



Temperature Effect on Fluorescence Intensity and Dipole Moment Using Thermochromic Shift Method of 7DA3MHBI-2HChromen-2-one Laser Dye in Highly Viscous Glycerol Solvent

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Received: 20 November 2023 / Accepted: 24 December 2023

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Abstract

The steady-state method is used to study the effect of temperature on the fluorescence characteristics of 7-(diethylamino)-3-(1-methyl-1H-benzo[d]imidazol-2-yl)-2H-chromen-2-one (7DA3MHBI-2HChromen-2-one) laser dye in glycerol solvent for the temperature range 293–343 K. Absorption and emission characteristics are affected by varying temperatures due to induced thermal effects. Transition probabilities mechanism of non-radiative and radiative are studied and frequency dependent parameters are estimated. Dipole moments in the ground and excited state are estimated using the thermochromic shift method over general solvatochromic methods.

Keywords Fluorescence intensity · Frequency dependent parameters · Inter-system crossing · Solvatochromic shift

Introduction

Spectral properties and dipole moments estimation are useful in understanding the molecular structure of organic laser dyes in excited states. Knowledge about dipole moments in excited state is useful in understanding the nature of excited states, photochemical transformation, and designing non-linear material, fabricating opto-electronic devices and thermal sensors. Spectral properties and dipole moments are changed by the immediate surrounding environment. Solvents and their properties (ϵ , n , E_T^N , Hydrogen bond donor (HBD), Hydrogen bond acceptor (HBA), viscosity, concentration, pH, temperature, quenchers, etc., affect the spectral characteristics of laser dyes [1–16]. Though various parameters affect the laser spectral properties, in recent years the effect of temperature has drawn the great attention of researchers. A large group of researchers extensively studied organic laser dye's spectral properties in different solvent environments by varying temperatures [17–27] and reported that

there is no much significant change in absorption and emission wavelength maxima; however fluorescence intensities decrease with an increase in temperature. These intensity changes are due to changes in probabilities of non-radiative and radiative transitions due to thermal effects initiated by varying temperature. To understand underlying deactivation mechanism of these temperature dependence transitions from an excited state. In this paper, we have studied the effect of temperature on spectral properties and estimated dipole moment of 7DA3MHBI-2HChromen-2-one laser dye in glycerol solvent. Also, spectral and decay rate parameters such as relative fluorescence intensities, lifetime, rate decay constant, activation energy, theoretically lifetime value at 20 °C are presented in this paper. Dipole moments are generally estimated using solvatochromic shift methods using different solvents with increasing polarities. However, in this paper we used advanced thermochromic shift method in estimating the dipole moment in single solvent. Glycerol solvent being associative and highly viscous solvent, we chose glycerol for our studies. This study will enhance the knowledge about the thermal sensitivity of laser spectral characters in different environments.

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Theory

Temperature Effect on Fluorescence Intensity

Fluorescence intensity varies with varying temperatures but fluorescence lifetime is found to be independent of it.

Fluorescence intensity decreases with an increase in temperature and can be explained by intersystem crossing between singlet (S_1) and triplet (T_1) states. However, any decrease in the fluorescence lifetime indicates an increase in the probabilities of non-radiative deactivation of the singlet excited state. Various mechanisms are used to explain the decrease in the fluorescence lifetime [22, 23, 27] with temperature such as internal conversion, inter-system crossing, disassociation, loss of molecular planar structure due to thermal energy, etc.

For 10–70 °C small temperature range it is assumed that only a non-radiative deactivation mechanism is dominant. The fluorescence lifetime dependence on temperature is given by [19]

$$\frac{1}{\tau(t)} - n_1 = n_2^0 \exp\left(-\frac{E}{RT}\right) \quad (1)$$

where, $\tau(t)$ – fluorescence lifetime at different temperatures, n_1 and n_2 – radiative and non-radiative transition probabilities respectively, n_2^0 – deactivation process factor, E – activation energy in de-activation process, T – absolute temperature, R – universal gas constant. The fluorescence rate of decay constant is given by

$$k_f = k^0 + k' \exp\left(-\frac{\Delta E}{RT}\right) \quad (2)$$

where, $k_f = \frac{1}{\tau(t)}$, k_f , and k' are fluorescence rate decay constant at different temperatures, k^0 – fluorescence rate decay constant at 0 °C, temperature independent rate decay constant, frequency assisted thermal process respectively, ΔE – free energy activation component. Fluorescence lifetime at different temperatures is given by

$$\tau(t) = \tau_0(20) \frac{I_0(t)}{I_0^{20}} \quad (3)$$

where, $\tau(t)$, $\tau_0(20)$, $I_0(t)$ and I_0^{20} are the fluorescence lifetime at different temperatures, fluorescence intensity at 20 °C, fluorescence intensities at different temperatures and at 20 °C respectively. A plot of $\ln k_f$ v/s T gives a straight line with $\ln k'$ as intercept and slope gives $(-\Delta E/RT)$. The magnitude of k' measures how the fluorescence intensity and lifetime vary with temperature.

Thermochromic Shifts and Dipole Moment

The dipole moment in the excited is estimated by using solvatochromic spectral shifts of absorption and fluorescence in different solvents of increasing polarity. The solvatochromic shifts method is suitable for estimating dipole moments of both small and big-sized molecules. In this method different interactions are involved and data spread is a bit quite large. Instead of using different solvents, an improved thermochromic solvatochromic method [28–35] suggests using a single

suitable solvent and varying the ϵ and n by varying the temperature. The experimental collected data spread is very small and specific interactions are eliminated. Thus, this method is found to be more accurate for the measurement of change in dipole moment in the ground and excited states.

