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Photochemical properties of (7-benzothiazol-2-yl-9, 9-didecylfluoren-2-yl)diphenylamine under one- and two-photon excitation

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Abstract

The photochemical properties of the fluorene derivative (7-benzothiazol-2-yl-9,9-didecylfluoren-2-yl)diphenylamine (**1**) in hexane and CH_2Cl_2 were studied under linear (one-photon) and nonlinear (two-photon) excitation. The quantum yield of the photochemical reaction, Φ, for **1** in hexane was in the range $(3.5–5) \times 10^{-5}$ for one-photon excitation (UVGL-25 and Xe-lamps) and was nearly the same under two-photon excitation (femtosecond laser with pulse duration 120 fs, average power ∼10 mW, repetition rate $f = 1$ kHz). The values of Φ in CH_2Cl_2 were $(2.5–4) \times 10^{-5}$ for one-photon excitation and increased 50–80 times under two-photon excitation. This increase can be explained by an additional one-photon absorption process from the first electronically excited state, resulting in the observed enhancement in photochemical decomposition.

Keywords: Fluorene derivatives; Photochemical quantum yield; Two-photon excitation; Photostability; Photodecomposition

1. Introduction

Two-photon absorbing (2PA) organic dyes with high thermal and photochemical stability remain a subject of great interest due to a wide array of potential applications, including two-photon microfibrication technologies [1,2], optical power limiting [3], two-photon photodynamic cancer therapy [4], and non-destructive two-photon fluorescence imaging [5]. Photochemical stability of organic dyes under one- and two-photon excitation is an important issue in the application of 2PA compounds, an issue that has arguably hindered the use of organic materials in nonlinear optical technologies.

Two-photon absorption processes can be conceptualized through considering a simplified electronic state diagram (Fig. 1) for S_0 (ground state) and S_1 (first excited state). As can be seen, both one- and two-photon absorption generally results in the same electronic level from which photoreactions may take place. Photobleaching processes, resulting from intense laser irradiation, can occur by one- or two-photon absorption, or two-step successive absorption of two photons within one pulse of laser excitation. High irradiation intensity can generate molecules excited to high electronic states that can undergo photochemical reactions such as photoionization and bond fission [6]. These photoprocesses may be different from reactions of the molecule in its lowest excited state, produced by low intensity excitation. The comparison of one- and two-photon excitation may help reveal the nature of the photobleaching processes of organic dyes and determine the peculiarities of their photochemical behavior. For example, Kao et al. [7] recently revealed the distinctions between one- and two-photon-induced photobleaching processes in protoplasts. It was shown that high intensity, multi-photon excitation impacted the molecules in ways much different than under low intensity irradiation conditions.

Recent developments in the design and synthesis of efficient 2PA molecules demonstrated that certain fluorene derivatives undergo strong nonlinear optical absorption [8,9], and are potentially useful for the applications mentioned above. Some aspects of the photochemical properties of fluorene derivatives were investigated earlier, with a focus on environmental pollutants [10,11]. The effect of solvents and substituents on the photooxidation of fluorene derivatives, and photochemical properties of fluorene at a

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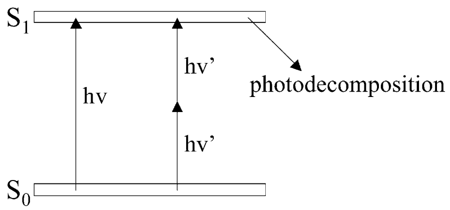

Fig. 1. Simplified diagram of the one- and two-photon excitation processes.

silica gel/air interface were reported [12,13]. The purpose of this paper is to report a comparative investigation of the one- and two-photon-induced photochemical reactions of new fluorene derivatives. We previously reported the synthesis [8,9] and photophysical characterization of these compounds [14]. Here, the quantum yields of the photoreactions were determined as a function of dye concentration solvent polarity. The results from this work are important both understand the photochemical reactivity of this class of compounds and elucidate their usefulness and limitations for emerging nonlinear optical applications.

