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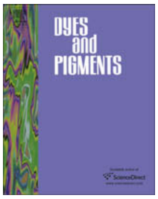

Photostability of phenothiazinyl-substituted ethylenes

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ABSTRACT

Photostability of phenothiazinyl-substituted ethylenes in both solution and in solid state (thin films) was investigated as a function of UV exposure time. Fluorescence intensity and spectral variation measurements in the presence and absence of ambient air were used to determine the effect of oxygen on the degradation of the compounds. The observed reduction in the fluorescence efficiency of air-saturated solutions and thin films subjected to UV was attributed to permanent photo-oxidative degradation. Photostability was higher in the cases of molecules that contained two bulky phenothiazinyl groups as compared to those containing only one phenothiazinyl group and a smaller phenyl substituent. Fully reversible fluorescence intensity decay in the degassed phenothiazine solutions was attributed to photo-isomerization, whereas the partially reversible changes in fluorescence intensity that were observed for films in the absence of air were ascribed to both photoexcited carrier capture by deep-traps acting as exciton quenching centres and degradation imparted by UV-induced bond scission.

1. Introduction

Photostability of dyes plays a very important role in nearly all aspects of their development and application [1,2]. Resistance to photodegradation is crucial for organic materials to be successfully applied in commercial optoelectronic devices, such as solar cells or light-emitting diodes (OLEDs) [3–5]. Photodegradation is considered to be a fundamental problem for luminescent organic materials and it occurs in OLEDs and organic lasers as a result of high optical fluence, which steadily destroys the material. It is well-known that stability issues that cause device failure include moisture and, in particular, oxygen, which must be eliminated from the immediate device environment by encapsulation [6,7]. It is generally accepted that the photodegradation of many organic materials is caused by photo-oxidation and, therefore, photodegradation is sometimes referred to as photo-oxidative decomposition [8,9]. Active oxygen species, such as singlet molecular oxygen or superoxide anion produced by the interaction of excited organic molecules with the ground-state molecular oxygen, attack materials to induce their degradation [10,11]. Therefore nowadays, commercially available OLEDs are being fabricated in high vacuum. High vacuum and inert gas atmosphere used in the device processing protect materials from ambient air, however, they considerably raise the cost of the device production. Thus, there is a constantly growing demand for high stability, less oxygen sensitive organic compounds with enhanced resistance to ambient effects. In spite of the oxygen, which severely degrades material stability, other intrinsic material properties like chemical stability of the molecule (strength of intramolecular bonds), susceptibility to photomodification of molecular structure, appearance of intrinsic fluorescence quenching or carrier trapping centers, defects are also important [4]. Poor chemical stability and increased susceptibility to irreversible photomodifications can significantly shorten the lifetime of even fully protected (from oxygen) optoelectronic devices.

In this work, photostability of four phenothiazinyl-substituted ethylenes has been studied. Derivatives of phenothiazines are best-known for their application in pharmaceutics [12]. Other potential applications include OLEDs [13], electrophotography [14], photorefractive materials [15], etc. The photostability was examined by monitoring fluorescence (FL) spectra and FL intensity changes in the dilute solutions and thin films of phenothiazine derivatives by

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continuously irradiating them with UV light. To distinguish the oxygen-induced material degradation (photo-oxidation) and that caused by poor stability of intramolecular bonds (in the absence of oxygen) air-saturated and degassed sample series were investigated.

2. Experimental

2.1. Materials

The photostability of four phenothiazine derivatives, namely, 1,2-di[10-(2-ethylhexyl)phenothiazin-3-yl]ethylene, 1,2-di[10-(p-methoxyphenyl)phenothiazin-3-yl]ethylene, 7-phenylethenyl-10-ethylphenothiazine and 3,7-di(phenylethenyl)-10-(2-ethylhexyl) phenothiazine, further referred to as **1**, **2**, **3** and **4**, respectively, was investigated. The structural formulas of the derivatives are shown in Fig. 1. The synthesis of compounds **1** and **2** is described elsewhere [16].

