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Analysis of Exciplex Emitters[†]

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In this study we present a guide to understanding of exciplex state: from physicochemical background to experimental procedures. Literature review is supported by original experimental results that exemplify the possible approaches. We show the experimental relationship between emission energy of solid-state exciplexes and electrochemically-derived ionization potential and electron affinity of donors and acceptors.

Keywords: exciplex, photophysics, TADF, electrochemistry, charge transfer

1. INTRODUCTION

Exciplexes have been investigated for many years, however their successful application in efficient OLED devices is an achievement of recent works. [1-4] It has to be said that exciplex is a bimolecular excited state that is considered to be an excited state complex. [5,6] There are other bimolecular excited states, such as excimer (excited state dimer). [5-7] Exciplexes have been used in OLEDs i.e. to obtain white electroluminescence by mixing blue exciton emission with broad yellow-red exciplex emission. [8] Nowadays, the research focuses on a possibility to increase OLED efficiency by the use of exciplexes. When the electronic levels of donor and acceptor are properly confined, the exciplex can become a TADF (Thermally Activated Delayed Fluorescence) emitter. [1-4,9]

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However the formation of donor-acceptor excited state complexes is observed even in enough concentrated solutions [5,10], solid state blends are the most common medium used in investigations. In case of OLED application, solid state is the only medium where exciplex state can be investigated. Exciplexes were found to form in donor-acceptor blends (bulk exciplex) as well as on an interface between the donor and acceptor thin layers (interfacial exciplex). [11]

The main reason of researchers' interest on exciplexes is the ease to screen them and tune their emission. In case of classical donor-acceptor OLED emitters, such as TADF emitters, [12-15] donor and acceptor must be chemically bound to each other. That implies a need to perform one or more chemical reactions to achieve this goal in case of one D-A pair. A donor-acceptor exciplex blend is formed just by mixing the components, therefore one D-A combination can be obtained in a time from seconds to minutes, depending on the deposition method. Using one donor (or acceptor) and mixing it with several different acceptors (donors) with different electron affinity (ionization potential) it is possible to obtain a number of different emissive blends of totally different emission color. [4] A strong advantage of an exciplex emitter in OLED application is that by changing the ratio of donor and acceptor the hole/electron transport properties of the layer can be tuned. [3]

Since exciplexes have been successfully applied to OLED devices with external quantum efficiencies EQE > 5% [1-4], interest on those blends has increased. The high EQE of OLED devices has been achieved by the aim of TADF phenomenon.

This study performs a brief literature review on theoretical aspects of exciplexes as well as presents an original work that is used to exemplify the practical aspects of exciplex formation and analysis. This work also contains an analysis of exciplex energy performed by the aim of electrochemical and photophysical methods.

2. PHYSICAL BACKGROUND OF EXCIPLEX FORMATION, ORIGINS OF DELAYED FLUORESCENCE

Exciplex is an excited state complex, therefore it should not be considered as a complex formed already in ground state. Bimolecular excited states such as exciplex or excimer are formed solely after excitation of one of interacting molecules (A*) that forms a complex with another molecule that is in a ground state (B) to form an excited state complex (A*-B). [5,7,16,17] In case of excimer the molecules A and B are identical, whereas in exciplexes they are different and called donor and acceptor, respectively. The concept of a donor-acceptor exciplex material is identical to a donor-acceptor molecule with non-conjugated D/A units. [18] The similarity is pronounced in theoretical and experimental approaches and is presented in this work. The main difference between donor-acceptor bi- and unimolecular emitters is that dilution strongly affects the first and does not affect the latter.

Exciplex can be formed solely between molecules of ionization potential and electron affinity enabling electron transfer from donor to acceptor (FIGURE 1). The minimal requirement is that $EA_A > EA_D$ and $IP_A > IP_D$ what means that acceptor (A) should have higher electron affinity (EA) and ionization potential (IP) than the donor (D). If that requirement is not met, there is no driving force for charge transfer. In other cases the formation of exciplex is not possible and is usually superseded by energy transfer. [3,5,16]

From a kinetic point of view the formation of an exciplex starts with excitation of D or A molecule to give A^* or D^* excited state species. The molecule forms a complex with a partner in a ground state ($[A^*D]$ or $[AD^*]$). Another step is a charge transfer from D to A resulting in a formation of an intermolecular charge transfer excited state $[D^+A^-]^*$. [5,16,17,19]

