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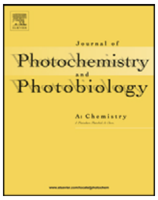

Photophysics and electrochemistry of quinoxaline chromophores decorated with thiophene or furane subunits

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ABSTRACT

The photophysical and the electrochemical behaviour of thienyl and furyl derivatives of quinoxaline and their covalently linked dimers have been studied in solution at room temperature and in glasses at low temperature. The obtained results are compared to those obtained for the 2,3-dimethyl quinoxaline and its 5,5′-dimer. All the compounds show quite negative reduction potentials and, except for the dimers, low fluorescence quantum yields and short lifetimes. The most likely deactivation pathway is through intersystem crossing to the triplet state, as the laser flash photolysis data show. The deactivation of the triplet state is proposed to be accelerated by the thiophene/furane triplet state mixing with that of the quinoxaline.

1. Introduction

In the last years there has been a lot of interest in thiophene polymers and co-polymers because of their potential applications in organic electronics [1]. The first compulsory step in their study is the characterization of the monomers photophysics and electrochemistry. Among the proposed compounds, many hetero-aromatic rings have been studied like pyrroles, pyridines, pyrazines or carbazoles. Indeed, any stable reducible organic compound with sufficiently low reduction potential is of special interest as possible alternative to fullerenes as *n*-type semiconductor material for all-organic solar cells [2]. Hetero-aromatic hydrocarbons usually undergo reversible electrochemical reduction at moderately negative potentials with the formation of stable radical anions contrary to their irreversible oxidation in solution occurring at very positive potentials. Quinoxalines have neither escaped this interest and their substituted thiophene based co-polymers have been recently used in solar cells [3,4] and proposed for other applications in other fields of organic electronics [5]. A very attractive and relatively easy pathway for the synthesis of such polymers is electro-polymerization under oxidative conditions. It can be realized by the attachment of any electro-polymerizable group (*e.g.*, thiophene or furane) to the hetero-aromatic kernel of interest. In fact, 2,3-dithienylquinoxaline has been successfully electro-polymerized in the past [6]. On the other hand, quinoxalines themselves are known for their high intersystem crossing rates and low fluorescence yields [7–9] what may limit their potential application as active materials for organic electronics devices.

In our laboratory we are searching for possible candidates for *n*-type semi-conductors for all-organic bulk-heterojunction solar cells. This paper describes the efforts made to understand the photophysical properties of the 2,3′-dithienyl and 2,3′-difuryl substituted quinoxaline and their dimers in solution in order to elucidate whether they are suitable for the above-mentioned applications. The difuryl and dithienyl monomers are compared to the 2,3-dimethylquinoxaline taken as a reference. Then, in order to check the behaviour of linked quinoxalines we have also synthesized dimers of all the three former compounds with direct linkage of both quinoxaline subunits. Their photophysical properties are discussed with special attention to the non-radiative processes responsible for the short emission lifetimes observed and of the deactivation of the triplet state. Additionally, the electrochemical polymerization has been tried for all the investigated molecules at different conditions.

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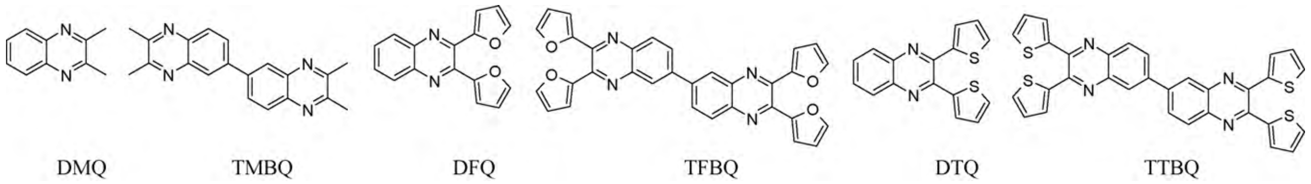

Fig. 1. Structures and short names of the investigated monomeric and dimeric quinoxaline derivatives.