7-phenylethenyl-10-ethylphenothiazine (**3**) and 3,7-di(phenylethenyl)-10-(2-ethylhexyl)phenothiazine (**4**) were synthesized by the Wittig reaction of diethylbenzylphosphonate and 10-ethylphenothiazin-3-carbaldehyde or 10-(2-ethylhexyl)phenothiazin-3,7-dicarbaldehyde, respectively, by the following general procedure. 10-Ethylphenothiazin-3-carbaldehyde or 10-(2-ethylhexyl) phenothiazin-3,7-dicarbaldehyde and diethylbenzylphosphonate were dissolved in anhydrous THF. NaH was added to the obtained solution using the molar ratio: 1.5 mol of diethylbenzylphosphonate and 3 mol of NaH for each formyl group. The reaction mixture was stirred at room temperature under a N_2 atmosphere until the starting compound was consumed (assessed using TLC). The product was precipitated in methanol, filtered and purified by column chromatography. A mixture of ethylacetate and hexane in a volume ratio of 1:7 was used as eluent.

The yield of crystalline compound **3** was 65%. $T_m = 132.5$–133 °C. ^{1}H NMR (300 MHz, $CDCl_3$, δ, m.d.): 1.45–1.50 (m, 3H, CH_3), 3.94–4.01 (q, 2H, $J = 7.2$ Hz, N–CH_2), 6.86–6.99 (m, 3H, Ar), 7.03 (s, 2H, CH=CH *trans*), 7.18–7.43 (m, 7H, Ar), 7.53–7.56 (m, 2H, Ar). IR (KBr, ν, cm^{-1}): 3049, 3035 (C–H – arom.), 2987, 2942, 2867 (C–H – aliph.), 1592, 1571, 1498, 1463 (C=C – arom.), 970 (C–H=CH *trans*), 744 (C–H – arom.). Elemental analysis calculated for $C_{22}H_{19}NS$: C 80.20%, H 5.81%, N 4.25%; found: C 79.5%, H 5.92%, N 4.27%. $M = 329.47$ g mol^{-1}. MS ($APCI^+$, 20 V), *m*/*z* (%): 330 ($[M + H]^+$, 100).

The yield of amorphous compound **4** was 49%. ^{1}H NMR (300 MHz, $CDCl_3$, δ, m.d.): 0.96–1.01 (m, 6H, CH_3), 1.38–1.59 (m, 8H, CH_2), 2.02–2.08 (m, 1H, CH), 3.82 (d, 2H, $J = 6.9$ Hz, N–CH_2), 6.92 (d, 2H, $J = 8.7$ Hz, Ar), 7.07 (s, 4H, CH=CH *trans*), 7.31–7.47 (m, 10H, Ar), 7.57–7.60 (m, 4H, Ar). IR (KBr, ν, cm^{-1}): 3079, 3022 (C–H – arom.), 2955, 2923, 2854 (C–H – aliph.), 1595, 1579, 1496, 1475 (C=C – arom.), 956 (C–H = CH *trans*), 749 (C–H – arom.). Elemental analysis calculated for $C_{36}H_{37}NS$: C 83.84%, H 7.23%, N 2.72%; found: C 82.99%, H 7.78%, N 3.05%. $M = 515.77$ g mol^{-1}. MS ($APCI^+$, 20 V), *m*/*z* (%): 516 ($[M + H]^+$, 100).

All of the compounds under study can be classified as stilbenes. Compounds **1** and **2** represent twin compounds, in which two differently substituted phenothiazine moieties are linked via an unsaturated ethylene bridge, whereas compounds **3** and **4** contain one alkyl-substituted phenothiazine moiety and either one or two phenyl-substituted ethylene groups, respectively. Compounds **1** and **4** were isolated after the synthesis as amorphous materials. Compounds **2** and **3** were obtained as crystalline materials, however they could be easily transformed to the amorphous state either by cooling from the melt or casting from the solution.