Basic Equations for Estimation of Dipole Moment

The following thermochromic solvatochromic equations are used to estimate the dipole moment in the ground and singlet excited state [28–35].

$$\bar{\nu}_A - \bar{\nu}_f = m_1 \cdot f_{BK}(\epsilon, n) + \text{constant} \quad (4)$$

$$\bar{\nu}_A + \bar{\nu}_f = -m_2 \cdot \phi_{BK}(\epsilon, n) + \text{constant} \quad (5)$$

where, $\bar{\nu}_A - \bar{\nu}_f$, $\bar{\nu}_A + \bar{\nu}_f$ are difference and sum of absorption and fluorescence maximum wave number respectively,

$$m_1 = \frac{2(\mu_e - \mu_g)^2}{hca^3} \quad (6)$$

$$m_2 = \frac{2(\mu_e^2 - \mu_g^2)}{hca^3} \quad (7)$$

$$\phi_{BK}(\epsilon, n) = f_{BK}(\epsilon, n) + 2g_{BK}(n) \quad (8)$$

$$f_{BK}(\epsilon, n) = \frac{\frac{\epsilon-1}{2\epsilon+1} - \frac{n^2-1}{2n^2+1}}{\left(1 - \frac{2\alpha}{a^3} \cdot \frac{\epsilon-1}{2\epsilon+1}\right) \left(1 - \frac{2\alpha}{a^3} \cdot \frac{n^2-1}{2n^2+1}\right)^2} \quad (9)$$

$$g_{BK}(n) = \frac{\frac{n^2-1}{2n^2+1} \cdot \left(1 - \frac{\alpha}{a^3} \cdot \frac{n^2-1}{2n^2+1}\right)}{\left(1 - \frac{2\alpha}{a^3} \cdot \frac{n^2-1}{2n^2+1}\right)^2} \quad (10)$$

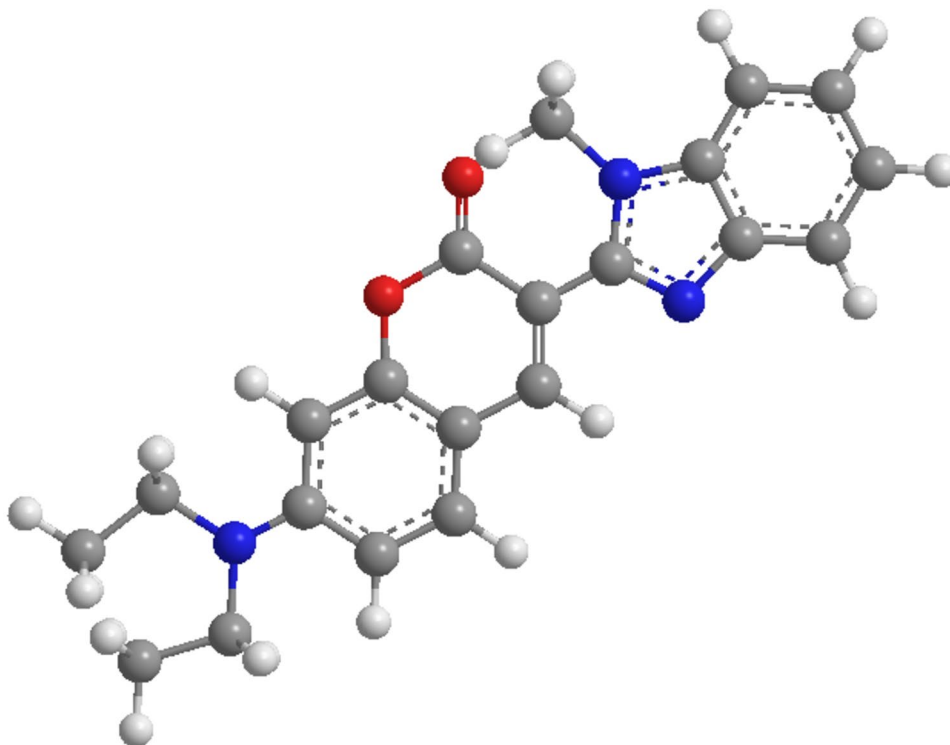
$$\mu_g = \frac{m_2 - m_1}{2} \cdot \sqrt{\left(\frac{hca^3}{2m_1}\right)} \quad (11)$$

$$\mu_e = \frac{m_1 + m_2}{2} \cdot \sqrt{\left(\frac{hca^3}{2m_1}\right)} \quad (12)$$

where, ϵ – dielectric constant, n – refractive index, m_1 , m_2 are the slopes determined using Eqs. (4) and (5), μ_e and μ_g – dipole moments in excited and ground states respectively, h – Planck's constant, c – velocity of light in vacuum, a – Onsager cavity radius, α – solute polarizability.

If the dipole moment of ground and excited is not parallel the angle between them is given by

Fig. 1 Optimized geometry of 7DA3MHBI-2HChromen-2-one molecule



$$\cos \varnothing = \frac{1}{2\mu_e\mu_g} \left[(\mu_g^2 + \mu_e^2) - \frac{m_1}{m_2} (\mu_e^2 - \mu_g^2) \right] \quad (13)$$

$f_{BK}(\epsilon, n)$ And $\phi_{BK}(\epsilon, n)$ values at different temperatures are calculated using

$$\epsilon(T) = \epsilon_0(T_0) - \alpha(T - T_0) - \beta(T - T_0)^2 - \gamma(T - T_0)^3 \quad (14)$$

$$n(T) = n(T_0) - a(T - T_0) - b(T - T_0)^2 \quad (15)$$

where, α , β and γ are the coefficients of T , T^2 , T^3 respectively of dielectric of varying temperature. a , b are coefficients of T , T^2 respectively of refractive index of varying temperature.

