

## Electronic Processes of Conjugated Polymers in Semiconductor Device Structures

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### Abstract

We report progress in the processing and application of poly(phenylene vinylene), PPV, as the emissive layer in electroluminescent diodes, LEDs. Photoluminescence efficiencies above 60% for solid films of PPV are now achieved, and single-layer EL diodes achieve luminous efficiencies above 2 Lumens/W and peak brightnesses up to 90,000 cd/m<sup>2</sup>. We demonstrate that PPV of this type can show stimulated emission in sub-picosecond pump-probe experiments, and can be used as the active lasing medium when incorporated in suitable microcavity structures.

Key words: Electroluminescence, Laser, Poly(phenylene vinylene), Photoluminescence

### 1. Introduction

Poly(phenylene vinylene), PPV, has fulfilled the role of prototypical luminescent conjugated polymer. PPV and its many derivatives have provided the best-performing emissive polymer layers in electroluminescent diodes, LEDs [1-5]. In spite of the very considerable work that has been done to characterise the electronic processes that determine operation of these devices, there is still considerable confusion about the nature of the semiconductor excitations in these materials. We discuss here the present understanding of the neutral excited state which is responsible for light emission, and which is generated either by electron-hole capture in LEDs, or directly by photoabsorption in photoluminescence experiments (PL).

### 2. Electroluminescence in PPV diodes

Electroluminescence from conjugated polymers was first reported in structures using poly(phenylene vinylene), PPV, as the single semiconductor layer between metallic electrodes [1], as is illustrated in figure 1. PPV has an energy gap between  $\pi$  and  $\pi^*$  states of about 2.5 eV, and produces luminescence in a band below this energy, with yellow-green emission. PPV is an intractable material with a rigid-rod microcrystalline structure, and excellent mechanical properties, with high elastic moduli and thermal stability to 400°C. We have worked with the sulphonium precursor [6-10] which is conveniently processed from solution in solvents such as methanol, and is converted to PPV by thermal treatment at temperatures typically near 200°C. The alternative strategy for polymer

processing is to attach flexible side-groups to the polymer chain so that the polymer is directly processible from solution. This is particularly convenient in that a single process step is required, but the trade-off is that the polymer film is softer and less stable thermally. The derivative of PPV that is most frequently used is poly(2-methoxy, 5-(2'-ethyl)hexyloxy-*p*-phenylene vinylene), MEH-PPV [2, 11].

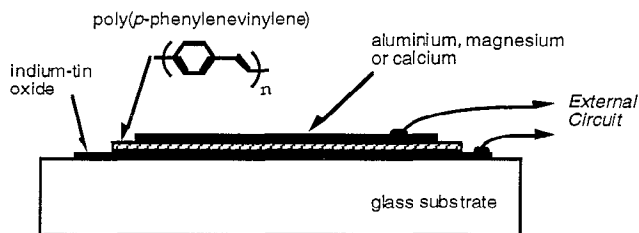


Figure 1 Structure of polymer LED

LED operation is achieved when the diode is biased sufficiently to achieve injection of positive and negative charge carriers from opposite electrodes. Capture of oppositely-charged carriers within the region of the polymer layer can then result in formation of the singlet exciton (which is also generated by photoexcitation across the  $\pi$ - $\pi^*$  gap [12]), and this can then decay radiatively, to produce the same emission spectrum as that produced by photoexcitation. The absorption rises rapidly above the onset of the  $\pi$ - $\pi^*$  threshold, and the emission spectrum appears on the low-energy side of the absorption. Both

absorption and emission spectra show broadening due to vibronic coupling, as is characteristic for optical transitions in molecular semiconductors where the excited state is a singlet exciton. Note that the similarity of the emission spectra produced by photoexcitation and by charge injection establishes that the excited state responsible for light generation in the LED is the same as that produced by photoexcitation.

The levels of efficiency of the first, simple LEDs based on PPV, which were fabricated with aluminium negative electrodes were relatively low, of order 0.01% photon generated within the device per electron injected [1]. External quantum efficiencies are strongly affected by the refractive indices of the layer that comprise the device, and the relationship between the two has been discussed by Greenham et al [13]. These values have risen rapidly over the past 5 years as improved understanding of the operation of these devices, aided in considerable measure by parallel developments made with sublimed molecular film devices [14-16], has allowed considerable optimisation of the device characteristics. The use of negative electrodes with lower work functions was shown by Braun and Heeger [2] to improve efficiency to as high as 1%, in devices made with ITO/MEH-PPV/Ca. The use of copolymers based on PPV with higher luminescence efficiencies also raised efficiencies to similar levels [17], and the use of heterostructure devices has brought efficiencies to 4% and above [3, 18].