2. Experimental

2.1. Synthesis

2,3-Dimethylquinoxaline (DMQ), 2,3-dithienylquinoxaline (DTQ) and 2,3-difurylquinoxaline (DFQ) have been synthesized according to procedures described in the literature [10–12] by means of condensation of *o*-phenylenediamine with butano-2,3-dione, 1,2-di(2-thienyl)-1,2-ethanedione, and 1,2-di(2-furyl)-1,2-ethanedione, respectively. Dimeric quinoxaline derivatives (TMBQ, TTBQ and TFBQ, correspondingly) have been synthesized in the same manner using 3,3′-diaminobenzidine as an active amine reactant. All the chemicals used in syntheses have been purchased from Aldrich and were used as received. Structures of the synthesized substances (Fig. 1) have been confirmed by means of MS and NMR spectra.

2.2. Electrochemical methods

Electrochemical measurements have been carried out with an AutoLab PGSTAT30 potentiostat (ECO-Chemie) in a standard three electrode electrochemical cell with a Pt disk of 2 mm diameter, a W wire, and a Pt solenoid wire served as working, quasi reference, and counter electrode, respectively. Electrochemical experiments have been performed in deoxygenated acetonitrile – ACN (Aldrich, 99.8% “water free” used as received) or dichloromethane – DCM (Aldrich, >99.5% ACS spectrophotometric grade) solutions containing 0.1 M of tetra-*n*-butylammonium hexafluorophosphate – $(n\text{-}C_4H_9)_4PF_6$ as supporting electrolyte (Aldrich, electroanalytical grade dried over night at 80 °C before use). All the reported potentials are referred to ferrocene/ferrocene$^+$ (FC/FC$^+$) redox system as internal reference.

2.3. Photophysical methods

All the spectroscopic measurements have been performed in solvents of ACS spectrophotometric grade purchased from Aldrich. Steady-state UV–vis absorption measurements were done with a Shimadzu UV3100 absorption spectrometer. Stationary emission, luminescence and phosphorescence spectra were recorded with a Jasny [13] or an Edinburgh FS 900 fluorescence spectrometer. Fluorescence quantum yields were determined using quinine sulphate in 0.05 M H_2SO_4 solutions as the standard with $\phi_{em} = 0.51$ [14]. The fluorescence decays in the nano-second time domain were recorded with an Edinburgh FS 900 CDT instrument operating in TCSPC mode with measurements limit about 200 ps.

Nano-second time resolved transient absorption spectra and kinetics were recorded with a home-built apparatus [15]. The spectrophotometer allows one to measure transient absorption spectra in the spectral range from 380 to 790 nm at delays ranging from −2 to 100 ns in relation to the excitation pulse (PTI PL 2300 nitrogen laser, FWHM = 0.6 ns, pulse energy about 1 mJ) with a temporal resolution better than 1 ns. Pico-second time resolved transient absorption spectra were recorded with a home-built apparatus described elsewhere [16,17]. Light pulses (1.5 ps duration, energy of 4 mJ, and repetition rate of 33 Hz) generated by a Light Conversion neodymium glass laser have been used (after splitting into two channels) for excitation (after conversion into the third harmonic with 352 nm wavelength and about 1 mJ energy) and for generation of the pico-second continuum used as a probing beam. As the detection system a home-built polychromator equipped with a Hamamatsu back-thinned, one-stage TE-cooled element (S7031-1007) was used. The apparatus allows measurements in the 400–750 nm range with temporal time resolution of 2.5 ps.