Quantum mechanical description of an exciplex state comprises of a linear combination of excited state complexes (as described above) with $[D^+A^-]^*$ and $[D^-A^+]^*$ charge transfer states (2.1, 2.2, 2.3). The first are considered as local excited (LE) the latter as a charge transfer (CT) state. Please note that letters a, b, c with subscripts 1, 2 represent the contributions of a respective state to the linear combination representing the overall, local, and charge

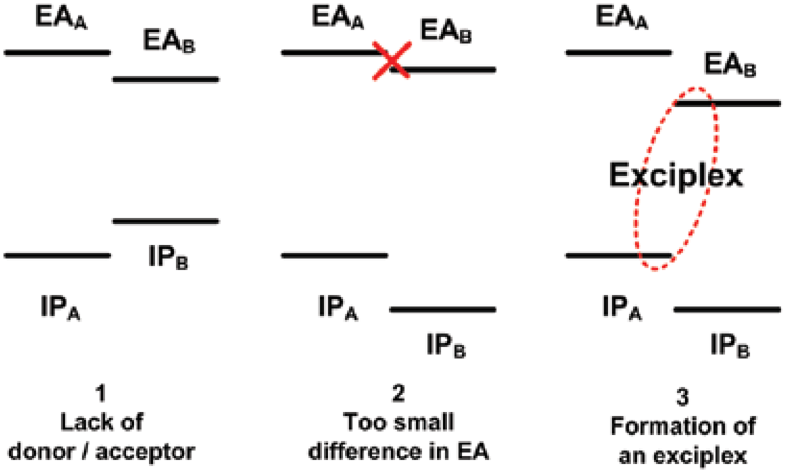

FIGURE 1

Typical relation of ionization potentials (IP) and electron affinities (EA) of organic materials. 1 – none of the materials is a donor / acceptor, charge transfer is not possible, energy transfer occurs; 2 – donor (A) and acceptor (B) have very similar electron affinities, what makes electron transfer inefficient; 3 – donor (A) and acceptor (B) with properly adjusted energy levels to facilitate efficient electron transfer, formation of an exciplex is possible.

transfer state, respectively. Knowing that charge transfer from acceptor (A) to donor (D) is unlikely, the expression describing this state can be removed from consideration. That means $b_2 = 0$ in equation (2.3). [5,7,19]

$$\left|DA\right\rangle^{*} = c_1\left|DA\right\rangle^{*}_{LE} + c_2\left|DA\right\rangle^{*}_{CT} \tag{2.1}$$

$$\left|\mathrm{DA}\right\rangle^{*}_{\mathrm{LE}} = \mathrm{a}_1\left|\mathrm{D}^{*}\mathrm{A}\right\rangle + \mathrm{a}_2\left|\mathrm{DA}^{*}\right\rangle \tag{2.2}$$

$$\left|DA\right\rangle^{*}_{CT} = b_1\left|D^{+}A^{-}\right\rangle + b_2\left|D^{-}A^{+}\right\rangle \tag{2.3}$$

The model of exciplex that explains its photophysical properties consists of local excited states of donor (LE_D) and acceptor (LE_A) as well as of an intermolecular charge transfer state (CT). [9] All considered excited states comprise respective singlet and triplet states. This description is actually identical to that of a typical TADF emitter [12-15,18] since there is no actual conjugation between donor and acceptor in such a molecule – like in an exciplex. It has already been proven that the concept of TADF in exciplex is identical to that of an unimolecular TADF emitter. [9] As a result the origin of a highly efficient E-type delayed fluorescence in such a blend is in a coupling between a triplet locally excited state of donor or acceptor and a charge transfer state of an exciplex (FIGURE 2). When the energy of a CT state is much lower or much higher than the energy of the lowest local triplet state then the coupling is ineffective and as a result there is no TADF observed or it is inefficient. Because formation of an exciplex requires a highly concentrated medium (solid state blend, concentrated solutions), therefore in the absence of E-type delayed fluorescence P-type delayed fluorescence (triplet-triplet annihilation) is likely to be observed. [3,9,20]

3. PHOTOPHYSICAL ANALYSIS OF EXCIPLEX BLENDS

As it has already been described, the ease of producing exciplex blends allows to screen numbers of mixtures in a reasonable time. The problem is that a donor-acceptor blend which is characterized in solid state might not be homogeneous. As a result observation of not only exciton but also excimer emission is very likely. [21]