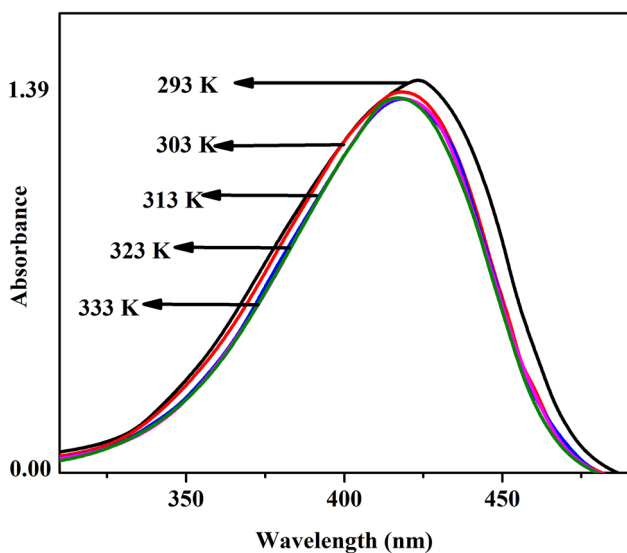


Fig. 2 Absorption spectra of 7DA3MHBI-2HChromen-2-one molecule in glycerol for different temperatures

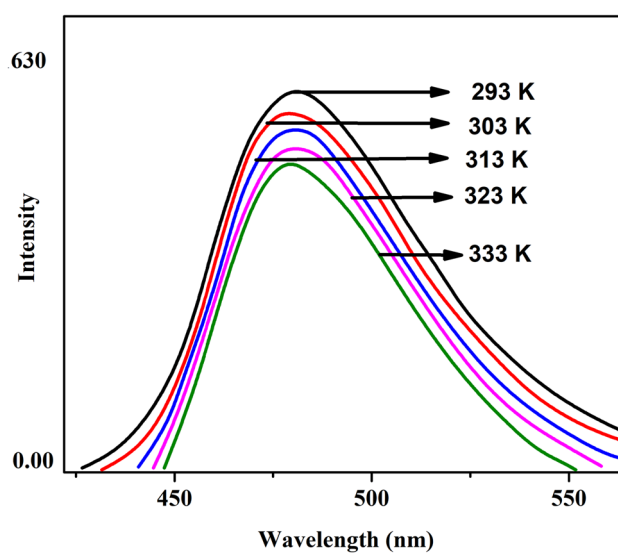
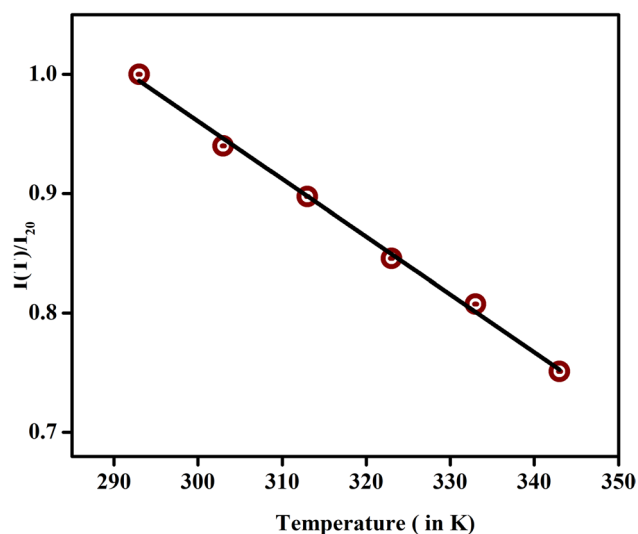
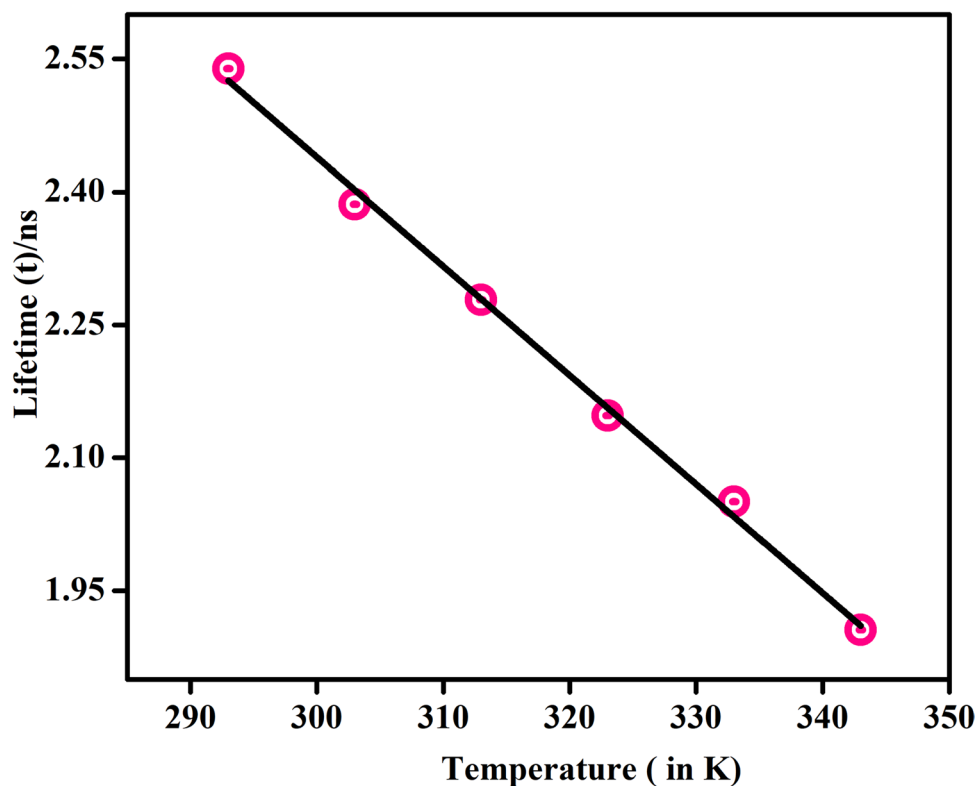