PPV has not generally been considered to be especially luminescent, and therefore not necessarily optimised for use as the emissive layer in LEDs. In particular, Rothberg and co-workers have found evidence that the majority of photoexcitations in the PPV that they studied are not the intrachain excitons which can give emission, but instead show optical absorption features characteristic of charged excitations [19], and these have been modelled as inter-chain polaron pairs [20]. However, as we discuss below, the PL efficiency of PPV is very sensitive to both the method of preparation, and also to the handling of the polymer film once made. PPV films as prepared in Cambridge were reported to show solid-state PL efficiencies (measured using an integrating sphere) of up to 27% [21].

These high PL efficiency values, combined with the desirable mechanical properties shown by precursor-route PPV provide a very attractive material for use in durable polymer LED products, and we have worked to improve further the properties of PPV produced in this way. PL efficiencies for standard HCl-eliminating precursors have now been raised to 40% and above, and, with modifications to the synthesis, up to 80%. These improvements in PL efficiency have a direct impact on the performance of PPV-based LEDs, and we illustrate this here for simple diode structures which use a single layer of semiconducting

polymer. Although such structures do not necessarily provide optimum balancing of electron and hole currents, very good efficiencies for devices made with MEH-PPV and related polymers have recently been reported, for devices made with calcium cathodes [22, 23]. LEDs made with structures similar to that shown in figure 1 using PPV with high PL efficiency now give luminous efficiencies of up to and above 2 Lumen/W, and can show high brightness at low drive voltages. This is illustrated in figure 2 for a device made with a low work-function cathode. Note that this device reaches a brightness of 400 cd/m<sup>2</sup> at a drive voltage of 4 V, comparable to results reported for MEH-PPV devices [22]. Other devices can be operated under CW conditions to 20,000 cd/m<sup>2</sup> and under pulsed mode, to 90,000 cd/m<sup>2</sup>.

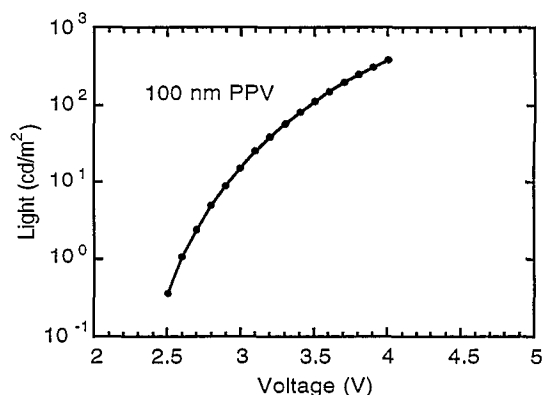


Figure 2 Light output versus drive voltage for a PPV-based single-layer LED

The significance of high peak brightnesses for polymer LEDs is that this is necessary for passive-matrix displays. A demonstrator with 16 x 60 pixels has recently been fabricated by Cambridge Display Technology [24].

### 3. Excitons in PPV

As mentioned in section 1, there is still a great deal of controversy surrounding the nature of the primary photoexcitation in PPV. Debate has centred on the binding energy of the photo-excited state which can arise from both coulombic and lattice distortion contributions. If the excitation does not have a significant binding energy then description within a band model description may be appropriate [25]; in contrast, if the electron and hole are

strongly bound they form an intrachain exciton [26-28]. We consider that the balance of evidence shows that an exciton model is more appropriate. As mentioned in section 2, it has been suggested that non-emissive spatially indirect excitons [19], or inter-chain polaron pairs [20] may be an alternative excitation route, but as previously mentioned, the high PL efficiency now measured for PPV made in Cambridge demonstrates that this is not the dominant excitation for these samples.

We draw attention to four sets of experiments recently carried out in Cambridge which provide information on these issues:

### 3.1 Photoconductivity Excitation Spectra

The measurement of the photoconductivity, PC, response of PPV as a function of photon energy has been reported by several groups, and it is generally agreed that the onset of the PC coincides with the onset of the  $\pi$ - $\pi^*$  absorption near 2.5 eV [25, 27, 29-32]. There is controversy however, as to the origin of this PC response. The Santa Barbara group consider that this energy is the onset for photogeneration of free electron-hole pairs (the single-particle gap) [25], whereas other groups, including Cambridge, have identified this energy as the energy of the singlet exciton, with the PC response arising via subsequent charge separation of the exciton [27, 31-33]. This latter process is often facilitated by the presence of extrinsic factors, such as adsorbed oxygen [34, 35].

