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Is Poly(vinylcarbazole) a Good Host for Blue Phosphorescent Dopants in PLEDs? Dimer Formation and Their Effects on the Triplet Energy Level of Poly(*N*-vinylcarbazole) and Poly(*N*-Ethyl-2-Vinylcarbazole)

Vygintas Jankus,* and Andrew P Monkman

The detailed measurement and analysis of the delayed emission from poly(vinylcarbazole) (PVK) and poly(*N*-ethyl-2-vinyl-carbazole) (P2VK) thin films is described. PVK has rapidly become a “polymer of choice” for hosting phosphorescent dopants in PLEDs, especially blue emitters. In this respect it is important to have a full understanding of the triplet properties of this host. It is concluded that in films, the electronic 0–0 peak energy of PVK phosphorescence is found at 2.88 eV (14 K). With an increase of temperature, >44 K, increasing emission from new long lived, lower energy species, previously ascribed to “trap states” in the literature, is observed. Increasing temperature enables thermally assisted triplet exciton hopping to these trap states. Critically it is shown that some of these triplet trap species are ground state triplet dimers in origin for both PVK (2.46 eV) and P2VK (2.1 eV), and not all of them are of excimer nature as previously thought. These species can quench the emission of blue heavy metal complexes doped in PVK and drastically effect performance over lifetime if the dimer formation increases over time and at elevated operating temperature. It is therefore concluded that PVK might not be such an ideal host material for blue phosphorescent emitters.

1. Introduction

Polymer light emitting devices (PLED) have come a long way since Burroughs et al.^[1] reported first devices using a soluble conjugated polymer. Since then the performance of PLEDs has increased enormously^[2–4] especially with the introduction of phosphorescent heavy metal complexes as emitting species.^[5,6] A major area of current interest for PLEDs is large area printed lighting panels, where low cost simple device structures with good efficiency are required.^[7] However, much work still needs to be done to enhance the efficiency of phosphorescent PLEDs for white light production. One major issue in improving the efficiency is finding suitable host polymers for deep blue phosphorescent dopants, as these require

high enough triplet energy to confine triplet excitons on the phosphorescent dopant. To date there have been several reported polymers with high triplet levels, but none have achieved a triplet energy greater than 2.6 eV,^[8–12] which is not high enough to prevent quenching of blue phosphorescence, emitting typically at 2.7–2.8 eV depending on the blue wavelengths required for different lighting applications or NTCS display colour gamuts. Poly(vinylcarbazole) (PVK) has been extensively used as a hole transporting host for green emitting devices^[13] reaching up to 31 cd A^{−1}^[14] or even up to ~40 cd A^{−1} current efficiency and ~12% external quantum efficiency^[15] and many people are turning to PVK as a host for blue phosphorescent dopants.^[16–17] However, PVK as a host for blue phosphorescent does not perform as well; for example, current efficiency in single active layer devices reaches not more

than 18.2 cd A^{−1}.^[17] This is even more evident if one compares devices manufactured in same laboratories and under similar conditions.^[14] The current efficiencies of more complicated PVK based blue devices^[18,19] (for example with additional electron blocking layer) reach slightly higher values; however, these additional layers might be very inconvenient when moving devices from scientific laboratories to larger scale manufacturing. Thus the question arises: why is PVK underperforming as a blue host?

The phosphorescence properties of PVK in solutions and in rigid isolated matrices has been studied in the past^[20–28] and the triplet level has been reported to reach 3 eV in solution which seems high enough for any blue dopant.^[21,22] In PVK films, it has also been reported that the triplet level is close to this value, ~2.9 eV.^[20] There are also contradictory publications in which the triplet level of PVK is suggested to be 2.5 eV.^[29,30] In addition, in PVK films, delayed emission from “traps” has been observed at ~2.5 eV.^[20,22,25,26,31,32] Reports suggest the existence of two trap states in PVK films – one related to a semi eclipsed pair of carbazole units and one fully eclipsed (face to face) carbazole pair.^[33] Carbazole has a ground state dipole moment which enhance the attraction between carbazole pairs over

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simple Van der Waals attractions. These trap states are apparent in device made simply with PVK emissive layers.^[34,35] We note however that Qian et al.^[35] also reported highly redshifted species which do not correlate to all of their PL measurements or those reported previously in literature. Poly(*N*-ethyl-2-vinyl carbazole) (P2VK) triplet properties have not been reported to such an extent yet and is included here for comparison purposes with its homolog PVK.