3. Results and discussion

3.1. Electrochemical behaviour

Monomers DMQ, DTQ and DFQ were studied in ACN and in DCM solutions while TTDQ and TFBQ (due to their low solubility in ACN) only in DCM. TMBQ has been investigated only in ACN solutions because of its reduction potential being too negative for the DCM electrochemical window. All the six studied compounds can be reduced reversibly (Fig. 2 and Table 1) and all of them show irreversible oxidation (not shown). The oxidation in the case of the DMQ and TMBQ compounds shows an amount of charge similar to their reduction processes. This is not the case for the thienyl (DTQ or TTBQ) and furyl (DFQ or TFBQ) derivatives that display very broad oxidation peaks with up to 10-fold in charge characteristic for their reduction processes. It can be also inferred that the quinoxaline moiety irreversibly oxidizes in the same potential regions (at only somewhat more positive values) as characteristic for the thiophene and furane moieties. This makes the electro-polymerization of the discussed compounds *via* oxidation of thiophene or furane subunits quite difficult because further and undesired oxidation of the quinoxaline core takes also place. Despite that, the successful production of the 2,3-dithienylquinoxaline electro-polymer seems to be possible as it has been already reported in the literature [9]. We could reproduce the reported electro-polymerization and observe an increase in the overall current concomitant to the deposition of a black solid on the Pt surface electrode, however only in DCM solutions. For the furane derivatives all efforts in this direction did not lead to any stable polymers.

As it can be appreciated from Fig. 2 and Table 1, the addition of thiophene or furane subunits to the quinoxaline kernel has a

Table 1
Redox potential for the investigated quinoxaline and 5,5-biquinoxaline derivatives. Values (in volts) *vs.* FC/FC$^+$ internal reference redox system in solutions containing 0.1 M $(n\text{-}C_4H_9)_4NPF_6$ as supporting electrolyte.

Compounds	Solvent	1st reduction step	2nd reduction step
Monomeric derivatives			
DMQ	ACN	– 2.28	
DFQ	ACN	– 1.90	
	DCM	– 1.99	
DTQ	ACN	– 1.94	
	DCM	– 1.99	
Dimeric derivatives			
TMBQ	ACN	– 2.13	– 2.33
TFBQ	DCM	– 1.86	– 2.05
TTBQ	DCM	– 1.87	– 2.02

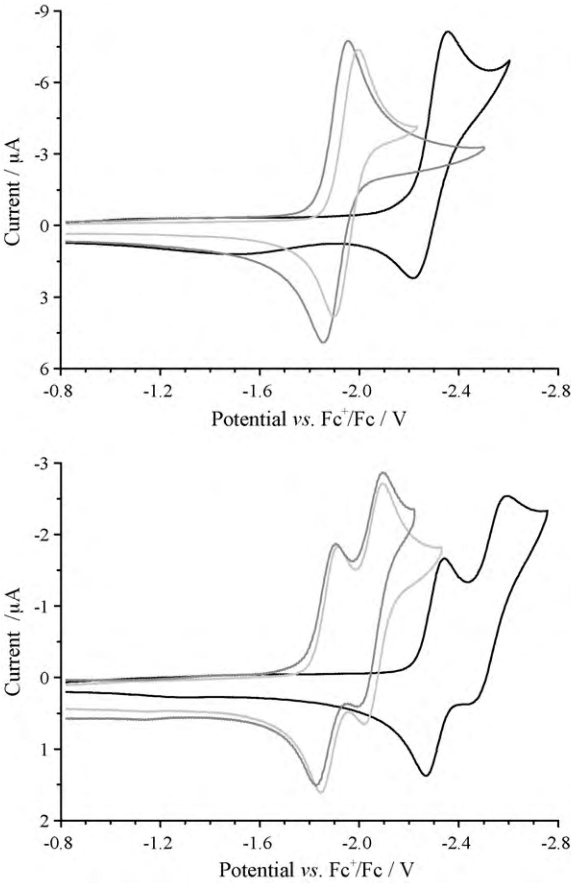

Fig. 2. Cyclic voltammetry curves recorded in 1 mM solutions of the investigated monomeric (top) and dimeric (bottom) quinoxaline derivatives in acetonitrile (DMQ, TMBQ, DFT and DTQ) or dichlormethane (TTBQ and TFBQ) solutions containing 0.1 M of $(n\text{-}C_4H_9)_4NPF_6$ as supporting electrolyte. Data for DMQ and TMBQ (black lines), DTQ and TTBQ (light grey lines), and DFQ and TFBQ (dark gray lines). Scan rate = 100 mV/s.