To obtain an exciplex blend, two materials must be mixed with each other. According to the theoretical approaches, the exciplex forms as a complex of two molecules, therefore an equimolar ratio of donor to acceptor is expected

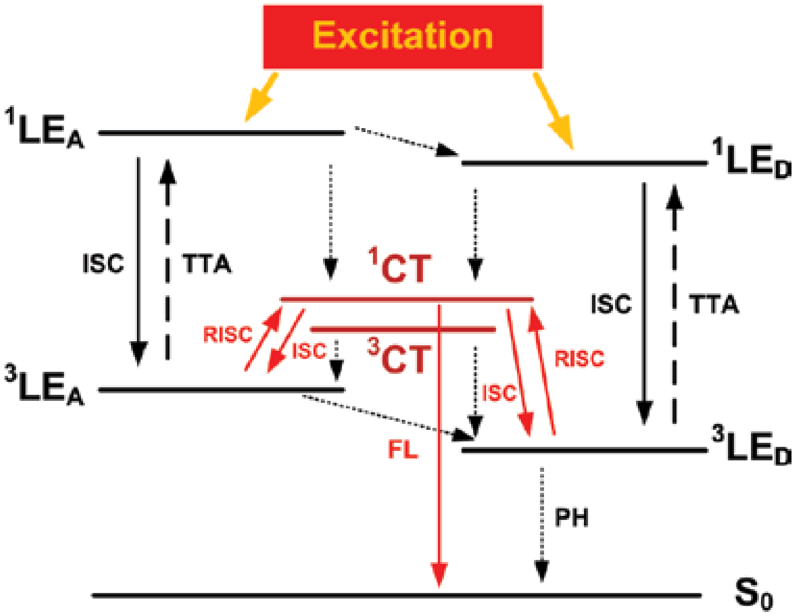

FIGURE 2
Electronic transitions diagram of an exciplex blend under photoexcitation. Red arrows represent the possible pathways of efficient TADF emission. Dashed arrows (TTA) indicate possible pathways of up-conversion giving rise to P-type fluorescence of an exciplex. Dotted lines represent energy transfer processes or phosphorescence (PH). Please note the ^{1}CT – ^{3}CT exchange is irrelevant and can be removed from consideration. LE – local excited state; CT – charge transfer excited state; S_0 – ground state; ISC – intersystem crossing; RISC – reverse intersystem crossing; FL – fluorescence; PH – phosphorescence. Numbers in superscript indicate singlet (1) or triplet (3) state. Letters in subscript indicate electronic states of donor (D) or acceptor (A). Scheme adapted from previous work [9].

to be optimal. It is worth to note, that the actual ratio of those components may be different in an OLED device to confine both hole and electron mobility. There are several ways to perform this, including wet and dry processes. In example drop-cast or spin-coat from a solution of two materials are an easy way to obtain exciplex blend. The best way is to use more concentrated solutions (2-30 mg/mL) to avoid crystalization during drying. Another approach is to prepare the blend by vacuum thermal evaporation or similar method. Researchers should be aware that the ratio of components obtained by coevaporation is neither a molar nor a mass ratio. In some cases the method of production of the blend may affect its photophysical properties, i.e. when the components are not stable in ambient conditions [21] or tend to form homoaggregates instead of mixing together. The method used for film production must always be considered accordingly to the properties of the layer.

The best way to actually find a promising donor-acceptor exciplex blend is to screen the materials that are potential donors and acceptors by mixing

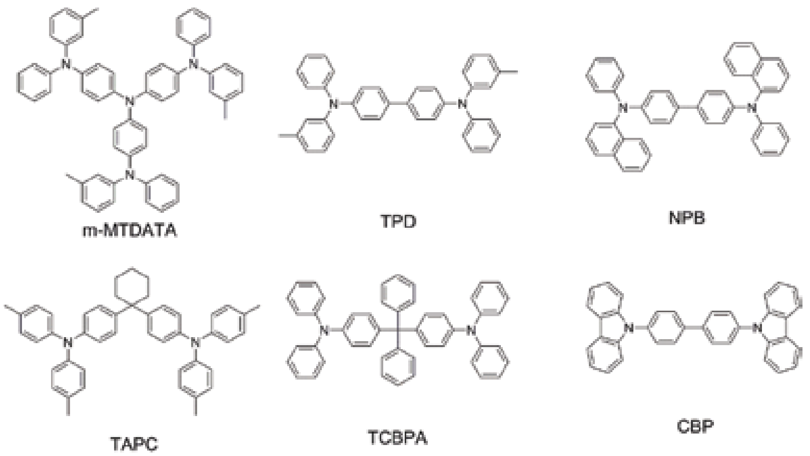

FIGURE 3
Commercially available electron-donating materials used in this work.