The exact nature of these trap states could be excimer (excited state dimer – nonexistent in a ground state) as conjectured in references^[20,27,32] or the triplet excited state of a ground state physical dimer or a mix of both. “Physical dimer”, according to Pope et al.,^[41] is a situation where two identical molecules in a ground state are closer in a spatial arrangement than to other molecules resulting in a large π - π^* overlap between them. This is different to “chemical dimer” where two identical molecules form chemical bonds. In this paper “physical dimer” is referred to as a “dimer”. The spectra of dimer emission normally is redshifted in comparison with the monomer, however it still keeps the majority excitonic features. A new redshifted absorption band normally arises with an increase of dimer concentration indicating ground state interaction. According to Pope et al.^[41] and the IUPAC definition, “excimer” is an electronically excited physical dimer consisting of the same species ground state molecule and excited state molecule. Most importantly, it is dissociative and non-bonding in a ground state, not unlike a dimer; hence, normally no new absorption band is observed with an increase of excimer concentration. Emission of excimers which are normally accompanied by charge transfer contributions is normally characterised by red-shifted broad unstructured spectra. Here it has to be mentioned that the terms excimer and dimer are used rather confusingly in the literature. Birks^[42] also defined excimer as a “dimer which is associated in an excited electronic state and which is dissociative (i.e., would dissociate in the absence of external restraints) in its ground electronic state”. This type of excimer or photodimer might be detected by ground state absorption; however it is still accompanied by excimeric charge transfer type broad emission spectra rather than excitonic well structured emission as is normally characteristic of dimers. There were a few studies where these type of states have been found.^[43] From the point of view of applications as a high

triplet host material for PLEDs, it is quite important to distinguish between these species. If it is true excimer, as described by Pope et al.^[41] or IUPAC (existing only in the excited state and dissociated in a ground state) then problems in PLEDs are caused only if there is charge recombination on PVK itself, i.e., only excitons residing on PVK might be trapped at these low energy sites, whereas triplets created on a dopant would still “see” the monomeric triplet level as ~ 2.9 eV. If, however, some of these traps are ground state dimers then this low triplet energy represents the lowest triplet energy of the system and excitons residing on dopants could be quenched by these host traps, therefore the effective triplet state of the host is ~ 2.5 eV. This could be the reason of underperformance of blue PLEDs. Consequently, in this article we set ourselves the task of proving or disproving the existence of ground state dimers having triplet character and confirming monomeric triplet levels of PVK and P2VK.

2. Results and Discussion

2.1. PVK

We confirm the native phosphorescence (PH) spectrum of PVK spin coated films, which is depicted in **Figure 1** observed at 14 K. A similar spectrum from PVK has been recorded previously by Burkhart et al.^[20] Highest energy electronic 0–0 emission peak is at 431 nm (2.88 eV) which is much higher than previously reported for PVK films by other groups (~ 2.5 eV).^[22,25,29,30] As already discussed,^[20,27,32] these lower energy features, at ~ 2.5 eV, are not inherent phosphorescence emission from PVK but arise from other low energy trap states. Furthermore, by using sensitized phosphorescence quenching Klopffer et al.^[22] have shown that these traps are populated via the triplet channel, probably by Dexter transfer, which is very reasonable in films where emissive sites can be within angstroms of each other, thus making this transfer much more efficient than in solutions, even if highly concentrated. The concentration of trap species might be extremely low, in the range of $5 \times 10^{-3}\%$ as conjectured by Klopffer et al.^[22] Hopping is very efficient in these types of films and triplets have long lifetimes

