Laser Dye Diffusion in Polymer Solutions Studied by Spectrophotometry*

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ABSTRACT: A mathematical approach based on the combined application of Fick's law of diffusion and the Beer–Lambert law was used to measure the diffusion coefficient of dyes viz., coumarin 515 [3-(1-methylbenzimidazol-2-yl)-7-diethylaminocoumarin], POPOP [1,4-bis (5-phenyloxazolyl-2-yl) benzene], and exalite 416 {1,1,1′,1′-tetraethyl-5,5′-diparaanisyl-[2,3:5,6] diindanobenzene} in solutions of polystyrene and poly(methyl methacrylate) in 1,4-dioxane. Diffusion data agreed well with the published results obtained using other techniques. © 2004 Wiley Periodicals, Inc. J Appl Polym Sci 93: 1157–1165, 2004

Key words: diffusion coefficient; polymer solution; laser dyes, Fick's law, Beer–Lambert law

INTRODUCTION

Advances in plastic scintillators have spurred exciting new innovations in a wide variety of areas such as nuclear and high-energy physics, medical imaging, and aerospace. In view of the widespread use of plastic scintillators in medical imaging, polymers like polystyrene, polyvinyl toluene, and methyl-substituted polystyrene, after doping with primary and secondary dyes, have been used.[1] The commercially available plastic scintillators contain polymer matrix doped with two fluorescent dyes viz., a primary dopant called the fluor and a secondary dopant called the wavelength shifter. The latter is usually added at a lower concentration than the primary dye. Among many compounds used as dopants, coumarin 515 [3-(1-methylbenzimidazol-2-yl)-7-diethylaminocoumarin], POPOP [1,4-bis (5-phenyloxazolyl-2-yl) benzene] and exalite 416 {1,1,1′,1′-tetraethyl-5,5′-diparaanisyl-[2,3:5,6] diindanobenzene} have been widely used. Diffusion of these dyes in polymer matrices used in scintillation processes is important. Therefore, in the present study, polystyrene (PS) and poly(methyl methacrylate) (PMMA) solutions in 1,4-dioxane are used to study the diffusion of the selected laser dyes mentioned above. Such studies are important to understand the physical and chemical processes occurring in polymer solutions containing dye molecules at different concentrations of polymer solutions and at different temperatures.[2–10] A PC-based spectrophotometer was used to record the absorption spectra of dye molecules diffusing across a plane in the solution column held in a standard cell. The recorded spectra were used to determine the solute concentration as a function of time to compute the diffusion coefficient of the dye.

EXPERIMENTAL SECTION

Materials

Coumarin 515 and exalite 416 laser dyes were supplied by Exciton. The scintillating grade POPOP dye was obtained from S.D. Fine Chemicals (Mumbai, India). 1,4-Dioxane, chloroform, and toluene solvents of HPLC grade supplied by S.D. Fine Chemicals were used as received. Polystyrene and poly(methyl methacrylate) were procured from Fluka (Germany). The diffusion cell, Q, is a rectangular quartz cell of 45 mm height and 10 mm sides (see Figure 1). In our experiment the solvent, M, was 1,4-dioxane, P was the polymer (PS or PMMA), and the solute, N, was a laser dye. Heights of the columns, MP and MN, were 30 and 10 mm, respectively.

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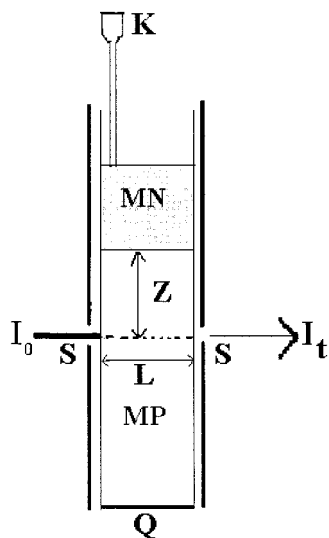
Figure 1 Schematic diagram of the experimental setup in the sample compartment of the spectrophotometer for measurement of the diffusion coefficient. Q, quartz cell; K, funnel with capillary tube; S, slit; MP, column of polymer solution; MN, column of dye solution; I_0, incident light beam; I_t, transmitted light beam; L, path length of light beam in the liquid; Z, distance of light beam from the interface.

Preparation of polymer and dye solution for diffusion measurements

Polymers and dyes were weighed in an electronic Mettler balance (Model AE 240, Switzerland). Coumarin 515 dye of concentration 2×10^{-3} M was prepared in 1,4-dioxane by dissolving 0.0070 g of the dye in 10 mL of 1,4-dioxane. POPOP and exalite 416 dye of concentration 2×10^{-3} M were prepared in 1,4-dioxane in the same manner. PS solutions of concentrations 3.75, 5.00, 7.50, and 10 g/dL and PMMA solutions of concentration 1g/dL were prepared in 1,4-dioxane.