2. Experimental

The unsymmetrical fluorene derivative (7-benzothiazol-2-yl-9,9-didecylfluoren-2-yl)diphenylamine (**1**) was studied in spectroscopic grade hexane and CH_2Cl_2 at room temperature. The structure of **1** is shown in Fig. 2a. Absorption spectra were recorded with a UV-Vis Cary 3 spectrophotometer in 10, 1, and 0.05 mm path length quartz cuvettes for dye concentrations in the range $3 \times 10^{-6} \leq C \leq 1.3 \times 10^{-3}$ M. Steady-state fluorescence spectra of **1** were obtained with a PTI Quantamaster spectrofluorimeter for dilute solutions ($C \leq 3 \times 10^{-6}$ M) in 10 mm fluorimetric quartz cuvettes. Photochemical parameters were measured by absorption and fluorescence methods, comprehensively described in [15].

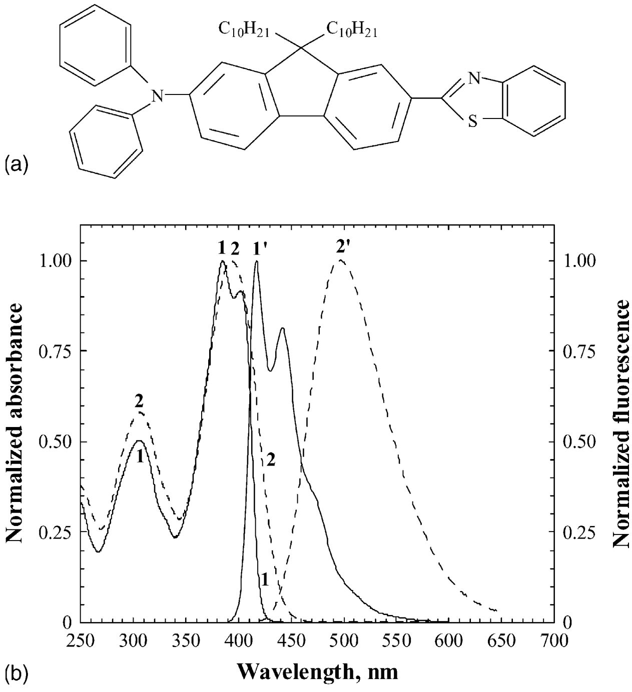

Fig. 2. (a) Structure of **1**. (b) Normalized absorption (1, 2) and fluorescence (1′, 2′) spectra for **1** in hexane (1, 1′) and CH_2Cl_2 (2, 2′).

2.1. Absorption method

The quantum yields of the photoreactions, Φ, for one-photon excitation (low intensity irradiation) over a wide range of dye concentrations were determined from the temporal dependences of the optical density at the maximum of the absorption band, $D(\lambda_{max}, t)$, of **1**. The values $D(\lambda_{max}, t)$ were measured during irradiation with a UV-lamp (UVGL-25; irradiation intensity, $I \approx 4\,\mathrm{mW/cm^2}$; excitation wavelength, $\lambda_{exc} \approx 360$ and FWHM ≈ 15 nm) in standard quartz cuvettes (10 mm × 10 mm × 35 mm). The entire volume of the cuvette was irradiated simultaneously, with the divergence of the light source taken into account. The intensity of the UV-lamp was measured with an IL-1400A powermeter (International Light Inc.). The quantum yield of the photobleaching was calculated for small temporal changes in the optical density, $D(\lambda, t)$, by [15]:

$$\Phi = \frac{[D(\lambda_{max}, 0) - D(\lambda_{max}, T)]}{\{T\varepsilon(\lambda_{max})\int_{\lambda} I(\lambda)[1 - 10^{-D(\lambda,0)}]\mathrm{d}\lambda\}}, \qquad (1)$$

where $\varepsilon(\lambda_{max})$ is the extinction coefficient of **1** in $M^{-1}\,cm^{-1}$; T the irradiation time in seconds; $I(\lambda)$ in phot $cm^{-2}\,s^{-1}$ and $D(\lambda, 0)$ are the spectral distribution of the UV-lamp and absorption spectrum of **1** before irradiation, respectively. The same method was used for the determination of Φ for deoxygeneted solutions, which were purged by N_2 for 20 min in the cuvettes.

