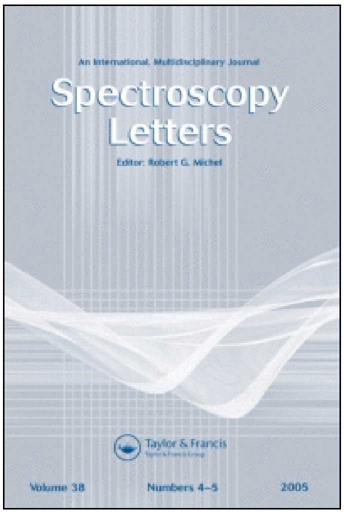

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Fluorescence Quenching Studies of 1,3-Diphenyl Benzene

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Fluorescence Quenching Studies of 1,3-Diphenyl Benzene

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Abstract: Fluorescence quenching of 1,3-diphenyl benzene (*m*-terphenyl) by carbon tetrachloride (CCl_4) at steady state in different solvents, namely *n*-hexane, *n*-heptane, cyclohexane, toluene, benzene acetonitrile, 1,4-dioxane, and with a transient method in benzene has been done at room temperature to understand the role of quenching mechanisms. The Stern–Volmer plot was found to be linear for all the solvents studied. The probability of quenching per encounter p was determined in all the solvents and was found to be less than unity. Further, from the studies of rate parameters and lifetime measurements in benzene at different temperatures (30–60°C), it was shown that the phenomenon of quenching is generally governed by the well-known Stern–Volmer (S-V) plot. The activation energy E_a (E'_a) of quenching was determined using literature values of activation energy of diffusion E_d, and it was found to be greater than E_d, which confirms the fact that the quenching mechanism is not solely due to material diffusion but there is also contribution from activation energy.

Keywords: Activation energy, material diffusion, *m*-terphenyl, steady state, S-V plot, transient fluorescence quenching

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INTRODUCTION

Fluorescence quenching of organic molecules in solution by various quenchers like aniline, bromobenzene, carbon tetrachloride, ethyltrithiocarbonate, halide ions, metal ions, and so forth, has been studied by several investigators.[1–13] The study has been mainly to understand the nature of bimolecular reactions taking place both under steady state and transient conditions. This research has been of importance not only in the physical sciences but also in the chemical, biological, and medical sciences.[14–20] The fluorescence yield in bimolecular liquid systems is hindered due to several mechanisms such as static and dynamic quenching, excimer and exciplex formation, charge transfer processes, and so forth. In literature, there is a dearth of data on photophysical and photochemical properties of 1,3-diphenyl benzene (*m*-terphenyl). Hence we have studied fluorescence quenching of *m*-terphenyl (i) in different solvents at room temperature and (ii) in benzene at different temperatures (30–60°C) by steady-state method with CCl_4 as quencher. One of the well-known experimental techniques used to study the fluorescence quenching is to determine the quenching rate parameter by using the Stern–Volmer (S-V) equation.

If the quenching mechanism is mainly due to dynamic process, then it will be largely due to diffusion in which case diffusion rate parameter k_o equals the quenching rate parameter k_q $(= K_\mathrm{sv}/\tau_\mathrm{o})$, where K_sv is the slope of the linear S-V plot and τ_o is the decay time of the solute in the absence of the quencher.

The phenomenon of quenching is generally governed by the well-known linear S-V equation given by[21]

$$\frac{I_o}{I} = 1 + k_q \tau_o [Q] \tag{1}$$

$$\frac{\tau_o}{\tau} = 1 + k'_\mathrm{q} \tau_o [Q], \tag{2}$$

where I_o and τ_o are the fluorescence intensity and fluorescence lifetime, respectively, in the absence of the quencher, I and τ are the fluorescence intensity and fluorescence lifetime, respectively, in the presence of the quencher at a concentration $[Q]$, and k_q (k'_q) is the quenching rate parameter.[22] The term $k_\mathrm{q}\tau_\mathrm{o}$ $(k'_\mathrm{q}\tau_\mathrm{o})$ is called the Stern–Volmer constant K_sv (K'_sv), which is obtained from the slope of the Stern–Volmer plot. The phenomenon of quenching is supposed to occur due to short-range collision between the excited molecules and the quencher molecules. If the close collision between the reactants should occur, then the reactants should occupy the contiguous position in the solution and separate due to diffusion after the collisional encounter.