The diffusion cell was prepared by taking a cell with 3 mL of polymer solution, MP, which was placed in the sample compartment of the spectrophotometer. It was then allowed to stabilize at room temperature for 10–15 min. Then, 1 mL of dye solution, MN, was gently introduced on top of the MP through a small glass funnel with a capillary tube, K, positioned at the corner of the cell (see Figure 1). The dye solution was lighter than the polymer solution and formed a colored band above the colorless transparent polymer solution. The temperature of the diffusion cell was controlled to an accuracy of ±0.1°C using a temperature controller unit attached to the instrument.

Spectral measurements

The PC-based Varian Model Cary 50 Bio UV-VIS spectrophotometer with Cary single cell Peltier accessory for temperature control was used within an absorbance accuracy of ±0.0004 and wavelength accuracy of ±0.2 nm. The sample was maintained at a constant temperature in the range from 20 to 60°C within an accuracy of ±0.1°C. To obtain the values of $A(Z,t)$, we chose $Z = 10$ mm and the light ray was allowed to pass through the diffusion cell at a height of 20 mm from the bottom of the cell or 10 mm below the interface between MN and MP by proper positioning of slits, S, on either side of the cell, Q. The slits also served to restrict the size of the light beam incident on the cell to a height of 0.5 mm in the vertical z-direction and a width of 3 mm in the horizontal direction. The path length, L, of the light beam through the solution, MPN, was 10 mm. To check the stability of the spectrophotometer, absorption spectra of the standard solution were recorded at 10-min intervals over a period of 3 h.

Viscosity measurements

Viscosities of polymer solutions were measured using a Canon Fenske viscometer with a capillary size of 300 (ASTM D 445, supplied by the Industrial Research Glassware Ltd.). Measurements were carried out at a constant temperature using a Schott-Gerate water bath (Model CT 050, Germany). Calibration details and viscosity measurements are the same as those given earlier.[11,12] By measuring viscosity as a function of concentration of PS in chloroform and PMMA in toluene at 30°C, viscosity average molecular weights of PS and PMMA were calculated to be 1.8×10^5 and 7.9×10^3, respectively.

THEORETICAL

Computation of diffusion coefficient of dye molecules in polymer solutions

Figure 1 schematically depicts the experimental set up. A column of polymer solution MP consisting of a polymer P dissolved in an organic solvent M held in a standard 1-cm quartz cuvette cell was used. On top of this column MP, a solution MN, consisting of a solute, N, dissolved in the same solvent, M, was introduced gently at time $t = 0$. The interface formed between the two solutions at this instant ($t = 0$) is taken as $z = 0$ plane. The column, MP, can be considered a semi-infinite slab; as time progresses, diffusion of M and N occurs along the z-direction. This is the standard case of one-dimensional diffusion. In unsteady state diffusion along the z-direction, a concentration gradient exists, whose profile changes with time. Thus, due to diffusion, the column, MP, becomes MPN in which the concentration of N, i.e., $C(Z,t)$, depends on the distance, z, from the interface and time, t, elapsed after
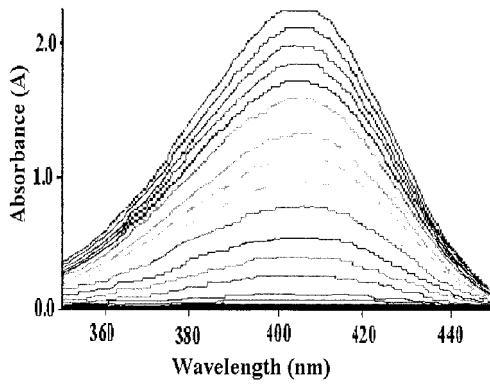
Figure 2 Absorption spectra of coumarin 515 dye diffusing in PS solution of concentration 7.5 g/dL at different time intervals after commencement of diffusion at 25°C.

TABLE I
Results of Viscosity and Diffusion Coefficient of Coumarin 515 Dye in Polystyréne Solutions at Different Concentrations at 25°C

Concentration, C (g/dL)	Viscosity, η (mPa.s)	Diffusion coefficient, $(D \times 10^5)$ cm^2/s
3.75	5.115	3.57
5.00	7.443	3.07
7.50	14.197	1.97
10.00	25.904	0.914

the commencement of diffusion. This type of system can be considered nearly ideal for the application of Fick's law of diffusion.[3]

We now consider the diffusion of solute, N, across the horizontal plane at a distance $z = Z$ from the interface. The concentration of N at any point, $C(Z,t)$, on this plane at any time, t, will be a function of distance of the plane from the interface, Z, and the time elapsed, t, after formation of the interface. To probe this concentration, let a narrow beam of monochromatic light of intensity $I_0(Z,t)$ be incident normal to MPN at Z at time t. Neglecting absorption by MP, the intensity of light transmitted, $I_t(Z,t)$, through the thickness, L, of the solution at Z at the instant, t, will

depend upon $C(Z,t)$. Consequently, absorbance, $A(Z,t)$ of N at Z at time t will also depend upon $C(Z,t)$.