very similar effect on the reduction potential of the molecules, shifting it *ca.* 300 mV to more positive values. In the monomeric derivatives the effect of these substituents is of similar magnitude as to that found for dimeric ones. It is also obvious that the double reduction peak of the latter comes from their ability to accept two electrons per molecule, one per quinoxaline unit. Differences between reduction potential values characteristic for the first and second reduction steps are rather small. Moreover, from the comparison of monomers with dimers, it can be also stated that the dimerization has no remarkable effect on the first reduction process. Both facts are suggesting only weak electronic interaction between both quinoxaline cores in the TFBQ or TTBQ dimers. None of the investigated molecules show appropriate electron affinity for their potential application as *n*-type semi-conductors in polymeric solar cells. Their reduction potentials are too negative for effective charge separation in the electron transfer reaction with most of the *p*-type counter polymers. Further extension of quinoxaline aromatic system seems to be absolutely necessary to consider using quinoxaline based materials in solar cell application (*cf.* [18]).

3.2. Stationary absorption and luminescence spectra

The absorption spectra of the monomeric quinoxaline derivatives in toluene solutions are placed in the near UV region as can be seen from Fig. 3 and Table 2. Upon substitution of the methyl groups by thienyl or furyl moieties, the first absorption band shifts bathochromically by almost exactly the same amount of 5400 cm^{-1}. The same trend was observed for the dimeric derivatives, though all the three are about 2000 cm^{-1} more red-shifted compared to the monomeric ones. Respect to the extinction coefficients the introduction of furyl or thienyl substituents doubles it, while the dimerization further increases this quantity roughly by a factor of two what seems to be understandable.

For DMQ and TMBQ molecules their luminescence bands are localized in the same spectral region contrary to that observed for the UV–vis absorption spectra. This finding can be rationalized assuming that the first absorption bands of DMQ and TMBQ molecules are a superposition of at least two electronic transitions which differently respond to dimerization (*e.g.*, due to different symmetry of the excited singlet states involved in the given transitions). In the case of DFQ or DTQ their fluorescence bands are bathochromically shifted by *ca.* 3000 cm^{-1} compared to DMQ. Emissions from dimeric TFBQ and TTBQ are further shifted by *ca.* 200–400 cm^{-1} with respect to that recorded for the monomeric DFQ and DTQ derivatives. This rather small shift in the emission maxima does not reflect the quite evident changes in band shapes. Taking into account short wavelength tails of these bands one can conclude that dimerization forces similar batochromic shifts in both, absorption and luminescence spectra. The above-described finding allows concluding that both, substitution and dimerization are working in the same direction lowering the energy of the first excited state in the investigated molecules. Replacement of methyl groups by furane or thiophene subunits shifts the energy of the emissive ^{1*}DFQ and ^{1*}DTQ states by *ca.* 0.4–0.6 eV whereas dimerization leads to a further shift by *ca.* 0.25 eV. Dimerization effects are distinctly less pronounced compared to the substitution what remains in nice agreement with the electrochemical data.

Measurements performed in more polar solvents (ACN for the monomeric and benzonitrile for the dimeric species) point to a lack of distinct changes in the absorption and fluorescence band positions. This reflects that the excited state dipole moment values must be comparable to those of the ground state and rules out the possibility of intrinsic charge transfer in the lowest excited states of the investigated molecules. In fact, a simple calculation taking into account the redox potentials of the quinoxaline and thiophene or furane moieties, leads to CT state energies larger than that of the singlet states by almost 1 eV.

