Photoluminescence studies study of a phenyl-substituted PPV

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Abstract

We report on the photophysics of a phenyl-substituted PPV both in solution and as a film. For both systems we have studied the decay of the photoluminescence and of the emission anisotropy for a large set of wavelengths spanning the entire photoluminescence spectrum. At long wavelengths the decay behavior is that of an interchain species. At the shortest wavelengths the decay of the photoluminescence from the film is observed to have a long-lived component, in addition to the rapidly decaying component usually associated with energy transfer. We attribute this slow component to emission by isolated intrachain excitons with reduced nonradiative relaxation.

1 Introduction

The first operation of a light-emitting diode based on electroluminescence from poly(p-phenylene-vinylene), PPV [1], has been followed by a worldwide effort to discover new and efficient organic electroluminescent materials, to develop practical devices, and to understand their conducting and luminescent properties.

Notwithstanding an intense effort, the photophysics of these materials is not yet completely understood. This is well exemplified by the recent work on, presumably, simple systems, i.e., low-concentration solutions of MEH-PPV [2, 3, 4]. These studies brought to light that the solvent quality has a major influence on the spectrum, decay and quantum efficiency of the photoluminescence. The effects are believed to be induced by solvent-induced conformational changes of the polymer, leading to aggregate formation in poor solvents.

In high-quality solvents, at low concentration the photoluminescence (PL) is primarily due to the decay of the primary exciton [2], the spectrum showing a 0-0 band and well-resolved vibronic sidemodes. The PL quantum yield is high (> 50%) and the decay time of the photoluminescence is of order 0.3-1.2 ns. The optical gain can easily be made sufficiently large to generate mirrorless lasing [5]. It decays at the same rate as the photoluminescence when the pumping strength is such that depletion of the excited state by stimulated emission can be neglected [5].

In the technologically relevant case that the polymer forms a thin film the photophysics continues to be a subject of debate. For instance, the exciton binding energy is advocated to be of order 0.1 eV [2], 0.5 eV [5, 6], or 1 eV [11]. This variability makes it virtually impossible to estimate the probability of exciton dissociation at any temperature. There is universal understanding, however, that interchain species play an important role in the emission process of conjugated polymer films, whether these interchain species emit themselves or not. It has also become clear that the luminescent properties of conjugated polymer films can be heavily influenced by the film-processing procedure, and, again, the quality of the solvent when the film is cast [11].

In comparison to high-quality solutions, films of, for instance, MEH-PPV (an alkoxy-substituted PPV) exhibit considerably reduced PL efficiency, a PL decay which is slower and nonexponential, and very rapid optical gain dynamics. Although these characteristics are found for many PPV-derivatives, they are not universal. For instance, in DP6-PPV, the gain dynamics of the film is as slow as that of the solution [12], making this material well-suited as gain material in a conjugated-polymer-film laser. Also, a substantial number of PPV-derivatives, notably the phenyl-substituted varieties, have roughly equal, high, PL quantum-efficiencies in the film and in solution. These phenyl-substituted PPVs deserve particular attention, being the first to be used in a commercial product that is on the market at the present time.

Notwithstanding their market introduction, the photophysics of such phenyl-substituted PPVs, developed to have a high electroluminescent efficiency in the condensed phase [13], has hardly been studied. To address that issue we report here results on PL spectra, spec-
Wavelength (nm)

Figure 1: Absorption and photoluminescence spectra of the phenyl-substituted PPV polymer under study. The dashed lines show the spectra for the polymer dissolved in chlorobenzene while the solid lines display the absorption and emission spectrum of a spin-coated polymer film.

Figure 2: Decay of the photoluminescence of the polymer as excited by an ultrashort pulse at $\lambda = 400$ nm. Curve (a) shows the emission at $\lambda = 472$ nm by the dissolved polymer while curves (b) and (c) give the results for the polymer film at a wavelength of $\lambda = 472$ nm and $\lambda = 600$ nm, respectively.

