

# Device spectroscopy of magnetic field effects in a polyfluorene organic light-emitting diode

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We perform charge-induced absorption and electroluminescence spectroscopy in a polyfluorene organic magnetoresistive device. Our experiments allow us to measure the singlet exciton, triplet exciton, and polaron densities in a live device under an applied magnetic field and to test the predictions of three different models that were proposed to explain organic magnetoresistance. These models are based on different spin-dependent interactions, namely, exciton formation, triplet-exciton polaron quenching, and bipolaron formation. We show that the singlet exciton, triplet exciton, and polaron densities and conductivity all increase with increasing magnetic field. Our data appear to be inconsistent with the exciton formation and triplet-exciton polaron quenching models.

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## I. INTRODUCTION

Recent years have seen a surge in interest in spin transport in organic semiconductor devices,<sup>1</sup> including the study of the organic magnetoresistive effect. Organic magnetoresistance (OMAR) is a recently discovered large, low-field, room-temperature magnetoresistive effect in *nonmagnetic* organic light-emitting diodes (OLEDs).<sup>2-4</sup> The effect can be as large as 10% relative change in resistance for a magnetic field  $B$  of 10 mT. To the best of our knowledge, the mechanism causing OMAR is not yet established with certainty. Very recently, a magnetoresistive effect in films of  $n$ -doped CdSe quantum dots,<sup>5</sup> whose line shape is identical to that of OMAR, has been observed, demonstrating that the physics behind OMAR is of importance in a wide class of hopping systems. Independently, the effect of small magnetic fields on spin dynamics in electron-hole pairs has also been studied for several decades in organics (for a review, see Ref. 6). The concepts that arose from these works in radical pairs have recently been applied to OLEDs. Magnetic field effects (MFEs) on photocurrent,<sup>7,8</sup> electroluminescence (EL),<sup>8-11</sup> and exciton dissociation at electrodes<sup>9</sup> were found experimentally and modeled based on spin dependence<sup>12</sup> between the singlet and triplet-radical pair channels of recombination. In addition, the interaction between triplet excitons and radicals was studied in anthracene crystals by Ern and Merrifield.<sup>13</sup> The question how relevant the concepts of radical pair and triplet-radical spin dynamics are to spin transport in organics naturally arises. We will study this question in the context of OMAR.

Three kinds of models have been put forward to explain OMAR. (i) The electron-hole pair (EHP) mechanism model based on concepts borrowed from the before mentioned MFE in radical pairs<sup>4,7-11</sup> (in this model, the spin-dependent reaction  $P^+ + P^- \rightarrow$  exciton between oppositely charged polarons to form an exciton (“recombination”) is of central importance); (ii) the triplet-exciton polaron quenching (TPQ) model<sup>14</sup> that is based on the spin-dependent reaction  $TE + P \rightarrow P + GS^*$  between a triplet exciton and a polaron to give an excited singlet ground state ( $GS^*$ ) (in principle, the excess energy could also be carried off by the polaron); and (iii) the

bipolaron (BP)<sup>15</sup> mechanism which treats the spin-dependent formation of doubly occupied sites (bipolarons)  $P^+ + P^+ \rightarrow BP^{2+}$  (and an analogous reaction for negative carriers) during the hopping transport through the organic film (we note that the BP model does not assume the formation of stable bipolarons but is merely based on the occurrence of doubly occupied hopping sites whose energy may be higher than that of two singly occupied sites). All three models are based on spin dynamics induced by the hyperfine interaction.<sup>16</sup> The involvement of hyperfine coupling is suggested by the characteristic magnetic field of  $\approx 5$  mT (Ref. 17) and confirmed by the observation that no OMAR effect is observed in  $C_{60}$  devices unless a polymeric hydrogen-containing electrode is used or a substituted  $C_{60}$  molecule with a hydrogen-containing sidegroup is used.<sup>18,19</sup> Models similar to BP, described in Refs. 5 and 20, were used to explain the magnetoresistive effect in CdSe quantum dot films and the relatively small positive and negative magnetoresistances<sup>21,22</sup> in amorphous inorganic semiconductors, respectively. Before further progress in the understanding of magnetotransport in organic semiconductors can be made, experiments must be completed to distinguish between these three directions. First, we note that the EHP and TPQ models require exciton formation (and therefore the presence of both majority and minority carriers), whereas BP can exist also in unipolar devices. Sheng *et al.*<sup>17</sup> found a significant dependence of the magnitude of OMAR on the minority carrier density, but this dependence is much weaker than the linear dependence that would be expected from EHP. Desai *et al.*<sup>14</sup> found that the onset of OMAR coincides with that of minority carrier injection. On the other hand, Nguyen *et al.*<sup>18</sup> showed that a small OMAR also exists in doped polythiophene derivatives and Guyot-Sionnest *et al.* observed<sup>5</sup> an effect phenomenologically very similar to OMAR in  $n$ -doped films of CdSe quantum dots, both of which are expected to be unipolar devices. We therefore conclude that the experiments so far did not provide a definite answer. Here, we therefore report on a different test that aims directly at observing the spin-dependent reactions.