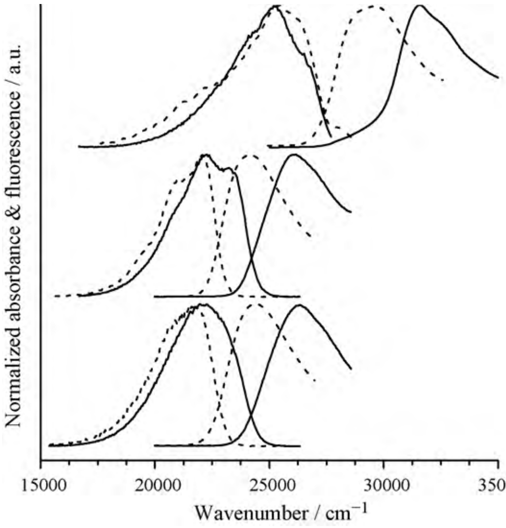

Fig. 3. Absorption and fluorescence emission spectra of the investigated monomeric (solid lines) and dimeric (dashed lines) quinoxaline derivatives in toluene solutions. Data for DMQ and TMBQ (top), DTQ and TTBQ (middle), and DFQ and TFBQ (bottom) pairs.

Table 2
Summary of spectroscopic and photophysical data for the investigated quinoxaline and 5,5′-biquinoxaline derivatives. UV–vis absorption maxima ($\tilde{\nu}_{abs}$), molar extinction coefficients (ε_{abs}), fluorescence maxima ($\tilde{\nu}_{flu}$), fluorescence quantum yields (ϕ_{flu}), fluorescence lifetimes (τ_{flu}), and phosphorescence maxima ($\tilde{\nu}_{phos}$). Data for room temperature and at 77 K (in parentheses).

Compounds/solvent	$\tilde{\nu}_{abs}$ (cm^{-1})	ε_{abs}[a] (10^4 M^{-1} cm^{-1})	$\tilde{\nu}_{flu}$ (cm^{-1})	ϕ_{flu}	τ_{flu} (ns)	$\tilde{\nu}_{phos}$ (cm^{-1})
Monomeric derivatives						
DMQ/toluene	31 550	0.65	25 250	0.004		(21 740, 20 500, 19 230)[b]
DMQ/acetonitrile	31 650		25 100	0.006		
DFQ/toluene	26 250	1.20	22 150	0.18	1.8 (2.2)	(No phosphorescence signal)
DFQ/acetonitrile	26 600		21 700	0.16		
DTQ/toluene	26 100	1.29	22 250	0.034	0.2 (0.5)	(17 360, 16 130)[b]
DTQ/acetonitrile	26 400		22 100	0.032	0.1 (0.1)	
Dimeric derivatives						
TMBQ/toluene	26 100	2.06	25 450	0.005		(20 000, 18 600)[c]
TFBQ/toluene	24 150	3.85	21 750	0.34	1.4 (1.5)	(No phosphorescence signal)
TFBQ/benzonitrile	24 100		21 400	0.30		
TTBQ/toluene	24 400	4.50	22 050	0.15	0.3 (0.5)	(No phosphorescence signal)
TTBQ/benzonitrile	24 300		21 700	0.13		

[a] ε_{abs} values at wave number of UV–vis absorption maxima (column under $\tilde{\nu}_{abs}$).
[b] Emission with well resolved wibronic structure.
[c] Emission with traces of vibronic structures.

3.3. Luminescence quantum yields and lifetimes

Much more interesting are the fluorescence quantum yield and lifetime measurements results obtained for the investigated molecules (Table 2). Both investigated methyl-substituted molecules (DMQ and TMBQ) show extremely small fluorescence quantum yields of fluorescence with lifetimes shorter than 100 ps (below the measuring limit of our TCSPC apparatus). The observation remains in nice agreement with the well known ultra-fast intersystem crossing characteristic for quinoxaline derivatives [7,8] resulting in nearly quantitative population of their strongly phosphorescent triplets (*vide infra*). Replacement of methyl groups by thiophene or furane subunits lead to well pronounced enhancement of luminescence yield for both investigated series. Whereas DTQ has a quantum yield about one order of magnitude larger than DMQ, their dimeric counterpart TTBQ emits markedly better. Still larger emission quantum yields have been found for DFQ and TFBQ. The latter one is the best emitter among the investigated molecules. Changes in the luminescence quantum yields are accompanied by longer emission lifetime values (Table 2). Noteworthy fluorescence lifetimes are weakly dependent on the sample temperature.