2.2. Fluorescence method

The comparison of the quantum yields, Φ, for compound **1** under one- and two-photon excitation was accomplished by the measurement of its fluorescence intensity, $F(t)$, with a PTI Quantamaster spectrofluorimeter (Fig. 3a). A full description of this method has been described [15]. One-photon excitation of fluorene **1** utilized the Xe-lamp of the spectrofluorimeter ($I \approx 5$–$10\,\mathrm{mW/cm^2}$ in the spectral range 350–400 nm). Dye solutions were placed in microcuvettes: ∅1 × 0.03, ∅1 × 0.1 and ∅1 × 0.5 mm (Fig. 3b). Each microcuvette was placed in a regular fluorescence cuvette (10 mm × 10 mm × 35 mm) filled with the corresponding solvent in order to ensure the small amount of dye solution (∼20–50 nl) did not evaporate. The diffusion of solvent into the microcuvette was negligible during irradiation. The entire volume of the microcuvette was irradiated simultaneously. The optical density of the dye solution was less than 0.1. The irradiation power was measured with a Laserstar powermeter (Ophir Optronics Inc.) with sensitivity in the nW range. The number of photobleached molecules was

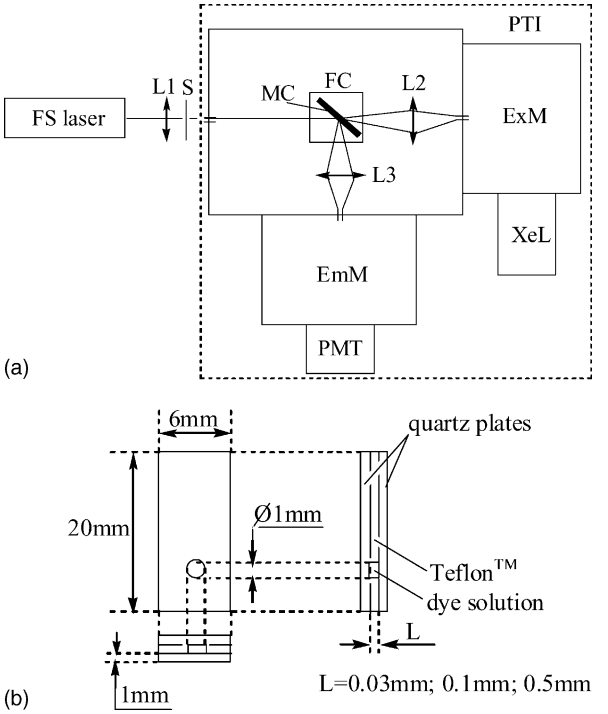

Fig. 3. (a) Experimental setup: PTI, Quantamaster spectrofluorimeter; XeL, Xe-lamp; ExM and EmM, excitation and emission monochromators, respectively; PMT, photomultiplier tube; L1, L2, L3, lenses; FC, fluorescence quartz cuvette 10 mm × 10 mm × 35 mm; MC, microcuvette (see scheme (b)); S switcher; FS laser, femtosecond laser. (b) Specifications of the microcuvette.

determined by measuring the temporal changes in the fluorescence intensity of **1.** The corresponding quantum efficiencies of the photoreactions, Φ, were calculated by [15]:

$$\Phi = \frac{(1 - F_T/F_0)}{[I(\lambda_{\mathrm{exc}})\sigma(\lambda_{\mathrm{exc}})\int_0^T (F(t)/F_0)\mathrm{d}t]}, \tag{2}$$

where F_0 and F_T are the fluorescence intensities in arbitrary units before and after irradiation, respectively; $I(\lambda_{\mathrm{exc}})$ the intensity of the Xe-lamp in phot cm^{-2} s^{-1}; $\sigma(\lambda_{\mathrm{exc}})$ is the absorption cross-section of **1** in cm^2; and T is total irradiation time in seconds. Eq. (2) is based on the following assumptions: the effect of diffusion processes on photochemical kinetics was negligible during irradiation; irradiation intensity $I(\lambda_{\mathrm{exc}}) \approx$ constant in the entire volume of the microcuvette; and photochemical products that were formed had no effect on the fluorescence intensity of **1** (for **1** in CH_2Cl_2 observed wavelength of the fluorescence $\lambda_{\mathrm{obs}} \approx 570$ nm).

In order to detect small temporal changes in the fluorescence intensity, $F(t)$, with sufficient accuracy, all possible instabilities of the experimental setup (Fig. 3a) were analyzed. The main instability was associated with the photomultiplier tube (PMT) and reached 3–5% h^{-1}. Taking into account the temporal changes in the background level of the PMT amplifiers, the accuracy of the relative fluorescence measurements was determined to be 0.3–0.5%, allowing the quantum efficiency of the photoreaction to be measured down to 10^{-6}.