In the present work, we put these models to a stringent test by measuring the dependence of the densities of the singlet, triplet excitons, and polarons on the applied magnetic

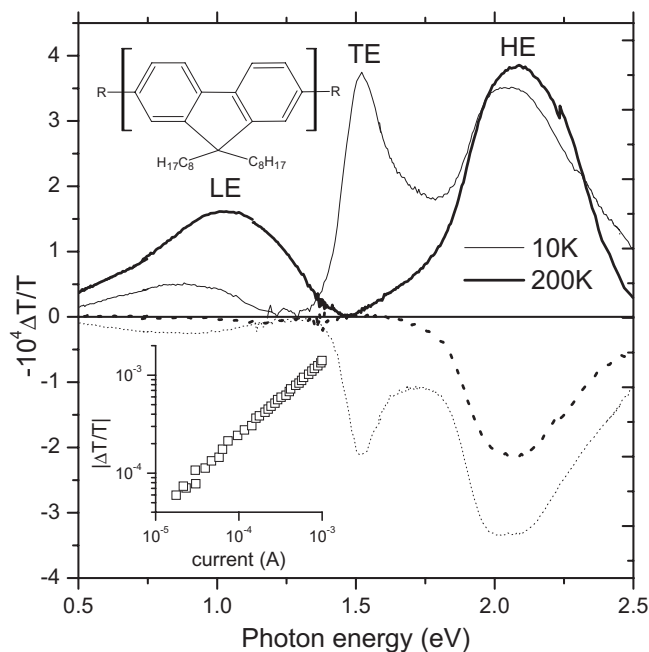


FIG. 1. The in-phase (solid) and quadrature (dotted) CIA spectra of a PFO device (100 nm thick) at 10 K (thin lines) and 200 K (bold lines) modulated at 1000 Hz. The TE and HE and LE peaks are assigned. The insets show the dependence of the magnitude of the TE band on device current and the chemical structure of PFO.

field. As we will show in Sec. IV, the three models make qualitatively different predictions for the MFE on these densities.

## II. EXPERIMENT

To measure the triplet and polaron densities, we use the charge-induced absorption (CIA) spectroscopy technique under an applied  $B$ . In this experiment, the changes in the device transmission spectrum resulting from induced absorption of the injected carriers and their recombination by-products are detected. The singlet excitons, which are too short lived for detection using CIA, are detected using EL spectroscopy. For these measurements we used a standard OLED architecture, as described in more detail previously:<sup>23</sup> A poly(3,4-ethylenedioxythiophene)-poly(styrenesulfonate) layer was spin coated onto an indium tin oxide covered glass slide as the hole-injecting electrode. The semiconducting polymer film, poly(9,9-dioctylfluorenyl-2,7-diyl) (PFO) (see Fig. 1 inset), purchased from American Dye Source, was spin coated onto the substrate from a chloroform solution with a concentration of 10 mg/ml to yield a thickness of  $\approx 100$  nm. Finally, the Ca cathode (with an Al capping layer) was deposited to complete the sandwich structure. The sensitive manufacturing steps were carried out in a nitrogen glove box. A device area of  $\approx 12$  mm<sup>2</sup> was chosen to match the size of the tungsten lamp filament used in CIA spectroscopy. For the CIA measurement, an applied ac voltage, modulated at about 1000 Hz between 0 V and a certain voltage level, was used resulting in a current of  $\approx 1$  mA. A monochromatized tungsten-halogen lamp (250 W) was used