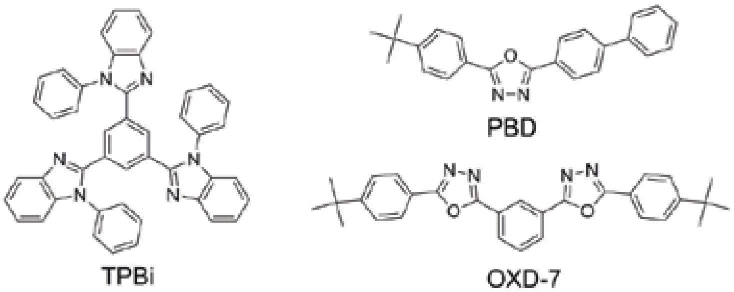

FIGURE 4
Commercially available electron-accepting materials used in this work.

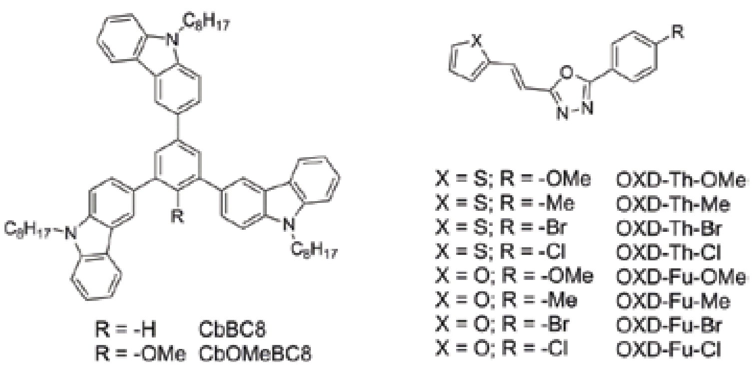

FIGURE 4
Commercially unavailable materials used in this work. These were described in previous works. [22,23]

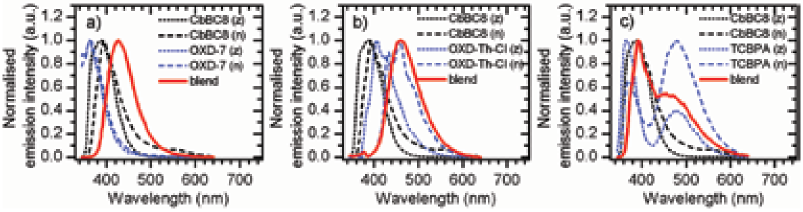

FIGURE 6
Steady-state fluorescence spectra of investigated materials: diluted in a transparent polymer Zeonex® (z), neat films (n), and an equimolar blend. Blends a) and b) show exciplex emission, whereas blend c) shows excimer emission of individual components and no exciplex is observed.

them together. It is possible to roughly predict that two molecules should form an exciplex, as it was described in previous text. In example, very many OLED electron transport materials form exciplexes with hole transport materials. [1,9,11,21] It is sometimes possible to reduce the number of screened blends i.e. by eliminating mixtures of two clearly electron-donating or electron-accepting materials that will not form an exciplex with each other, since there is no driving force for charge transfer.

Screening procedure is relatively fast if exciplex blends are produced by a wet process. Typically the absorption of such a blend is a sum of the absorption of both compounds before mixing them, but fluorescence shows large Stokes shift if exciplexes or excimers are formed. [1-5,21] Some exciplexes might not be very emissive, therefore it is recommended to note the relative intensity of blends' emission as a first approximation of their photoluminescence quantum yield. In the second stage the most luminescent exciplexes should be investigated, the rest can be removed from consideration if the aim of the work is to find promising OLED emitters.

To start screening a number of materials, researcher must firstly know fluorescence spectra of the compounds in a diluted solution (in a solvent or a polymer) and in pristine film (FIGURE 6). This knowledge is important to properly interpret the results derived from investigation of blends. When the two materials are mixed there are several species that can be found in the fluorescence spectra: **excitons**, **excimers**, and **exciplexes**. Sometimes excimer emission can be similar to the expected exciplex emission and it is very important to know not only the wavelength range of this emission, but also its shape. Exciplex emission has a charge transfer origin and is featureless, with Gaussian-like shape. [1-4] Any bands that show vibronic structure have exciton or excimer origin. In example, FIGURE 6b presents a situation where excimer emission of the acceptor almost overlaps with the emission of an exciplex, however the excimer emission is clearly vibronic and differs from featureless exciplex emission.

