Excited state behaviour of acridinedione dyes in PMMA matrix: inhomogeneous broadening and enhancement of triplet

V. Thiagarajan, C. Selvaraju, P. Ramamurthy*

Department of Inorganic Chemistry, University of Madras, Guindy Campus, Chennai 600025, India

Abstract

Photophysics and photochemistry of acridinedione (ADD) dyes doped in PMMA matrix have been studied using fluorescence and flash photolysis technique. The absorption and emission spectra of acridinedione dyes in PMMA matrix are broad. A pronounced influence of λ_{exc} on the red shift of ADD fluorescence spectra was found which is independent of the various substituents in the ADD dyes. This observed shift was explained by the inhomogeneity in the PMMA matrix site results in the migration of the electronic excitation energy from the blue centres to red ones. The inhomogeneous distribution of ADD dyes in PMMA matrix causes the non-exponential fluorescence decay. The triplet lifetimes of these dyes in polymer matrix is found to be in millisecond time domain, whereas in solution the triplet lifetime is in microseconds. Laser flash photolysis shows that the photoionisation of ADD dyes in PMMA matrix results in the formation of an electron and a cation radical. Steady state photolysis of ADD dyes in PMMA matrix was carried out. ADD dyes have higher photostability in PMMA matrix than in solution.
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Keywords: Acridinedione dyes; PMMA matrix; Inhomogeneous broadening; Triplet enhancement; Higher photostability

1. Introduction

The influence of molecular structure and motion of polymer matrices on the photochemical and photophysical processes of molecularly doped chromophores is a topic of current interest. Over the last decade, important advances in the preparation of inorganic glasses have taken place through the application of the sol–gel method [1]. Organic molecules can be trapped within the ceramic matrix if they are dissolved in the medium during gelation [2]. Dyes have been used as probes to monitor the gelation process and as permanent guests imparting to the glass specific photoreactivity, such as photoconductivity [3], photochemical hole-burning capabilities [3] and photochromism [4]. One of the important advantages of transparent polymers compared with inorganic glasses and crystals are that they exhibit superior optical homogeneity, which is extremely important in fabricating conventional optical elements for various laser applications [5–7]. The most frequently used polymeric material is poly(methyl methacrylate) (PMMA) because of its better optical transparency and its superior resistance to laser damage [8].

Polymer matrices have been considered as a convenient media for spectroscopic investigation of excited triplet states over wide temperature ranges and the use of photophysically detectable probes allow the investigation of the structure of polymer matrices and of photophysical transitions connected with changes in the mobility of certain structural units. Such studies are connected with the practical interests connecting the reactivity of low molecular compounds embedded in polymer matrices in photomemory and photosensitive polymer systems, and of the reactivity of additives admixed to polymers as stabilisers against photodegradation and thermal degradation [9].

The lifetime of the excited state depends on a number of factors such as environment, energy difference between the ground and excited states, type of chromophore, etc. The solid state environment enables the molecule to enhance the lifetime at its excited state than the liquid state and gaseous phase and is due to its rigid nature of the solid surroundings. Difference between the solid state reactions and those in solution is that of the molecular motion due to restriction of mobility of reactant in solids. It is best studied by using a polymer matrix such as poly(methyl

* Corresponding author. Present address: National Centre for Ultrafast Processes, University of Madras, Taramani Campus, Chennai 600113, India. Tel.: +91-44-24480962; fax: +91-44-24926709.
E-mail address: prm60@hotmail.com (P. Ramamurthy).

methacrylate) and polystyrene, which form a transparent glassy substances even at room temperature. Poly(methyl methacrylate) is an organic glass and usually regarded as an inert matrix for photochemical and photophysical processes.

Polymer matrices have much influence on the triplet lifetime of organic molecules. Non-exponential decay of triplet state was reported for various aromatic hydrocarbons, doped in polymer matrices [10–13]. The photophysics of pyrene in organic glassy polymers was examined by Avis and Porter for PMMA glasses [14]. Fujiwara et al. studied the drastic changes of fluorescence dynamics in pyrene doped PMMA latex microspheres [15].

Acridinedione dyes have been reported as a new class of laser dyes with lasing efficiency comparable [16–19] to that of coumarin-102 and these dyes have structural similarities with NADH. These dyes have been shown to mimic the NADH analogs to greater extent because of its tricyclic structure, which is capable of protecting the enamine moiety [20]. The photophysical and photochemical properties of acridinedione dyes in solution have been extensively studied [21–27]. In the present investigation, acridinedione dyes were doped into the PMMA matrix. The flash photolysis of acridinedione dyes was carried out using conventional flash photolysis and laser flash photolysis techniques. The following dyes were selected for the investigation.
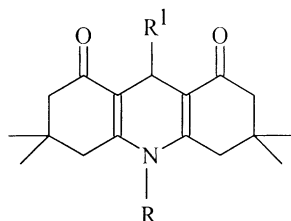
ADD-1	:	R = H,	R^1 = CH_3
ADD-2	:	R = H,	R^1 = C_6H_5
ADD-3	:	R = CH_3,	R^1 = CH_3

2. Experimental

The dyes 1–3 used in the study were prepared following the methods described in the literature [25]. The experiments were carried out using methanol as solvent (HPLC grade, Qualigens, India Ltd.). The methyl methacrylate (MMA) was purchased from E. Merck. Azobisisobutyronitrile (AIBN) was purchased from Fluka.