2 Experiment

The conjugated polymer under study is the phenyl-substituted PPV, poly[2-phenyl-(2'-decyloxy)-1,4-phenylene-vinylene]. It exhibits good solubility in standard organic solvents and has a high electroluminescent efficiency when made into a thin film [13].

Our base material is a solution of the polymer in chlorobenzene at a concentration of 1 g/l. We fill a small cuvette with the solution to measure the PL spectrum and PL decay of the solution. Additionally, we form thin films by spin coating the solution on a BK7 substrate in a dry nitrogen atmosphere. The samples are kept and studied permanently under such atmosphere to avoid degradation due to the presence of oxygen and/or water. The optical density of the films is measured to be approximately 2 at a wavelength of 400 nm.

We excite the polymer, in solution or as a thin film, with the frequency-doubled output ($\lambda = 400$ nm) of an ultrafast (60 fs pulse duration) Ti:sapphire laser (Kapteyn-Murnane Laboratories) operating at a repetition rate of 82 MHz. The excitation pulses from the laser are heavily attenuated in order to remain in the linear-response regime and to avoid degradation of the sample. The laser beam is vertically polarized and is focussed to a spot of 1 mm diameter at the sample. The photoluminescence emitted by the polymer is collected with a lens and imaged on the input aperture of our detection equipment. Steady-state PL spectra are recorded with a simple fiber-optic spectrometer (Ocean Optics S2000), while we use time-correlated single-photon counting to measure PL in the time domain. With a multichannel-plate based photomultiplier (Hamamatsu model R3809U-52) our setup has a temporal resolution of $\approx 40$ ps. In the time-domain measurements we use spectral filters (bandwidth 10 nm, 10$^{-7}$ out-of-band transmission) to select specific wavelength bands in the range 472-600 nm. We include a polarizer in our setup to separately record the polarized and sensitized luminescence.

Figure 1 shows the steady-state absorption spectrum together with the time-integrated PL spectrum of the polymer, both in solution (dashed curves) and as a thin film (solid lines). For the solution the absorption spectrum peaks at $\lambda = 430$ nm, whereas the PL spectrum has its maximum at $\lambda = 490$ nm. The absorption spectrum of the film is similar to that of the solution, peaking at $\lambda = 428$ nm. As compared to that of the solution, the PL spectrum of the film is red shifted, relatively broad and structureless, with a peak at $\lambda = 538$ nm. When excited with more powerful pump pulses, generated by an amplified laser system, we observe amplified stimulated emission from our film, centered around $\lambda = 545$ nm.
3 Photoluminescence decay

For the polymer in solution the temporal evolution of the PL is almost wavelength independent and is well described by a single exponential:

$$ S = S_0 \exp(-\gamma t), \quad (1) $$

where $\gamma$ represents the PL decay rate. The decay takes place due to a combination of radiative (spontaneous emission) and nonradiative processes, which have, for the polymer at hand, roughly equal rates. Curve (a) in Fig. 2 shows the decay of the PL of the dissolved polymer as measured in a narrow interval around $\lambda = 472$ nm, i.e., in the blue wing of the pure singlet-exciton transition (see Fig. 1).

The PL decay of the thin film shows a strong wavelength dependence, and can not be described by a single exponential. Rather, the PL decay shows both a fast and slow component, the latter becoming slower with increasing wavelength. Additionally, for the longer emission wavelength, one observes that the PL rise time is no longer instrumentally limited as is the case for shorter emission wavelengths. To illustrate the wavelength dependence of the PL decay we include in Fig. 2 decay curves for the thin film (solid lines), as recorded in wavelength bands around $\lambda = 472$ nm (curve(b)) and $\lambda = 600$ nm (curve(c)). To simplify the comparison of the various decay curves in Fig. 2 they have all been normalized to unity at their respective maxima.