It has already been shown [9] that an efficient TADF emission arising from an exciplex requires a localized triplet state to be energetically close to the

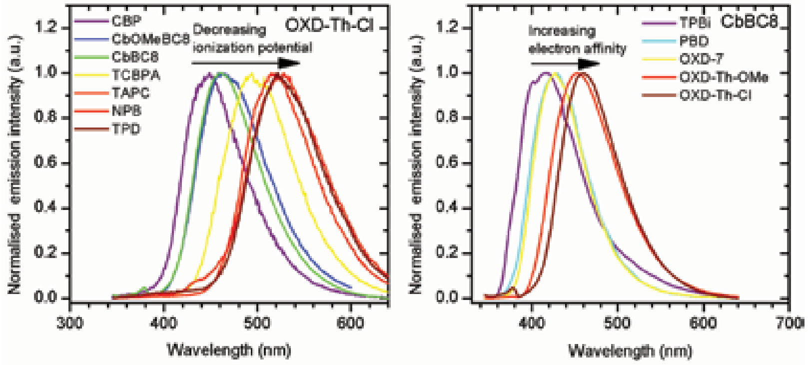

FIGURE 7
Steady state fluorescence spectra of exciplex blends obtained by various donors mixed with one acceptor (left) and various acceptors mixed with one donor (right).

CT state. Therefore, to have a full view it is recommended to screen the starting donor/acceptor materials to be aware of their triplet energy both in diluted samples (i.e. in polymer matrix) and in pristine films (possible excimer phosphorescence). If one of the components shows green or yellow phosphorescence (triplet energy < 2.5 eV) it is rather unlikely to observe TADF from an exciplex layer showing blue fluorescence (singlet energy ≈ 3 eV).

In some cases it is possible to form a broad range of different exciplexes using one material and changing the counterpart. I.e. when an electron accept-

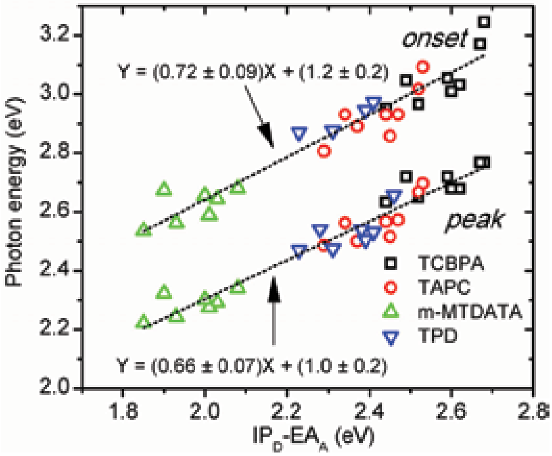

FIGURE 8
The relation between *onset* / maximum energy of exciplex fluorescence and electrochemical properties of donors / acceptors. This study was performed on oxadiazole acceptors (FIGURE 5) and triphenylamine-based donors (FIGURE 3). The graph legend shows which electron-donating compound was used to form each group of exciplexes.

ing material is used, the donors of increasing ionization potential should form different exciplexes, accordingly to the energy difference between electron affinity of the acceptor and ionization potential of the donor.

4. DETERMINING THE ENERGY OF EXCIPLEX STATE BY THE AIM OF ELECTROCHEMISTRY

Researchers have already noticed [3,5,19] that the energy of an exciplex derived from the emission spectrum and the expected energy calculated as a difference between the IP (ionization potential) of the donor and EA (electron affinity) of the acceptor obtained from electrochemical measurements do not correlate. To explain that difference many researchers use a simple equation (4.1), assuming that the difference can fully be explained by coulombic attraction of donor and acceptor in a charge transfer state. [5,24] The use of equation (4.1) requires an assumption that the exciplex is actually formed in gas phase and has pure charge transfer character. [5] The studies performed years ago [5,25] show that the charge transfer in an exciplex is not necessarily full in all cases, since there are exciplexes with a weak charge transfer, so called heteroexcimers. The work of Weller [5] shows that the actual exciplex energy in solution depends on several parameters (4.2). Interestingly, the energy of an exciplex is calculated either from an *onset* or maximum of the fluorescence spectrum, however the latter seems to be used more often. Nevertheless, the work [5] shows that the energy of an exciplex is not the energy of the photons at fluorescence maximum, neither *onset*. The exciplex energy cannot be derived directly from its fluorescence spectrum without considering the dissociation enthalpy of the exciplex (4.3). According to the study presented below, this enthalpy cannot be assumed to be ≈0 in solid state. If this assumption was true for solid state, the relation presented on FIGURE 7 should be proportional, as for heteroexcimers. [25] The dissociation enthalpy can be derived from the Van’t Hoff plot of exciplex to local emission ratio. [26]