Optical absorption in PPV and its derivatives shows considerable structure at energies well above the  $\pi$ - $\pi^*$  absorption onset, and several groups have considered how this can arise [28, 36, 37]. According to the calculations of the Mons group, at the INDO/SCI level, the higher-lying optical absorption bands involve both delocalised  $\pi$  bands and also the  $\pi$  wavefunctions localised on each of the phenylene units along the chain. Such excitations account for the absorption bands seen in dialkoxy-PPV's at 3.8 and 4.6 eV.

We have studied the spectral dependence of the photoconductivity for a range of PPV derivatives, using a simple sandwich cell geometry, of silica-glass substrate/semi-transparent gold/polymer/semi-transparent aluminium. Short-circuit currents have been measured as a function of photon energy up to and beyond 5 eV. Detailed results are presented elsewhere in these proceedings [35]. We find that the photocurrent rises at each of these optical absorption bands. In detail, the size and spectral position of the response depends on the atmospheric exposure and direction of illumination (through the Au or Al electrodes), but the general result is that there is a strong increase in photocurrent as the photon energy reaches each of these absorption bands (e.g. 2.2, 3.8 and 4.6 eV for dialkoxy-PPV).

This association of structure in the photocurrent spectra with features in the optical absorption associated with the molecular structure of the polymer chain is supported strongly by calculations performed by the Mons group, reported in [35], which show the excited state wavefunctions for these higher-lying excitonic excitations. A strongly increasing excited-state dipole moment is found at these higher-lying transitions; this is expected to allow increasingly easy charge separation after initial generation of the excited state as the photon energy is raised.

We note that the energy difference between successive absorption/photocurrent features is determined by the interplay of the localised and delocalised states involved in the transition. Chandross et al. [28] also observed the first of these higher-lying transitions for a dialkoxy-PPV, at 3.8 eV, but considered that this was the energy of the separated electron-hole pair (the so-called  $n$ - $B_u$  state) associated with the delocalised  $\pi$  bands. From this they derive the binding energy for the singlet exciton in excess of 1 eV. We consider that the explanation advanced above provides an alternative description - one which says nothing about the size of the exciton binding energy, which we consider in the section below to be considerably smaller than 1 eV.

### 3.2 Photovoltaic Diodes

Though photoconductivity can be measured in single-layer structures of the type considered in section 3.1 above, it is found that it can be considerably enhanced in devices which incorporate an extrinsic mechanism for exciton ionisation (adsorbed oxygen plays such a role in these single-layer devices [35]). The addition of fullerene,  $C_{60}$ , to soluble dialkoxy-PPV's in solid solution was reported to reduce strongly the luminescence, and to produce more efficient charge generation [38-40]. Though solid solutions of this type show efficient luminescence quenching, they are not optimised for charge collection at the two electrodes formed in standard diode structures of the type shown in figure 1.

We have investigated the properties of 2-layer diodes formed with precursor-route PPV deposited on ITO/glass, with evaporated films of electron acceptors formed onto the PPV layer, and with a final top electrode of thermally-evaporated aluminium [41, 42]. We find that these devices can show efficient charge generation when incident light is absorbed by the PPV layer, with quantum efficiencies for charge collection under short-circuit conditions of up to 9% for devices made with fullerene as electron acceptor. The action spectrum under short circuit conditions is shown in figure 3 for such a diode, and we have shown that this can be modelled quantitatively using a model in which only light absorbed close to the heterojunction between PPV and fullerene results in charge generation. We equate the width of this 'active' region to the diffusion range of excitons

which are photogenerated in the PPV, and we have determined that this length is about 8 nm for the PPV we have used for this work [41]. The match between model and experiment is shown in figure 3. Results for diodes made using a tetra-carboxyimideperylene derivative as electron acceptor give similar results [42].