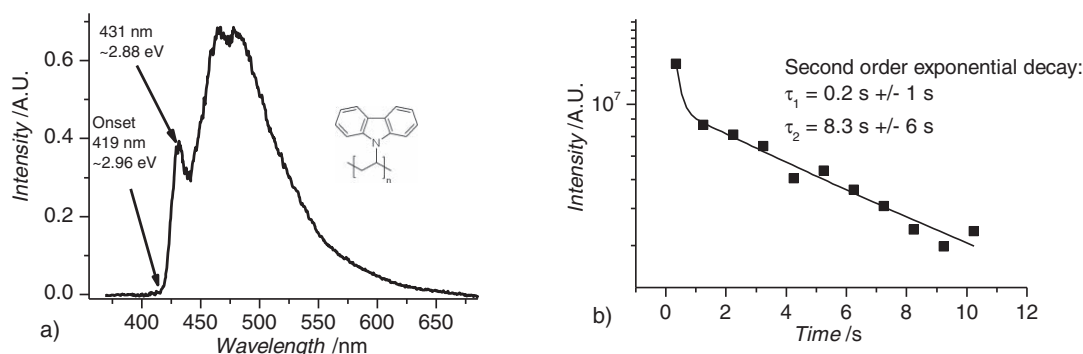


Figure 1. a) Phosphorescence spectrum of PVK film (structure depicted) recorded 800 ms after excitation at 14 K with 355 nm laser. Highest energy electronic 0–0 peak is at 2.88 eV. The relative ratio of all peaks do not change (up to ~ 10 s after excitation). b) Decay of phosphorescence of PVK film (at 430 nm) at 14 K excited at 355 nm. Two exponential lifetime constants could be fitted (0.2 s and 8.3 s).

to enable them to find a trap easily.^[36–38] However Chakrabarty et al.^[23] estimate that nearly all carbazoles are paired at 10 K. Bente et al.^[39] have performed calculation on bridged carbazole pairs, pointing out that the large red shift of the monomer to dimer phosphorescent cannot be simply attributed to excitonic delocalisation; substantial charge resonance effects must contribute, with large orbital overlap between the pairs. Thus, these triplet trap states will have significant charge transfer character and so be subject to solvatochromic effects. This will lead to strong broadening environmental effects in films.

Given the conjecture, the phosphorescence spectrum of PVK films should be highly temperature dependent as higher kT enhances triplet hopping. This behaviour we show in Figure 2. At 44 K, we observe a dramatic change in spectrum (in comparison with 14 K) with the delayed emission spectrum red shifting to an energy peak at ~2.55 eV. Increasing the temperature to 85 K we see further changes in spectral profile with increased red shift to ~2.41 eV.

Although it is difficult to prove the purity of the PVK used down to the ppm level, we think that this is not due to impurities as very similar spectra (especially those at ~44 K and above 85 K) have been recorded by other groups in the past.^[25,27] Furthermore, the PVK was synthesised by the groups which published the spectra^[22] and they used different polymerisation methods but measured similar PH emissions. Also, the lifetime of these different emissions is extremely long, in the range of seconds at low temperatures (Figure 1) and this long lived emission at such low temperatures could not be explained by carbonyl compounds which frequently are formed by degradational processes.^[27] Thus, we rule out the impurity or degradational product emission option in the case of low temperatures. However, at room temperature, new late emission bands from PVK film are observed which has been recorded 40 ms after excitation (see Supporting Information for spectra). Emission peaking at similar wavelengths (600 nm) at room temperature have been observed by Qian et al.^[35] in electroluminescence (EL) and photoluminescence (PL) spectra and assigned to excimer emission. As it is observed only at room temperature, at very

late times, one would expect it to be dominant at higher temperatures. This emission is however different for high molecular weight PVK (1100000) and low molecular weight PVK (90000) (see Supplementary Information). These facts and the very unconventional shape of the spectrum leaves us with the conclusion that it arises due to impurities or some remaining chemical reaction products not excimer or dimer states.