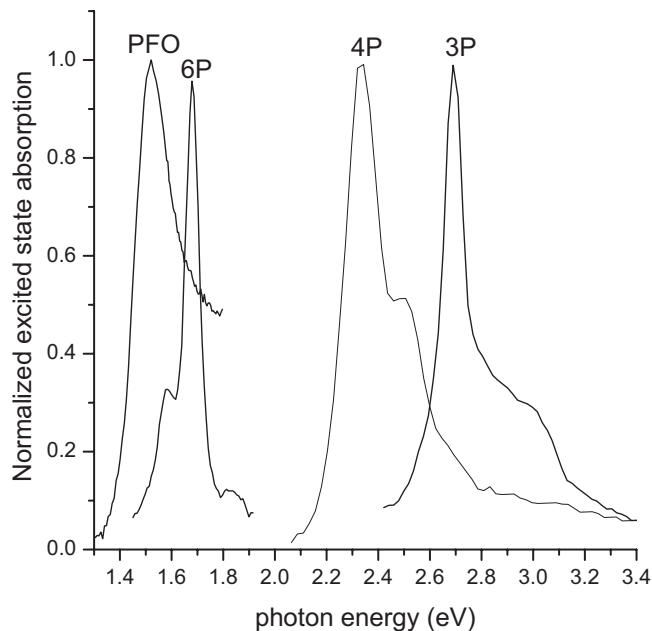


FIG. 2. A collection of excited state absorption spectra of oligophenyls (length assigned) and PFO. The absorption bands of the oligophenyls were assigned to triplet-triplet absorption. The data for the oligophenyls were reproduced from Ref. 24.

as the probe beam. The CIA spectra were obtained by plotting the negative fractional change in transmission,  $-\Delta T/T$ , versus the probe photon energy.  $-\Delta T/T$  is proportional to the induced change in absorption coefficient. The MFE on CIA,  $\delta T/\Delta T \equiv (\Delta T(B) - \Delta T(0))/\Delta T(0)$ , and that on EL,  $\delta(EL)/EL \equiv (EL(B) - EL(0))/EL(0)$ , were measured under identical conditions together with the magnetoconductance.

## III. EXPERIMENTAL RESULTS

Figure 1 shows the in-phase (solid) and quadrature (dotted) CIA spectra of a PFO device (100 nm thick) at 10 and 200 K. Three absorption bands were observed, a narrow band at 1.52 eV [triplet exciton (TE)] and two broader bands [a high-energy (HE) band at 2.1 eV and a low-energy (LE) band that shifts its peak position with temperature]. The TE band is believed to result from triplet exciton absorption.<sup>25</sup> For the interested reader, we now briefly summarize several prior experiments that led to this assignment. A large number of continuous wave photoinduced absorption experiments showed that these spectra in phenylene- and phenylene-vinylene-based polymers are dominated by a band in the spectral region between 1 and 1.5 eV (see, e.g., Refs. 26 and 27). In phenylene-based polymers (a class to which PFO belongs), this band was assigned to triplet absorption by studying a number of oligophenyls with increasing length,<sup>24</sup> where it was shown that the band assigned to triplets in terphenyl evolves into the band observed in poly(phenylene). In Fig. 2, we reproduced these data and show that the triplet band in PFO can indeed be understood from the conjugation-length dependence of the triplet band in oligophenyls. Furthermore, the triplet energies and triplet-triplet absorption en-