3.4. Transient absorption

The above-described findings can be hardly rationalized in terms of the energy gap law for the non-radiative rate constant [19] or the Strickler–Berg approximation for the radiative one [20]. Thus the observed changes in the measured quantum yields may be rationalized by differences in intersystem crossing efficiencies for the investigated molecules.

To check the above hypothesis, we tried to determine the intersystem crossing efficiencies by measuring the transient absorption of the investigated quinoxaline derivatives in toluene solution. It was done using two different set-ups for the transient absorption experiments: the first one allows us to measure in the nano-second domain up to 100 ns, while the second in the pico-second domain. None of the recorded spectra shows any appreciable changes in the 100 ns time span measured. From the long time measurements (Fig. 4), one can definitively conclude that in all cases a very long living species can be observed, most likely attributable to the triplet state. In fact the recorded DMQ transient absorption spectra have been found to be very similar in shape to the already reported in the literature [21]. A similar spectral pattern is characteristic for TMBQ molecule. For DTQ and DFQ the T–T absorption spectra are shifted to the red respect to DMQ. In all the cases a long tail up to 800 nm is observed. The spectra for dimeric TTBQ and TFBQ are somewhat more complicated due to the overlap of the ground state bleaching with the T–T band. The intersystem crossing quantum yields can be roughly estimated from the bleaching of the ground state in the case of TFBQ and TTBQ. In both cases a minimum yield of 0.5 can be assessed. For the monomeric dimeric derivatives, similar minimum values of 0.5 can be given by comparing the T–T absorption signals obtained for DMQ ($\Phi_{ISC} = 0.99$) with that for DFQ or DTQ.

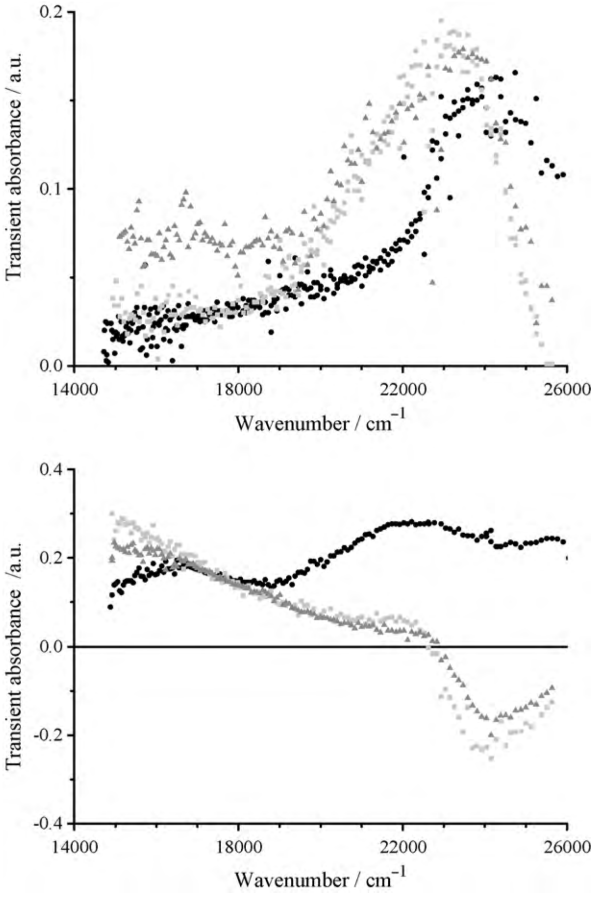

Fig. 4. Transient absorption spectra of the investigated monomeric (top) and dimeric (bottom) quinoxaline derivatives in toluene solutions. Data for DMQ and TMBQ (black circles), DTQ and TTBQ (light grey squares), and DFQ and TFBQ (dark grey triangles). Excitation wavelength: 337 nm and delay range of 5–10 ns.