2.1. Purification of MMA

The methyl methacrylate was extracted with 2M NaOH solution, washed several times with water and dried over CaCl$_2$ and then distilled twice under reduced pressure and kept at low temperature to prevent polymerisation.

2.2. Preparation of ADD dyes in MMA polymer matrix [28]

The ADD dyes were dissolved in 25 ml of purified monomer to give a solution with concentrations of 1×10^{-4} to 7×10^{-4} M. After the addition of 0.015 g AIBN to the solution in a stoppered pyrex tube of diameter 2.5 cm covered with aluminium foil, was immersed in a water bath at 50–60 °C for 12–18 h. This method gives reproducible high quality PMMA polymeric glasses. The solid samples were then annealed at 110 °C for 2 h before cut and polished to appropriate shapes. The absorption spectra of ADD dyes were found to be independent of the AIBN addition which confirms that there is no degradation of ADD dyes during the polymerisation processes.

Absorption spectra were recorded on a Hewlett–Packard 8452A diode array spectrometer. Steady state photolysis experiments were carried out using a Spectroline low-pressure mercury pencil ray lamp with a maximum output at 365 nm. The intensity of light reaching the matrix has been measured to be 3.04×10^{15} photons per second. Conventional flash photolysis experiments were carried out using an Applied Photophysics KN-020 series flash kinetic spectrometer fitted with a Czerney Turner monochromator and a Hamamatsu R928 photomultiplier tube as detector and equipped with an IE-567 Digitest Lab, India oscilloscope for signal capture and display. Laser flash photolysis experiments were carried out using the third harmonic output from a Quanta Ray GCR-2 Nd-YAG laser (355 nm, 8 ns). The monitoring source was a 250 W pulsed xenon lamp. The transient signals were captured with a 54201A Hewlett–Packard digital storage oscilloscope interfaced to a PC via GPIB-IEEE 488 board and the analysis of the data were done using an in-house developed software described elsewhere [29].

Fluorescence spectra were recorded using a Perkin-Elmer LS5B luminescence spectrometer. Fluorescence decay was recorded using TCSPC method using the following set-up. A diode pumped millennia CW laser (Spectra Physics, 532 nm) was used to pump the Ti–Sapphire rod in Tsunami picosecond mode locked laser system (Spectra Physics). The 750 nm (82 MHz, 2.4 ps FWHM) was taken from the Ti–Sapphire laser and passed through pulse picker (Spectra Physics, 3980 2S) to generate 4 MHz pulses. The second harmonic output (375 nm) was generated by a flexible harmonic generator (Spectra Physics, GWU 23PS). The vertically polarised 375 nm laser was used to excite sample. The incident power at the sample compartment is 0.3 mW. The fluorescence emission at magic angle (54.7°) was dispersed in a monochromator (f/3 aperture), counted by a MCP PMT (Hamamatsu R 3809) and processed through CFP, TAC and MCA. The instrument response function for this system is ∼52 ps. The fluorescence decay is obtained which is further analysed using IBH software (DAS6).
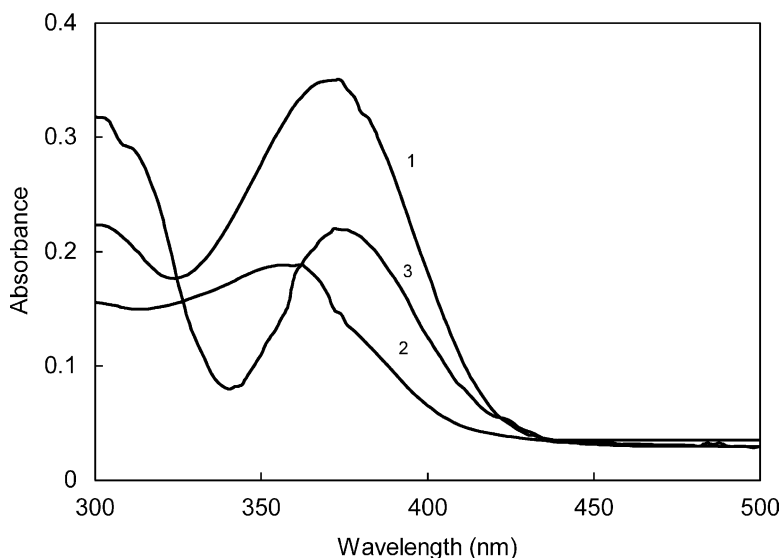
Fig. 1. Absorption spectrum of ADD dyes in PMMA matrix: (1) ADD-1; (2) ADD-2; (3) ADD-3.