Fig. 3 Emission spectra of 7DA3MHBI-2HChromen-2-one molecule in glycerol for different temperatures

Table 1 Absorption and Fluorescence spectral data of 7DA3MHBI-2HChromen-2-one in glycerol from 293 to 343 K

Temperature (in ° K)	Viscosity ^a η (m/Pa S)	Absorption λ_{max}^{abs} (nm)	Absorbance	Fluorescence λ_{max}^{ems} (nm)	Fluorescence Intensity
293	934	420.09	1.381	482.21	1881
303	738	419.07	1.377	480.91	1768
313	561	418.95	1.374	480.62	1688
323	376	418.89	1.367	480.42	1591
333	202	418.81	1.349	480.12	1519
343	24	418.76	1.348	479.81	1412

^adata from CRC Handbook 95th Edition**Fig. 4** Relative intensities v/s temperature of 7DA3MHBI-2HChromen-2-one molecule in glycerol**Fig. 5** Temperature dependence of the fluorescence lifetime of 7DA3MHBI-2HChromen-2-one molecule in glycerol

Experimental Details

Materials

7DA3MHBI-2HChromen-2-one laser dye was purchased from sigma Aldrich Chemicals Pvt. Ltd., HPLC grade glycerol solvent from S.D. Fine Chemicals Ltd., India. Solute and solvent were used without any additional purification.

Computational Method

For solute properties 7-(diethylamino)-3-(1-methyl-1H-benzo[d]imidazol-2-yl)-2H-chromen-2-one (7DA3MHBI-2HChromen-2-one) laser dye is geometrical optimized (Fig. 1) using DFT-B3LYP/6-31 basis set and R-closed wave function using Gaussian Software 0.9 version. Solute

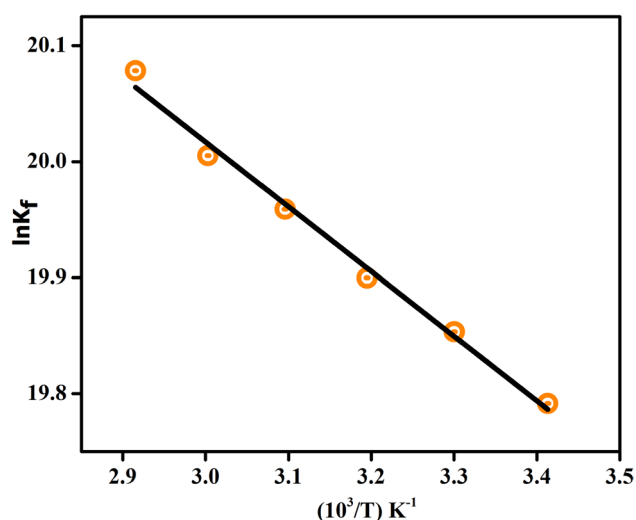


Fig. 6 Plot of $\ln K_f$ versus $10^3/T$ of 7DA3MHBI-2HChromen-2-one molecule in glycerol

polarizability $\alpha = 40.32 \times 10^{-30}$ and the ratio of α/a^3 is found to be 0.536.

Steady State Measurements

For stable and reliable spectra, the concentration of the solute in the solvent is kept low (1×10^{-6} mol/L). The absorption spectra were recorded using UV–VIS spectrophotometer Hitachi U-3310. The absorption maximum wavelength is chosen for excitation wavelength.

Fig. 7 Absorption and fluorescence spectra of 7DA3MHBI-2HChromen-2-one molecule in glycerol

