), but had no effect on its emission lifetime. It was further confirmed that intrachain excitons on the acceptor polymer can be regenerated via thermal excitation of such interfacial CT states.⁸ Experiments on poly(*p*-phenylenevinylene) (PPV)-based blend systems led to a

similar picture, although with some important variations in the detail.^{5,9,15} In particular, application of an electric field led to a reduction of the CT-emission lifetime, suggesting that such states are subject to field-assisted dissociation.³ A more recent study by Veldman et al. on the steady-state and time-resolved emission properties of a mixture of a polyfluorene copolymer with a soluble fullerene derivative revealed a strong decrease of the CT-emission lifetime with external bias, implying that the electric field directly dissociates the emissive interfacial excitation.⁵ By comparing the efficiency of field-dependent emission quenching with the normalized photocurrent, these authors proposed that CT excitons may be the precursor states to all free charge carriers extracted from the device. However, to our knowledge, none of the previous publications documented a quantitative analysis of the relationship between the field-induced quenching of steady-state and/or transient PL and the quantum efficiency of free carrier generation.

In this letter, we present a comprehensive study of the optoelectronic properties of a blend comprising the donor polymer poly[2,5-dimethoxy-1,4-phenylene-1,2-ethynylene-2-methoxy-5-(2-ethylhexyloxy)-(1,4-phenylenevinylene-1,2-ethynylene)] (M3EH-PPV)¹⁶ and the small molecule acceptor 4,7-bis[2-(1-hexyl-4,5-dicyanoimidazol-2-yl)vinyl]benzo[c][1,2,5]-thiadiazole (HV-BT).¹⁷ An initial study on the optical and photovoltaic properties of this blend revealed a long wavelength emission attributed to the radiative decay of CT

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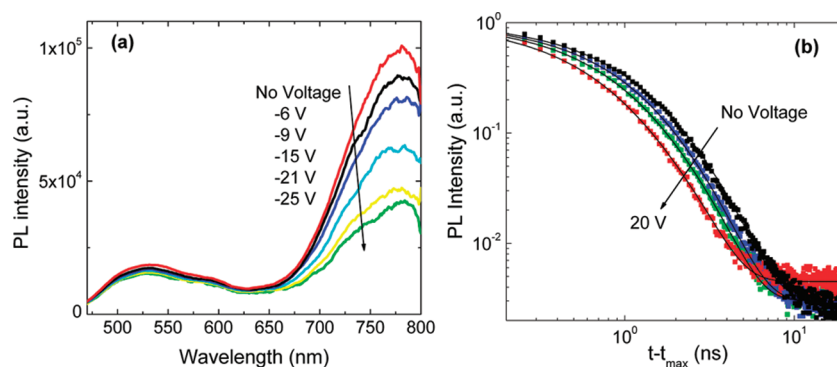


Figure 1. (a) Electric field-induced quenching of PL from a 180 nm thick 1:1 M3EH-PPV:HV-BT layer excited at 450 nm through the transparent ITO. Voltage is applied in the reverse direction and increased until -25 V. “No voltage” is equivalent to open-circuit conditions. (b) Normalized time-resolved PL transients detected at 800 nm. Solid lines show multiexponential fits.

excitons.¹³ At the same time, the emission from individual components is strongly quenched, by more than a factor of 10, in the 1:1 (weight ratio) blend film. This suggests a high degree of intermixing between the two components and a small size of the donor- and acceptor-rich domains. In addition, the low fill factor of solar cells fabricated from such blends was attributed to a strong field-dependence of free carrier formation from bound interfacial GPs. By performing photocurrent studies with various illumination intensities and bias, the conditions for which the device performance is determined solely by monomolecular recombination of such bound pairs could be clearly identified.¹⁸ Therefore, this system appears to be highly suitable for a conclusive investigation of the CT-exciton emission and dynamics in relation to free carrier formation.

Figure 1a shows absolute PL spectra of a blend layer sandwiched between an indium tin oxide (ITO)/poly(3,4-ethylenedioxythiophene) poly(styrenesulfonate) (PEDOT: PSS) anode and a Ca/Al cathode, measured at different bias. As discussed earlier, the spectrum measured at open-circuit conditions displays a broad long wavelength emission centered at 780 nm, which has been attributed to the radiative decay of CT-excitons (see Figures S1 and S2 of the Supporting Information).