2.2. Sample preparation and instrumentation

^{1}H NMR spectra were recorded with Varian Unity Inova (300 MHz) spectrometer. All the data are given as chemical shifts δ (ppm) downfield from $(\mathrm{C{-}H_3})_4\mathrm{Si}$. IR-spectroscopy measurements were performed on a Perkin Elmer Spectrum GX

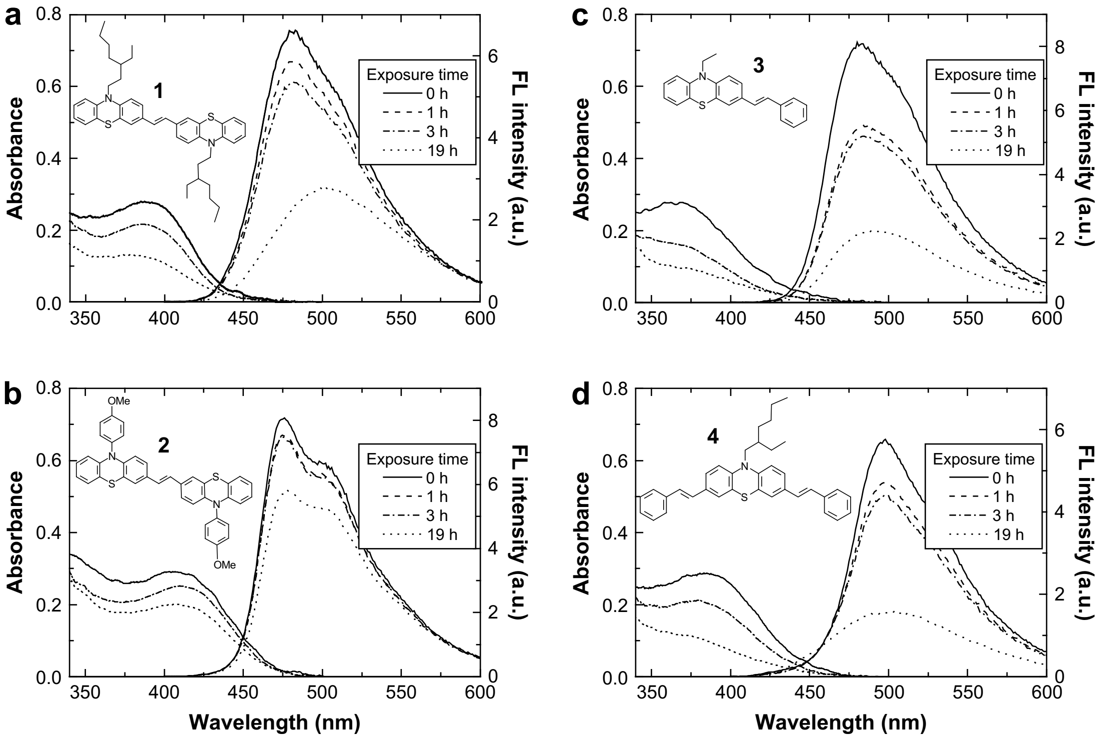

Fig. 1. Evolution of absorption and fluorescence spectra of air-saturated 10^{-5} M toluene solutions of phenothiazine compounds (a) **1**, (b), **2** (c), **3** and (d) **4** after 0, 1, 3 and 19 h of 1.5 mW/cm^2 UV exposure. The structural formulas of the compounds are indicated.

spectrophotometer, using KBr pellets. Mass spectra were obtained on a Waters Micromass ZQ mass spectrometer. Elemental analysis (C, H, N) was conducted using the Elemental Analyzer CE-440.

To estimate the influence of oxygen on the photostability of solutions of the phenothiazine derivatives described above two series of 10^{-5} M toluene solutions were prepared and hermetically sealed in cylindrical glass ampoules. One series of the solutions was air-saturated and the other was degassed. Degassing of the solutions on purpose to remove dissolved oxygen was achieved by bubbling the solutions for about 10 min with high-purity argon gas prior to sealing the ampoules.

Photostability measurements were also performed on about 100 nm-thick films of the phenothiazine derivatives prepared from solutions on the glass substrates by spin-casting technique. The spin-casted films were dried for 16 h in a vacuum chamber. After the vacuum treatment half of the films were covered by 300 nm-thick aluminum layer to protect the films from oxygen in the air. Both protected-from-air and unprotected parts of the films were subjected to UV exposure to discern oxygen-induced FL intensity changes.