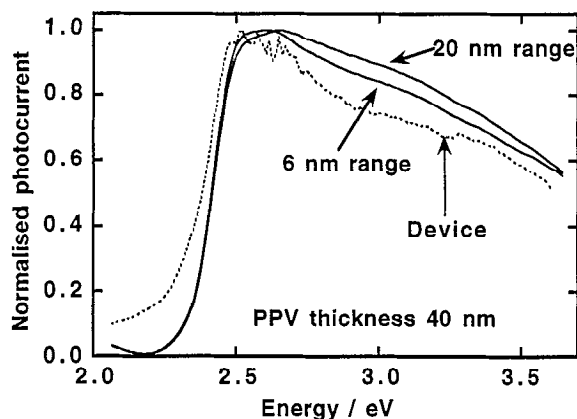


Figure 3 Short-circuit current versus incident photon energy for a 2-layer photodiode made with 40 nm of PPV and 40 nm of fullerene [41]. Also shown are modelled responses for two different exciton diffusion ranges, of 6 and 20 nm.

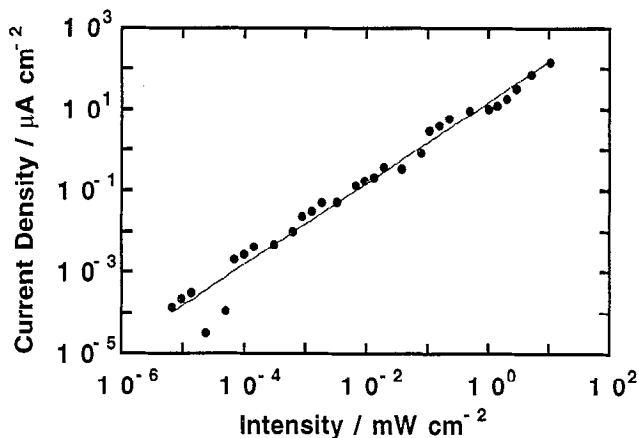


Figure 4 Variation of short-circuit current with incident light intensity for a photodiode of the type shown in figure 3.

These 2-layer diodes show good fill-factors, and also show excellent linearity of short-circuit current with incident light intensity, as is shown in figure 4. This indicates that recombination and charge trapping are not important, as is expected for diodes in which holes and electrons are generated in separate layers following the exciton dissociation at the heterojunction.

Efficiency of these devices is limited by the low exciton diffusion range in relation to the optical absorption depth, and the values reached here (up to 9%) are difficult to improve further using this device architecture. We and also the Santa Barbara group have investigated an alternative structure in which the heterojunction is distributed throughout the film, and is formed by the phase separation of two soluble polymers which can act as electron and hole accepting components. This is achieved using soluble dialkoxy-PPV's in combination with cyano-derivatives of PPV [43, 44]. Considerably higher efficiencies can be obtained using such structures, with quantum yields of up to 80% under applied bias.

### 3.3 Photoluminescence Excitation Spectra

We have mentioned above that the high PL efficiency we have reported for PPV synthesised and measured in Cambridge is apparently at odds with the evidence for a low efficiency for exciton generation as has been suggested from results for stimulated emission [19]. These latter measurements have taken as evidence that non-emissive spatially indirect excitons, or inter-chain polaron pairs [20] are the dominant photoexcited species.

We have recently found that the PL excitation spectrum for PPV is very sensitive to the presence of a surface-photooxidised layer. If this is present, then light absorbed in this region results in low PL efficiency (though possibly high charge generation efficiency), and the PLE spectrum peaks close to the absorption edge, at the value of absorption coefficient which maximises the fraction of incident light which is absorbed within the deeper-lying regions of the film which show efficient PL. These results are reported elsewhere in these proceedings [45].

We have been able to measure PLE spectra for non-oxidised PPV films which show a constant PL efficiency throughout the wavelength range 250-500 nm. We consider that these results indicate that the dominant photoexcitation product is the singlet intra-chain exciton when absorption takes place in pristine PPV. This is consistent with the high PL efficiencies found for these samples [21, 45].

### 3.4 Transient Stimulated Emission

Following the work which we discuss in section 3.3 above, we have carried out measurements of transient absorption following photoexcitation, using sub-picosecond light pulses. The object of these experiments was to determine if the absence of stimulated absorption reported for PPV by Yan et al. [19] might be related to the nature of the PPV used. We were particularly interested to see how PPV which was characterised to show high PL efficiency would behave under these conditions.