The probability of quenching per encounter is represented by p, which is less than unity, and frequency of encounter is represented by k_o:

$$p = \frac{k_q}{k_o} \tag{3}$$

$$p' = \frac{k'_q}{k_o}. \tag{4}$$

The value of k_o can be calculated from the theoretical equation giving the bimolecular reactions controlled by material diffusion[23]

$$k_o = 4\pi NDR\ 10^{-3}\{1 + R/(2D\tau_o)^{1/2}\}, \tag{5}$$

where N is Avogadro's number, D $(= D_Y + D_Q)$ is the sum of the diffusion coefficients of solute (D_Y) and quencher (D_Q), and R $(= R_Y + R_Q)$ is the sum of the molecular radii of solute (R_Y) and quencher (R_Q), respectively, and τ_o has its usual meaning as explained earlier. The degree to which material diffusion controls the quenching and also efficiency of quenching can be determined by comparing the values of k_o and k_q. In the current work, we have used both steady-state and transient experimental setup to investigate the fluorescence quenching of *m*-terphenyl with carbon tetrachloride (CCl_4) as quencher in order to understand the role of diffusion in quenching mechanism involved in these systems. The diffusion coefficients are calculated by using the Stokes–Einstein relation,[24]

$$D = \frac{kT}{a\pi\eta r}, \tag{6}$$

where k is the Boltzmann's constant, T the absolute temperature, η the viscosity of the medium, r the radius of the molecule (calculated according to Edward[25]), and a is the Stokes–Einstein number. For self-diffusion, $a = 3$,[26] and for diffusion of larger molecules in a liquid of smaller molecules, $a = 6$.[27] The various rate parameters responsible for fluorescence quenching have been determined. In the light of these rate parameters, lifetime measurements at different temperatures (30–60°C) without quencher, and dependence of quenching rate parameters on the temperature of the medium, the possible quenching mechanisms are discussed.

If the probability per encounter p is equal to unity, then the quenching mechanism is controlled purely by material diffusion,[23] otherwise, if it is less than unity, the reaction of quenching is not solely controlled by material diffusion and may depend on the activation process. The activation energy for quenching reaction can be given by

$$E_a = E_d + RT \ln\left[\frac{1}{p} - 1\right], \tag{7}$$

where E_d is the activation energy for diffusion whose values are taken from literature. If $E_a < E_d$ ($E'_a < E_d$), the reaction of quenching is controlled purely by material diffusion, and if $E_a > E_d$, ($E'_a > E_d$), the reaction of quenching may not be controlled by diffusion alone.