In the present treatment, Fick's law of diffusion gives the relationship between $C(Z,t)$ and diffusion coefficient, D, while the Beer–Lambert law is used to calculate $C(Z,t)$ in terms of absorbance, $A(Z,t)$, to establish the relationship between $A(Z,t)$ and D. According to Fick's second law of diffusion for one-dimensional diffusion along the z-axis, we have[3]

$$\left(\frac{\partial C}{\partial t}\right)_z = D \left(\frac{\partial^2 C}{\partial z^2}\right)_t. \qquad (1)$$

Employing the boundary conditions, Eq. (1) can be solved to give

$$C(Z,t) = \frac{C_0}{\sqrt{\pi D t}} \exp\left(\frac{-Z^2}{4Dt}\right). \qquad (2)$$
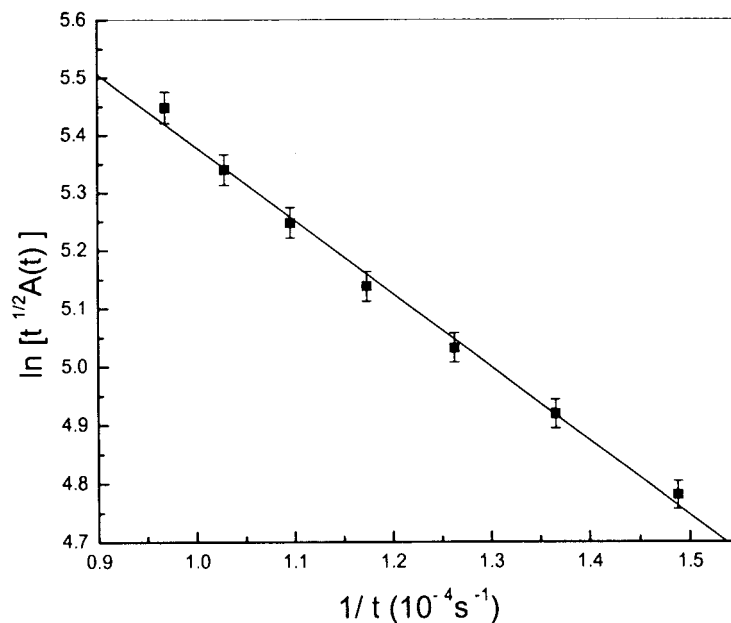
Figure 3 Variation of $\ln[t^{1/2} A(t)]$ with $1/t$ for coumarin 515 dye diffusing in PS solution of concentration 7.5 g/dL at different time intervals, t, after commencement of diffusion at 25°C.
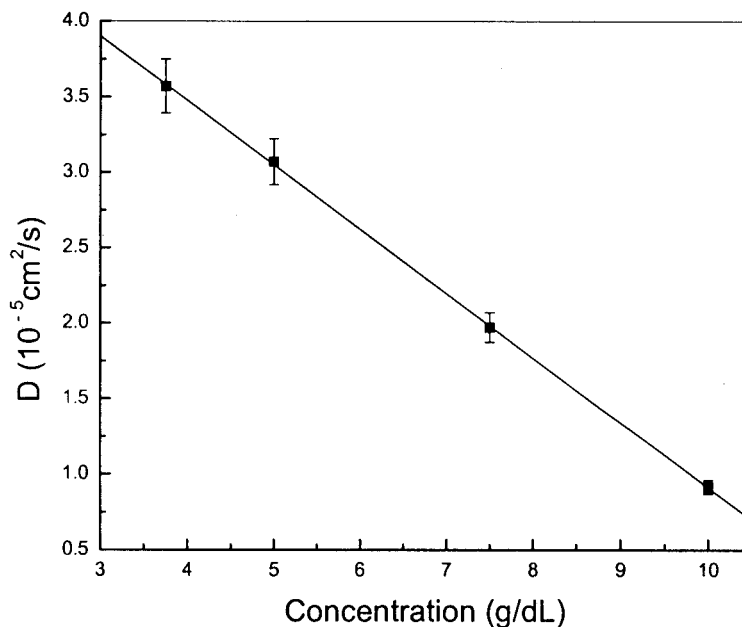
Figure 4 Variation of the diffusion coefficient of coumarin 515 dye in PS solution at different concentrations at 25°C.

