Spectral Behavior and Photophysical Parameters of Dihydrophenanthro[9,10-e][1,2,4]Triazine Derivative Dyes in Sol–Gel and Methyl Methacrylate Polymer Matrices

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Abstract
This paper deals with the optical and photophysical properties of dihydrophenanthro[9,10-e][1,2,4]triazine fluorescent dyes doped in Silicate based sol–gel and homo-poly methyl methacrylate (PMMA). Solid hosts were found to effect on the optical and photophysical parameters such as molar absorptivity, cross sections of singlet–singlet electronic absorption and emission spectra, excited state lifetime, quantum yield of fluorescence. The dipole moment of electronic transition, the length of attenuation and oscillator strength of electronic transition from $S_0 \rightarrow S_1$ have been calculated. The dyes were pumped with different powers using 3rd harmonic Nd:YAG laser of 355 nm and pulse duration 8 ns, with a repetition rate at (10 Hz). Good photostability for compounds 1 and 2 were attained. It was decreased to 49% & 54% and 46% & 40% of the initial ASE of dyes in sol gel and PMMA, respectively, after 55,000 pumping pulses at (10 mJ/pulse). The dyes in sol–gel showed improved photostability compared with those in organic polymeric matrices.

Keywords Optical property · Solid state laser dye · Photophysical parameters · Photostability · Silicate based sol–gel · Polymeric matrices

Introduction
In recent years, much attention has been paid to the synthesis of new high-performance dyes as well as the implementation of new ways of incorporating these dyes into the solid matrix. This have resulted in significant advances towards the development of practical tunable solid-state dye lasers [1–3], due to their high efficiency. Moreover, they do not contain volatile solvents, so they are nonflammable and nontoxic. They are also compact in size and mechanically and thermally more stable [4]. On the other hand, using liquid dye lasers causes several disadvantages like poor thermal stability of dye solutions, requirement of large volume of solutions, the need of significant maintenance, as well as high rate of non-radiative processes due to the high flexibility of dye in solution. [5–8]. To overcome these difficulties, doping dye molecules into restricted host matrices like polymers, silica gels, xerogels and sol–gel glasses was performed as an efficient technique [8, 9]. The sol–gel method is a technique for incorporating organic dye molecules into an inorganic silica host [10]. This process showed some advantages, e.g., compactness, better manageability and highly porous materials, transparent in Uv visible–near IR regions. Its reaction can be controlled easily by chemical methods. It also allows introducing permanent organic groups to form inorganic–organic hybrid materials [11] and the process takes place at low temperature [12].

Fused heterocyclic systems that contain nitrogen were reported to exhibit fluorescent activity [13]. They are also widely applied as LEDs, lasers of semiconductors, probes, and fluorescent sensors. In the development of organic LED (OLED) technologies trends are focused primarily on optimizing existing devices and developing new emission materials.
In this respect we recently reported the synthesis of bis(dihydrophenanthro[9,10-e][1,2,4]triazine) dyes and investigated their optical, photophysical and solvatochromic properties [16].

In continuation of this work, the present study discussed the spectral behavior and photophysical parameters of dihydrophenanthro[9,10-e][1,2,4]triazine derivative dyes doped in various solid hosts matrices including sol–gel and PMMA.

### Experimental

#### Materials

Spectroscopic grade solvents and all chemicals were obtained from Sigma Aldrich and used without further purification. Active chromophores 1 and 2 were prepared as recently described by our group [16].

![Compound 1](image1)

12a-dihydrophenanthro [9, 10-e][1,2,4]triazine-3(4H)-thione

![Compound 2](image2)

2,6-Bis(((4b,12a-dihydrophenanthro[9,10-e][1,2,4]triazin-3-yl)thio)methyl)pyridine

Silicate Sol–gel matrix and polymer (using methyl methacrylate MMA as monomer) were prepared as described in references [17] and [18, 19], respectively.