MATERIALS AND METHODS

The solute *m*-terphenyl was obtained from Sigma-Aldrich (St. Louis, MO, USA) and was of scintillation grade and used without any further purification. The quencher CCl_4 was obtained from BDH Laboratory (Glaxo, Mumbai, India) and was of HPLC grade. All the solvents of HPLC grade were obtained from S.D. Fine Chemical Ltd (Mumbai, India) and were used without any further purification. The solutions were prepared keeping the concentration of *m*-terphenyl fixed (1×10^{-4} M) and varying the concentration of the quencher (0.02 to 0.1 M). The absorption maxima for *m*-terphenyl corresponds with 275 nm. Hence the solute was excited at 275 nm, and the fluorescence spectrum was recorded in all the solvents. The fluorescence maxima corresponds with around 335 nm in all the solvents studied. Fluorescence intensities of all the solutions were measured by using fluorescence spectrophotometer F-2000 (Hitachi, Japan; bandwidth 10 nm). Fluorescence measurements were made with perpendicular geometry by taking fresh solution each time in a rectangular quartz cell having an airtight stopper. First the fluorescence intensity I_o was measured without the quencher, and then the fluorescence intensity I was measured at different quencher concentrations and at a fixed solute concentration. Fluorescence lifetime of the solute with and without quencher was measured at different temperatures by using single photon counting time-domain spectrometer (Model EI-199; Edinburgh Instrument, UK). Hydrogen gas was used to fill the lamp of the single photon counting (SPC) system at 0.6 atm pressure, keeping the electrode separation at 0.7 mm, applying the discharge voltage of about 7 kV, and operating the lamp at a frequency of 30 kHz. With this setting, lamp pulses having pulse width of about 1 ns at FWHM (full width at half maximum) were obtained. A nonlinear least square iterative reconvolution mathematical procedure was used here. The fluorescence lifetime data were analyzed by considering reduced chi-square (χ^2_r) value, which is nearly equal to one. The experimental values are reproducible within 5% of the experimental error. Description of the experimental setup is presented elsewhere.[14]

RESULTS AND DISCUSSION

The fluorescence intensities I_o and I were measured without and with quencher, respectively, at different quencher concentrations and at a fixed solute concentration. The typical absorption spectra of *m*-terphenyl at different concentrations of quencher CCl_4 in hexane is obtained, and it is found that there is no apparent shift in absorption wavelength. The typical emission spectra of *m*-terphenyl at different concentrations of quencher CCl_4 in 1,4-dioxane was also obtained. It is observed that there is no apparent shift in emission wavelength at different concentrations of quencher. This fact rules out the possibilities of ground state complex formation. The S-V plots were obtained by using experimentally determined values of I_o and I in each solvent and are shown in Fig. 1, and we see that S-V plots (I_o/I) against [Q] are linear with intercept nearly equal to unity. This clearly indicates that the steady-state quenching phenomenon in all solvents follows the S-V relation. Further, the fluorescence lifetimes τ_o and τ for the solute were measured in benzene, without and with quencher. The total fluorescence decay was biexponential, and it is observed that τ_1 is less than τ_2. However, τ_1 value cannot be neglected in view of its relative amplitude contribution.

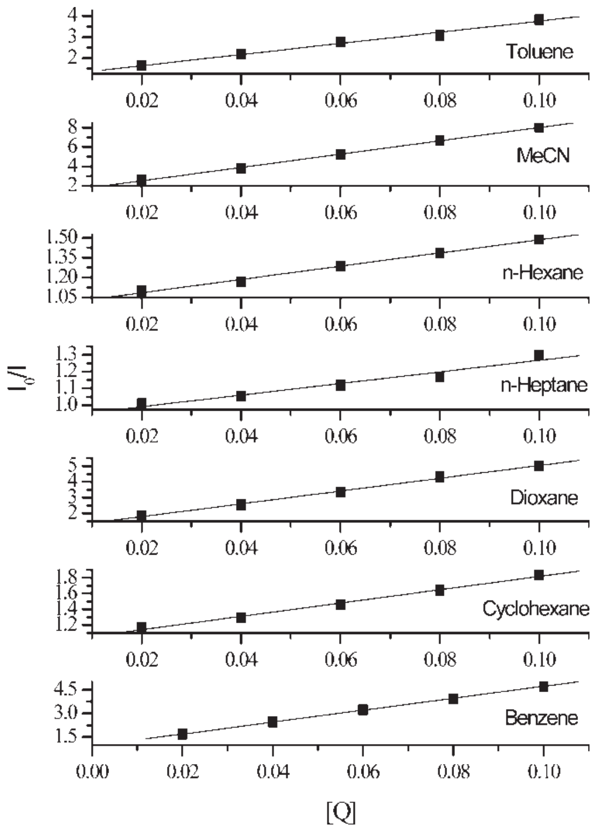

Figure 1. S-V plots of I_o/I against [Q] for *m*-terphenyl in different solvents.