Increasing the bias reduces the intensity of the long wavelength emission, which is indicative of field-assisted dissociation processes. The quenching at -25 V, relative to the emission measured without external voltage applied (open-circuit condition), amounts to 50 % at the maximum of the CT-exciton emission. Interestingly, the field has no effect on the emission at shorter wavelengths (being attributed to the radiative decay of excited states on either the donor or the acceptor molecules). Furthermore, the results of time-resolved PL studies did not reveal any effect of the field on the emission lifetime at ca. 550 nm (see Figure S3 of the Supporting Information). Evidently, exciton regeneration via thermal excitation of interfacial states as discussed above for blends based on polyfluorene copolymers is insignificant in our M3EH-PPV:HV-BT D/A blends. As a consequence, exciton recycling does not compete with other decay channels of the CT-exciton, which would otherwise have to be taken into consideration in the quantitative analysis of the results.

Transient PL traces recorded at 800 nm at varying bias are shown in Figure 1b. These normalized traces could be satisfactorily fitted to a multiexponential decay with three components:

$$I_{\text{PL}}(t) = a_1 e^{-t/\tau_1} + a_2 e^{-t/\tau_2} + a_3 e^{-t/\tau_3}, \quad a_1 + a_2 + a_3 = 1 \quad (1)$$

(see Figure S4 for two exemplary fits and Table S1 for the extracted values of decay times). In addition to a short decay component with a lifetime τ_1 of ca. 80–100 ps, the fits yield decay times between several hundreds of picoseconds for τ_2 and a few nanoseconds for τ_3 . Note that the representation of PL traces in a log–log plot does not show discrete decay steps, as has been previously observed for blends of M3EH-PPV with the high molecular weight electron acceptor poly[oxa-1,4-phenylene-1,2-(1-cyano)-ethylene-2,5-dioctyloxy-1,4-phenylene-1,2-(2-cyano)-ethylene-1,4-phenylene] (CN-ether-PPV).¹¹ Evidently, the intermediate and longer decay times, τ_2 and τ_3 , as deduced from the multiexponential fit are not related to distinct physical processes, but are representative for a rather broad distribution of decay times.

Decay times deduced from the triexponential least-squares fits are plotted as a function of the electric field in Figure 2a. Clearly, the application of a field shortens the PL lifetimes, indicative of field-assisted dissociation of the interfacial CT excitons. To determine how the emission intensity from each decay component is affected by the electric field, the emission intensity relative to the total intensity at zero field, A_i , was calculated according to $A_i(E) = R(E) \cdot f_i(E)$ with $R(E) = I(E)/I(0)$ being the relative emission intensity at field E taken from the steady-state PL data at 800 nm and

$$f_i(E) = \frac{a_i(E)\tau_i(E)}{a_1(E)\tau_1(E) + a_2(E)\tau_2(E) + a_3(E)\tau_3(E)} \quad (2)$$

being the relative intensity contribution of the i th decay component in the transient PL data at an electric field E . It can be seen in Figure 2b that A_1 is rather small, less than 10 %, and only weakly dependent on electric field. Evidentially, the fast decay component originates from a strongly bound or highly localized state, possibly intrachain excitations or

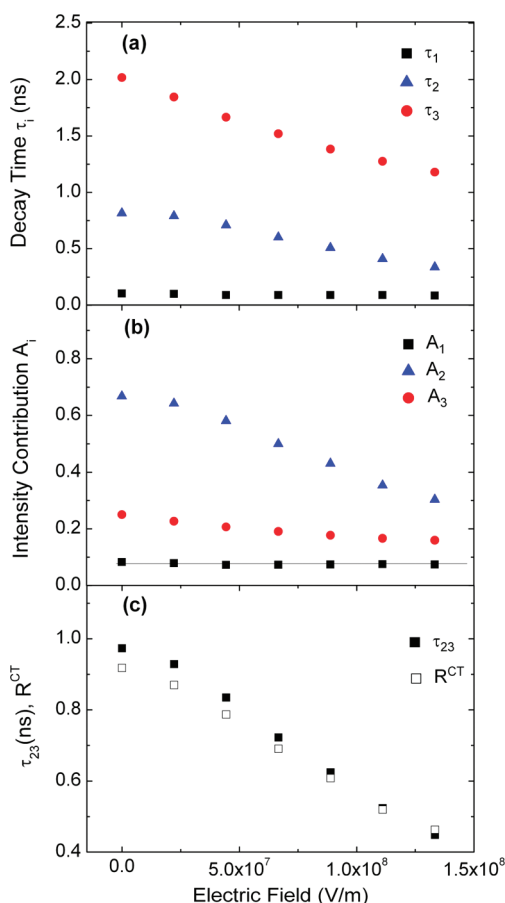


Figure 2. Result of the analysis of the PL decay traces in Figure 1b with a triexponential decay fit (see text for details).