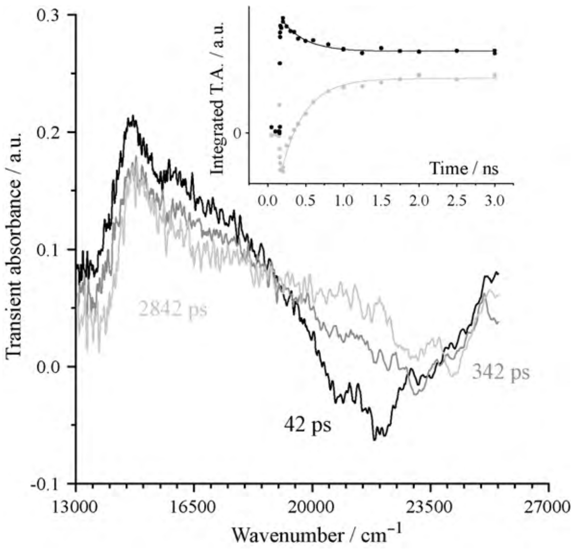

Fig. 5. Room temperature transient absorption spectra of TTDQ in toluene solutions at different delay times. Inset presents kinetic evolution of the transient absorption signals in the 450–500 nm region (grey circles) and in the 600–700 nm region (black circles). Excitation wavelength: 337 nm. The region of 13 000–18 000 cm^{-1} corresponds to the $T_1 \rightarrow T_x$ absorption, whereas the region of 20 000–25 000 cm^{-1} to mixture of the $S_0 \rightarrow S_1$ absorption bleaching and stimulated $S_0 \leftarrow S_1$ emission.

More precise values cannot be given as the extinction coefficients are unknown.

In the pico-second time resolved transient absorption experiments three signals at least can be observed: ground state bleaching in the more energetic spectral zone, stimulated emission and T–T absorption (Fig. 5). The kinetics extracted from the integration of the latter two bands show that the excited singlet and the triplet are kinetically linked by a single component (*e.g.*, 300 ps for TTBQ) in agreement with their fluorescence lifetimes. The decrease of the T–T absorption on this time scale can be understood as the disappearance of the excited singlet state which spectrum must overlap with that of the triplet. The resulting T–T spectra (measured at delay times longer than necessary for the excited singlet state decay) are comparable to those obtained in the nano-second scale time-resolved experiments.

3.5. *Phosphorescence spectra*

In order to further infer the differences in the photophysics of the investigated quinoxalines, we have also performed phosphorescence measurements at low temperatures in toluene (Fig. 6) and propanol glasses at 77 K. As expected and already reported in the literature [9], DMQ yields a very strong phosphorescence signal. The same has been observed for its dimeric counterpart TMDQ. However, somewhat surprisingly, this is not the case for the rest of the compounds. In fact TTBQ, DFQ and TFBQ did not show any phosphorescence at all, while DTQ only residually. Instead, fluorescence can be still observed under low temperature conditions. The fact that fluorescence lifetimes are nearly independent of the measurement temperatures (Table 2) allows concluding that the lack of the phosphorescence emission can be only hardly explained by changes in the ISC efficiencies. If this was a case, one could expect distinct longer luminescence lifetimes at 77 K. These troubling observations suggest that the furane and thiophene moieties attached to the quinoxaline or the 5.5′-biquinoxaline kernels have a very strong influence either on the radiative and/or the non-radiative rate constants that deactivate the triplet state. In fact, it is known that the excited triplet lifetime of thiophene is only of 1 μs (due to coupling between the triplet and the ground singlet *via* ring torsion) [22] and that it must be even shorter for furan [23]. Quinoxaline itself has a triplet lifetime of 0.25 s at low temperatures [9]. The only reasonable explanation for this enormous change upon introduction of furyl or thienyl substituents is that their triplet states are involved in the deactivation of the investigated DTQ and DFQ as well as of TTBQ and TFBQ molecules. It is somewhat an unexpected result because the lowest excited triplet states of furane or thiophene (with energies *ca.* 3.5 eV [24,25]) are located at energies *ca.* 1.0 eV higher than characteristic for quinoxaline or 5,5′-biquinoxaline triplets. On the other hand, an increase of the non-radiative decay of 3–4 orders of magnitude can be expected if a triplets states mixture with a contribution as low as 1% from the thiophene/furane moiety is invoked. This is because for the mixed states their effective non-radiative deactivation rate constant can be estimated taking into account the contribution from the component with shorter lifetime. In the investigated cases non-radiative rate constants for thiophene and furane components, are six orders of magnitude faster than that characteristic for quinoxaline. Thus only 1% contribution of the former suffices to reduce dramatically the phosphorescence quantum efficiencies.