Table 1. Fluorescence lifetime as a function of quencher concentration at a fixed solute concentration in benzene at room temperature

Quencher concentration [Q] M	τ_1 (ns)	a_1	τ_2 (ns)	a_2	τ_{av} (ns)	τ_0/τ
0.00	0.97	0.13	11.80	0.87	10.39	—
0.02	0.95	0.17	9.82	0.83	8.33	1.24
0.04	0.81	0.20	8.08	0.80	6.62	1.56
0.06	0.75	0.42	3.82	0.58	2.52	4.12
0.08	0.73	0.55	2.70	0.45	1.61	6.45
0.10	0.70	0.57	2.08	0.42	1.28	8.11

Hence, we considered the average value for studying the quenching phenomena. The average lifetime value is given in Table 1. The typical fluorescence decay profile of *m*-terphenyl in benzene with CCl_4 as quencher at different concentrations (0.00–0.10 M) is shown in Fig. 2. In order to check whether these reactions are diffusion limited, the temperature dependence of rate constant k_q is examined. The fluorescence quenching was carried out in the temperature range 30–60°C by steady-state method and by transient method at room temperature in benzene. The fluorescence lifetime τ of *m*-terphenyl in benzene (without CCl_4) at different temperatures is measured by using SPC. The decay curve for *m*-terphenyl in benzene in the temperature range 30–60°C is shown in Fig. 3. The red curve in Fig. 3 represents the lamp profile. The fluorescence lifetime values were obtained by fitting experimental decay profiles to double exponential function to get χ^2 values very close to unity and are listed in Table 2. The fluorescence lifetime decreases slightly with increase in temperature. The S-V plots from steady-state measurements in the temperature range 30–60°C show linear

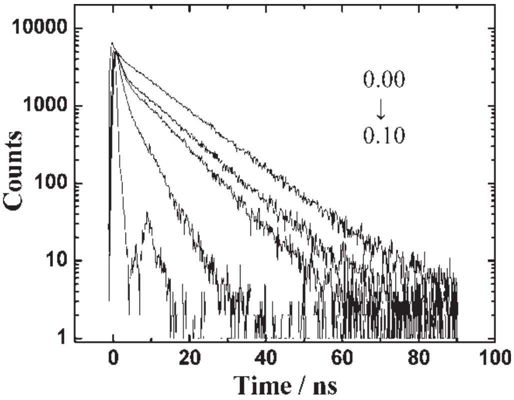

Figure 2. Fluorescence decay of *m*-terphenyl in benzene with CCl_4 quencher.

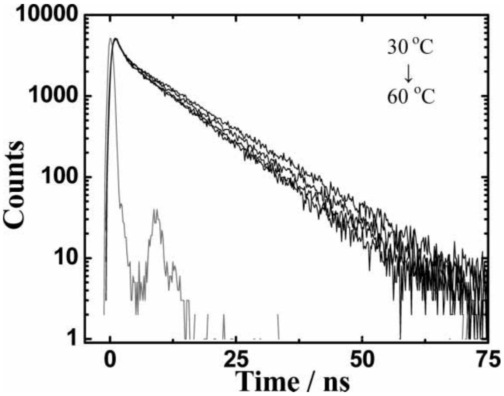

Figure 3. Fluorescence decay curves for *m*-terphenyl at different temperatures (30–60°C) in benzene.