Here, C_0 is a constant equal to concentration of N per unit area at $Z = 0$ and $t = 0$. Equation (2) in the logarithmic form can be given as

$$\ln\left[\sqrt{t}C(Z,t)\right] = \ln\left[\frac{C_0}{\sqrt{\pi D}}\right] - \left[\frac{Z^2}{4D}\right]\frac{1}{t}. \qquad (3)$$

Thus, the plot of $\ln\left[\sqrt{t}C(Z,t)\right]$ versus $1/t$ would be a straight line with slope $= (Z^2/4D)$. Therefore, from a set of values of $C(Z,t)$ measured as a function of time, t, the diffusion coefficient, D, can be calculated. In the present method, D is calculated by measuring absorbance, $A(Z,t)$ corresponding to $C(Z,t)$.

According to the Beer–Lambert law, transmitted intensity, $I_t(Z,t)$ related to incident intensity, $I_0(Z,t)$, and concentration, $C(Z,t)$, is given by

$$I_t(Z,t) = I_0(Z,t)\exp[-\varepsilon C(Z,t)L], \qquad (4)$$

TABLE II
Results of Viscosity and Diffusion Coefficient of coumarin 515 Dye in Polystyrene Solutions at Different Temperatures

Temperature (°C)	Viscosity, η (mPa.s)	Diffusion coefficient, $(D \times 10^5)$ cm²/s
25	25.905	0.914
30	21.618	1.498
35	18.349	3.090
40	15.705	6.378

where ϵ is the molar extinction coefficient of the dye molecule. Equation (4) can be rearranged to give

$$C(Z,t) = \frac{2.303}{\varepsilon L}A(Z,t). \qquad (5)$$

Combining Eqs. (3) and (5) we get

$$\ln\left[\sqrt{t}A(Z,t)\right] = \ln\left[\frac{C_0\varepsilon L}{2.303\sqrt{\pi D}}\right] - \left(\frac{Z^2}{4D}\right)\frac{1}{t}. \qquad (6)$$

The linear plot of $\ln\left[\sqrt{t}A(Z,t)\right]$ versus $[1/t]$ can be constructed with a slope $= -(Z^2/4D)$. Thus, in the experimental geometry of Figure 1 and from the measured values of $A(Z,t)$ at known times, t, D can be calculated.

In the present method, $A(Z,t)$ is obtained as a function of t by placing the diffusion cell in the sample compartment of the spectrophotometer and then recording the absorption spectra at different intervals. Before the introduction of solution MN over MP, the absorption spectrum of MP is taken as the reference spectrum. After the introduction of solution MN over MP, absorption spectra are automatically scanned and saved at the preprogrammed intervals. Since absorption spectrum of MP is subtracted from each of the absorption spectrum of MPN, the recorded spectra represent the absorption spectra of N in MPN.

The absorption spectrum represents the plot of absorbance, $A(Z,t)$, versus wavelength, λ, which has a broad peak, λ_m, and is a characteristic of the absorbing
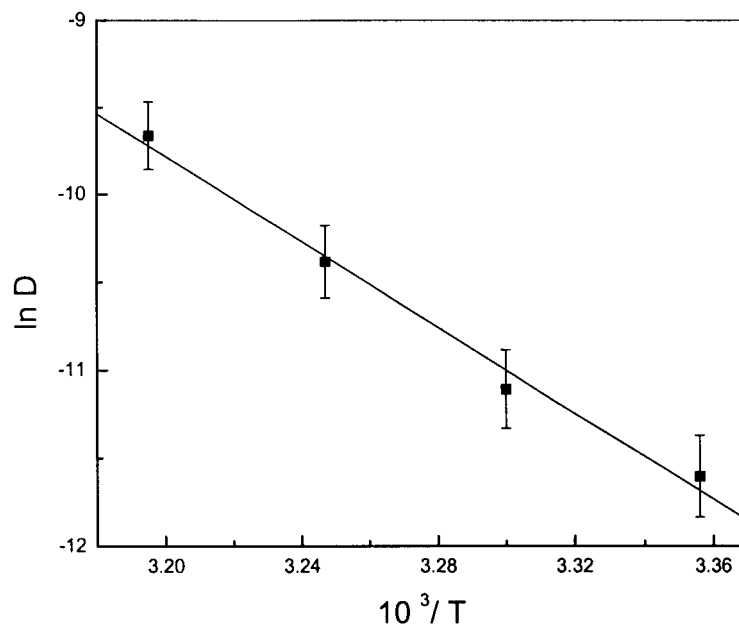
Figure 5 Arrhenius plot for coumarin 515 dye diffusing in PS solution of concentration 10 g/dL in 1,4-dioxane.

molecule. This is used to identify the diffusing dye molecule, N. In Eq. (6), $A(Z,t)$ is used for the wavelength, λ_m. From the set of spectra recorded at different times, t, and using Eq. (6), the diffusion coefficient of the dye molecule can be calculated.