Two-photon excitation of **1** was performed with a Clark-MXR 2001, Ti:Sapphire amplified, second harmonic of an erbium-doped fiber ring oscillator system (output 775 nm) that pumped an optical parametric generator/ amplifier (TOPAS, Light Conversion), providing pulse duration $\tau_{\mathrm{P}} \approx 120$ fs (FWHM) at a repetition rate $f = 1$ kHz with tunable wavelengths from 560 to 2100 nm. Photochemical decomposition of **1** was observed in the microcuvette (Fig. 3b) under two-photon excitation at $\lambda_{\mathrm{exc}} = 760$ nm. The quantum efficiency of the photochemical reaction, $\Phi_{2\mathrm{PA}}$, was determined by the following equation [15], based on the assumptions mentioned above:

$$\Phi_{2\mathrm{PA}} = \frac{(1 - F_T/F_0)}{\{2f\int_0^T (F(t)/F_0)\mathrm{d}t[1 - \exp(-\sigma_{2\mathrm{PA}}\alpha P_0^2(Sf\beta^{-2}))]\}}, \tag{3}$$

where $f = 1$ kHz is the repetition rate; $\sigma_{2\mathrm{PA}}$ the two-photon cross-section of **1** in cm^4 s phot^{-1}; P_0 is the average laser power in photon per second; S the cross-section of the microcuvette in cm^2; $\alpha = \int_{-\infty}^{+\infty}\exp[-(t/\tau_{\mathrm{P}})^4]\mathrm{d}t$, and $\beta = \int_{-\infty}^{+\infty}\exp[-(t/\tau_{\mathrm{P}})2]\mathrm{d}t$. For $\tau_P = 120$ fs $\alpha \approx 9 \times 10^{-14}$ and $\beta \approx 1.28 \times 10^{-13}$ s. The long-term stability of the femtosecond laser (5–10% h^{-1}) was insufficient for the determination of very small changes in the fluorescence intensity. In this case, the number of photobleached molecules was determined by probing the steady-state fluorescence of **1** using weak Xe-lamp irradiation (intensity $I \leq 5 \times 10^{-6}$ W/cm^2) after 760 nm two-photon excitation conditions that did not affect the photobleaching processes. An additional signal of the average fluorescence intensity, $F_{2\mathrm{PA}}$, as a result of two-photon excitation was determined by the time resolution of PMT (~5 ns) operating in the photon counting regime. This was equal to the repetition rate of the femtosecond laser, e.g. $F_{2\mathrm{PA}} \approx 10^3$ counts per second. All of these experimental caveats were taken into account. The quantum yields of the photochemical reactions of **1** obtained for one-photon excitation by the absorption (UV-lamp irradiation) and fluorescence (PTI Xe-lamp irradiation) method were in a good agreement with one another.

All of the experimental methods described in this paper were also evaluated with compounds possessing well-known photochemical parameters (e.g. 5,7-dimethoxycoumarin (DMC) in acetonitrile and CH_2Cl_2 [16]).

3. Results and discussion

The absorption and corrected fluorescence spectra of fluorene **1** in hexane and CH_2Cl_2 are presented in Fig. 2b. The maxima of the fluorescence spectra of **1** (curves 1′, 2′) exhibited a strong dependence on solvent polarity, according to the Lippert equation [17]. This is in contrast to the solvent insensitive absorption spectra (curves 1 and 2). Hence, no specific interactions of **1** with solvent molecules occurred. Comprehensive spectral investigations of **1** were previously