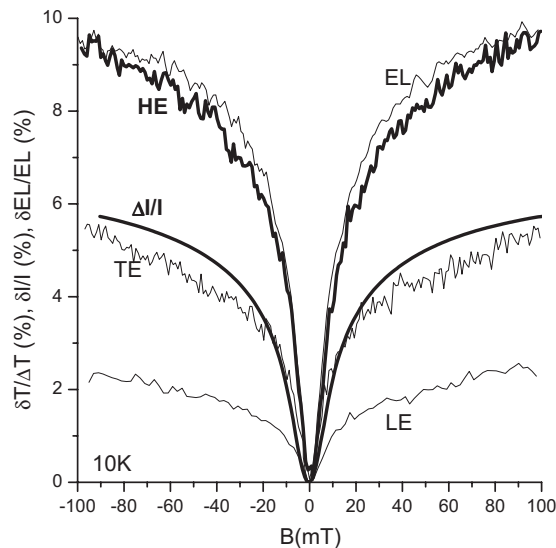


FIG. 3. The magnetic field effect on the various CIA bands ( $\delta T/\Delta T$ ), current ( $\delta I/I$ ), and EL ( $\delta(\text{EL})/\text{EL}$ ) at 10 K.

ergies in many materials have also been studied using pulse radiolysis.<sup>28</sup> In pulse radiolysis, the polymer chain is excited by energy transfer from the solvent, which is excited by the electron beam, via a suitable acceptor intermediate. The dynamics of this excitation scheme is such that only triplets are efficiently generated on the polymer, in distinction from optical excitation where singlets are the dominant initial excitation. Their result for PFO is in good agreement with our assignment. The same group also published a triplet transient absorption spectrum in excellent agreement with our TE band.<sup>29</sup> Recent theoretical results are also in agreement with our assignment.<sup>30</sup> In the case of poly(phenylene-vinylene) the spin associated with this band was directly measured by optically detected magnetic resonance spectroscopy.<sup>31</sup>

Dhoot and Greenham<sup>32</sup> previously studied CIA in PFO devices similar to ours and reported a very similar spectrum. They also assigned TE to triplet excitons. Furthermore, they “tentatively” assigned the HE band to polaron absorption. The assignment of the HE band to polarons has, to the best of our knowledge, not yet been established with certainty, but, since it is the dominant band in CIA where the primary effect is the injection of carriers, this assignment seems plausible. Furthermore, Dhoot and Greenham<sup>32</sup> assigned LE also to polarons, since the HE and LE bands “were found to have the same dependence on voltage, driving frequency, and temperature, indicating they belong to a single species.” However, we obtained different results. Figure 1 shows that these two bands have a different ratio between in-phase and quadrature signals (and therefore a different lifetime) and a different temperature dependence. These two bands therefore belong to two different species. The assignment of LE is therefore, to the best of our knowledge, not known at present.

Next, we turn to the main experiment in this paper, the study of the MFE on the various bands in the CIA spectrum together with that of the singlet EL and the current. Figure 3 shows  $\delta T/\Delta T$  measured at three fixed probe photon energies,

namely, 1.0, 1.5, and 2.1 eV. The magnetoconductivity,  $\delta I/I$ , and  $\delta(\text{EL})/\text{EL}$  are also shown. All five quantities increase with  $B$  (i.e., the fractional MFE is positive). First, we note that the measured spectroscopic MFE must be caused by a change in population rather than a change in the optical cross section. This is because all the transitions we studied are fully allowed, spin-conserving transitions which are therefore well described by the electric dipole approximation. Spin states and magnetic fields therefore do not factor into the cross sections. Furthermore, the Zeeman shift, which is on the order of  $1 \mu\text{eV}$ , is absolutely negligible compared to the observed linewidths. Our results therefore show that the singlet exciton density (measured using EL), the triplet exciton density, and the polaron density all simultaneously increase with  $B$  together with the device current. Since all these traces share the same dependence on  $B$  (within experimental accuracy), they are clearly caused by a single common MFE. In agreement with our earlier results,<sup>3</sup>  $\delta(\text{EL})/\text{EL}$  is somewhat larger than  $\delta I/I$ .  $\delta T/\Delta T$  of TE is similar in magnitude as  $\delta I/I$ , whereas  $\delta T/\Delta T$  of HE is similar in magnitude as  $\delta(\text{EL})/\text{EL}$ . LE is significantly less magnetic field dependent than the other bands. Finally, we note that the measurement times for taking such traces are rather long, at least several hours. Therefore, drift due to thermal effects and device degradation occur during the measurements, which can lead to a distortion in the measured line shape. We kept the effect of the drift to a minimum by measuring a large number of sweeps, with each sweep taking about 20 min. From the individual traces and a comparison to their average, we conclude that the estimated distortion at any point of the reported data is less than 1% absolute.