RESULTS AND DISCUSSION

Diffusion of coumarin 515 in polystyrene solutions

The absorption peak of coumarin 515 dye was observed at 405 nm, which is rather widely separated from the absorption peak of 1,4-dioxane at 210 nm and that of polystyrene at 252 nm.[13] Since we are interested in the diffusion of coumarin 515 in PS solution, the range of wavelength for display was taken from 350 to 450 nm, which is the absorption region for coumarin 515. Just before MN was introduced over MP, the spectrum recorded gave a flat baseline. After the introduction of MN, the absorption spectrum of N having a broad peak at 405 nm and steadily increased with time, indicating an increase in the concentration of N due to the progressive diffusion of N through MP.

Typical absorbance spectra of coumarin 515 in PS solution of 7.5 g/dL concentration at 25°C, recorded at intervals of 10 min over a period of 3 h, are shown in Figure 2. Absorbance data, $A(Z,t)$, at 405 nm as a function of t were used to plot the graph of $\ln[\sqrt{t}A(Z,t)]$ versus $[1/t]$ as shown in Figure 3. From the slope of the straight line (deviation < 2%), the

value of D $(1.97 \pm 0\ 0.02) \times 10^{-5}$ cm^2/s was calculated.

To study the concentration dependence of diffusivity, the spectra of coumarin 515 dye were recorded at 25°C. Values of D calculated in PS solution concentration of 3.5, 5.0, and 10 g/dL in 1,4-dioxane are presented in Table I. Values of D vary linearly with polymer concentration, C, according to the relationship[14]

$$D = D_0(1 + kC), \tag{7}$$

where k is a constant and D_0 is the concentration-independent diffusion coefficient. Thus, a plot of D versus C is a straight line within a deviation of 1.5%, as
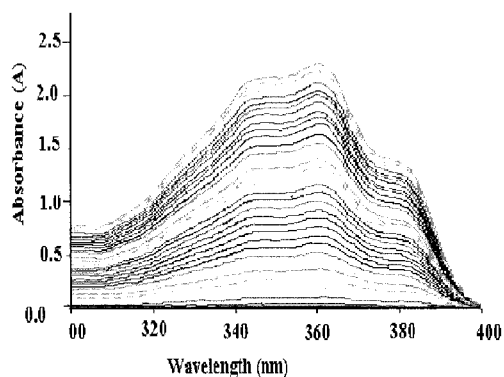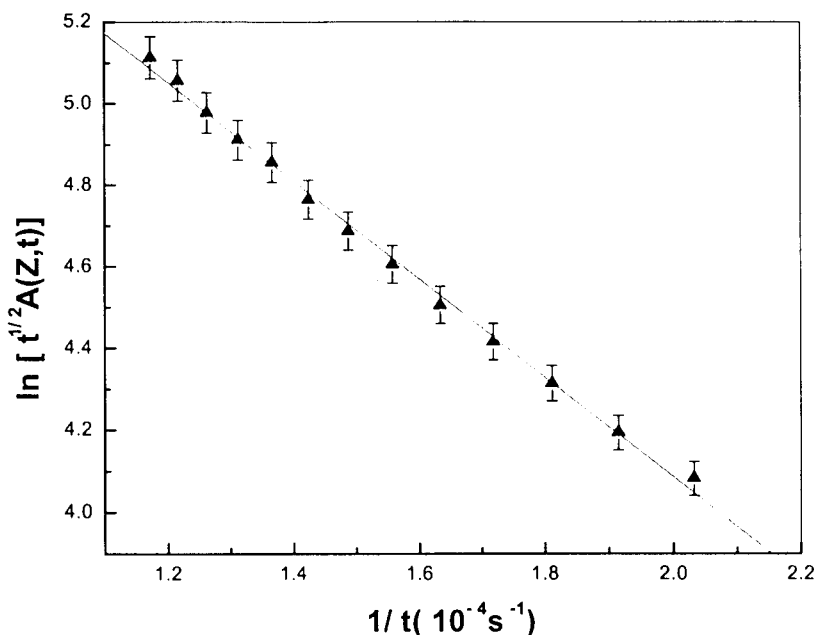

Figure 6 Absorption spectra of POPOP dye diffusing in PS solution of concentration 7.5 g/dL in 1,4-dioxane at different time intervals after commencement of diffusion at 25°C.

Figure 7 Variation of $\ln[t^{1/2} A(Z,t)]$ with $1/t$ for POPOP dye diffusing in PS solution of concentration 7.5 g/dL at different time intervals, t, after commencement of diffusion at 25°C.

shown in Figure 4. The value of D_0 in pure solvent is found to be $(5.18 \pm 0\ 0.03)10^{-5}$ cm²/s.