1.5 mW optical power and 365 nm wavelength UV light-emitting diode NSHU590-B (Nichia) was used to continuously irradiate the air-saturated and degassed solutions of the phenothiazine derivatives. A special care was taken to ensure UV–LED irradiates the whole ampoule to avoid thermal convection in the solution. Absorption spectra of the phenothiazine solutions were measured with the fiber optic spectrometer AvaSpec-2048 (Avantes). Spectrometer LS50B (Perkin–Elmer) was employed to measure FL excitation spectra. The photostability of the phenothiazine solutions exposed to UV light were measured with the CCD spectrometer (Hamamatsu PMA-11). The long-term stability of the whole system comprising the excitation and detection channels was evaluated by measuring photon flux fluctuations of the UV–LED with the same CCD spectrometer over the 20 h period. The fluctuations were found to be less than 0.5%, i.e. negligible as compared with experimentally observed FL intensity changes.

The photostability of the protected-from-air and unprotected phenothiazine films was investigated with the F900 spectrometer (Edinburg Instruments). 0.2 mW average optical power picosecond diode laser EPL-375 emitting 50 ps pulses at 375 nm was used for the sample UV irradiation and fluorescence excitation. All the measurements were carried out at room temperature.

3. Results and discussion

3.1. Photostability in solutions

Fig. 1 shows absorption and fluorescence spectra of air-saturated toluene solutions of the phenothiazine derivatives measured after 0, 1, 3 and 19 h irradiation by UV light. The lowest-energy absorption maxima (370–420 nm) of the derivatives are shifted to lower energies in respect of that of 10-hexylphenothiazine (312 nm) [17], thus indicating extended conjugation length of the compounds due to the ethylene linkage. The FL spectra of the phenothiazine solutions feature clearly asymmetrical fluorescence bands peaking in the blue-green spectral range (475–500 nm). A general tendency observed for all the compounds studied is the decrease of absorbance and FL intensity with UV exposure time. A similar decrease of absorbance and FL intensity was previously reported for PPV solutions and thin films [18]. FL intensity integrated over the spectrum for each compound is depicted in Fig. 2 as a function of UV exposure time. A rapid decrease of FL intensity is observed for all the phenothiazine derivatives in solutions within the first several hours. The decrease becomes much slower at a later stage. FL intensity of the air-saturated solutions of compounds **3** and **4** decreases faster as compared to that of the compounds **1** and **2**. The decrease of absorbance and FL intensity with UV exposure in air-saturated phenothiazine solutions may be attributed to the material degradation due to oxygen-induced scission of intramolecular bonds. As verified by FTIR measurements in conjugated polymer PPV, photo-oxidation involves carbonyl substitution on the vinyl group and scission of the vinyl double bond [19,20]. Due to the strong electron affinity carbonyl defect (C=O) is known to act as an exciton dissociation site by trapping the electron in the lower energy excited state of the carbonyl group, and thus, quenching fluorescence. It has been confirmed that the presence of the C=O groups inversely correlates with FL yield [19]. The reported data imply that photo-oxidation can indeed contribute to the decrease of FL intensity with UV exposure time observed in Fig. 2.

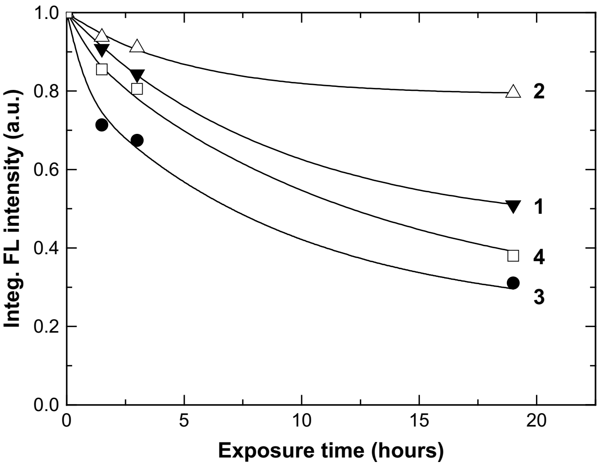

Fig. 2. Integrated FL intensity of air-saturated 10^{-5} M toluene solutions of the phenothiazine derivatives as a function of UV exposure time with the power density of 1.5 mW/cm^2. Lines serve as guides for the eye.