In the standard description of light emission by conjugated polymers intrachain energy migration and reexcitation play an important role [11]. The singlet exciton, generated through the photo-excitation process, is thought to migrate from regions of short conjugation length to regions of longer conjugation length, thereby reducing its energy. This process takes place on a time scale of order 100 ps [15]. Additionally, there can be interchain energy transfer due to dipole-dipole coupling, for instance through the Förster process [10]. This model is generally believed to be incomplete. Experimental evidence abounds that one has to assume the presence of at least two types of excitation: intrachain excitons and long-lived, red-emitting interchain species, such as excimers. These excitations are coupled through a one-way transfer from exciton to excimer. This transfer takes place in a time comparable to the response time of our setup ($\approx 40$ ps). Note that not all primary excitons need to follow this path. The presence of a long-lived component in the film PL at $\lambda = 472$ nm suggests that at least some of the primary excitons survive.

We parametrize the PL by a biexponential:

$$ S(t, \lambda) = S_{\text{fast}}^\lambda \exp(-\gamma_{\text{fast}}^\lambda t) + S_{\text{slow}}^\lambda \exp(-\gamma_{\text{slow}}^\lambda t), \quad (2) $$

where $S_{\text{fast}}^\lambda$ and $S_{\text{slow}}^\lambda$ are proportional to the population of the fast-decaying and the slow-decaying species that emit at wavelength $\lambda$, respectively. These species decay at rates $\gamma_{\text{fast}}^\lambda$ and $\gamma_{\text{slow}}^\lambda$, respectively. Figure 2 shows that the slowly decaying component can indeed be described by an exponential. Our parametric description of the luminescence fits our experimental data quite well, particularly at longer wavelengths.

For $\lambda > 515$ nm we associate the slowly decaying component of the PL with the excimer, and the component with fast decay with the exciton, each emitting at that specific wavelength. In the extreme blue wing of the PL spectrum we make a different connection; there we associate the fast decaying component of the PL with the (nonradiative) relaxation of the bulk of the exciton population, while the slow decay represents the evolution of a class of high-energy excitons that have strongly reduced nonradiative decay; henceforth we will refer to these excited states as “isolated excitons”. The rapid decay of the bulk of the population of high-energy excitons is due to nonradiative processes such as intrachain and interchain energy transfer and coupling to excimer states. In that light it does not come as a surprise that Eq. (2) does not fit our data so well at short wavelengths. One can hardly expect the complicated energy transfer process to be described by a biexponential.

![Figure 3: Upper frame: PL spectra measured for a solution (dashed line) and for a film (solid line). The triangles indicate the midband positions of the narrowbandwidth (10 nm) filters to selectively measure the wavelength dependent PL decay. Lower frame: The dashed curve shows the wavelength dependence of the decay time of the PL from a solution; the solid curve displays the behavior of the decay constant associated with the long tail of the PL emitted by a film.](image-url)
From the fits one can extract values for the two decay rates $\gamma_{\text{fast}} \leq 5 \times 10^9$ s$^{-1}$ and $\gamma_{\text{slow}}$. In Fig. 3 we show the values of $\tau_{\text{slow}}(\lambda) = (\gamma_{\text{slow}})^{-1}$ as a function of emission wavelength. For reference purposes we have included the decay time for the polymer in solution as obtained from a fit of the appropriate data with a curve described by Eq. (1). Figure 3 also shows that the film PL (at least its slow component) decays at a rate that is appreciably slower than that of the solution. This observation supports our assumption that an interchain species is involved in the slow decay of the film PL.

4 Photoluminescence anisotropy

Additional information about interchain interactions can be obtained from a time-domain study of the photoluminescence anisotropy \[ r(t, \lambda) = \frac{S_{\parallel}(t, \lambda) - S_{\perp}(t, \lambda)}{S_{\parallel}(t, \lambda) + 2S_{\perp}(t, \lambda)}. \] (3)

Here $S_{\parallel}(t, \lambda)$ and $S_{\perp}(t, \lambda)$ represent the strength of the polarized and sensitized PL signals at wavelength $\lambda$ as a function of time.