#### Spectra Measurements

Dyes samples of concentrations ranging from $2 \times 10^{-6}$ M to $2 \times 10^{-4}$ M were doped in transparent sol–gel and of concentrations ranging from $4 \times 10^{-5}$ M to $2 \times 10^{-4}$ M in PMMA. The electronic absorption and fluorescence properties of these dyes in sol–gel and PMMA polymer matrices were studies using a Camspec M501 UV/Vis spectrophotometer and a PF-6300 spectrofluorometer, respectively. The wavelengths of excitation were adjusted at absorption maximum wavelengths for each fluorescence measurement of dye samples. Laser induced fluorescence of dye samples as function of different input pumping energies were carried out by 3rd harmonic Nd: YAG laser using homemade setup as previously reported [20]. The photostability of the dye was also determined using the same experimental setup [20]. The input energy was kept constant at (10 mL) by pumping with 355 nm of 3rd harmonic Nd: YAG laser.

#### Photophysical Parameters Calculations

Depending upon the absorption and emission spectra, important and significant photophysical parameters could be determined according to standard equations. The oscillator strength values were calculated from the Eq. (1) [21].

$$f = 4.32 \times 10^{-9} \int \epsilon (\tilde{\nu}) d \tilde{\nu}$$  \hspace{1cm} (1)

where the wave number $\tilde{\nu}$ is expressed in cm$^{-1}$ and $\epsilon$ is the molar absorptivity (1 mol$^{-1}$ cm$^{-1}$). Scientifically the value of ($f$) is dependent on the molecular structure and the type of electronic transition. The attenuation length $\Lambda(\lambda)$ (the distance at which the original light intensity $I_0$ is reduced to $I = I_0/e$, is given by Eq. (2) [22].

$$\Lambda(\lambda) = \frac{1}{\epsilon(\lambda) c \ln(10)}$$  \hspace{1cm} (2)

where the molar absorptivity is $\epsilon(\lambda)$ and $c$ is the concentration. The $\Lambda$ values are mainly dependent on the wavelength.
The dipole moment transition \( \mu_{12} \) was calculated according to Eq. (3) [22].

\[
\mu_{12}^2 = \frac{f}{4.72 \times 10^{-7} E_{\text{max}}}. \tag{3}
\]

where \( E_{\text{max}} \) is the maximum absorption energy in cm\(^{-1}\). The rate of radiative decay constant (\( k_r \)) of a fluorophore can be theoretically calculated from Eq. (4) [23].

\[
k_r = \frac{1}{\tau_0} = 2.88 \times 10^{-9} n^2 \int \frac{F(\tilde{\nu})d\tilde{\nu}}{F(\tilde{\nu})^{\frac{3}{2}} d\tilde{\nu}} \int \frac{\tilde{\epsilon}(\tilde{\nu}) d\tilde{\nu}}{\tilde{\nu}} \tag{4}
\]

where \( F(\tilde{\nu}), \tilde{\nu}, \epsilon, (\tilde{\nu}) \) are the intensity of fluorescence, the wave number and the molar absorptivity at wave number \( (\tilde{\nu}) \), \( n \) is the index of refraction of solid host.

The absorption cross-section \( \sigma_a \) is given by Eq. (5) [24].

\[
\sigma_a = 0.385 \times 10^{-20} \epsilon(\lambda). \tag{5}
\]

For the quantum yield of a compound relative to a standard probe, the following relationship (6) was applied: [25, 26].

\[
\phi_f = \phi \times \frac{I_u}{I_s} \times \frac{A_u}{A_f} \times \frac{n_{\nu}^2}{n_{\lambda}^2} \tag{6}
\]

where \( \phi_u \) and \( \phi_f \) are the fluorescence quantum yields of the unknown and the standard, respectively. \( I_u \) and \( I_s \) are the areas under emission curves for the unknown and the standard, respectively. and are the absorbance of the unknown, \( A_u \) and \( A_f \) the standard, respectively. \( n_{\nu} \) and \( n_{\lambda} \) are the refractive indices of solvents used. In determination of fluorescence quantum yields \( \phi_{u,0} \) (determined relative to the fluorescein laser dye in ethanol as reference) care was taken to keep the concentration of all the samples at levels low enough to avoid re-absorption of the emitted photons [25, 26].

Fluorescence lifetimes (\( \tau_f \)) were calculated using Eq. (7).

\[
\tau_f = \tau_0 \phi_f \tag{7}
\]

where \( \tau_0 \) is the natural lifetime (\( \tau_0 = 1/k_r \)). The rate constant of intersystem crossing (\( k_{isc} \)) is related to the quantum yield \( \phi_f \) for (\( \phi_f \approx 1 \)) by the relationship (8) [27]:

\[
K_{isc} = \frac{(1 - \phi_f)}{\tau_f} \tag{8}
\]

where \( \phi_f \) is the quantum yield.