To account for the degradation of phenothiazinyl-substituted ethylenes in solutions due to photo-oxidation process the FL intensity decay was monitored in the air-saturated and degassed solutions. The solutions were exposed to UV light with 1.5 mW/cm^2 power density over the 12 h period with the 4 h interruption of exposure in between. Fig. 4a shows such a dependence of the integrated FL intensity on UV exposure for the solution of **1**. FL intensity of the degassed solution (solid line) decreased by 7% within the first 20 min and then saturated. After 4 h interruption of exposure, a full recovery of the FL intensity was observed clearly demonstrating no permanent degradation of the material in the degassed solution. Quite different behavior of the FL intensity was observed in the air-saturated solution of **1** (dashed line in Fig. 4a). The FL intensity exhibited a sudden drop by almost 90% within the first hour and then continued to decrease further at a slower rate with no indications of saturation. After 4 h of continuous exposure FL intensity dropped to 6% of its initial value at 0 min. Switching UV exposure off for 4 h and switching it on again resulted only in partial recovery of the FL intensity to 10% of its initial value, however, the intensity continued to decrease as it did before the interruption of exposure. These results confirm the photo-oxidative mechanism of the air-saturated solutions and additionally indicate some reversible process taking place both in air-saturated and degassed solutions. We suggest the photo-isomerization of the phenothiazinyl-containing stilbenes to be responsible for the reversible FL decay. Since all the investigated phenothiazine derivatives possess ethylene linkage *cis-* and *trans-* conformations of the derivatives are possible. Similar to stilbenes [21] or azobenzenes [22], where the presence of *cis-* and *trans-* isomers results

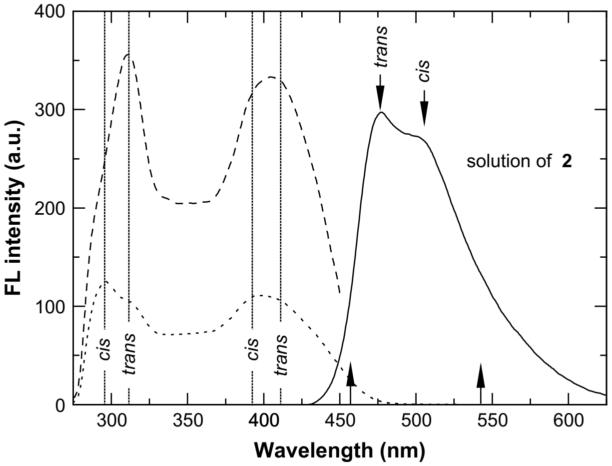

Fig. 3. FL (solid line) and FL excitation spectra of air-saturated 10^{-5} M toluene solution of compound **2** probed at 460 nm (dashed line) and 540 nm (dotted line), respectively.

in the appearance of two different intensity FL bands, the asymmetrical shape of FL spectra of the phenothiazine derivatives may be a consequence of overlapping isomer-related bands. The double band structure can be particularly well resolved for the solution of compound **2** (Fig. 1b). Coexistence of two isomeric forms is supported by FL excitation data provided for the compound **2** in Fig. 3. It shows two FL excitation spectra probed at 460 nm and 540 nm wavelengths with dominating fluorescence originating from *cis-* or *trans-* forms. Probing FL at 460 nm results in the two dominating absorption bands located for about 15 nm to longer wavelengths than those probed at 540 nm. These data support the existence of the two distinct states, although a strong overlap of their absorption spectra prevents their clear resolution. By analogy with similar absorption and fluorescence spectra of other stilbene class compounds [21,23] and azobenzenes [22] we assign the higher energy fluorescence band and lower energy absorption bands to *trans-* isomer and the lower energy fluorescence band and higher energy absorption bands to *cis-* isomer. UV exposure of solutions evidently distorts equilibrium concentration of *cis-* and *trans-* isomers shifting it to the isomeric form with higher absorbance at the excitation wavelength. This phenomenon is known as photochromism. The photochromic transition taking place on tens of minutes time scale decreases the sample absorbance at the excitation wavelength and consequently reduces the integrated FL intensity. The two isomeric forms may also feature different FL yields, which can further enhance FL decay effect. Switching UV exposure off establishes initial equilibrium concentration of *cis-* and *trans-* forms. Since FL of the *cis-* isomer is lower-in-energy than that of the *trans-* isomer, the FL band may exhibit a red shift with UV exposure time, which indeed is observed in the phenothiazine solutions, in particular for the compound **1** (Fig. 1a).