![Figure 4: Time-dependent anisotropy of the emission from a solution (curve (a) for $\lambda = 472$ nm and curve (b) for $\lambda = 580$ nm), and from a film (curve (c) for $\lambda = 472$ nm and curve (d) for $\lambda = 600$ nm).](image)

Modeling the conjugated polymer, both in solution and in the film, as an ensemble of randomly oriented dipoles one finds $r(0) = 0.4$ (that is $S_{\parallel}(0)/S_{\perp}(0) = 3$), if the absorbing and emitting dipoles are parallel. Beyond $t = 0$, the photoluminescence anisotropy (PLA) decays due to orientational relaxation. In a polymer solution this happens through orientational diffusion of the polymer chains. In the film, the decay of $r(t, \lambda)$ is due to intrachain and interchain excitation transfer, or creation of interchain states.

In our experiments we find that $r(t, \lambda)$ never exceeds the value 0.3, even for the solution at $t = 0$. This implies either misalignment of the absorbing and emitting dipoles, or very fast depolarization during the energy relaxation process from the photo-excited to the luminescent level of the singlet manifold.

Because of the importance of the instrumental response during the rising part of the PL signal, one cannot arrive at a reliable value of $r(t, \lambda)$ while either $S_{\parallel}(t, \lambda)$ or $S_{\perp}(t, \lambda)$ increase rapidly. We therefore calculate $r(t, \lambda)$ only during the decay of the PL signals, with $t = 0$ being defined as the time at which the PL signals reach their maximum. Figure 4 shows our results for the time-dependent anisotropy for the same set of wavelengths as in Fig. 2. In all measurements the PLA decays as a function of time, not reaching a steady-state value within our time window (1 ns). Additionally, the PLA of the solution is, at all times, substantially larger than that of the film. For $t \geq 0.2$ ns the PLA is well described by an exponential time dependence; this applies to all wavelengths both in solution and in the film. By fitting our data for $t \geq 0.2$ ns with an exponential we obtain the associated decay times, of order $1 - 3$ ns.

Figure 5: Wavelength dependence of the PL anisotropy at $t = 0$ for the polymer solution (dashed curve), and for a polymer film (solid curve).

Figure 5 shows the values of $r(0)$, i.e., the maximum value of the photoluminescence anisotropy. For the solution we find a, basically, wavelength independent value of $r(0)$, except at the shortest wavelengths. Curve (a) of Fig. 4 shows that the increased value of $r(0)$ at $\lambda = 472$ nm is due to an enhanced contribution of the fast-decaying component of $r(t)$. The value of $r(0)$ for the film is strongly wavelength dependent.
5 Discussion

Reviewing all our experimental results for the solution we note that the photophysics in that system is essentially wavelength independent except at the shortest wavelengths. This is summarized succinctly by the relevant curves of Figs. 3 and 5. The photoluminescence decay can be described by a single exponential, yielding a PL decay time of \( \approx 0.65 \) ns. The photoluminescence anisotropy is substantial and has a long-lived component that is well described by an exponential time dependence, with a time constant of a few ns. This component of the PLA decay can be attributed to orientational diffusion of the polymer in its solvent [19]. The weak, fast decaying component of the anisotropy is associated with dipole-dipole-coupling-induced loss of orientation during (intrachain) energy migration along the polymer backbone [2] [15] [20]. This is supported by our data that show that this fast-decaying component is quite a bit larger at the shortest wavelength than at longer wavelengths (see Fig. 3). The photoluminescence at short wavelengths is entirely due to excitons that have not migrated, whereas at longer wavelengths the emission is due to excitons that have been populated directly or through an intrachain energy transfer process. Interchain energy transfer can be excluded because of the low probability of interchain overlap in solution.

The results for the film are in sharp contrast with those of the solution. Here we observe a strong wavelength dependence (see Figs. 4, 5, and 6) and decay curves that can not be described by a single exponential. As discussed earlier this behavior is due to interchain processes, all-important in the film, and virtually absent in the solution.

Combining the results of Fig. 5 with those of Fig. 1 one notices that the anisotropy is very weak \( r(0) < 0.06, S_{\parallel}/S_{\perp} < 1.2 \) and essentially featureless over the main body of the emission spectrum of the film. This small value of the anisotropy supports the earlier made assumption that, in this wavelength interval, the emission originates predominantly in species that have not been populated directly in the photo-excitation process. In the extreme red wing, where the photoluminescence decays slowest (see Fig. 3), these have interchain character and are collectively called excimers. These interchain species are generally believed to have reduced oscillator strengths, hence the slow decay of the photoluminescence (see Fig. 3). Less far in the red wing the emission may well be due to both excitons and excimers, the excitonic emitting states (partly) having been populated through an intrachain energy transfer process.