$$E_e = IP_D - EA_A - C \tag{4.1}$$

$$E_e = E_D^{OX} - E_A^{red} + U_{dest} - U_{stab} + \Delta H_e^{sol} + 0.32 \tag{4.2}$$

$$E_e = hv_{0-0} - \Delta H_d \tag{4.3}$$

Where: E_e – exciplex energy, eV; IP_D – ionization potential of donor, eV; EA_A – electron affinity of acceptor, eV; C – coulomb attraction energy at equilibrium distance, eV; E_D^{ox} – electrochemical oxidation potential of donor, V; E_A^{red} – electrochemical reduction potential of acceptor, V; U_{dest}, U_{stab} – destabilization and stabilization energy, eV; ΔH_e^{sol} – solvatation enthalpy, eV. $h\nu_{0\text{-}0}$ – energy of the 0-0 transition, can be estimated by *onset* energy from the fluorescence spectrum, eV; ΔH_d – dissociation energy, eV.

In this section we present an original study of exciplexes formed by oxadiazole-based acceptors and triphenylamine-based donors in solid state to show the relationship between electrochemical data and photophysically-derived energy of exciplex fluorescence. This is to show a way to analyze that aspect and avoid traps and misunderstandings. The authors would like to stress that the study can be performed exclusively when both ionization potential and electron affinity of donor and acceptor, respectively, are recorded using the same experimental method. Since different methods might give slightly different results, therefore actual comparison of IP and EA obtained in alternative ways already bears an error. The use of equation (4.4) is not recommended in this case, since the use of optical band gap generates another error. [27] Usually, the acceptor can be reduced in a specified electrolyte, therefore EA can be obtained directly.

$$EA = IP - E_g^{op} \tag{4.4}$$

Where: EA – electron affinity, eV; IP – ionization potential, eV; E_g^{op} – optical energy gap, eV.

As it is seen on FIGURE 7 the slope of the relation is definitely different than 1 and is roughly equal to 0.7 for the photon energy derived from either *onset* or peak. Some works however show [5,10] that in case of some heteroexcimers, in solution, the proportional relation can actually be achieved. Another study, also performed in solution, showed that the experimental data corrected by the factor of dissociation enthalpy presented linear, proportional relation of exciplex energy vs. electrochemical oxidation/reduction potential difference of donor/acceptor. [5,26] Assuming that this is also the case in solid state exciplexes (FIGURE 7), the ΔH_d increases with the increase of exciplex energy. This study presents consistence with previous experimental investigations on heteroexcimers [5,26], since the closer the exciplex energy gap is to the energy gap of its components, the weaker is the intermolecular CT, and the exciplex actually becomes closer to the concept of heteroexcimer.

It has been observed [26] that exciplexes in solution undergo two exponential temperature-dependent processes: currently we can interpret these results that the first one is related to delayed fluorescence of the exciplex (increase of the CT emission with temperature in low-temperature region), the latter with the process of exciplex dissociation that occurs in higher temperatures and causes decrease in the CT emission with increasing temperature. From this high-temperature profile the dissociation enthalpy can be derived.

5. CONCLUSIONS

We have presented a guide for those who are not familiar with exciplexes to understand the basic theoretical and experimental challenges. This is only a guide and the work is not exhaustive, thus it is highly recommended to refer to the cited literature for details. This work shows possible results obtained during screening of materials in the search for exciplex blends. It has been stressed that an efficient TADF exciplex requires a local triplet state lying energetically close to the charge transfer state. An experimental study, supported by literature data, showed that proper determination of exciplex energy requires dissociation enthalpy to be considered also in the solid state, whereas other, simplified approaches, usually lead to misleading results.

6. ACKNOWLEDGMENT

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