#### IV. DISCUSSION

Next, we separately discuss the experimental findings within the framework of the three models. The interpretation of the experimental data shown in Fig. 3 in an EHP-type model would occur along the following lines:<sup>17</sup> In an OLED device, electrons and holes are injected from the cathode and anode, respectively, into the organic layer. During the recombination of free carriers into singlet and triplet excitons, they form intermediate electron-hole pairs, either singlet pairs (SPs) or triplet pairs (TPs). SPs finally form singlet excitons, which emit fluorescent light, and TPs form triplet excitons, which are usually nonradiative. However, because of the negligible exchange interaction, the spin states of SP and TP are mixed by the hyperfine interaction. In particular, in the absence of  $B$ , the singlet mixes with the entire triplet manifold. However, an applied magnetic field lifts the triplet degeneracy, and for a field strong compared to the hyperfine strength, the mixing remains only between the singlet and the triplet state with magnetic quantum number zero. It has been shown by several authors<sup>4,7,9,17</sup> that the application of  $B$  therefore leads to enhanced singlet exciton formation together with reduced triplet exciton formation if the triplet exciton formation rate is larger than that of singlet excitons and vice versa. This is because the more slowly recombining pair, which therefore has a larger population, transfers its excess population into the faster recombining pair. In any

case, one type of exciton is formed at the expense of the other, in clear disagreement with our experimental data (Fig. 3). Reufer *et al.*<sup>33</sup> recently showed that in a (weakly) phosphorescent OLED, the magnetic field dependence on singlet emission and that on triplet emission, i.e., electrofluorescence and electrophosphorescence, are exactly equal to each other (within the measurement inaccuracy) and that both have a positive MFE, in agreement with our results. These authors also drew the conclusion that this observation suggests that the EHP model is not a feasible candidate for explaining OMAR. However, we believe that their experiment is not 100% conclusive, since in phosphorescent devices, it is not possible to distinguish between phosphorescence that occurs as a result of intersystem crossing from a singlet exciton or from a triplet exciton, directly formed from polaron pairs.

So far, we have discussed the effect of the EHP spin mixing on the singlet and triplet exciton populations, but the effect on the conductivity must still be addressed. The above described spin mixing also leads to an overall faster polaron pair recombination rate. This is a second order effect,<sup>34</sup> meaning that if the effect on the singlet exciton population is roughly 10%, as in our case, then the effect on the overall recombination rate is expected to be of order 1%, in any case much less than the effect on the singlet:triplet ratio.<sup>4</sup> Since  $B$  reduces this spin mixing, it leads to an overall decrease in polaron recombination and exciton formation. It is this second order MFE on the total recombination rate that Prigodin *et al.* used as the basis for their explanation of OMAR.<sup>35</sup> In this model, the reduction in polaron recombination leads to larger space-charge cancellation in the region of the device where the positive and negative carrier populations overlap. Since the space-charge potential codetermines the applied voltage, this reduction in space charge leads to a reduced operating voltage at a constant current or more current at a constant voltage.

In the TPQ model, the mobility of polarons is affected by spin-dependent scattering processes obeying the reaction equation  $TE+P \rightarrow P+GS^*$ . In addition to the triplet population, the mobility of polarons can be affected by this reaction, but the number of polarons is conserved. This is of course dictated by charge conservation: the number of polarons can only change as a result of either recombination with an oppositely charged polaron or because of bipolaron formation between two equally charged polarons. Therefore, our result that the polaron population changes with magnetic field is inconsistent with this model. There exist further contradictions as well. This model is based on a bimolecular reaction whose strength is proportional to the triplet exciton density times the polaron flux (current). As is shown in Fig. 1 (inset), the triplet exciton density in the devices increases with a fitted exponent of 0.77 with increasing current. OMAR due to the TPQ mechanism should therefore increase with increasing current, in contradiction with the experimental result that OMAR traces in many materials, e.g., in PFO (see Ref. 23, Fig. 1), actually decrease with increasing current. Furthermore, Fig. 1 shows that the triplet density is measurably large only at low temperature (see also Ref. 32, Fig. 2), in contradiction with the relative temperature insensitivity of OMAR.<sup>23</sup> This reduction of TE population with temperature is known to result from a decrease in intrinsic