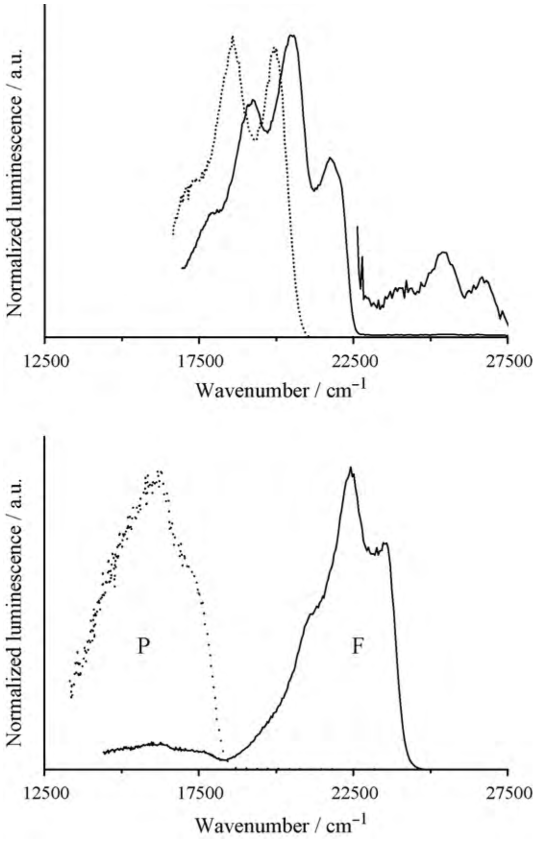

Fig. 6. Low temperature (77 K) luminescence spectra of DMQ (top, dash), TMBQ (top, solid) and DTQ (bottom) at low temperature in toluene (DMQ and TMBQ) and propanol glasses (DTQ). F and P stand for fluorescence and phosphorescence components, respectively. DMQ fluorescence has been multiplied by a factor of 50. The phosphorescence spectrum of DTQ was chopped from the overall luminescence.

Additionally, the observed batochromic shift between the phosphorescence spectra of DMQ and DTQ, suggests that the electronic interaction between the excited triplet states of both subunits is quite efficient leading to a ^{3*}DTQ excitation delocalization over the whole DTQ molecule instead of being constrained to the quinoxaline moiety. Comparing photophysical properties of the investigated molecules one can also infer that the electronic interaction within the triplet manifold is much more pronounced as compared to the singlet one. A more detailed discussion of the above-proposed explanation needs advanced quantum-mechanical calculations that are out of the scope of this communication.

4. Conclusions

Attachment of furyl or thienyl subunits to quinoxaline or 5,5′-biquinoxaline kernels affects in a similar way the electrochemistry and photophysics of these molecules. Both substituents shift the overall molecule reduction potential to more positive values. Similarly batochromic shifts are observed in the singlet excited state position. The most interesting change has been observed in the nature of the triplet state though because non-radiative deactivation of the triplets of quinoxaline as well as 5,5′-quinoxaline derivatives is dramatically enhanced by the attached furane or thiophene moieties. Most probably the effect is caused by strong mixing of the excited triplet states localized on subunits resulting in delocalized excitation. One can expect that similar effects may take place in other donor–acceptor systems based on thiophene or furane subunits resulting in a shortening of their triplet lifetimes. Therefore, in the design of new molecular compounds this should be taken into account if properties of their triplet states are important.

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