3. Results and discussion

3.1. Absorption and fluorescence

Acridinedione dyes have the absorption maximum around 360–374 nm in PMMA matrix. The absorption spectra of ADD dyes in PMMA matrix are shown in Fig. 1. The emission spectra of ADD dyes in PMMA matrix were recorded by exciting at the absorption maximum. The emission spectrum of ADD dyes in PMMA matrix are shown in Fig. 2. The absorption and fluorescence spectral data of ADD dyes in PMMA matrix and methanol are compiled in Table 1. In comparison with methanol, the absorption and fluorescence maximum of ADD dyes are blue shifted with broadening (around 10 nm measured at FWHM) in the respective spectrum. Solvent effect on the absorption and emission spectral properties of ADD dyes have been thoroughly studied and reported by Srividya et al. [26]. On decreasing the solvent polarity the absorption and emission maximum of ADD dyes are blue shifted. Based on the solvent effect, the absorption and fluorescence of ADD dyes are assigned to intramolecular charge transfer. The blue shift in the absorption and
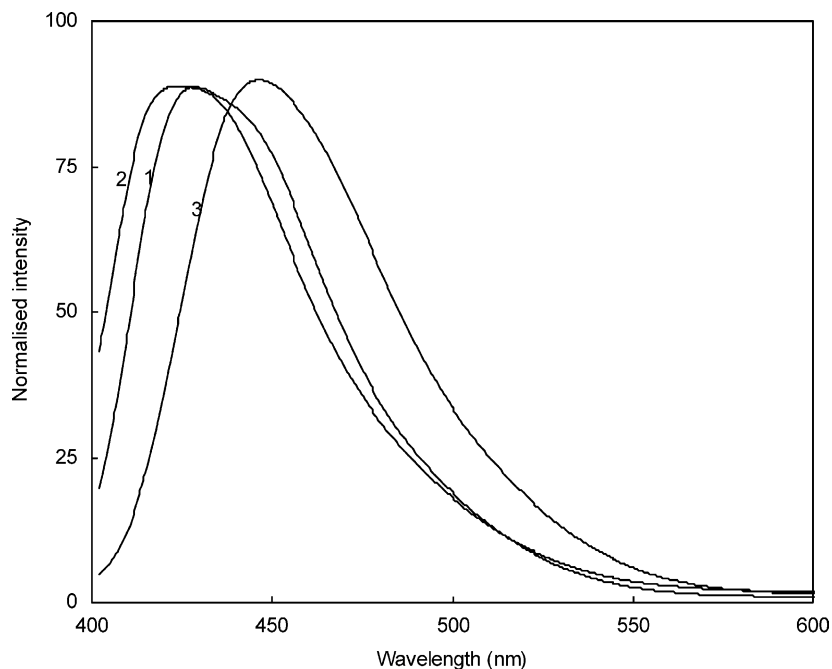

Fig. 2. Emission spectrum of ADD dyes in PMMA matrix: (1) ADD-1; (2) ADD-2; (3) ADD-3.

Table 1
Absorption and emission maxima of ADD dyes in PMMA matrix and in methanol

Dyes	Absorption		Emission	
	PMMA matrix (nm)	Methanol (nm)	PMMA matrix (nm)	Methanol (nm)
ADD-1	371	380	425	447
ADD-2	364	375	420	437
ADD-3	374	384	443	464

fluorescence maximum of ADD dyes in PMMA matrix indicates that the dyes experience a less polar environment compared to methanol. The fluorescence maximum are greatly influenced by solvents than the absorption maximum which indicates that the excited state is more polar than the ground state.

3.2. Inhomogeneous broadening

The absorption and fluorescence spectra of ADD dyes in PMMA matrix are broad when compared to the same in solution. The fluorescence maximum of ADD dyes depends on the excitation wavelength. The fluorescence spectra of ADD-1 in PMMA matrix at different excitation wavelength are shown in Fig. 3. A red shift of the fluorescence maximum by 50 nm (from 402 to 452 nm) is observed while shifting the λ_{exc} from 340 to 400 nm in PMMA matrix. The substituent in the 9th position and on the nitrogen of the ADD dyes does not play any role in the above observation.

The observed shift in the fluorescence maximum with λ_{exc} can be explained by the trapping of ADD dyes in different environments of the PMMA matrix. Their relaxation during the excited state lifetimes is limited by the free volume (mobility in the matrix cage) present in these sites. During the

polymerisation process it is known that the dyes are trapped in different sites having different free volume and environment [30,31]. Excitation of ADD in fluid media will lead to fast relaxation to a most stable solvent relaxed state and the emission originates from this state. So in fluid media, the emission maximum is independent of excitation wavelength. In polymer matrix, the relaxation rate constant is smaller than the fluorescence rate constant and hence the emission will occur from a excited state corresponding to the solvation site specifically excited. In the rigid polymer matrix, the dynamic equilibrium between the different solvation sites is lost and excitation of ADD dyes in a given solvation site will lead to an emission of the ADD in that solvation site, which results in the excitation wavelength dependence of fluorescence of ADD in polymer matrix [30,31]. The magnitude of the shift depends on the environment in the matrix generating a local electric field [32].