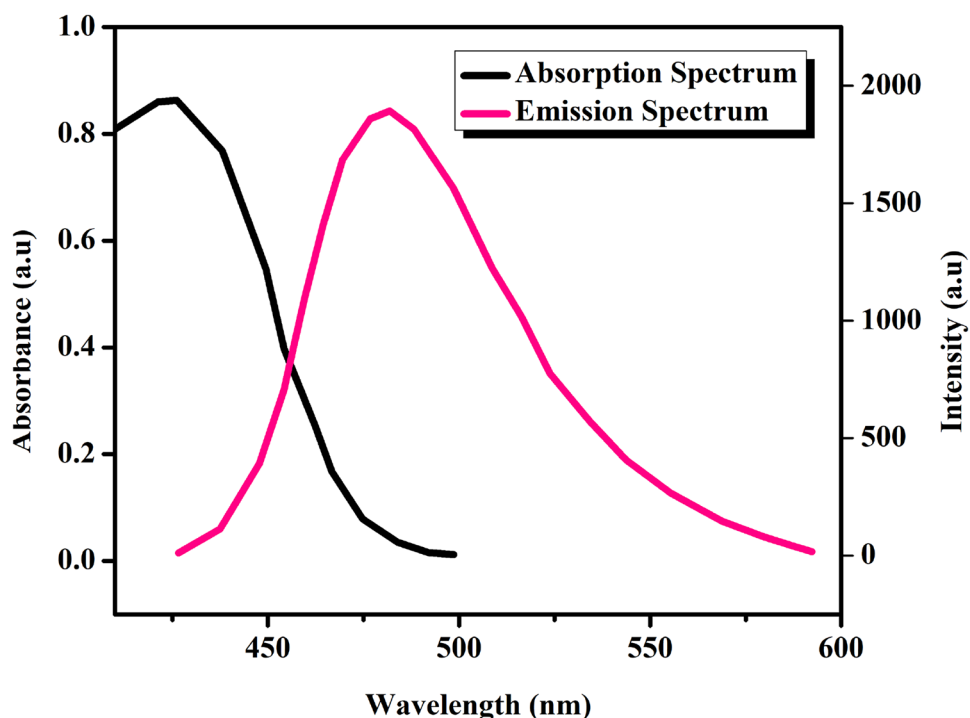


Table 2 Relative fluorescence intensities, lifetime, and rate constant of 7DA3MHBI-2HChromen-2-one in glycerol as a function of temperature

Temperature (in °K)	$1/T \times 10^3 \text{ K}$	I_f/I_{20}	τ (ns)	$\ln K$ (s^{-1})
293	3.4130	-	2.539	19.7915
303	3.3003	0.940	2.386	19.8535
313	3.1949	0.897	2.278	19.8998
323	3.0960	0.846	2.148	19.9589
333	3.0030	0.808	2.050	20.0052
343	2.9155	0.751	1.906	20.0783

Fluorescence intensity was recorded using Hitachi F-7000 fluorescence spectrophotometer. For temperature studies, cuvette temperature is controlled by using thermocouple within ± 0.3 °C between 20–70 °C. Fluorescence lifetime at 20 °C is measured using Edinburgh instrument model EI-199.

Results and Discussion

Fluorescence intensities of 7DA3MHBI-2HChromen-2-one in glycerol solvent as a function of temperature have been measured. The typical absorption and fluorescence spectra of 7DA3MHBI-2HChromen-2-one in glycerol solvent as a function of temperature (20–70 °C) are shown in Figs. 2 and 3. It is found that absorbance wavelength maxima (418.2 ± 2.5 nm) more or less remain the same and doesn't

Table 3 Actual and radiative lifetimes, activation energy for the radiationless transition and the correlation coefficient of 7DA3MHBI-2HChromen-2-one in glycerol

τ_0 (ns)	k' ($\times 10^9$ s)	S_1 Energy (cm^{-1})	Activation energy E_2 (kJmol^{-1})	T_2 Energy (cm^{-1})	Correlation coefficient
2.539	2.65	21885.2	4.641	22273.15	0.9904

change much with increase in temperature as shown in Table 1. Fluorescence maxima shift slight towards lower wavelength of 0–1 nm as shown Table 1, this specify that excited singlet emission state is slightly modified on heating and compound basicity in the excited state is increased [36]. Many thermal induced phenomena's affects fluorescence intensity, decrease in intensity with increase in temperature is either due to increase in non-radiative deactivation process involvement i.e. intercrossing from singlet state (S_1) to triplet state (T_1) or molecular dissociation. For any small changes in fluorescence intensity with less than 25% with temperature is explained in terms of conversion of electronic to vibrational energy. If it's greater than 25% its essential to look into non-radiative deactivation mechanism from singlet to triplet state. Relative fluorescence intensity (I_f/I_{20}) is estimated for different temperature and I_f/I_{20} vs temperature is plotted as shown in Fig. 4 with linear

Table 4 Permittivity and refractive index parameter for glycerol solvent

ϵ at 293 K	α	β	γ	n at 293 K(a)	b
46.501	77.503	-0.037984	-0.00023107	1.47557	0.00045

fit (Adj. $R^2 = 0.996$) Figs. 5 and 6. It shows I_f/I_{20} linearly decrease with increase in temperature. To understand the underlying mechanism of non-radiative deactivation i.e. intersystem crossing mechanism, values of S_1 and T_2 are to be determined using crossing point for absorption and fluorescence spectra and $T_2 = \Delta E + S_1$ respectively [22]. S_1 is found by intersecting point between absorption and emission spectra of 0–0 band (Fig. 7) whose value is found to be $21,885.2 \text{ cm}^{-1}$. The value of T_2 is estimated using the estimated values of decay rate parameters. The decay rate parameter K_f at different temperature is found by taking the reciprocal of $\tau(t)$ (using Eq. 2) by using τ_0 at 20°C which is found experimentally as shown in Table 2. The fluorescence lifetime and K_f of 7DA3MHBI-2HChromen-2-one decrease with increase in temperature. A plot of fluorescence lifetime and $\text{Ln } K_f$ vs temperature is plotted as shown in Figs. 5 and 6 with good linear fit who's Adj. R^2 values are found to be 0.996 and 0.990 respectively. Using linear least square fit method the value of k' and ΔE were calculated and represented in Table 3