As can be seen from Figure 2, if one subtracts the spectrum at 14 K from the spectrum at 44 K, one obtains a spectra, which is very similar to the spectrum of PVK isolated in an inert matrix.^[21] Thus, we conclude that, even at 14 K, we have both emission from monomeric PVK phosphorescence as well as emission from “trap” states. This means that, at 14 K, the traps are still present. At 14 K, it is very unlikely there is excimer type emission given the requirement of activation energy needed to form the excimer.^[41]

Hence, we recorded absorption spectra of PVK in toluene at different concentrations at room temperature. Results depicted in Figure 3 indicate that at very high solution concentrations (~38 mg mL⁻¹) another absorption band (from 375 nm to 475 nm) is emerging, indicating the creation of ground state dimer species rather than dissociative in a ground state excimers as suggested previously. We would like to note that in most cases, when fabricating PLEDs, PVK is spincoated from comparable concentrations.

To confirm this, we excited PVK dropcasted film with a 450 nm laser and indeed observed emission peaking at ~2.5 eV (Figure 3) similar to the trap emission spectra in Figure 2. This is firm evidence confirming the existence of ground state dimers. Also, we point out that the spectra of this dimer has well resolved excitonic structure and clearly shows two vibronic peaks both at low temperatures and at room temperatures (Figure 3b and Figure SI2). This rules out that this emission comes from Birks^[42] type excimer which is dissociative in the ground state but not dissociated as it should be accompanied by very broad unstructured emission as demonstrated for example by Hayer et al.^[43] or Gierschner et al.^[44] Also, the triplet trap state spectra detected by Burkhart et al.^[25] show similar

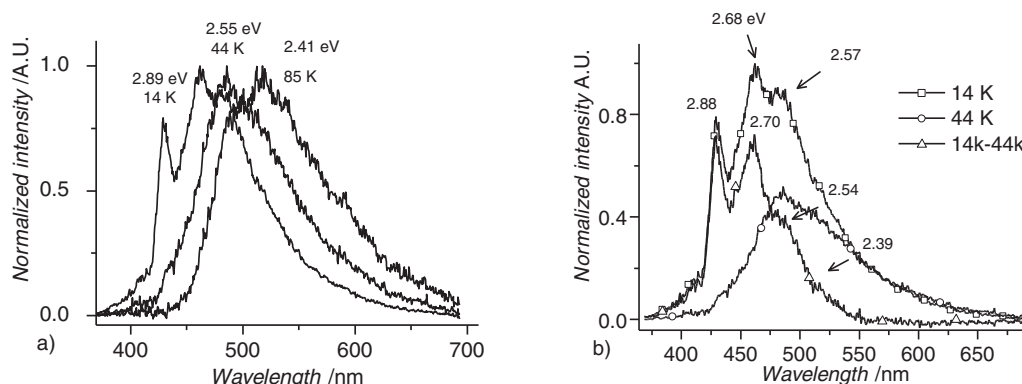


Figure 2. a) Delayed emission spectra of PVK film recorded 70 ms after excitation at 14 K, 44 K, and 85 K (excited with 355 nm laser). At 14 K, ~400–410 nm the leftovers of delayed fluorescence is still seen which changes the peak to peak ratio of the 14 K spectrum (compare with Figure 1). b) The tail from ~550 nm to 675 nm of 44 K spectrum is aligned with the tail of 14 K spectrum and then subtracted. The resultant spectrum, very similar to PVK in dilute matrix spectrum recorded by Pina et al.,²¹ clearly indicates that at 14 K, emission from trap species is observed. Peak energy values are indicated (in eV) on the graph in order to emphasize that after subtraction difference between vibronics of spectrum is closer to ~0.17 eV which could be C–C double bond vibrations.