The emission cross-section \( \sigma_e \) was calculated according to Eq. (9) [28].

\[
\sigma_e = \frac{\phi_f \lambda_e^2 E(\lambda)}{8 \pi c \tau_f n^2 \int E(\lambda) d\lambda} \tag{9}
\]

where (c) is velocity of light, (\( \phi_f \)) is fluorescence quantum yield, (\( \lambda_e \)) is wavelength of the emission, (\( \tau_f \)) is fluorescence life time, (\( n \)) is refractive index of the solvent and (\( E(\lambda) \)) is normalized fluorescence spectrum since (\( \int E(\lambda) d\lambda = 1 \)).

Results and Discussion

Photo-physical Parameters in Different Matrices

Figures 1 and 2 show the UV–vis absorption and fluorescence spectra of the dyes (Cpd 1 and Cpd 2) in sol–gel and homo polymer PMMA polymer matrices. There is a minimum overlap between the dye’s absorption and emission spectra in the two solid matrices. This is important as far as reabsorption of emitted photons is concerned.

Figure 1(A, B) shows that the absorption maximum peaks were at 372 nm and 350 nm for compounds 1 and 2 in PMMA, respectively. Figure 1 shows also that the emission maximum peaks were at 455 nm and 440 nm for compounds 1 and 2 in PMMA, respectively. Inset Fig. 1(A, B) shows that the emission of compounds 1 and 2 of different concentrations in PMMA at excitation wavelengths 372 nm and 350 nm, respectively. It showed a red shift with increasing concentration as represented in inset of Figs. (1A, 1B). The optimum dye concentration was 7 \( \times \) 10\(^{-5}\) M and 4 \( \times \) 10\(^{-5}\) M in PMMA for Cpd 1 and Cpd 2, respectively.

Figure 2(A, B) shows that the absorption maximum peaks were at 375 nm for compounds 1 and 2 in sol–gel host. It also shows that the absorption maximum peaks were at 465 nm and 475 nm for compounds 1 and 2 in sol–gel host, respectively. Inset Fig. 2(A, B) shows that the emission of compounds 1 and 2 at different concentrations in sol–gel host at excitation wavelength 375 nm, respectively. The dye emission spectra with different concentrations doped in sol–gel host was shown in inset Fig. 2(A, B). The dye fluorescence peak wavelength showed a large red shift as the concentration increased and the dye fluorescence peak intensity increased till 7 \( \times \) 10\(^{-5}\) M in case of Cpd 1 and 4 \( \times \) 10\(^{-5}\) M in case of Cpd 2 then it decreased with increasing concentrations which might be attributed to molecular aggregations of dye molecules. Also, the optimum dye concentration was 7 \( \times \) 10\(^{-5}\) M and 4 \( \times \) 10\(^{-5}\) M in sol–gel for Cpd 1 and Cpd 2, respectively. Comparing the dye fluorescence peak wavelengths and intensities in different solid matrices showed that, the fluorescence emission wavelengths are higher red shifted in sol–gel compared to polymer matrices. This behavior indicates a more relaxed, excited state due to dyes host interaction within excited state lifetime. Some important photo-physical parameters of the dye were calculated and summarized in the table (1), which proved their future applicable action.
in advanced optical applications. However, the absorption cross-section ($\sigma_a$) is the ability of the molecule to absorb a photon of a certain polarization and wavelength. Emission cross-section ($\sigma_e$) measures the probability of the excited photon of a certain polarization and wavelength. Emission of a compound 1 in sol–gel is higher than that in polymer matrices for a compound 2. Hence, the effective number of electrons transferred from the ground to excited states in polymer matrices is higher than those in sol–gel matrix for a compound 1 and in sol–gel matrix is higher than that in polymer matrices for a compound 2. Therefore, the effective number of electrons transferred from the ground to excited states in polymer matrices is higher than that in polymer matrices for a compound 2.