The excitation spectrum of ADD-1 dye in PMMA matrix monitoring at different emission wavelength is shown in Fig. 4. The excitation maximum shifts towards red region on shifting the monitoring emission wavelength from 425 to 500 nm. Depending on the environment the excitation energy of ADD dyes varies. The monitoring emission wavelength dependence of the excitation spectrum of ADD dyes confirms the inhomogeneous environment in PMMA matrix. Based on the above results, the broadening of absorption spectrum is attributed to the inhomogeneous distribution of ADD dyes in PMMA matrix. Similar to the excitation wavelength dependency of the emission spectra, emission wavelength dependency of the excitation spectra also independent of the substituent in the 9th position and the nitrogen of the ADD dyes.

The emission spectral broadening of ADD dyes in matrix is due to the inhomogeneous environment in the PMMA matrix which leads to the migration of the excitation
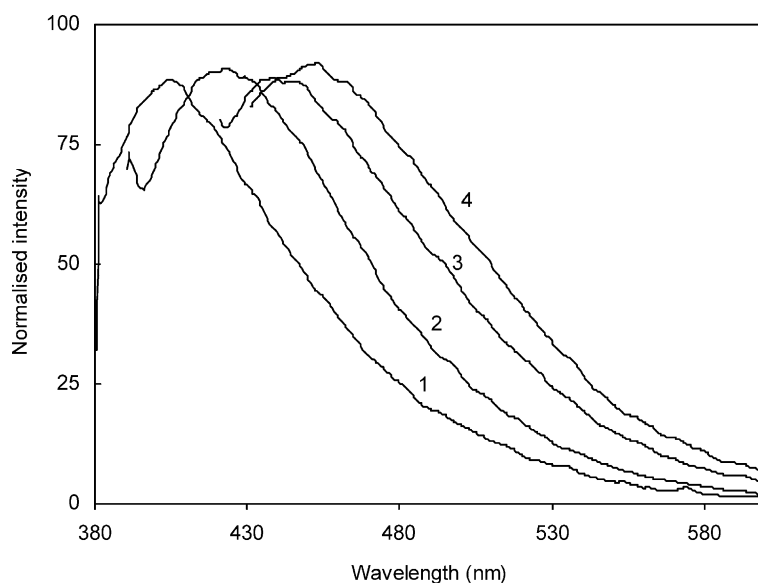

Fig. 3. Emission spectrum of ADD-1 at different excitation wavelengths: (1) 340 nm; (2) 360 nm; (3) 380 nm; (4) 400 nm.
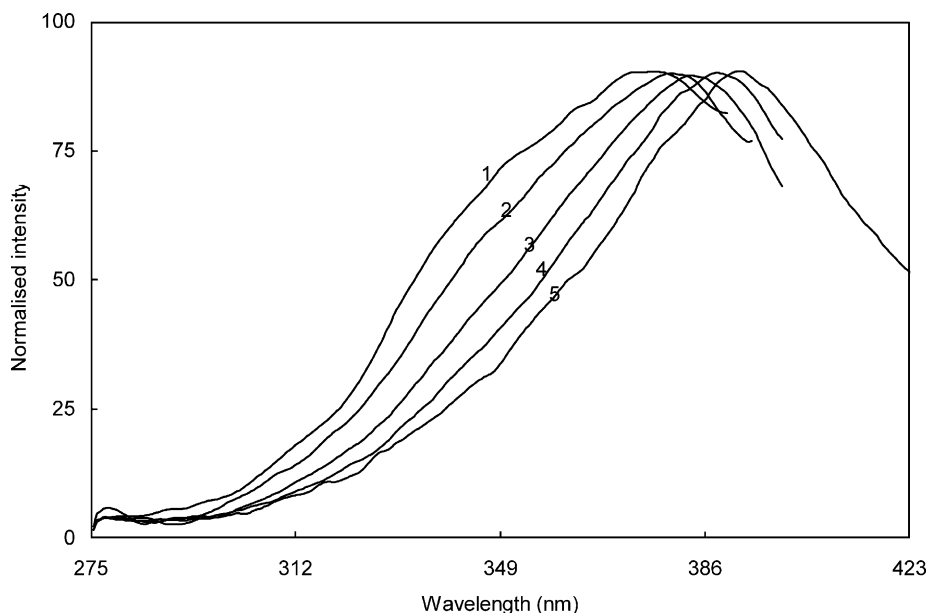
Fig. 4. Excitation spectrum of ADD-1 at different emission wavelengths: (1) 425 nm; (2) 430 nm; (3) 450 nm; (4) 475 nm; (5) 500 nm.

energy from the blue centres to the red one. Zurkowska and co-workers reported this type of energy migration in a set of flavomononucleotide (FMN) in PVA films [33,34]. Fig. 5 shows the scheme of energy levels of fluorescent centres of ADD molecules in PMMA matrix. The photoselection of the blue centres with highest energy E_0^A is followed by non-radiative energy transfer to the centres with low energy E_0. Similarly each respective group of centres emit characteristic fluorescence with a red shifted maximum. The excitation of the system at $\lambda_{exc} = 360$ nm does not show the fluorescence contribution of the groups A and the excitation at $\lambda_{exc} = 380$ nm does not show the contributions of the group B in the total fluorescence.