variation with intercept nearly equal to unity as shown in Fig. 4. The values of K_{sv} and k_q were determined at all temperatures and are listed in Table 2. From this table, it is clear that the values K_{sv} and k_q increase with temperature. It has been assumed that k_q is proportional to exp $^{(-E_q/RT)}$,[6] where E_q is the activation energy for collisional process, R the gas constant, and T the absolute temperature. Thus, the value E_q can be determined from the slope of the plot of ln k_q versus inverse of the absolute temperature T. Figure 5 shows the plot of ln k_q versus $10^3/T$ for *m*-terphenyl, which is linear. From the least square fit value of the slope of this plot, the value of E_q has been determined and is given in Table 2. The value of E_q is 32.67 kJ mol^{-1}, being approximately equal to the values obtained by others suggesting that the rate of reaction is limited by diffusion of the reactants through the solvent. Hence we may conclude that the process of quenching is due to close collision between the excited solute and quencher molecules controlled by material diffusion in this system. The S-V plot τ_o/τ versus [Q] using Eq. (2) in benzene is

Table 2. The fluorescence lifetime τ, Stern–Volmer quenching constant K_{sv}, and quenching rate parameter k_q as a function of temperature and the activation energy for collisional quenching process E_q.

Temperature (°C)	Lifetime τ (t) (ns)	K_{sv} (M^{-1})	$k_q \times 10^{-9}$ (M^{-1} s^{-1})	E_q(kJ mol^{-1})
30	9.8	33.0	3.36	32.67
40	8.9	36.4	4.08	
50	8.4	37.0	4.40	
60	8.0	40.0	5.00	

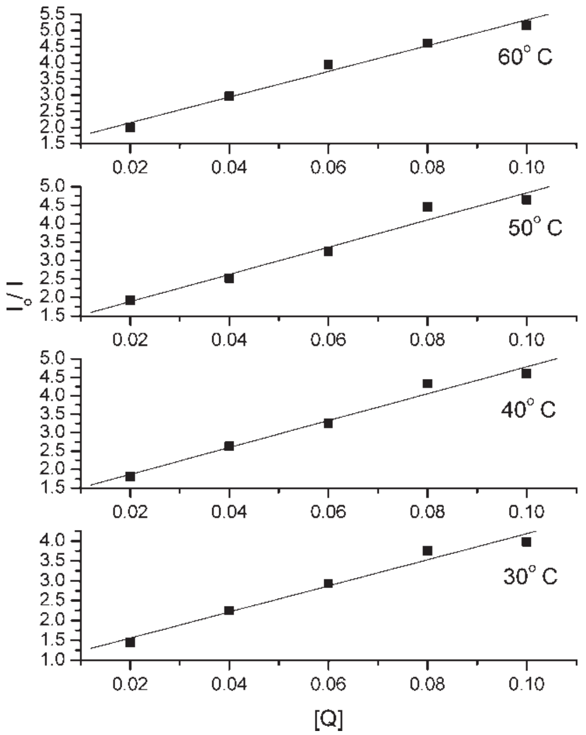

Figure 4. S-V plots from steady-state fluorescence emission intensity measurements for *m*-terphenyl + CCl_4 system in benzene at different temperatures.

shown in Fig. 6, which is found to be nonlinear. It indicates that transient effect is playing a role in overall quenching process. The least square fit values of the slopes ($k_q\tau_o$) were obtained in each solvent medium and are given in Table 3. Further using the experimentally determined value of τ_o= 10.39 ns, the quenching rate parameter k_q was obtained for each solvent, and the values are given in Table 3. From transient method, the values of K'_{SV}, p', and k'_q were determined and are given in Table 3. In order to calculate the rate parameter k_o according to Eq. (5), the sum of the diffusion coefficients D of solute and quencher was calculated using Eq. (6), and the sum of the molecular radii R of solute was calculated by using the method suggested by Edward.[25] The values of k_o calculated using Eq. (5) for all the solvents are given in Table 4. The term in the larger bracket of Eq. (5) is neglected, as its contribution is negligible.