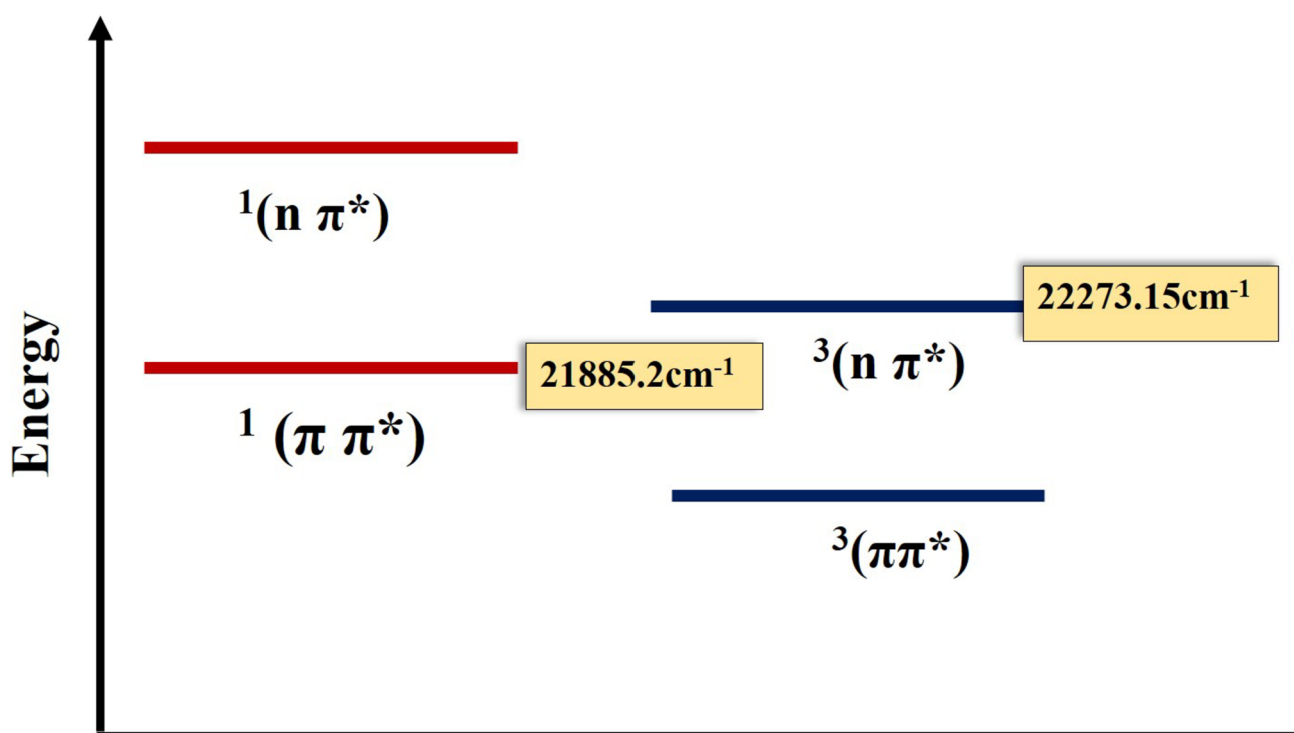
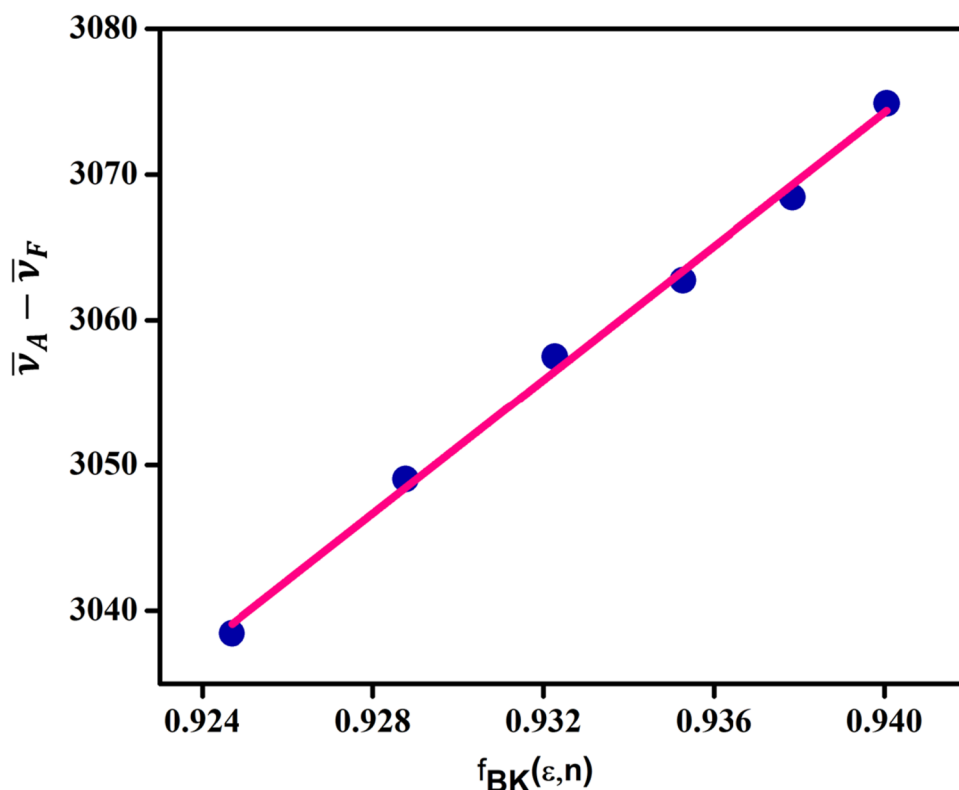
**Fig. 8** Energy levels of the 7DA3MHBI-2HChromen-2-one molecule in glycerol

Fig. 9 Plot of $\bar{\nu}_A - \bar{\nu}_F$ v/s $f_{BK}(\epsilon, n)$ of 7DA3MHBI-2HChromen-2-one molecule in glycerol at different temperatures T



with correlation value of 0.990. Using the value of ΔE and S_1 , T_2 is estimated whose value is less than S_1 (Table 3) Henceforth, T_2 state lies above S_1 indicating that transition to ground state (S_0) from the excited singlet state occurs via triplet T_2 state [22, 23, 27]. With increasing in temperature ΔE is found to be small indicating that T_2 energy level lies closely to S_1 (Fig. 8).