3.3. Fluorescence lifetime

The fluorescence decay curve of all the ADD dyes in methanol show single exponential on excitation at 375 nm. In PMMA matrix, the fluorescence decay curve of ADD dyes does not obey single exponential fit, but they are satisfactorily fitted by the triexponential function according to the equation

$$I(t) = A + \sum_i B_i \exp\left(\frac{-t}{\tau_i}\right)$$

where B_i and τ_i are the pre-exponential factors and fluorescence lifetimes. The multiexponential analyses of the
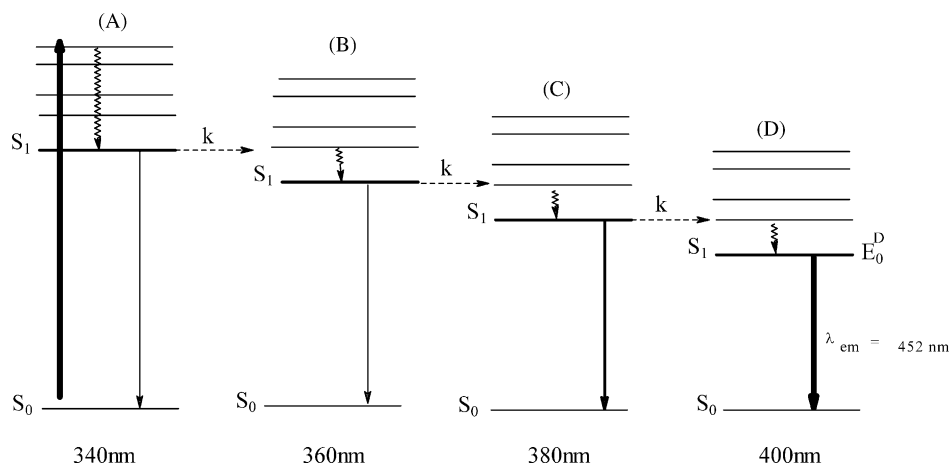

Fig. 5. Illustration of the effect of inhomogeneous broadening of energy levels on the mechanism of energy migration in ADD dyes in polymer matrix; k rate constant for energy transfer.
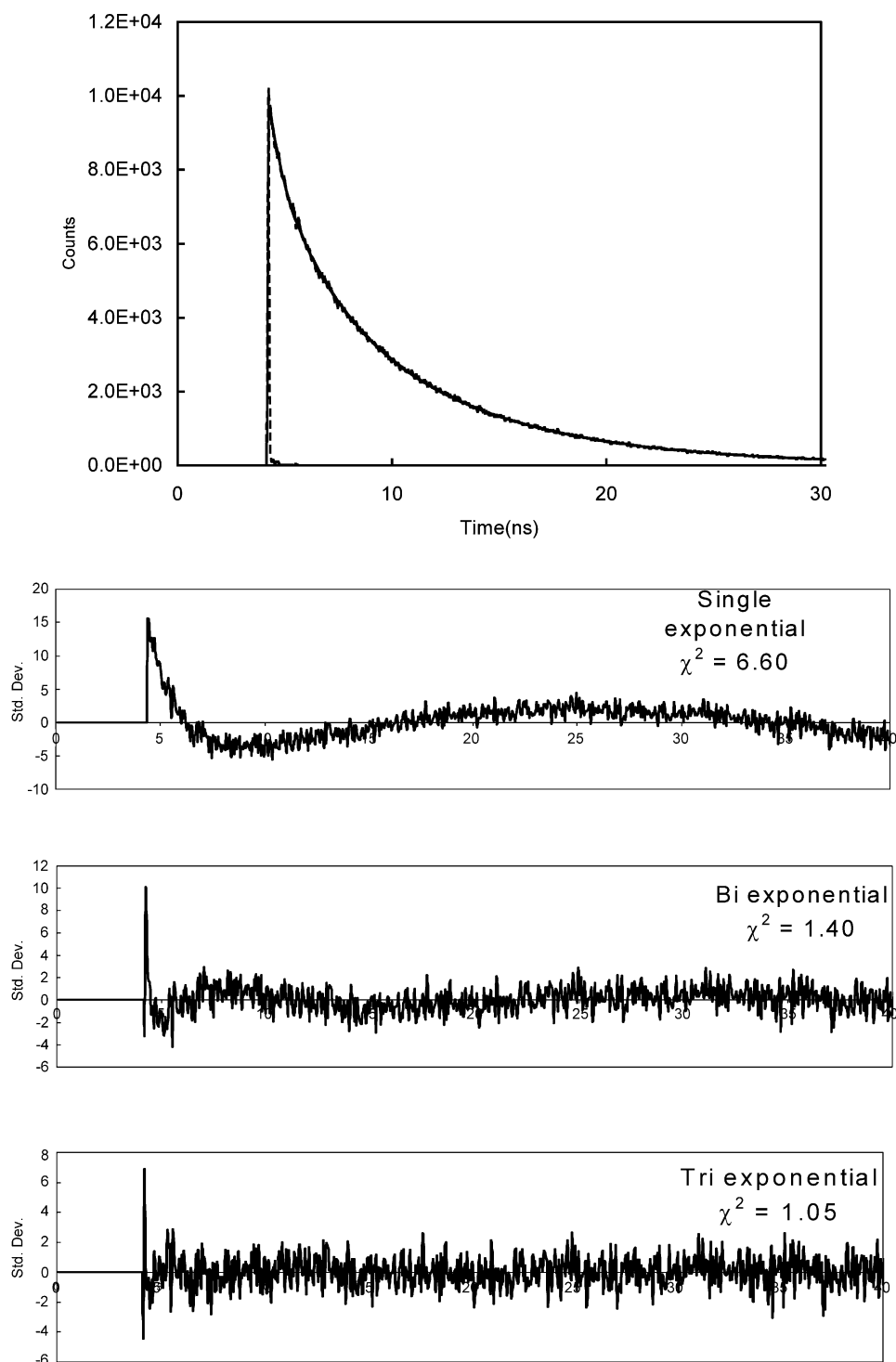
Fig. 6. Fluorescence decay of ADD-1 in PMMA matrix, emission monitored at 430 nm.