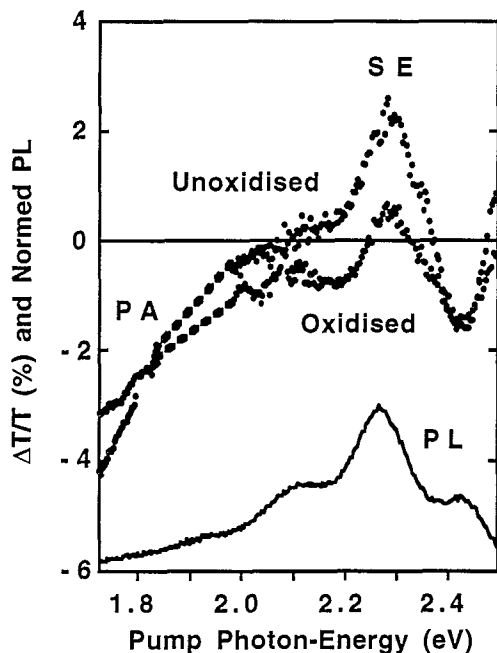


Figure 5 Comparison of transient optical transmission through pristine (unoxidised) and photo-oxidised PPV at 1 psec after photoexcitation. The CW PL spectrum is shown for comparison.

For these measurements, the pump and probe pulses were derived from the amplified output of a synchronously pumped dye laser operating at 650 nm (1.9 eV) with a pulse duration of 100fs. The amplified pulses are focussed into a quartz plate to produce white-light continuum which is then split, with part being used as the probe beam; the rest is spectrally filtered to leave only the 650 nm component, amplified and frequency doubled in a beta barium borate crystal. This beam at 325 nm (3.8 eV) acts as the pump and is focussed onto the sample surface, coincident with the probe beam. Both beams were horizontally linearly polarised. Spectrally-resolved

measurements of the fractional change in probe transmission in the presence of the pump ( $\Delta T/T$ ) were made using a Peltier-cooled CCD camera. Chirping of the probe pulses was numerically corrected using an empirical determination of the dispersion.

We obtain strong stimulated emission in the region of the cw-detected PL spectrum, but this is reduced greatly in oxidised material [46]. We show that the presence of defects caused by photo-oxidation can explain some of the key results of previous time-resolved studies of PPV which were assumed to be intrinsic effects. Additionally, our measurements indicate that pristine PPV exhibits a considerably higher efficiency of photogeneration of singlet polaron-excitons than the figure determined by Yan et al. [19]. These results are indicated by those shown in figure 5. Note that the unoxidised sample shows strong stimulated emission, SE, close to the maximum of the CW PL spectrum, but that this is strongly suppressed in the oxidised sample. Induced absorption, PA, is seen at lower photon energies, in agreement with Yan et al. [19].

We conclude from these measurements, see also [46], that SE is observed from PPV which shows strong PL. This SE can be strongly enhanced in a cavity structure, as is discussed in section 4 below.

### 4 Optical Microcavities and Waveguides

Lasing from conjugated polymers in the solid state has been a challenge ever since the first report of electroluminescence in PPV [1]. We have made use of the improved forms of PPV now available, as used for example in the efficient EL diodes discussed in section 2.

The use of a planar microcavity introduces feedback into the optically-pumped polymer film, and readily gives rise to enhanced values of SE. This is illustrated in figure 6, for a microcavity formed with PPV sandwiched between two semitransparent silver mirrors. This cavity has been constructed so that there is a resonant cavity mode close to the CW PL emission peak [46]. The transient optical transmission in the region of the PL peak is shown in figure 6(a), and shows a response due both to SE and also to the modulation of the cavity mode wavelength as a result of the refractive index change (reduction) following photoexcitation. The latter response can be removed, to leave the SE response as shown in figure 6(b). Note that the peak SE is now equivalent to a 25% change in fractional transmission, considerably higher than as shown in figure 5 above for a PPV film on glass. Lasing is produced when the quality factor of the cavity is improved so that the fractional transmission rises to 100%. We have achieved this in devices formed with one dielectric mirror and one silver mirror, and have reported on this elsewhere [47, 48].