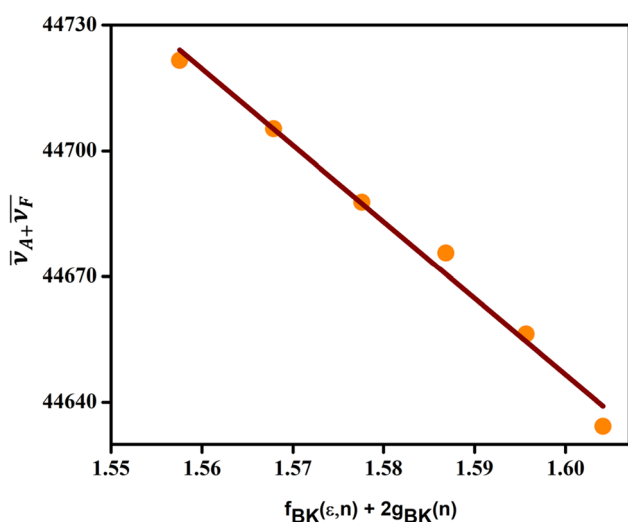


Fig. 10 Plot of $\bar{\nu}_A + \bar{\nu}_F$ v/s $f_{BK}(\epsilon, n) + 2g_{BK}(\epsilon, n)$ of 7DA3MHBI-2HChromen-2-one molecule in glycerol at different temperatures T

Advance thermochromic solvatochromic method is used to estimate dipole moments of 7DA3MHBI-2HChromen-2-one laser dye. Table 1 and Figs. 2 and 3 show that with increase in temperature a blue shift in the absorption and emission wavelength. It is found that solvent polarities (ϵ, n) and solvent polarity parameters $f_{BK}(\epsilon, n)$ and $\phi_{BK}(\epsilon, n)$ decrease with increase in temperature. Neglecting higher orders the decrease in ϵ and n values are calculated using Eqs. 14 and 15 and Table 4. Solvent polarity parameters also depend on mean polarizability and Onsager cavity radius of the solute. Edward's increment method [37] is used to estimate the cavity radius and solute polarizability by Gaussian 9.0 software ($\alpha/a^3 = 0.543$). Solvent polarity parameters $f_{BK}(\epsilon, n)$ and $\phi_{BK}(\epsilon, n)$ are estimated (Eqs. 9 and 10) by using $\alpha/a^3 = 0.536$. A plot of difference and sum of thermochromic band shifts ($\bar{\nu}_A - \bar{\nu}_F$ and $\bar{\nu}_A + \bar{\nu}_F$) wave number versus solvent polarity parameters $f_{BK}(\epsilon, n)$ and $f_{BK}(\epsilon, n) + 2g_{BK}(n)$ are plotted as shown

Table 5 Dipole moments (in Debye) in the lowest excited singlet state determined from thermochromic shifts of 7DA3MHBI-2HChromen-2-one in glycerol for $\alpha/a^3 = 0.536$

m_1 (cm ⁻¹)	m_2 (cm ⁻¹)	μ_g (D)	μ_e (D)	$\Delta\mu$ (D)	Cos ϕ (radians)
229.86	1825.04	0.60	4.54	3.94	5.45
		1.41 ^b	4.51 ^b	3.10 ^b	

^bExperimental data using solvatochromic shift method Renuka et al. [13] for comparison