Fig. 1 (A, B): Normalized absorbance and emission intensity of A) compound 1 and B) compound 2 in PMMA, respectively. Inset figure: Emission of compounds 1 and 2 at different concentrations in PMMA at excitation wavelength A) 372 nm and B) 350 nm, respectively.

Fig. 2 (A, B): Normalized absorbance and emission intensity of A) compound 1 and B) compound 2 in sol–gel, respectively. Inset figure: Emission of A) compound 1 and B) compound 2 at different concentrations in sol–gel at excitation wavelength 375 nm, respectively.
states also allow for exciton trapping [22] adding to factors causing fluorescence quenching. However, for the compound 2, it is noted that the role of fluorescence quenching in polymer matrices comes from the higher intersystem crossing rate constants ($k_{ISC}$) values in polymer matrices compared with sol–gel glass.

Laser-Induced Fluorescence of Dihydrophenanthro[9,10-e][1, 2, 4] Triazine Derivative

The spontaneous fluorescence intensities and wavelengths of compounds 1 and 2 in PMMA polymer and sol–gel were varied after pumping with 3rd harmonic Nd:YAG ($\lambda = 355$ nm). They were pumped with different input powers of 5, 10 and 20 mJ as shown in Figs. 3 and 4(A, B).

By pumping using 3rd harmonic Nd: YAG pulsed laser with $\lambda_{ex} = 355$ nm excitation wavelength the emission intensity of ASE peak wavelength of two different dyes 1 and 2 in sol–gel matrix ($\lambda_{f} = 495$ nm and $\lambda_{f} = 480$ nm) respectively, showed a large red shift than that of the dyes 1 and 2 in PMMA ($\lambda_{f} = 480$ and $\lambda_{f} = 465$ nm) as shown in Figs. 3 and 4(A, B). The Figs. 3 and 4(A, B) showed the emission intensity of the two dyes in solid hosts at different input power at 5, 10 and 20 mJ. The increasing of the peak intensity of ASE of the dye may be attributed to the increasing of the number of excited molecules (increase the population of $S_1$ state) which yields more emitted photons. These changes in wavelengths may be due to the interaction of the structure of the two different dyes molecules with different solid matrices as shown in Figs. 3 and 4(A, B).

The photostability, as an important photochemical parameter, was studied by the evaluation of the output fluorescence as a function of number of pulses in the same position of the samples as outlined in Fig. 5(A, B). This study was carried out for the samples of the dyes which the repetition rate of 355 nm Nd: YAG laser with 8 ns pulse duration was kept at (10 Hz) and the energy was kept constant at (10 mJ/pulse). The output energy gradually decreased due to the photodegradation progressive and thermal degradation of the dye’s molecules. This

| Sample / solvent | Cpd1 | Cpd2 |
|------------------|------|------|
| Cpd1 Sol–gel     | 8.52 | 10.91|
| PMMA             | 11.52| 9.58 |
| $\sigma_a$       | 3.2  | 4.8  |
| $\sigma_e$       | 1.94 | 2.35 |
| $\Lambda$        | 0.05 | 0.039|
| $\tau_f$         | 0.32| 0.31 |
| $\tau_0$         | 0.52| 0.45 |
| $\mu_{12}$       | 6.53| 8.94 |
| $E_f$            | 0.49| 0.55 |
| $K_{sp}$         | 1.92| 2.22 |
| $K_{isc}$        | 1.20| 0.55 |
| $F$              | 0.65| 0.96 |
| $\phi_f$         | 0.62| 0.70 |

Table 1: Photo-physical parameters of $7 \times 10^{-5}$ M and $4 \times 10^{-5}$ M of Cpd 1 and Cpd 2 in different hosts, respectively; (a) molecular extinction coefficient; $\sigma_a$ and $\sigma_e$: cross section of absorption and emission; (A) the attenuation length; ($\tau_f$) fluorescence life time, ($\tau_0$) calculated fluorescence life time, $\mu_{12}$ (D) the dipole moment transition, ($E_f$) energy yield of fluorescence, ($K_{sp}$) the radiative decay rate, ($K_{isc}$) the rate of intersystem crossing, ($f$) oscillator strength, $\phi_f$ fluorescence quantum yield, in different hosts
decreasing occurred at a faster rate for the dyes in polymer than in sol gel, and the peak ASE dropped to 46%, 40% and 49%, 54% of the initial ASE of the dyes (Cpd 1 and Cpd 2) in PMMA and sol–gel, respectively by pumping with 355 nm 3rd harmonic Nd: YAG pulsed laser at 10 mJ. Since the mechanism of photodegradation occurs by the interaction molecules of the dye in the excited state with other species such as impurities, other dye molecules and singlet oxygen. Through the process of doping dyes into a solid medium, the photochemical pathways including bimolecular reactions can be suppressed by caging or trapping the dye within a solid host [29].