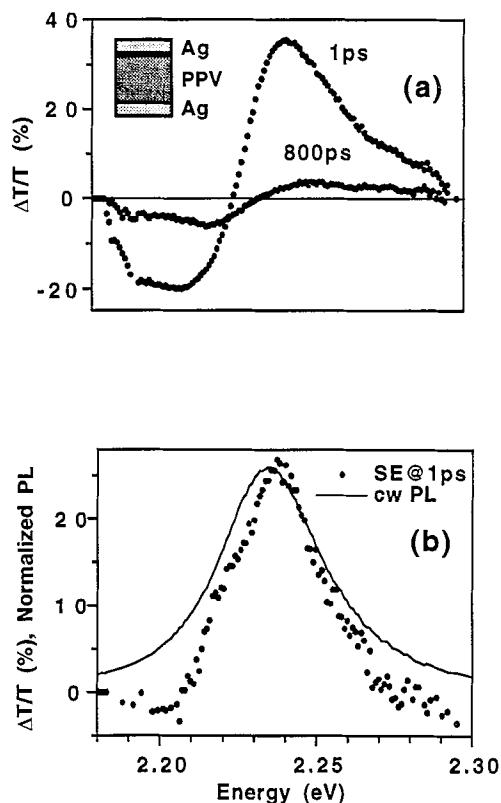


Figure 6 (a)  $\Delta T/T$  spectra measured on the microcavity structure at 1 and 800 ps after excitation. Inset: structure of microcavity and PPV. (b) Dots: Result of subtraction of the scaled 800 ps  $\Delta T/T$  spectrum from that obtained at 1 ps. The solid line is the cw-detected PL from the structure.

We have also noticed that emission from PPV films formed on glass substrates can show strong line narrowing above a threshold excitation intensity, as has also been reported elsewhere in this conference [49, 50]. Figure 7 shows PL spectra obtained from a Spectrosil-mounted PPV film using different incident power levels. At the lowest power, we observe a typical PPV emission spectrum displaying vibronic structure and a FWHM of about 50 nm. At the next power level, it is apparent that the weight of the spectrum has shifted to the central vibronic peak, resulting in a reduction in the line-width to 30 nm. With a further increase in power by a factor of 5, the emission spectrum changes to become a single emission line, peaked close to 550 nm and with a FWHM of 4.5 nm. To investigate this effect further, we repeated the experiment on a thinner film (below half a wavelength in thickness) and found no evidence for spectral narrowing, in agreement with other measurements reported recently.

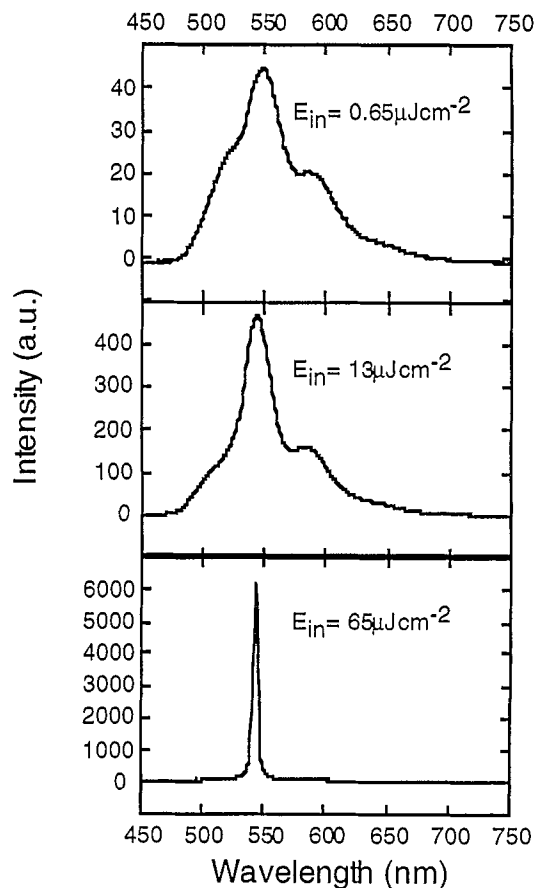


Figure 7 Time-integrated PL spectra measured from a PPV film on a silica glass substrate at different pump fluences,  $E_{in}$ , as shown on the figure. Excitation was provided at 355 nm with pulses of 200 psec duration.

These structures are not obviously capable of providing the feedback necessary for lasing. One proposed explanation of this effect is that PL is waveguided in the film and experiences gain (via amplification of spontaneous emission) over a relatively long optical path-length. Alternatively, superradiance has been invoked as a possible cause [50].

## 5 Conclusion

We have presented a range of measurements performed on PPV which shows high luminescence efficiency. The conditions necessary for efficient charge photogeneration have been discussed, under which conditions the PL is quenched. We have highlighted the importance of extrinsic

factors in this process. In contrast, for PPV which shows efficient PL, we find that many of the properties, for example associated with stimulated emission, are consistent with photogeneration of intra-chain excitons.

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