Further using the experimentally determined values of k_q and the calculated values of k_o, the probability of quenching per encounter p was determined according to Eq. (3) for all the solvents, and the values are given in the Table 3. The values of p are less than unity for all the solvents, indicating that the probability of quenching per encounter is always less

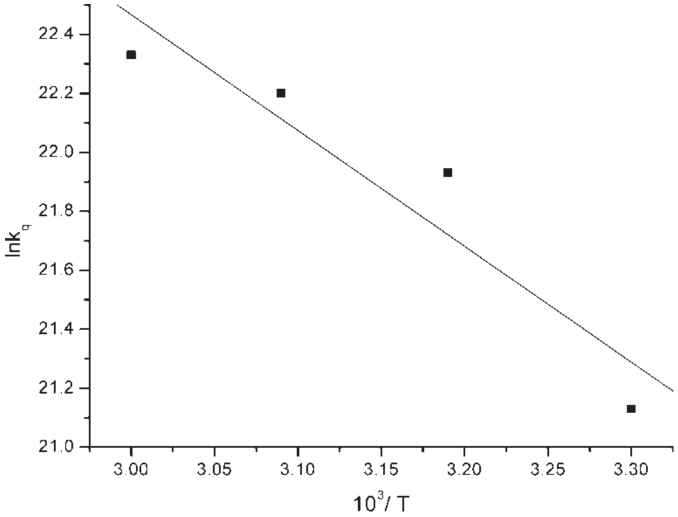

Figure 5. Plot of ln k_q versus $10^3/T$ for *m*-terphenyl + CCl_4 system.

than unity, a fact hitherto observed by others,[14,15,22] and hence it is only due to close collision between the solute and quencher molecules. Hence the reaction of quenching is not solely controlled by material diffusion, in which case p would be unity. The variation of p with different solvents confirms this fact. From Fig. 7, we see that although the frequency of

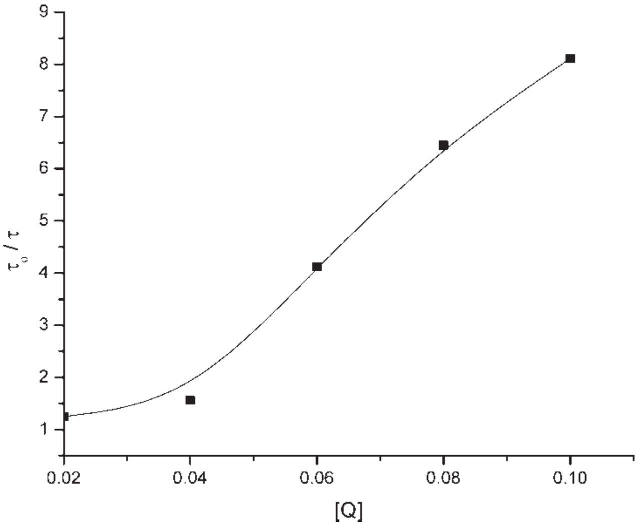

Figure 6. S-V plot of τ_0/τ versus $[Q]$ in benzene for *m*-terphenyl + CCl_4 system.

Table 3. The values of inverse of viscosity η^{-1}, slope ($k_q\ \tau_o$), quenching rate parameter k_q, and quenching probability per encounter p

Solvent	$\eta^{-1} \times 10^{-2}$ (P^{-1})	$k_q\ \tau_o$ (M^{-1})	$k_q \times 10^{-9}$ ($M^{-1}s^{-1}$)	p
n-Hexane	3.35	5.00	0.48	0.01
n-Heptane	2.52	3.44	0.33	0.01
Toluene	1.81	26.50	2.48	0.13
Benzene	1.66	37.93	3.65	0.20
		[$K'_{SV} = 0.23$]	[$k'_q = 0.22$]	[$p' = 0.32$]
Acetonitrile	1.34	68.76	6.62	0.46
Cyclohexane	1.11	8.47	0.82	0.07
1,4-Dioxane	0.83	40.57	3.90	0.44

collisional encounter k_o increases as the viscosity decreases, the rate constant k_q does not depend on the viscosity of the solvent. Hence we may infer that the phenomenon of quenching is not solely controlled by material diffusion. Therefore, in addition to diffusion, it may also depend on the activation process.