The temperature dependence of D was studied using an Arrhenius-type relationship,[15]

$$\ln D = \ln D_A - \left(\frac{E_D}{RT}\right), \qquad (8)$$

where D_A is the Arrhenius frequency factor, E_D is the activation energy for diffusion, and R is the universal gas constant taken as 8.314 mol/K. The D values for coumarin 515 dye calculated in PS solution of concentration 10 g/dL at 25, 30, 35, and 40°C are presented in Table II. An Arrhenius plot of the diffusion coefficient with temperature for PS solution of concentration 10 g/dL is shown in Figure 5. From this plot, the value of E_D was calculated to be 2.23 ± 0.11 kJ/mol.

Diffusion of POPOP dye in polystyrene solutions

The absorption peak for POPOP is observed at 360 nm, which is rather widely separated from the absorption peak of 1,4-dioxane at 210 nm and that of PS at 252 nm.[15] Since we are interested in the diffusion of POPOP in PS solution, the range of wavelength was taken between 300 and 400 nm, which is the absorption region for POPOP. Typical absorbance spectra of POPOP in PS solution of 7.5 g/dL concentration at 25°C, recorded at intervals of 10 min for 2 h and 10 min, are shown in Figure 6. Absorbance data, $A(Z,t)$, at 360 nm as a function of t were used to plot the graph

of $\ln[\sqrt{t}A(Z,t)]$ versus $[1/t]$ as shown in Figure 7, from which the value of D of $(2.07 \pm 0\ 0.02) \times 10^{-5}$ cm²/s was calculated. The D values of POPOP calculated in PS concentrations of 5 and 10 g/dL in 1,4-dioxane at 25°C are given in Table III. The plot of variation of D of POPOP dye with concentration of PS is shown in Figure 8. The value of D_0 in 1,4-dioxane is found to be $(2.18 \pm 0\ 0.02) \times 10^{-5}$ cm²/s.

Diffusion of exalite 416 dye in polystyrene solutions

The absorption peak of exalite 416 was observed at 355 nm, which is rather widely separated from the absorption peak of 1,4-dioxane at 210 nm and that of polystyrene solution at 252 nm. Since we are interested in the diffusion of exalite 416 in PS solution, the range of wavelength for display was taken from 300 to 400 nm, which is the absorption region for exalite 416. Typical absorbance spectra of exalite 416 in PS solution of 10

TABLE III
Results of Viscosity and Diffusion Coefficient of POPOP Dye in Polystyrene Solutions at Different Concentrations, at 25°C

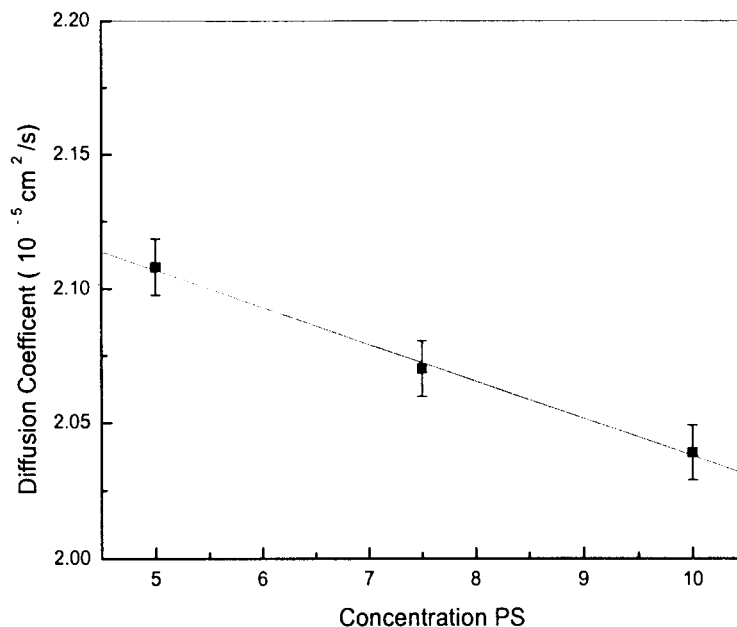| Concentration, C (g/dL)	Viscosity, η (mPa.s)	Diffusion coefficient, $(D \times 10^5)$ cm²/s
5	7.443	2.108
7.5	14.197	2.070
10	25.904	2.039

Figure 8 Variation of diffusion coefficient values of POPOP dye in PS solution of different concentrations at 25°C.

g/dL concentration at 25°C, recorded at intervals of 10 min over a period of 2 h, are shown in Figure 9. Absorbance data, $A(Z,t)$, at 355 nm as a function of time were used to plot the graph of $\ln[\sqrt{t}A(Z,t)]$ versus $[1/t]$ as shown in Figure 10, from which D was calculated to be $(1.52 \pm 0.02) \times 10^{-5}$ cm^2/s.