fluorescence decay of ADD-1 in PMMA matrix are shown in Fig. 6. The lifetimes of all the ADD dyes monitored at the respective emission maximum (Table 1) is shown in the Table 2. The triexponential fit is only an approximation to the non-exponential decay of ADD dyes trapped in different sites of the PMMA matrix.

3.4. Flash photolysis

Conventional flash photolysis of ADD dyes in PMMA matrix were carried out using a cylindrical matrix of 5 cm length. The transient absorption spectrum of ADD dyes in PMMA matrix is shown in Fig. 7. All the ADD dyes exhibit

Table 2
Fluorescence lifetimes of ADD dyes in PMMA matrix monitored at respective emission maxima

Dyes	τ_1 (ns)	τ_2 (ns)	τ_3 (ns)	χ^2
ADD-1	1.4 ± 0.2 (4.76)	3.9 ± 0.4 (32.81)	8.1 ± 0.5 (62.43)	1.049
ADD-2	1.3 ± 0.2 (6.40)	3.2 ± 0.4 (31.46)	7.7 ± 0.5 (72.14)	1.036
ADD-3	1.5 ± 0.2 (1.41)	4.2 ± 0.4 (24.47)	9.7 ± 0.5 (74.11)	1.039

transient absorption maximum in the region of 610–650 nm and the transient decay follows first-order kinetics.

Srividya et al. reported a similar transient absorption maximum in the laser flash photolysis of ADD dyes in organic solvents such as methanol and acetonitrile [27]. The triplet lifetime of ADD in PMMA matrix is found to be in the order of milliseconds, whereas in solution (methanol) the triplet lifetime is found to be in the microsecond range. The triplet lifetime and absorption maximum of ADD dyes in PMMA matrix and in methanol are given in Table 3. The increase in the triplet lifetime is attributed to the stabilisation of triplet state by the rigid environment of the polymer matrix. In homogeneous solution, collisions between the solvent molecules and ADD dyes can effectively deactivate the triplet state of ADD via non-radiative pathway. Such collisions are not be possible in PMMA matrix.

Table 3
Triplet absorption maxima of ADD dyes in PMMA matrix and in methanol

Dyes	In PMMA matrix		In methanol	
	Absorption maximum (nm)	Lifetime (ms)	Absorption maximum (nm)	Lifetime (μs)
ADD-1	620	54.9 ± 0.2	620	1.5 ± 0.2
ADD-2	640	112.0 ± 0.2	640	17.3 ± 0.2
ADD-3	640	44.4 ± 0.2	640	7.9 ± 0.2