The improved photostability of the dyes within solid hosts has been attributed for caging and molecules immobilizing of the dyes, minimizing thereby excited-state interaction with other species including molecular oxygen. So, the dye photo-degradation in a solid host depends on the dye’s molecule nature, the host composition and structure, the host impurities as well as presence of molecular oxygen. Another possible factor that may explains the reduced rate of degradation in the rigid matrices may be that the dyes molecules are more tightly confined within the pores of the matrix, limiting rotational and translational freedom. A mobile molecule, as in solution, will be more likely to encounter an oxygen molecule and undergo degradation. Less freedom, as defined by the restrictive pores of the matrix, may make the dyes molecule less likely to interact with molecular oxygen leading to photodegradation or molecular oxygen fluorescence quenching [29]. The micro-viscosity environment around dye molecules in the solid matrix affects their

Fig. 4 (A, B): Emission intensities of ASE of A) compound 1, B) compound 2 in sol–gel, with excitation wavelength 355 nm by 3rd harmonic Nd: YAG pulsed laser at different input pumping power 5 mJ, 10 mJ and 20 mJ
photodegradation. The net photo deterioration would be slow if the irradiated molecules were swiftly replaced by fresh molecules. As a result, photodegradation in sol–gel samples is negligible, with the longest half-life values. On the other hand, photodegradation occurs at a faster pace in the copolymer samples. This is because dye molecules in polymer samples are surrounded only by polymer matrix with very little solvent around them, whereas dye molecules in sol–gel matrix are rapidly replenished because sol–gel samples contain ethylene glycol, which may aid in the mobility of the embedded dye molecules, resulting in minimal photo-degradation and the longest half-life values.

**Conclusion**

The optical absorption and emission properties of dihydrophenanthro[9,10-e][1,2,4]triazine dyes have been studied in different solid hosts such as sol–gel and PMMA polymer. Their respective spectroscopic and photo physical parameters meet the best requirements for a good laser dye such as high molar absorption coefficients at the wavelength of the pump laser, broad spectral region of fluorescence and high fluorescence quantum yield, short fluorescence decay time, large Stokes shift, little overlap of the fluorescence and triplet absorption spectral regions, photochemical stability. Pumping the samples using 3rd harmonic Nd:YAG laser (λ = 355 nm) showed different emission wavelength of ASE peak. It was nearly in PMMA polymer (λf = 495 nm), (λf = 465 nm) and in sol–gel matrix (λf = 495 nm), (λf = 480 nm) of compounds 1 and 2, respectively. The nature of solid host has significant effect on spectroscopic properties of dye. The new dyes exhibited good photostability. It decreased to 46%, 40% and 49%, 54% of the initial ASE of the dyes (Cpd 1 and Cpd 2) in PMMA and sol–gel, respectively, after pumping with 355 nm 3rd harmonic Nd:YAG laser of 8 ns pulse duration, with a repetition rate (10 Hz). The energy was kept constant at (10 mJ/ pulse) after 55,000 pulses.

**Author Contributions** All authors contributed to the design and implementation of the research, analysis of the results, and the manuscript’s preparation.

**Credit Authorship Contribution Statement** Mahmoud E. M. Sakr: Methodology, writing—review and editing. Maram T. H. Abou Kana: Conceptualization, investigation, supervision, writing- original draft. Ahmed H. M. Elwahy: Conceptualization, supervision, investigation, synthesis, resources. Samy A. El-Daly: Review, editing and supervision. El-Zeiny M. Ebeid: Supervision.

**Declarations**

**Ethical Approval** This article does not contain any studies with human or animal subjects.

**Conflicts of Interest** The authors declare that they have no conflict of interest.

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