Diffusion of POPOP dye in poly(methyl methacrylate) solutions

The absorption peak of POPOP was observed at 360 nm, which is rather widely separated from the absorption peak of 1,4-dioxane at 210 nm and that of PMMA at 228 nm.[15] Since we are interested in the diffusion of POPOP in PMMA solution, the range of wavelength for the display was taken from 300 to 400 nm, which is
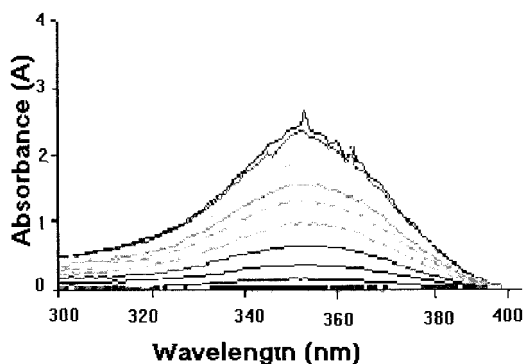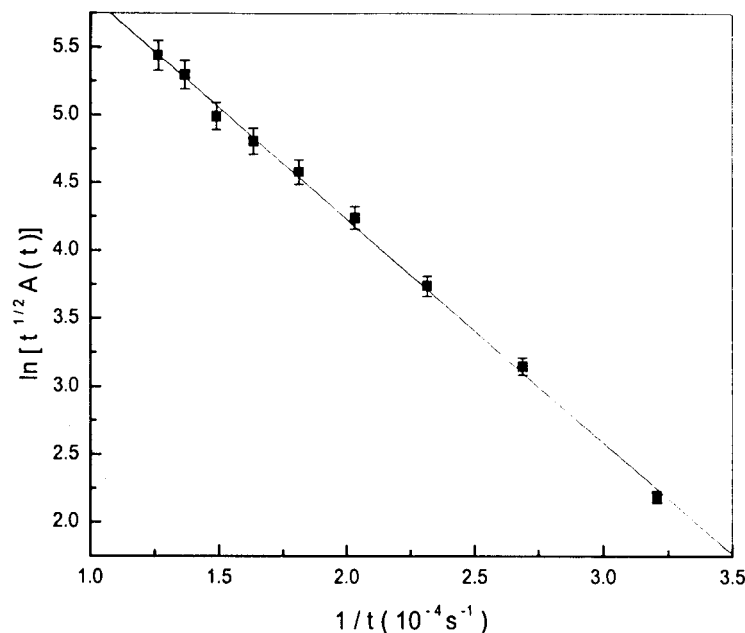

Figure 9 Absorption spectra of exalite 416 dye diffusing in PS solution of concentration 10 g/dL at different time intervals after commencement of diffusion at 25°C.

the absorption region for POPOP. The viscosity of PMMA solution at 25°C was 19.21 mPa·s. Typical spectra of POPOP in PMMA solution of 1.0 g/dL concentration at 25°C, recorded at intervals of 10 min over a period of 2 h and 20 min, are shown in Figure 11. Absorbance data, $A(Z,t)$, at 360 nm as a function of time were used to plot the graph of $\ln[\sqrt{t}A(Z,t)]$ versus $[1/t]$, as shown in Figure 12, from which the value of D was found to be $(1.31 \pm 0.02) \times 10^{-5}$ cm^2/s.

From our experimental results it may be noted that shapes of absorption peaks displayed in Figures 6 and 11 for POPOP dye in PS and PMMA solutions, respectively, are slightly skewed when compared to coumarin 515 and exalite 416 dyes. This can be attributed to energy transfer mechanisms occurring in organic scintillators; i.e., the final step of energy transfer in organic liquid scintillators from PS or PMMA solutions in 1,4-dioxane to the unexcited fluorescent solute may take place in two different ways, one involving long-range dipole–dipole interactions and the other involving short-range collisions[16] when the excited polymer molecule and the unexcited fluorescent dye molecule come sufficiently close to each other. Observed anomalous variations in the spectra of POPOP in PS and PMMA solutions (Figures 6 and 11) indicate that there is more possibility of involvement of long-range dipole–dipole interactions between POPOP in PMMA than POPOP in PS solutions because PS is less polar than PMMA.