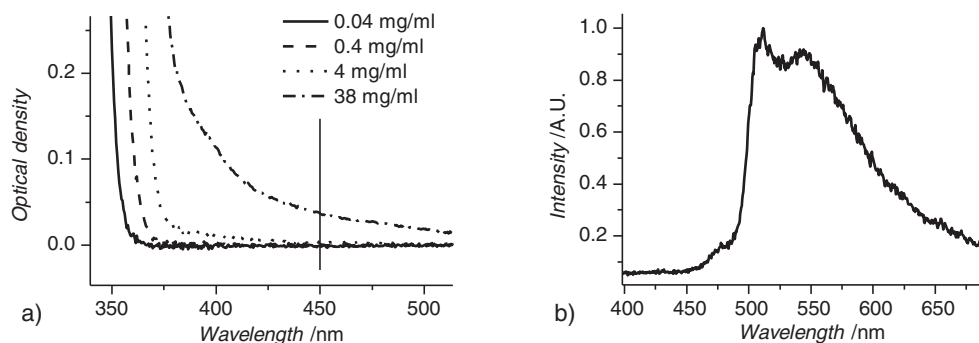


Figure 3. a) Absorption spectra of PVK in toluene at various concentrations at room temperature. b) Time resolved spectrum of PVK dropcasted film recorded 12 μ s after excitation at 14 K. Excited with 450 nm wavelength into the emerging dimer – ground state – band as shown in (a). Similar spectra in microseconds after excitation are observed at all temperatures up to room temperature although having different lifetimes (see Figure 4). Similar spectrum has been previously observed^[29] and assigned to monomeric triplet emission of PVK; here we assign it to dimer triplet emission (see text for more details).

excitonic features more characteristic of dimer emission rather than excimer emission. Finally, it has to be stated that the aim of this paper to prove the existence of ground state triplet traps capable of trapping phosphorescent blue dopants in PLEDs where PVK is used as a host has been achieved, irrespectively of nomenclature.

Now we will turn our attention to the question of how those dimer states are populated. These dimer species decay mono-exponentially at 295 K, 100 K, and 85 K (980 ns, 2190 ns, and 2640 ns respectively) and biexponentially at 44 K and 14 K (Figure 4). These lifetimes clearly indicate the triplet character of this emission. Thus spectra of PVK at 14 K, 44 K, and 85 K recorded in millisecond time region when excited at 355 nm (Figure 2) is a mixture of PVK monomeric triplet emission and triplet emission from dimer states. There are two pathways for the population of triplet dimer states. When exciting PVK film at 355 nm into the main PVK monomer absorption band (at 44 K) similar dimer spectra to that shown in Figure 3b is observed in the microsecond time region having very similar

lifetimes to that found in PVK film excited at 450 nm directly to the dimer absorption band (Figure 4b). We suggest the following scenario – PVK monomer singlet is excited at 355 nm, excitation is transferred to the dimer singlet and later to the dimer triplet via intersystem crossing, i.e., monomeric PVK singlet > dimer singlet > dimer triplet (gives emission in microsecond regime similar to the “natural” dimer emission when exciting at 450 nm to dimer absorption band). Another possible pathway is that excitons in directly excited monomer PVK singlet state are intersystem crossed into the triplet level of monomer PVK. These triplets are longer lived (Figure 1b) thus they can hop in a film and with time the probability to find and populate the dimer triplet state increases (i.e., monomeric PVK singlet > monomeric PVK triplet > dimer triplet). Consequently, at very late millisecond-range times, emission from both species is observed (monomer triplets and dimer triplets), with very similar lifetime and mixed spectra with ratio of dimer emission and PVK monomer emission depending on hopping rate, i.e., on temperature (Figure 4b and Figure 2). If dimer