excimers. In contrast, A_2 and A_3 decrease continuously with field in a quite similar fashion, in accordance to the observed shortening of the corresponding decay times. It is, therefore, meaningful to define an effective emission ratio (R^{CT}) taking into account only the two longer decay components:

$$R^{\text{CT}}(E) = \frac{A_2(E) + A_3(E)}{A_2(0) + A_3(0)} \quad (3)$$

Furthermore, as τ_2 and τ_3 appear to be representative of a broader distribution of decay times, it is plausible to consider only the average decay time as defined by

$$\tau_{23}(E) = \frac{a_2(E)\tau_2(E) + a_3(E)\tau_3(E)}{a_2(E) + a_3(E)} \quad (4)$$

As shown in Figure 2c, R^{CT} and τ_{23} decay likewise with the applied field. Consequently, the decrease in PL intensity must be caused by the direct field-induced dissociation of the CT excitons rather than that of a dark precursor state feeding the CT excitation. Under these conditions, the temporal evolution of the density of the CT excitons, η^{CT} , can be written as

$$\frac{d\eta^{\text{CT}}}{dt} = G - (k_r + k_{\text{nr}} + k_{\text{diss}}(E))\eta^{\text{CT}} \quad (5)$$

with G being the field-independent rate for CT exciton formation, k_r and k_{nr} being the field-independent coefficients for

radiative and nonradiative decays, respectively, and k_{diss} being the rate for field-assisted CT exciton dissociation. Then, the effective efficiency for CT-emission quenching (Φ^{CT}) defined by $\Phi^{\text{CT}} = 1 - R^{\text{CT}}$, can be expressed by the efficiency of charge carrier generation, η^{CT} , via dissociation of the CT excitons:¹⁹

$$\Phi^{\text{CT}}(E) = \frac{(\eta^{\text{CT}}(E) - \eta^{\text{CT}}(0))}{(1 - \eta^{\text{CT}}(0))} \xrightarrow{\eta^{\text{CT}}(0) \ll 1} \Phi^{\text{CT}}(E) \approx \eta^{\text{CT}}(E) \quad (6)$$

Equation 6 is correct for any combination of k_r and k_{nr} . This implies that the efficiency of carrier generation via field-assisted exciton dissociation can well exceed the quantum efficiency for radiative recombination. Note that there is evidence from experimental and theoretical studies that $\eta^{\text{CT}}(0) \ll 1$ may not be valid for high mobility materials such as phenyl-C61-butyric acid methyl ester (PCBM).⁵ However, since the photocurrent efficiency in our blend device is only 5% at short-circuit conditions, one expects an even smaller dissociation efficiency at open-circuit conditions. Consistent with that, calculations with eq 6 and $\eta^{\text{CT}}(0) > 5\%$ gave unrealistic field-dependencies of η^{CT} (see Figure S5 of the Supporting Information). On the basis of these considerations, the maximum internal quantum efficiency (IQE) for the photogeneration of charge carriers via the field-induced dissociation of CT excitons can be well approximated by

$$\text{IQE}^{\text{CT}}(E) = \eta^{\text{CT}}(E) \approx \Phi^{\text{CT}}(E) \quad (7)$$

presuming that all of the initially excited states form CT excitons.

In order to determine the efficiency of charge carrier generation as a function of bias, photocurrent experiments were performed on the same devices as used for the steady-state and time-resolved PL measurements. For the blend devices studied in this work, the photocurrent increases linearly with illumination strength up to an irradiance of ca. 10 mW/cm² (see Figure S6 of the Supporting Information). The deviation from the linear behavior at higher intensities has been attributed to the onset of space charge effects and bimolecular recombination processes, meaning that illumination under AM1.5 conditions with irradiance of 100 mW/cm² will cause a significant electric field redistribution inside our devices.¹⁸ Therefore, all experiments described in the following have been performed with an irradiance below 10 mW/cm².

Current–voltage (I – V) characteristics recorded in the dark and at an irradiance of 2.5 mW/cm² are shown in Figure 3a. Obviously, the dark current remains rather low up to a voltage of ca. –6 V. At low voltages, the photocurrent increases nearly linearly with bias, consistent with the results of earlier measurements on thinner devices.¹³ The external quantum efficiency (EQE) measured as a function of wavelength at different bias is shown in Figure 3b. At zero bias (short-circuit conditions), the EQE of ca. 5% coincides with previous reports on the same blend system.¹³ Raising the external bias to –9 V increases the EQE to nearly 50%. Note that the shape of the IPCE spectrum is essentially independent of the applied voltage, meaning that the increase in the photocurrent with bias under white light illumination resembles the voltage dependence of the EQE.