Laser flash photolysis of ADD dyes were carried out in methanol and also in PMMA matrix. The transient absorption spectrum of ADD-3 in methanol and PMMA matrix is shown in Fig. 8c. Both in methanol and PMMA matrix the triplet absorbs around 610–650 nm. The triplet decay of ADD-3 in PMMA and methanol monitored at 620 nm is shown in Fig. 8b. In methanol triplet decays within 100 μs and in PMMA matrix decay lives upto millisecond. ADD
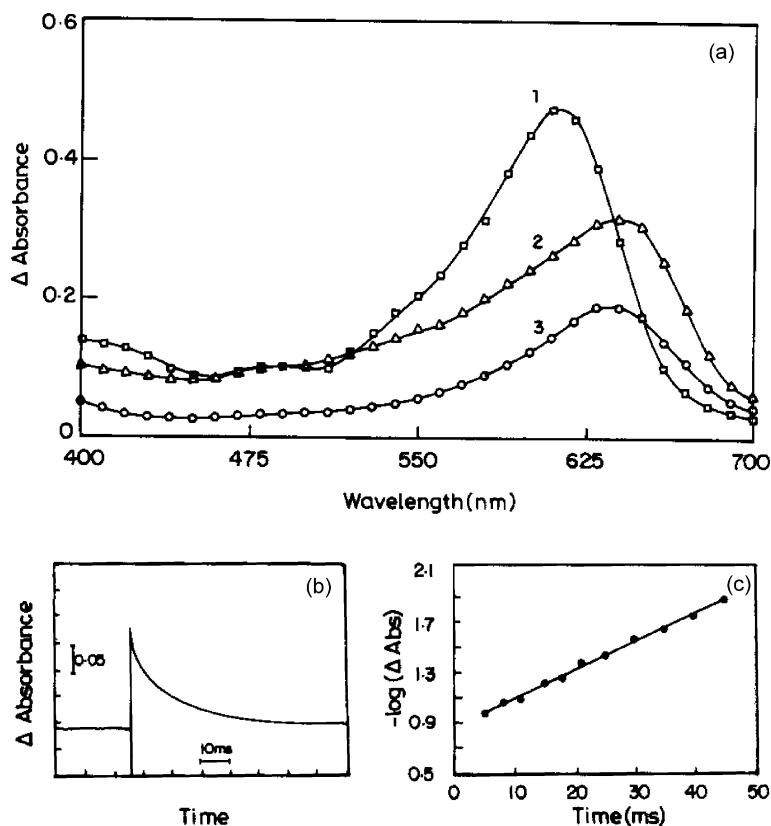

Fig. 7. Conventional flash photolysis of ADD dyes in PMMA matrix: (a) transient absorption spectrum of ADD dyes in PMMA matrix recorded immediately after flash: (1) ADD-1, (2) ADD-2, (3) ADD-3; (b) transient decay of ADD-3 monitored at 640 nm; (c) kinetic analysis of ADD-3 of the transient decay monitored at 640 nm.
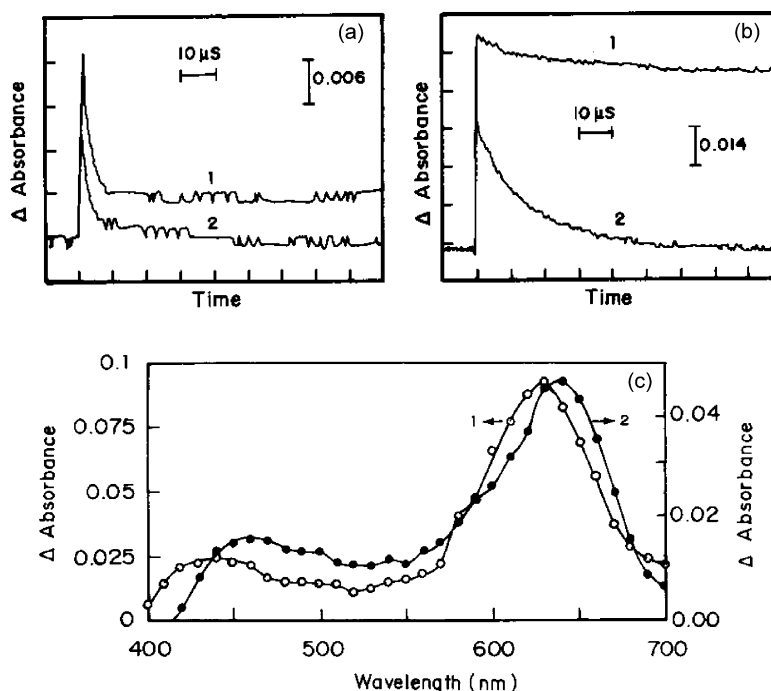
Fig. 8. Laser flash photolysis of ADD-3 in PMMA matrix (1) and in methanol (2): (a) transient decay monitored at 440 nm; (b) transient decay monitored at 640 nm; (c) transient absorption spectrum of dye recorded 2 μs after the laser flash.

dyes show another transient absorption maximum at 440 and 460 nm in PMMA matrix, methanol, respectively.