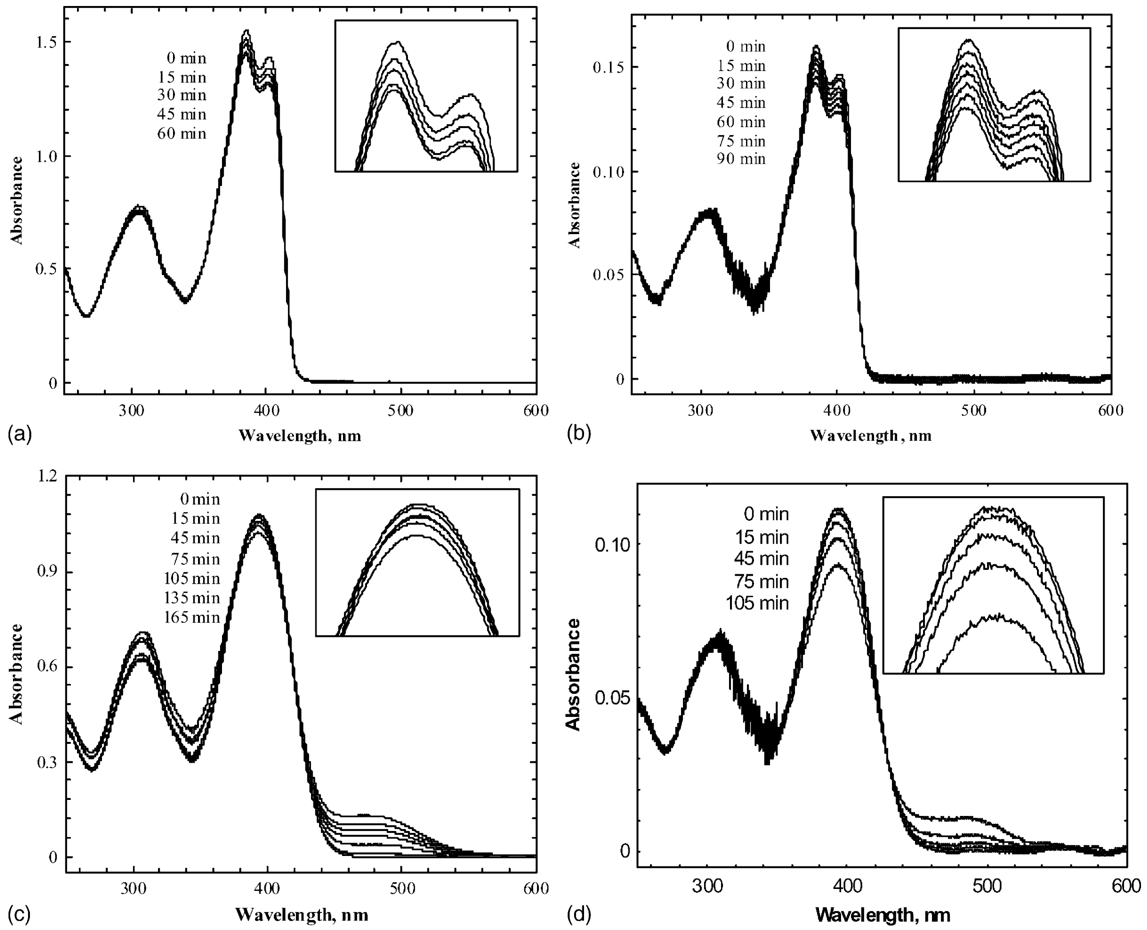

Fig. 4. Time-dependent absorption spectra of **1** in hexane (a and b) and CH_2Cl_2 (c and d) under UV irradiation ($\lambda_{exc} = 360$ nm, $I \approx 4$ mW/cm^2, 10 mm quartz cuvettes). The dye concentrations were: 10^{-3} M (a and c) and 10^{-6} M (b and d). Inserts are the changes in the main absorption peak.

reported [14]. The photochemical stability of **1** was measured by the absorption and fluorescence methods described above.

3.1. One-photon excitation

The absorption method was used for the determination of the photoreaction quantum yields in air-saturated and deoxygeneted solutions under one-photon excitation. The changes in the absorption spectra of **1** in hexane and CH_2Cl_2 under UV irradiation (UVGL-25, $\lambda_{exc} \approx 355$ nm) are presented in Fig. 4 as a function of dye concentration. From these spectra, it was observed that the photobleaching processes of **1** are complex, resulting in variable rates of photobleaching upon irradiation (Fig. 4a,c and d), with the exception of low concentrated hexane solution in which the photobleaching rate was essentially constant (Fig. 4b). Therefore, based on these data, the quantum yields of the photoreactions, Φ_i, were calculated by Eq. (1) for each small intermediate change in the optical density, ΔD_i, at the corresponding irradiation time, $\Delta t_i = 15$ min or 30 min ($i = 1 - n$, n is the number of measurements). The averaged values of $\Phi = \Sigma\Phi_i/n$ for the

Table 1
Quantum yields of the photoreactions for **1** in air saturated, Φ, and deoxygenated, Φ^d, solutions with different concentrations, C, under one- (Φ, Φ^d) and two-photon (Φ_{2PA}) excitation