These results indicate that FL intensity behavior of degassed solution of compound **1** is mainly governed by photo-isomerization, whereas in air-saturated solution permanent material degradation due to photo-oxidation occurs. Comparison of the FL intensity quenching in air-saturated and degassed solutions of **1** (see Fig. 4a) justifies the material degradation to account for about 90% drop of FL intensity with the rest 10% belonging to reversible conformational changes. Although FL intensity dependences on the UV exposure time for solutions of all four phenothiazine derivatives were found to be somewhat different implying concurrent action of photo-isomerization and photo-oxidation, the photo-oxidation in air-saturated solutions of all the compounds played a dominant role. Thus, a comparison of the FL decay of the air-saturated phenothiazine solutions presented in Fig. 2 revealed that the compounds **1** and **2** containing one ethylene moiety and two rather bulky substituted phenothiazinyl groups per molecule were more stable against photo-oxidation than **3** and **4** with considerably smaller phenyl substituents. This observation is in agreement with the improved stability of polysilane-based copolymers achieved by addition of bulky alkyl or aryl side groups [4].

3.2. Photostability of thin solid films

Since thin organic films are the constituents of optoelectronic devices the investigation of the film photostability is of high practical importance. Fig. 4b illustrates integrated FL intensity of protected-from-air and unprotected thin films of the phenothiazine derivative **1** as a function of UV exposure time at the UV power density of 20 mW/cm^2. Similarly to air-saturated solution of **1**, FL intensity in the unprotected film of **1** decreased with the UV exposure time showing no signs of saturation. Moreover, interruption of UV exposure resulted in almost no recovery of FL intensity, which is also in close resemblance to the air-saturated solution of **1**. These features can be attributed to permanent material degradation due to photo-oxidation. In contrast, FL intensity of the protected-from-air film of **1** exhibited a tendency towards saturation after about 55 min of exposure. Additionally, a recovery of the FL intensity from 70% to 90% of its initial value was observed after 20 min interruption of UV exposure in the protected film. Incomplete recovery of the FL intensity after interruption of UV exposure clearly points out partial degradation of the film.

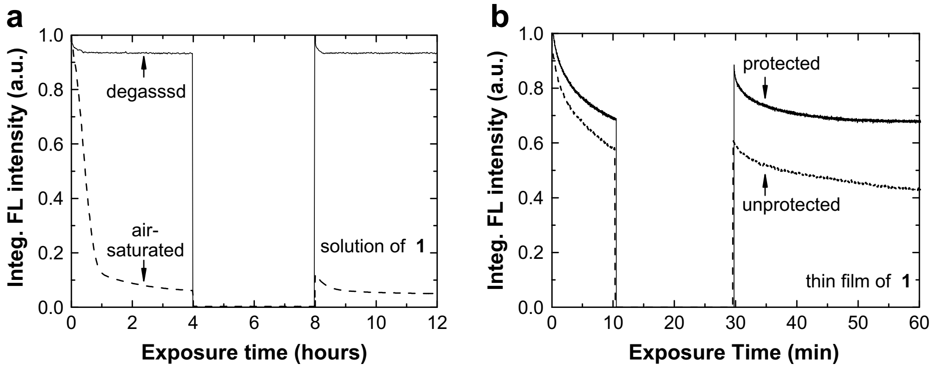

Fig. 4. Integrated FL intensity of (a) air-saturated (dashed line) and degassed (solid line) 10^{-5} M toluene solutions as a function of UV exposure time with the power density of 1.5 mW/cm^2, and of (b) protected-from-air (solid line) and unprotected (dashed line) thin films as a function of UV exposure time with the power density of 20 mW/cm^2 for the phenothiazine derivative **1**.