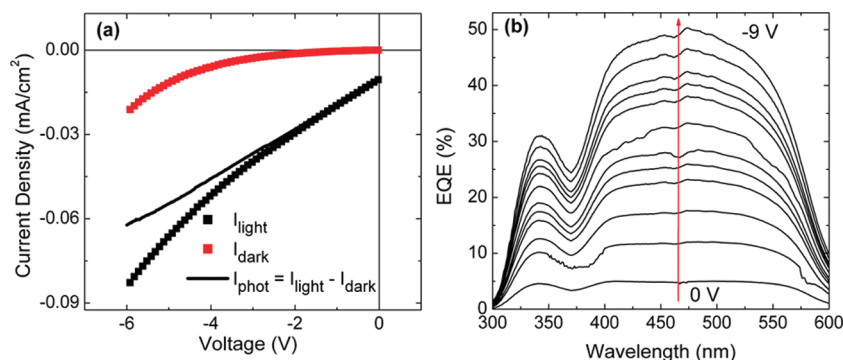


Figure 3. (a) I – V characteristics recorded for a M3EH-PPV:HV-BT blend solar cell under white light illumination of 2.5 mW/cm² intensity. (b) EQE spectra recorded at applied voltages varying from 0 to –9 V.

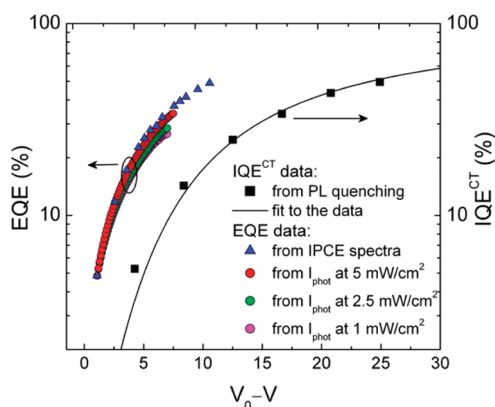


Figure 4. EQE versus internal bias compared to values of the maximum IQE^{CT} as calculated from the efficiency for field-induced quenching of the CT-exciton emission. The solid line through the IQE^{CT} data is the fit of the Onsager–Braun model to the free carrier generation efficiency of the dissociated CTexcitons (see text and Supporting Information for details). The internal bias in the device is $V_0 - V$, where $V_0 = 1.1$ V is the voltage where the photocurrent is equal to zero.

Figure 4 summarizes values of EQE taken from the maximum of the corresponding spectra in Figure 3b and the values calculated from photocurrent measurements for three different intensities (assuming that the photocurrent at short-circuit conditions corresponds to an EQE of ca. 5%). Within the accuracy of the experiment, all these measurements yield an identical dependence of the efficiency for free charge carrier generation on internal bias. Therefore, illuminating the sample during the photocurrent experiments should have no effect on the field-distribution inside the device. For all voltages considered here, the EQE for photo-generation of charges is significantly larger than the IQE^{CT} calculated with eq 7 from the field-induced quenching of the CT excitons. This discrepancy suggests that the dissociation of radiative CT excitons does not constitute the main channel for free carrier formation in the blend considered here.

The Onsager–Braun model is commonly used to describe the electric field-dependent dissociation of electron–hole pairs in organic media.²⁰ When applying this model to our data, the mobility of the fastest charge carrier and the rate of

CT exciton recombination was taken from the experiment, leaving the initial electron–hole pair distance as the only adjustable parameter (see Supporting Information for details and the parameters used in the model). As shown in Figure 4, a very good description of the field-dependent quenching of the CT-exciton emission is obtained for a CT-exciton radius of 0.7 nm. This value is well comparable with typical intermolecular distances. On the contrary, the model was not applicable to describe the photocurrent data when only the initial electron–hole distance is varied. In accordance with earlier publications, a carrier lifetime in excess of 20 ns, which is much longer than the lifetime of the transient CT-emission or, alternatively, a much higher carrier mobility, needs to be used to obtain a proper fit.^{5,21,22}

In conclusion, the results from our study of field-induced emission quenching and of the field-dependence of photo-generation are not consistent with existing models describing the interrelation between interfacial excitations and free carriers for the M3EH-PPV:HV-VT blend.^{5,7} While steady-state and transient PL data obtained at different bias unambiguously prove that interfacial CT excitons are prone to direct field-induced dissociation, the split-up of these states cannot account for the observed photocurrents of the very same devices. These results provide convincing evidence that the formation of the majority of free carriers in M3EH-PPV:HV-BT blend devices involves a nonemissive precursor and that the recombination of this precursor into a more tightly bound electron–hole pair is not emissive, i.e., it does not proceed via the formation of CT excitons.

SUPPORTING INFORMATION AVAILABLE Sample preparation, experimental methods, steady-state and time-resolved PL spectra of the neat and the blend films, decay transients of the blend film detected at 800 nm, the field-dependence of IQE^{CT} in the case of a nonzero CT exciton dissociation efficiency, the dependence of photocurrent on light intensity, and the parameters to fit IQE^{CT} data using the Onsager–Braun model. This material is available free of charge via the Internet at <http://pubs.acs.org>.

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