Recently, Mohan and Mittal [35] reported the pulse radiolysis of ADD dyes in organic matrix. Pulse radiolysis of ADD in an organic matrix shows transient absorption at 420–440 nm. The transient in this region is assigned to the cation radical of ADD. Photoionisation of acridinedione dyes in PMMA matrix results in cation radical and an electron. The mobility or diffusion of electron is prevented in PMMA matrix and this prevents the further reaction of electron with ADD molecules. The 420–440 nm transient in PMMA matrix is due to the cation radical of ADD. The recombination of electron and cation radical occurs predominantly in PMMA matrix. The recombination reaction favours the triplet induction when the ion pair energy is higher than the triplet state of ADD. The enhancement of triplet absorption in PMMA matrix is nicely explained by the triplet induction by the ion pair recombination. The recombination
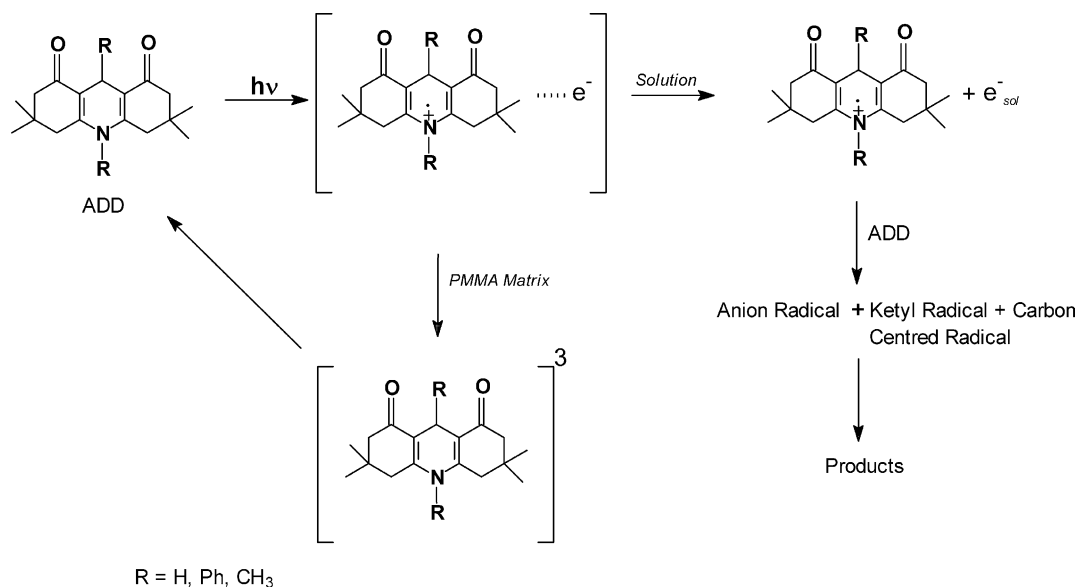
Scheme 1.

results in the formation of triplet state of ADD which is reflected in the enhancement of triplet absorption of ADD dyes in PMMA matrix (Fig. 8b). Scheme 1 illustrates the photochemical reactions of ADD dyes in PMMA matrix and in solution.

The photochemical oxidation of ADD dyes have been reported [27]. ADD dyes undergo photoionisation in the excited state to result a solvated electron and a cation radical. The solvated electron reacts with the ground state molecule to form an anion radical. The anion radical of ADD dyes absorb at 460 nm, which is supported by the pulse radiolysis of ADD dyes in the presence of one electron reductant [36]. The 460 nm transient observed in methanol is due to the anion radical of the ADD dyes.

In solution, steady photolysis of ADD leads to the formation of different products depending on the substituents and the conditions of photolysis. The intermediate anion radical, ketyl radical and carbon centred radical are responsible for the formation of photoproducts [27]. On steady photolysis of ADD in matrix, there is no change in the absorbance even after photolysis for 5 h and this observation support the following discussion. In PMMA matrix, these intermediates are not formed and there is no product formation of ADD on photolysis. ADD has higher photostability in PMMA matrix and this enables the usage of these dyes for dye laser application in the matrix.

4. Conclusion

The absorption and emission spectra of ADD dyes in polymer matrix were studied. The pronounced influence of λ_{exc} on the longer wavelength shift in the fluorescence spectra of ADD dyes in PMMA matrix were observed. This is due to the inhomogeneous distribution of ADD dyes in different sites of PMMA matrix leads to the electronic energy migration from the blue centres to red ones. The deviation of single exponential decay of fluorescence lifetime is due to the different distribution of ADD energy levels in PMMA matrix. The above said photophysical properties do not depend on the substituents present in the ADD dyes where as it depends only on the heterogeneity of the PMMA matrix. The ADD dyes have long lived triplet lifetime in the PMMA matrix and this is due to the rigid structure of the matrix. Flash photolysis shows that the photoionisation of ADD dyes in PMMA matrix results in the formation of cation radical and an electron. In PMMA matrix, the recombination of the electron and the cation radical results in the formation of triplet, thus the formation of an anion radical is prevented in the matrix. ADD dyes have higher photostability in PMMA matrix compared to that of in solution.

Acknowledgements

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