TE lifetime with increasing temperature, from  $\approx 10$  ms at 10 K (Ref. 36) to  $\approx 25 \mu\text{s}$  at 300 K (Ref. 37) in a representative material.

In a BP model, the MFE does not act on excitons but affects the carrier mobility directly by shifting the equilibrium between the mobile polarons and less mobile bipolarons. This leads, in the present case, to an increase in current. In addition, this increase in mobility is also expected to result in a corresponding increase in the by-products of carrier recombination, such as singlet and triplet excitons. We have discussed this particular point in a previous publication.<sup>38</sup> In short, carrier recombination in OLEDs is believed to occur via the Langevin recombination mechanism for which the recombination rate is proportional to the mobility. Of course, this argument holds true more generally for all diffusion limited recombination processes. We note that the magnitude of the MFE on singlet and triplet excitons need not necessarily be equal to that of the magnetoconductance<sup>3</sup> because carrier recombination is bimolecular. Furthermore, if the magnetoconductance of minority and majority carriers can be different, then the MFE on excitons and current need not show a simple relation to each other at all. However, the experimental observation that the MFE on the singlet excitons is about twice as large as that for the triplets appears at first sight to be at odds with the BP model, since in this model, the excitons are assumed to form independently of their spin state. However, possible explanations for this discrepancy exist. It is straightforward to show (using a rate equation treatment) that this 1:2 ratio is expected for the scenario that singlet excitons recombine monomolecularly, whereas the triplet excitons recombine bimolecularly (the factor of 2 comes from the exponent of the bimolecular recombination term). This indeed appears to be the case. Whereas the continuous wave photoluminescence in organic films depends linearly on the laser power, the triplet photoinduced absorption signal is proportional to the square root of the laser power at near steady state densities and modulation frequencies.<sup>39,40</sup> Since the employed modulation frequency is similar to that used by us in CIA and because the triplet densities are similar (since the observed induced absorption magnitudes are similar), it appears reasonable that bimolecular recombination is also active in our CIA measurements. A bimolecular recombination of triplets appears to be at odds with the observed exponent of 0.77 between triplet population and current, where an exponent of 0.5 is expected. However, an exponent larger than 0.5 would be expected in the case of bimolecular formation of triplets from the charge carriers. Altogether, we believe that none of the presented measurements are in clear contradiction to the bipolaron model. However, our experiments cannot prove the validity of the bipolaron model. For such a proof, the bipolaron density would have to be measured, e.g., by detecting its induced absorption band (whose spectral position, shape, and intensity are, to the best of our knowledge, not known at present). Finally, its magnetic field dependence would have to be studied and compared to model predictions.

## V. CONCLUSION

We reported the charge-induced absorption spectrum of a polyfluorene OLED with the goal of elucidating the mecha-



nism behind the recently discovered organic magnetoresistive effect. The spectrum contained bands that were previously assigned to triplet excitons and polarons together with a low-energy band whose assignment is presently unknown. We measured the dependence of the densities of the singlet and triplet excitons, as well as polarons, on the applied magnetic field. Our results show that the singlet exciton, triplet exciton, and polaron densities simultaneously increase together with the device current upon application of a magnetic field. We discussed the experimental findings within the framework of all three models of the organic magnetoresis-

tive effect that we are aware of: (i) the electron-hole pair model, (ii) the triplet-exciton polaron quenching model, and (iii) the bipolaron mechanism. Our experimental findings appear to be in disagreement with mechanisms (i) and (ii).

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