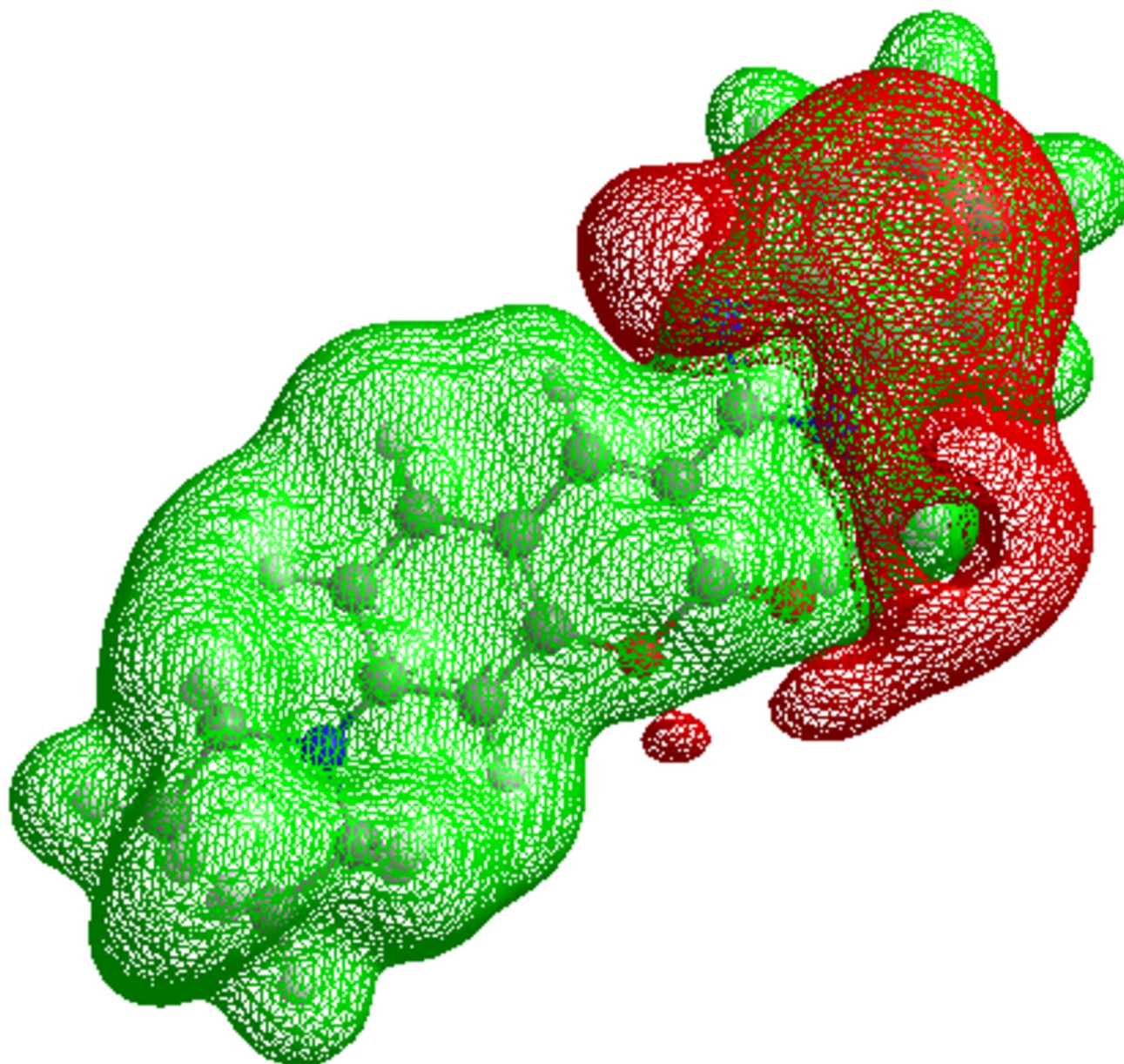


Fig. 11 Electrostatic potential of 7DA3MHBI-2HChromen-2-one molecule in glycerol solvent

in Figs. 9 and 10 respectively. A linear regression fit of these points was carried out. Shows a good correlation of adj. $R^2=0.99$ and 0.98 respectively. The slopes m_1 and m_2 from graphs Figs. 9 and 10 are tabulated in Table 5. Using m_1 and m_2 values μ_e is estimated using Eqs. 11 and 12. Ground state and excited state dipole estimated values are found to be 0.6 D and 4.54 D respectively. For same $a=4.221$ \AA^0 Renuka et al. [38] it is reported that in a series of alcohols solvents using solvatochromic shift method the dipole moments are μ_g and μ_e values are found to be 1.41 D and 4.51 D respectively. For same $a=4.221$ \AA^0 using thermochromic shift method the value of μ_e is in close agreement with the reported value and μ_g value

shows a deviation of 0.81 D. The cosine angle between the μ_e and μ_g is found to be 5.45 radians (using Eq. 13). To understand the type of interaction and locate optimal site of interaction between solute and solvent, molecular electrostatic potential of 7DA3MHBI-2HChromen-2-one molecule in glycerol solvent using Gaussian software 9.0 is calculated and plotted as shown in Fig. 11. Green color indicates positive region or nucleophilic active region and red indicates negative region or electrophilic active region. Negative region is localized over 1-methyl-4,5-dihydro-1H-pyrazole region of compound and suggests that the specific hydrogen bonding interaction occurs between $-\text{OH}$ group of glycerol and 1-methylpyrrolidine [39].

Conclusions

The spectral characters of 7DA3MHBI-2HChromen-2-one laser dye have been studied in glycerol solvent by varying the temperature (20–70 °C). Fluorescence maxima negligibly shift towards shorter wavelength (0–1 nm) and relative intensity decreases linearly with increase in temperature. Increase in temperature results in decrease in fluorescence lifetime of 7DA3MHBI-2HChromen-2-one molecule suggesting involvement of non-radiative transition phenomenon. Fluorescence frequency decay parameters are used to evaluate k' and ΔE . Using ΔE and S_1 , T_2 energy level is evaluated. It is noted that T_2 energy levels lies very close to singlet state (S_1) indicating that deactivation from excited singlet to ground state involves in intersystem crossing ($S_1 \rightarrow T_2$). The advanced method thermochromic shifts over solvatochromic shifts is used to estimate the dipole moment in the ground and excited state. The excited state dipole moment estimated using thermochromic shifts is nearly same and deviation in ground state dipole moment of about 0.81 D in excited state is observed when compared with solvatochromic shifts method for same value of Onsager cavity radius. Also, ground state and excited state dipole moment are nearly parallel. This study helps in determining dipole moments in singlet excited state using single solvent for varying temperature over normal solvatochromic shift method and also to understand the thermal sensitivity of laser dyes with temperature.

Author Contributions A.K. Prepared - Conceptualization, Methodology, Data Analysis, Figures, Tables A.K. Prepared - Original draft, C.G.R. Reviewing, Supervision

Funding None.

Availability of Data and Materials Not applicable.

Declarations

Ethical Approval Not applicable.

Competing Interest The authors declare no competing interests.

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