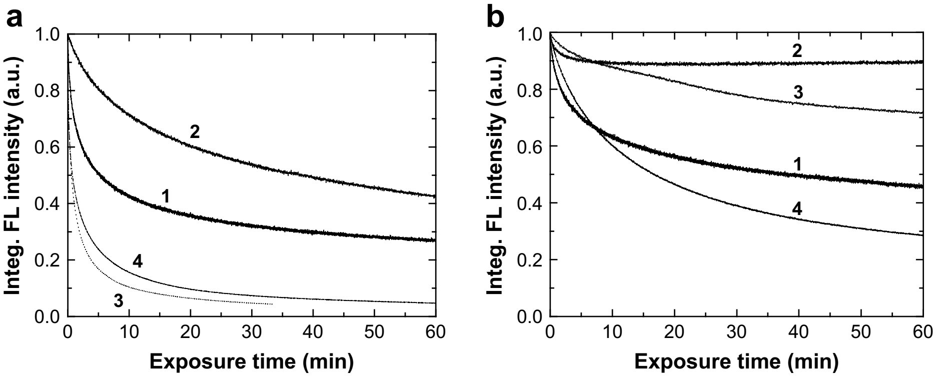

Fig. 5. Integrated FL intensity of (a) unprotected and (b) protected-from-air thin films of the phenothiazine derivatives as a function of UV exposure time with the power density of 20 mW/cm^2.

Though not completely impossible, isomerization of the molecules in the films (rigid matrix) is highly suppressed, and thus, it can not account for larger FL intensity decay/recovery than that in solutions (see Fig. 4a). Additional FL quenching mechanisms associated with generated charge carriers are known to be present in molecular solids [24–26]. Here even low density of excitation quenching centers of the order of 10^{16}–10^{17} cm^{-3} is capable to induce significant quenching effect due to efficient excitation energy migration. We attribute reversible FL quenching observed for the thin films to result from photogenerated charge carriers captured into deep traps. Occupation and release of the deep-traps taking place on a second and minute time scales explain the FL intensity recovery observed after interruption of UV exposure. Importance of the carrier traps in organic semiconductors has been confirmed [27,28]. It was emphasized that formation of the deep-traps can arise due to impurities as well as structural defects, which are in particular favorable in the films prepared from solutions. Exciton quenching by trapped charge carriers has been unambiguously determined in pentacene-doped tetracene [29] and in single polymer chains [30]. According to this interpretation the tendency towards saturation of FL intensity in the protected film of **1** after 55 min of exposure (see Fig. 4b) is evidently a result of the saturation of the deep-traps. So, the intricate FL intensity behavior in the protected films is caused by both effects, i.e. carrier capture to the deep-traps acting as exciton quenching centers and permanent material degradation. Note that partial degradation due to photo-oxidation even in protected-from-air films is possible because of oxygen penetration through defects in the aluminum layer. Small amount of oxygen may also remain in the film after vacuum treatment.

A comparison of the FL intensity decays of the unprotected and protected phenothiazine films with UV exposure time is presented in Fig. 5. As discussed above, the initial rapid decrease of FL intensity in the protected films is affected by the carrier capture to deep-traps. The carrier capture in the unprotected films is completely dominated by material degradation due to photo-oxidation. The unprotected films of **1** and **2** were found to be more stable against photo-oxidation than films of **3** and **4** (Fig. 5a). In the most photostable film of **2** FL intensity drop over 60 min was 60% as compared to 95% in the least stable films of **3** and **4**. The same order of the phenothiazine derivatives in accordance with their photostability was also attained for air-saturated solutions (see Fig. 2). For the protected films of the phenothiazinyl-substituted ethylenes the