To test the validity of the present method, we measured the diffusion coefficient of two organic liquids viz., aniline and benzene, for which the accepted val-
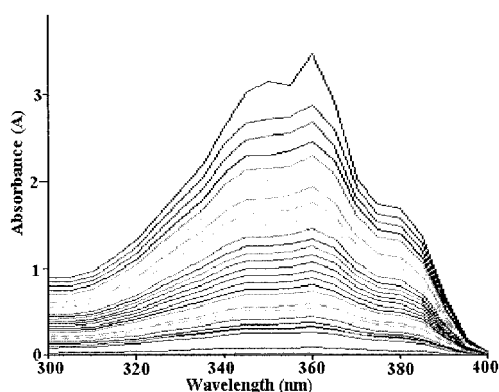
Figure 10 Variation of $\ln[t^{1/2} A(t)]$ with $1/t$ for exalite 416 dye diffusing in PS solution of concentration 10 g/dL at different time intervals, t, after commencement of diffusion at 25°C.

ues of D obtained by other standard methods are available. Replacing MP with aniline and MN with benzene, we studied the diffusion of aniline in benzene. Because aniline is denser than benzene, a clear interface was observed. However, in this case, the geometry of Figure 1 is reversed, i.e., the column of aniline is below that of benzene so that aniline diffuses in the upward direction. As before, heights of the columns of aniline and benzene are 10 and 30 mm, respectively, but $Z = 10$ mm is the distance above the interface between two liquids. From a plot similar to Figure 3 (not drawn to avoid overcrowding), we have obtained the D value of $(1.93 \pm 0.02) \times 10^{-5}$ cm^2/s for aniline in benzene at 25°C, while its accepted literature

value[17] is 1.96×10^{-5} cm^2/s. Such a close agreement is clear proof of the fact that the present method is accurate.

CONCLUSION

In this study, inert solvents were chosen with scintillating dye solutes so that one can study the energy transfer from polymers dissolved in inert solvents like 1,4-dioxane. It is concluded that solute, being a scintillator, might have aided in energy transfer from the polymer solution to the scintillating dye. The method used is relatively simple and rapid for the measurement of diffusion coefficient of dyes in polymer solutions or liquids when compared to rather elaborate conventional methods. The method is free from errors associated with the manually recording spectroscopic methods since here all absorption spectra were recorded using a fully automated instrument at the desired time intervals. The initial formation of a sharp boundary between columns MN and MP is essential, and this was achieved without any appreciable turbulent mixing by gently applying the lighter solution of the dye over the heavier polymer solution using a capillary tube. Moreover, the diffusion cell is kept at a constant temperature and is free from mechanical disturbances. Finite vertical dimensions of the incident beam introduce an error in experimental diffusion coefficients, which was reduced to about 2% in the present experiment by restricting the vertical width of the light beam to 0.5 mm by the slits on either sides of

Figure 11 Absorption spectra of POPOP dye diffusing in PMMA polymer solution of concentration 1 g/dL at different time intervals after commencement of diffusion at 25°C.
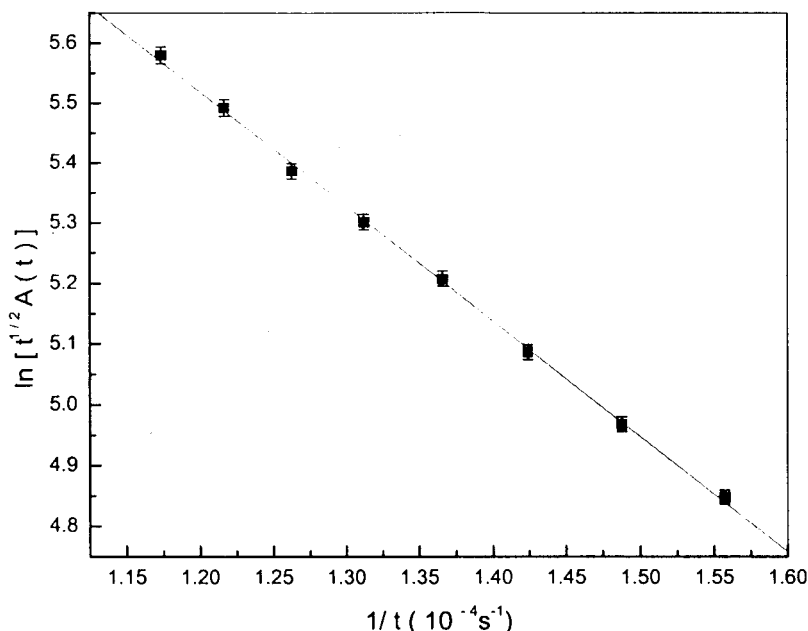
Figure 12 Variation of $\ln[t^{1/2} A(t)]$ with $1/t$ for POPOP dye diffusing in PMMA solution of concentration 1 g/dL at different time intervals, t, after commencement of diffusion at 25°C.

the cell. The method might also be useful for investigating the migration of dyes or plasticizers in polymer solutions, with applications in polymer processing industries as well as in scintillation studies in medical imaging.

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