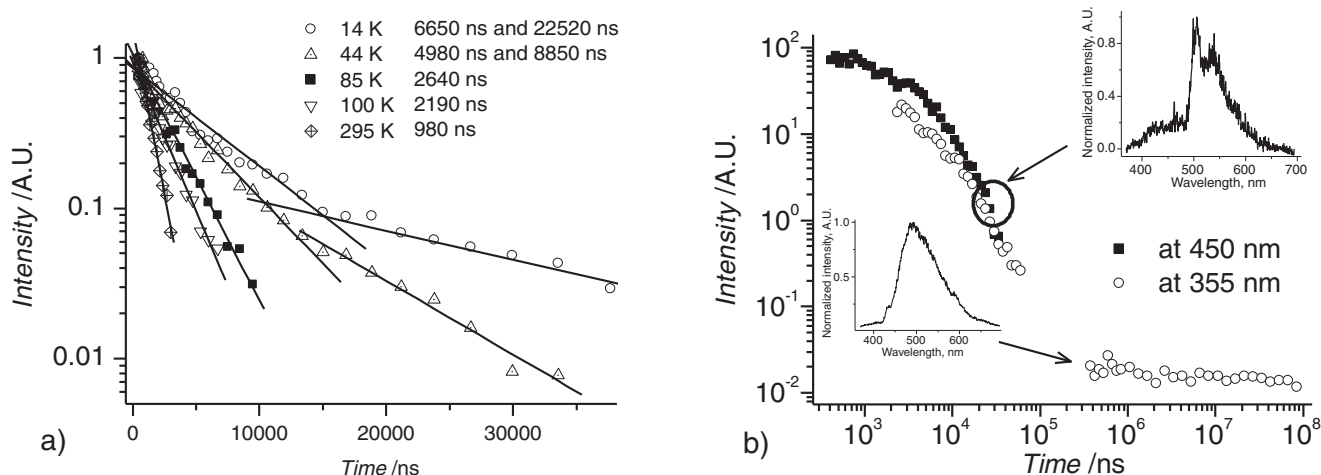


Figure 4. a) Decay curves of dimer emission shown in Figure 3b when excited at 450 nm. Straight lines are linear fits in log–lin scale and fitted lifetimes are indicated at each temperature. Intensity is normalized for clarity. b) Decay times of dimer emission at 44 K shown in Figure 3b when excited at 450 nm and at 355 nm (collected at 550 nm). Time resolved spectra for each time regime indicated with arrows. Top is a spectrum in microsecond regime – solely dimer emission (for both excitation wavelengths) and bottom is in millisecond regime – mixed emission from dimer and monomeric PVK emission.

triplet states were populated via the singlet dimer channel *only* (not via monomeric PVK triplet) we would not see emission from dimer sites at such late times, as their natural decay is in the microsecond range at these temperatures (Figure 4a). And indeed, review of literature confirms this notion – Klopffer et al.^[22] using guest quenchers in PVK proved that those triplet trap states, which here is ascribed to dimer species, are at least partly populated via triplet channel.

The existence of ground state is confirmed by the independent study by Bente et al.^[39] on model systems. They synthesised triply bridged carbazophanes where the dihedral angle and the separation distance between fully overlapped pairs of carbazole rings was changed systematically from nearly parallel to oblique by controlling the number of methylene groups in between carbazoles.^[39] They found that when the angle between the carbazole planes is changed from oblique to parallel the “phosphorescence emission” in rigid diluted glass shifts from ~2.88 eV to ~2.5 eV. Furthermore, they observe a new absorption band increasing with increasing planarity of the carbazole pairs what clearly shows that some of those triplet states exist in the ground state.^[39]

2.2. P2VK

If these trap states are the result of a ground state interaction between adjacent pairs of carbazole units on a single backbone, or with units on neighbouring chains, presumable from some form of interdigitation, changing the substitution point of the carbazole ring connection to the backbone of polymer or adding substitution on the carbazole units should affect dimer formation. This we explore using the PVK homolog, poly(*N*-ethyl-2-vinylcarbazole) (P2VK).

In Figure 5, we compare the temperature dependent and matrix isolated P2VK phosphorescence spectra with that previously found for PVK. The phosphorescence spectrum of dilute