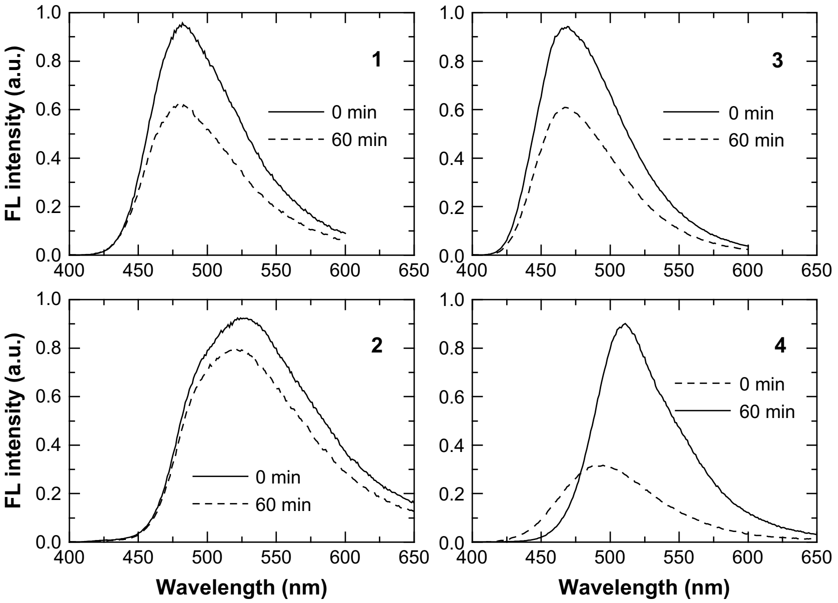

Fig. 6. FL spectra of protected-from-air thin films of the phenothiazine derivatives (a) **1**, (b), **2** (c), **3** and (d) **4** after 0 (solid line) and 60 min (dashed line) of UV exposure with the power density of 20 mW/cm^2.

decrease of FL intensity with UV exposure time is significantly slower than for the unprotected ones. For instance, 60 min of exposure induced up to 6 times smaller drop of FL intensity for the protected films than for their unprotected counterparts (in the case of **2**). A decrease of the FL intensity by only 10% with the subsequent saturation of the intensity of the protected film of **2** observed after 10 min of UV exposure indicates the highest photostability of this compound as compared to the rest studied in this work. Here, the saturation shows the filling of deep-traps by carriers, meanwhile a continuous decrease and non-saturation of the FL intensity in the protected-from-air films of other phenothiazine derivatives (**1**,**3**,**4**) after 60 min of UV exposure are the signatures of the constant material degradation resulting from the UV-induced scission of covalent bonds or from other irreversible molecular structure modifications. The results obtained for the films of phenothiazinyl-substituted ethylenes are consistent with those obtained for the solutions and imply enhanced photostability of the phenothiazine compounds bearing more bulky groups.

UV exposure-induced spectral changes of the protected films of phenothiazinyl-substituted ethylenes are revealed in Fig. 6, where FL spectra measured after 0 and 60 min of UV exposure are displayed. Exposure of the films to UV irradiation for 60 min results in a slight blue shift of the spectral maxima. The blue shift is especially strongly pronounced for the least stable phenothiazine film of **4**, which contains two ethylene groups. We attribute this blue shift as well as the decrease of FL intensity with UV exposure to a reduction of the conjugation length similar to that reported for conjugated polymer films [18,19]. By analogy with conjugated polymers we suggest the scission of ethylene double bond to be responsible for the reduction of the conjugation length resulting in the smaller (phenothiazinyl and phenyl) molecular fragments, which give rise to additional FL at shorter wavelengths. The absence of long-wavelength emission from the deep-traps (or defects) responsible for UV-induced reversible FL intensity changes in the protected-from-air films identifies the traps in phenothiazines as being non-emissive.

4. Conclusions

In conclusion, photostability of the ethylene-linked phenothiazine derivatives bearing either two identical and bulky phenothiazinyl groups or significantly smaller phenyl substituents has been studied. UV-induced irreversible FL intensity decay for the air-saturated phenothiazine solutions and films of phenothiazinyl-substituted ethylenes is governed by permanent material degradation due to photo-oxidation. Completely reversible FL intensity variations in the degassed solutions of phenothiazinyl-substituted ethylenes are evidently caused by molecular photo-isomerization, whereas only partly reversible intensity changes in the protected-from-air films are attributed to both photoexcited carrier capture by deep-traps and material degradation due to UV-induced bond scission. Our results imply that ethylenes substituted by rather bulky phenothiazine groups are more photostable as compared to those substituted by smaller phenyl groups, and thus, if employed can improve the stability of optoelectronic devices such as OLEDs.

Acknowledgements

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