Concentration,	Hexane			CH_2Cl_2		
C (M)	$\Phi \times 10^5$	$\Phi^d \times 10^4$	$\Phi_{2PA} \times 10^5$	$\Phi \times 10^5$	$\Phi^d \times 10^4$	$\Phi_{2PA} \times 10^3$
1.3×10^{-3}	5 ± 2	–	5 ± 3	4 ± 1.5	–	2 ± 1
3×10^{-4}	4.5 ± 1.5	1.2 ± 0.4	4.5 ± 2	3.5 ± 1	1.2 ± 0.4	1.5 ± 0.5
8×10^{-5}	4 ± 1.3	1.5 ± 0.5	3 ± 1	4 ± 1.2	1.3 ± 0.4	2 ± 1
2×10^{-5}	5 ± 1.5	1.2 ± 0.4	3.5 ± 1.2	3 ± 1.3	1 ± 0.3	0.8 ± 0.4
3×10^{-6}	3.5 ± 1	1.4 ± 0.4	3 ± 1	2.5 ± 0.8	0.8 ± 0.25	0.9 ± 0.45

entire irradiation time are given in Table 1. The photochemical quantum yield was (2.5–5) × 10^{-5}, and exhibited a weak dependence on dye concentration for both solvents. This suggests that the photochemical processes of **1** in hexane and CH_2Cl_2 correspond primarily to first order photoreactions, especially at low concentrations in hexane ($C \leq 10^{-5}$ M), when a constant rate of photobleaching was observed.

As a result of photoreaction, an absorption band of the photoproducts of **1** appeared at ≥430 nm in CH_2Cl_2 (Fig. 4c and d). This is in contrast to what was observed in hexane solutions (Fig. 4a and b), where changes occurred in the spectral range corresponding to the main absorption band (<420 nm). The appearance of the longer wavelength absorption, in the spectral region ≥430 nm, is likely due to an electron transfer reaction of **1** (for example, electron transfer from the nitrogen of the diphenylamino group to polar CH_2Cl_2), resulting in the formation of a stable cation radical [18]. In hexane, no long wavelength absorbing photoproducts were observed, consistent with electron transfer only occurring in electron accepting solvents. In this case, photoproducts were generated that only absorbed in the shorter wavelength spectral region ($\lambda \leq 350$ nm).

Deoxygenation of the solutions led to an increase in the photochemical quantum yields of **1** by two to five times (Table 1), i.e. a decrease in photostability. These results suggest an unexpected role of molecular oxygen in the photostability of **1**, possibly quenching a potentially reactive excited state species, e.g. a triplet state leading to efficient return to ground state.

Nearly the same values of photochemical quantum yields, Φ, for **1** in hexane and CH_2Cl_2 were obtained under one-photon excitation by the previously described fluorescence method. The Φ values determined from the fluorescence method (Xe-lamp irradiation) were in a good agreement with those obtained by the absorption method (UV-lamp UVGL-25).

3.2. Two-photon excitation

The quantum yields of the photochemical reactions for **1** in hexane and CH_2Cl_2 under two-photon excitation ($\lambda_{exc} = 760$ nm; $\tau_P \approx 120$ fs; $f = 1$ kHz) were obtained by the fluorescence method (Fig. 3). The temporal dependences of the fluorescence intensity of **1**, $F(t)$, were measured under two-photon irradiation, and the quantum efficiencies of the photoreactions, Φ_{2PA}, were calculated using Eq. (3). The values of Φ_{2PA} for different dye concentrations are listed in Table 1. The analysis of these data reveals a weak dependence of Φ_{2PA} on dye concentration, very similar to the photochemical behavior of **1** under one-photon excitation. Thus, a dominant role of first order photoreactions can also be assumed.

The Φ_{2PA} for **1** in CH_2Cl_2 increased 30–50 times relative to those obtained for low intensity one-photon irradiation, reaching a maximum of $\Phi_{2PA} \approx 2 \times 10^{-3}$. In contrast, nearly the same photochemical quantum yield for **1** was observed for both one- and two-photon excitation in hexane, i.e. $\Phi \approx \Phi_{2PA} \approx (3–5) \times 10^{-5}$. In order to understand this photochemical behavior, the influence of the excitation intensity, I, on the efficiency of the photobleaching was studied. Fig. 5 illustrates the temporal changes in the optical density, $\Delta D/\Delta t$, on I^2 for the solutions of **1** in hexane and CH_2Cl_2 under two-photon excitation. For purely a two-photon excitation process, a quadratic dependence of $\Delta D/\Delta t$ on I can be expected.