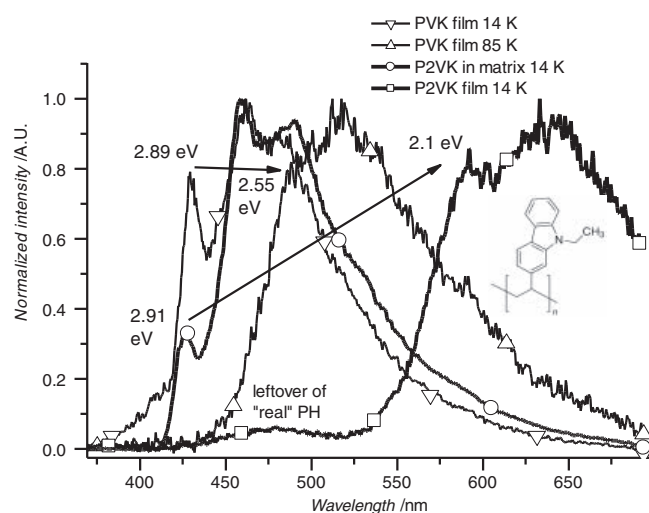


Figure 5. Delayed emission spectra of PVK film recorded at 14 K and 85 K, P2VK film at 14 K and P2VK (structure depicted) dispersed in zeonex matrix at 14 K (shape similar to the spectra recorded at 44 K and 85 K). Recorded 70 ms after excitation.

P2VK in zeonex matrix shows the characteristic peak at ~2.91 eV which is similar to parent PVK which can be recorded at 15 K. However, the spectrum of spin coated films of P2VK even at 14 K shows a dramatic red shift of the peaks to ~2.1 eV, which is a surprising result. Also we note that this late emission spectrum has at least two vibronic features. We think that in this case we have similar triplet character dimer states to PVK but the carbazole unit is more easily accessed and the degree of freedom for the unit to reorientate is increased in the solid state enabling better phase to phase dimer formation having better orbital overlap and subsequently even lower dimer triplet energy than that of the parent PVK.

We also have measured the change of absorption spectra with increasing concentration. Figure 6 shows clear evidence for the formation of new ground state absorption at high concentration. The concentration at which this new band arises is much lower than the concentration for parent PVK (compare with Figure 3).

Additionally, we have recorded steady state emission spectra of low concentration solution of P2VK in toluene (0.01 mg mL⁻¹) and highly concentrated solution (4.7 mg mL⁻¹) as a function of excitation wavelength (Figure 6b). If excited at 355 nm, low concentration solution emits at ~390 nm and does not emit when excited at 450 nm. At high concentration, we observe the same emission at 390 nm when excited at 355 nm; however, if we then excite at 450 nm we also observe a new band at ~525 nm. This clearly indicates that it must exist as a ground state to be able to be directly excited. When we record the emission spectrum of spincoated film of P2VK, we observed emission only at 525 nm, irrespective of which wavelength we excited, indicating increased energy transfer via migration. This is very similar to previously observed behaviour of polyfluorene^[40] or 4,4'-*N,N'*-dicarbazolyl-1,1'-biphenyl^[38] containing low energy trap states.

As we suggest these dimer states to be due to intrachain dimer formation, a question might arise as to why they are not observed at low concentrations. This probably happens due to the fact that, at lower concentration, molecules are better solvated and solvent molecules are present between the neighbouring carbazole rings, hence preventing dimer formation. Whereas, if the concentration is higher, P2VK (or PVK) do not solvate so well and dimers are more likely to be formed, which is confirmed by the additional absorption band emerging at higher concentrations. The low possibility for triplets to hop via Dexter transfer is probably the reason why the triplet spectrum of isolated P2VK (or indeed PVK) in dilute host matrixes was not quenched as host (zeonex) inhibits the freedom of carbazole rings.

3. Summary

In conclusion, we show conclusively the intrinsic phosphorescence spectrum for PVK film and verify that the triplet level of this popular host and hole transport material is 2.88 eV. However, we also observed triplet trap species to which mobile PVK triplets can migrate and are trapped at temperatures >44 K. We also conclusively show that some of these triplet trap states are formed as a result of a ground state interaction between neighbouring pendant carbazole units and so the lowest energy