In order to see whether activation process is playing a role, we have calculated the activation energy for quenching reaction E_a using the literature values of E_d, activation energy for diffusion, and the experimentally determined values of p, probability per encounter, according to Eq. (7). From Table 4, it is clear that $E_a > E_d$ ($E'_a > E_d$ in benzene) in all the solvents; this clearly proves the fact that the quenching reaction is not controlled by material diffusion alone. Further for pure dynamic quenching $I_o/I = \tau_o/\tau$.[28] This means that the values of quenching rate parameters k_q and k'_q determined

Table 4. The values of diffusion coefficients D_Y and D_Q of the solute and quencher, respectively, diffusion rate parameter k_o, activation energy for diffusion E_d, and activation energy for quenching E_a

Solvent	$D_Y \times 10^5$ ($cm^2\ s^{-1}$)	$D_Q \times 10^5$ ($cm^2\ s^{-1}$)	$k_o \times 10^{-9}$ ($M^{-1}\ s^{-1}$)	E_d (Kcal mol^{-1})	E_a (Kcal mol^{-1})
n-Hexane	1.93	5.40	36.25	2.07	12.86
n-Heptane	1.45	4.05	27.25	2.19	13.18
Toluene	1.04	2.91	19.54	2.66	7.47
Benzene	0.96	2.67	17.95	3.00	6.41
					[$E'_a = 8.36$]
Acetonitrile	0.77	2.16	14.49	—	—
Cyclohexane	0.59	1.64	11.02	3.23	9.54
1,4-Dioxane	0.48	1.34	8.98	3.06	6.31

$R_Y = 3.81$ Å; $R_Q = 2.73$ Å.

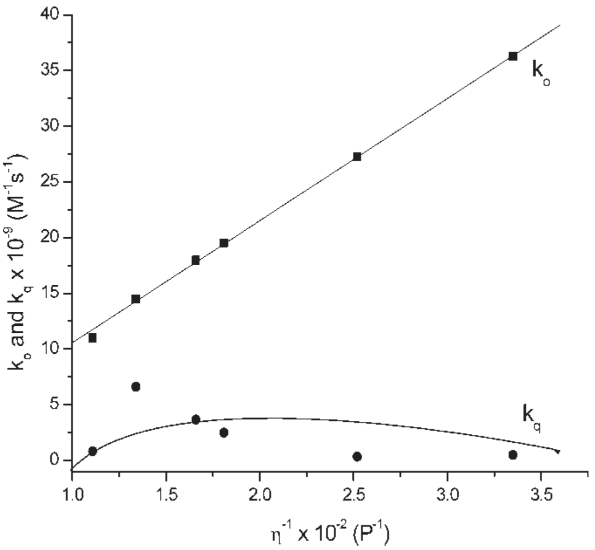

Figure 7. Variation of k_q and k_o as a function of inverse of viscosity $\eta^{-1} \times 10^{-2} P^{-1}$ of the solvents.

from Eqs. (1) and (2), respectively, are same. However, from Table 3 we observe that in case of benzene for this system, $k'_q < k_q$. This indicates that fluorescence quenching of *m*-terphenyl by CCl_4 in benzene is not purely dynamic. This supports our experimental result that quenching reaction is not solely controlled by material diffusion but there is also contribution due to some activation processes.

CONCLUSIONS

From the ongoing discussion, we find that S-V plots are linear in all the solvents. The value of probability p is less than unity in all the solvents. The value of E_a (E'_a), activation energy for quenching process, is greater than E_d, activation energy for diffusion, in all the solvents, which confirms the fact that the fluorescence quenching of *m*-terphenyl by CCl_4 is not solely controlled by material diffusion. Fluorescence lifetime (τ) decreases slightly with increase in temperature, whereas fluorescence quenching rate parameter k_q increases with increase in temperature.

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