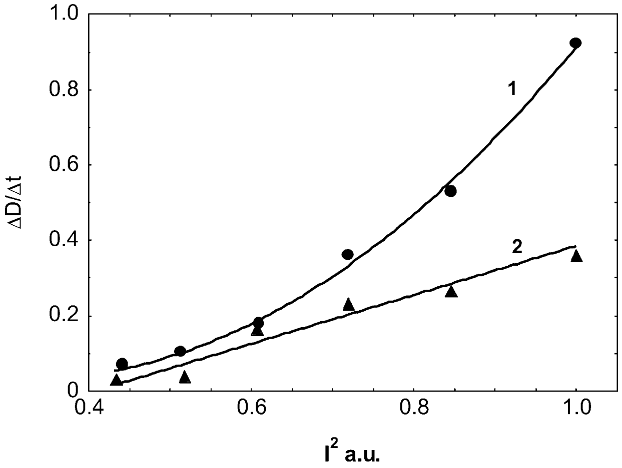

Fig. 5. Temporal changes in the optical density, $\Delta D/\Delta t$, of **1** as a function of the square of excitation intensity, I^2, in CH_2Cl_2 (1) and hexane (2).

In general, the value of $\Delta D/\Delta t$ can be expressed as:

$$\frac{\Delta D}{\Delta t} \sim \sigma_{2PA} I^2 (k_1 + \sigma_{1n} I\, k_n \tau_n)\tau_1, \tag{4}$$

where k_1, k_n and τ_1, τ_n are the rate constants of the photochemical decomposition and lifetimes of the first, S_1, and higher excited, S_n, electronic states of **1**, respectively; σ_{1n} is the one-photon absorption cross-section for the transition $S_1 \rightarrow S_n$ at $\lambda_{exc} = 760$ nm. In Fig. 5, a quadratic dependence of $\Delta D/\Delta t$ on I in hexane can be seen (i.e. $\Delta D/\Delta t \sim I^2$), suggesting the dominance of photoreaction from the first excited state, S_1, i.e. $k_1 \gg \sigma_{1n}\, I\, k_n\, \tau_n$ (since 760 nm radiation is only energetic enough to produce two-photon absorption into S_1 or the longer wavelength absorption band). In contrast, there was a substantial increase in the photochemical decomposition of **1** in CH_2Cl_2, relative to hexane, and corresponded to a cubic dependence $\Delta D/\Delta t \sim I^3$ (Fig. 5, curve 1). This dependence can be explained by an additional one-photon absorption processes from S_1 at $\lambda_{exc} = 760$ nm, and effective photochemical reaction from the higher excited state S_n, when $k_1 \ll \sigma_{1n}\, I\, k_n\, \tau_n$. Hence, more efficient photochemical decomposition of **1** was observed from higher excited electronic states.

4. Conclusions

The photochemical decomposition of fluorene 1 in hexane and CH_2Cl_2 under one-photon (linear) excitation resulted in the quantum yield of the photoreaction $\Phi \approx (2.5–5) \times 10^{-5}$. Under these conditions, the photodecomposition was approximately a first order process. In air-saturated solutions, the one-photon photodecomposition of **1** is suppressed two to five times relative to that in deoxygenated solutions, revealing an important role of molecular oxygen in the photostability of compound **1**.

Under two-photon excitation, the quantum yield of the photodecomposition for **1** in hexane was nearly the same, $\Phi \approx (3–5) \times 10^{-5}$ as under single-photon excitation and independent of irradiation intensity. This can be ascribed to a dominant photoreaction occurring from the first excited state of **1**, and to photoprocesses similar to those observed for one-photon excitation. In contrast, the quantum yields of the photochemical reactions of **1** in CH_2Cl_2 under two-photon excitation increased by 30–50 times ($\Phi_{2PA} \approx 2 \times 10^{-3}$) relative to single-photon excitation, and exhibited a clear dependence on the irradiation intensity. This behavior can be explained by an additional one-photon absorption from the first excited state, followed by subsequent photoreaction from higher electronically excited states.

Overall, **1** exhibited relatively good photostability, making it suitable for use in linear and nonlinear optical applications.

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