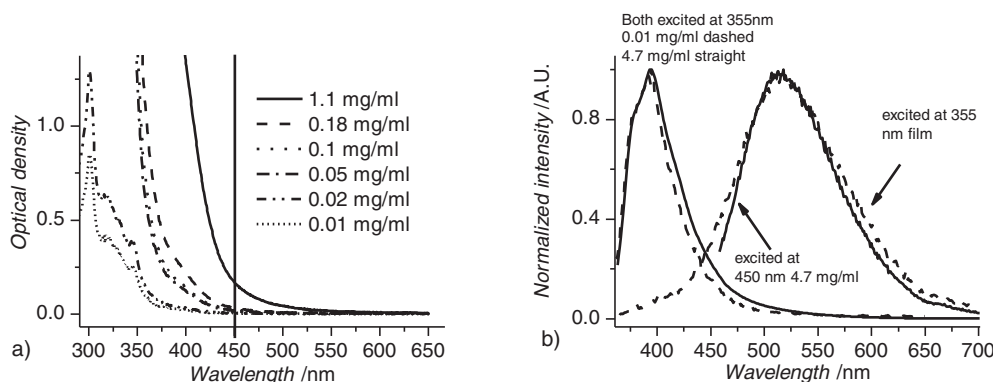


Figure 6. a) Absorption spectra of P2VK in toluene at various concentrations. b) Steady state spectra of P2VK in toluene solution excited at 355 nm (0.01 mg mL^{-1} dashed and 4.7 mg mL^{-1} straight on the left) and excited at 450 nm (4.7 mg mL^{-1} – straight line indicated by arrow). Steady state spectra of P2VK film excited at 355 nm (dashed line indicated by arrow).

triplet state in PVK films are these dimers with a triplet energy of 2.5 eV. Changing the chemical structure of the pendant carbazole group can cause further lowering of this triplet energy, as in the case of the analogue P2VK. These dimer traps are readily populated by triplet migration which turns on at very low temperatures. This is why the trap emission dominates at temperatures $>44\text{K}$ and so intrinsic PVK film phosphorescence has not been previously seen before as most of the spectra have been recorded at 77 K .^[25] In regard to using PVK and analogues as host materials for blue phosphors, it is clear that the mechanism of charge recombination is vital. If recombination occurs on the host then there will be a direct competition for exciton capture by the intentional phosphor dopant and the dimer traps. If, on the other hand, recombination occurs via charge trapping on the phosphor dopant site then it may be possible to prevent quenching of the dopant excitons by the host dimer traps if their concentration can be kept to a very low level. However, it seems likely that this will not be the case and the dimer traps will quench a proportion of the phosphors, especially as the energy of the phosphor triplet reaches closer to that of the PVK host. During the PLED lifetime, more dimers may form (through effective thermal annealing) further reducing the device efficiency. Thus, PVK and its analogues may cause a new set of problems when used as a host for deep blue phosphorescent emitters.

4. Experimental Section

All samples for phosphorescence measurements were spin coated or drop casted (to achieve higher absorption when exciting with 450 nm) from toluene solutions of 20 mg mL^{-1} concentration on quartz or sapphire substrates of 12 mm diameter. PVK was received from Acros Organics (MW 90000) or Aldrich (MW >1000000). P2VK was received from Aldrich. Gated luminescence and lifetime measurements were made using a system consisting of excitation source, pulsed YAG laser emitting at 355 nm (EKSPLA and from CryLas GmbH). Samples were excited at a 45° angle to the substrate plane and the energy of each pulse was $\sim 30 \text{ }\mu\text{J}$. Coumarin 450 laser dye was pumped with 355 nm laser to get excitation at 450 nm (typically $\sim 20 \text{ }\mu\text{J}$ per pulse). Emission was focused onto a spectrograph and detected on a sensitive gated iCCD camera (Stanford Computer Optics) with sub nanosecond resolution. For low temperature measurements (down to 12 K), samples were

placed in the duplex cryostat. Details of these measurements can be found elsewhere.^[36] Steady state absorption and luminescence emission spectra of the solutions and films were recorded using a UV-vis spectrophotometer (UV-vis 3600 from Shimadzu) and a commercial spectrofluorimeter (Fluoromax from Jobin Yvon), respectively.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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