In the extreme blue wing of the emission spectrum the PLA is strongly wavelength dependent, as shown by Fig. 5. Here the value of \( r(0) \) is comparable to that in solution (see Fig. 4). This suggests that, at these wavelengths, the same species (a singlet exciton) is responsible for the emission by both the film and the solution. Nonradiative processes such as intrachain transfer rapidly deplete this exciton population, inducing the fast initial decay of the PL. The dipole-dipole coupling that is believed to be responsible for (part of) these transfer process causes the observed rapid loss of anisotropy at these wavelengths. The slowly decaying tail of the luminescence attests to the presence of excited species that emit at these wavelengths at much later times. We believe that these are not excimers or other low-energy interchain species. Rather, we attribute this emission to the isolated excitons, introduced earlier. These communicate only weakly with their environment, as shown by their slow nonradiative decay and slow depolarization.

It is interesting to make a connection to measurements of the transient gain in thin films of this and similar PPVs. Initially, the transient gain can be quite considerable but is, almost universally, seen to decay very rapidly, on a time scale of tens of picoseconds. It is tempting to connect the fast decay of the gain in polymer films with the rapid decay of the initially excited species [11]. Hence, we postulate that the gain is exclusively related to the exciton population, while a considerable part of the photoluminescence is related to the population of excimers. The mere fact that the photoluminescence quantum efficiency of a conjugated polymer is particularly high thus does not guarantee that it is especially suitable as a gain material in a laser. Rather, in order to make a good optically pumped polymer laser, one has to reduce the interchain coupling in the film. This discussion puts into context the recent extensive efforts to control and reduce interchain effects in conjugated polymers [21] [22] [15] [23] [24] [25] [26]. Note, however, that interchain coupling is essential for any kind of charge transport, i.e., for the development of injection-type polymer lasers.

It is clear that a description of the lasing process in thin films of conjugated polymers in terms of a standard four-level system is, from this perspective, not to the point. An interesting issue to pursue is the question why the excimers in conjugated polymers do not give rise to lasing. A curious aspect of excimer lasing in conjugated polymers is that the energy transfer process that populates the excimer states enhances the Stokes shift. In standard laser physics, a large Stokes shift is considered to be advantageous. The reduced oscillator strength of the excimers should not pose a problem in view of the many laser systems that are
based on very low oscillator-strengths optical transitions. Obviously, the crucial question concerns whether the excimer emission is self-absorbed by the material, i.e., whether (photo-induced) absorption by excimers or other interchain species will overwhelm the gain.

6 Conclusions

We have studied the photophysics of a phenyl-substituted PPV, dissolved in a common organic solvent, and as a thin film by performing time-domain measurements of the spectrally resolved photoluminescence and its anisotropy across the full emission spectrum of the material. As for many conjugated polymers, the emission by the film is red-shifted as compared to that of the solution. The overall picture is that the measured properties for the dissolved polymer are wavelength independent, while the luminescent properties of the film vary considerably with wavelength. These observations thereby confirm the results obtained by others in that the photoluminescence in the film has, for the most part, a different origin as that in solution. While in solution the emission is dominated by the radiative decay of singlet excitons, in the film the main contribution comes from interchain species such as excimers.

We have paid particular attention to the blue wing of the photoluminescence spectrum where the differences between film and solution are smallest. In that spectral region the film photoluminescence (and its anisotropy) is dominated by a quickly decaying component, the decay being caused by intrachain relaxation. Once this component has decayed away, we measure an emission that slowly gets weaker. We attribute this emission to isolated excitons that only weakly interact with their surroundings.

Finally, we speculate on the connection between photoluminescence and gain in films of conjugated polymers. We discuss whether the long-living emitter in the film can be exploited for lasing.

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