Studies on Molecular Transport of *n*-Alkanes Through Poly(tetrafluoroethylene-*co*-propylene) Elastomeric Membrane

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ABSTRACT: Molecular transport of a series of *n*-alkanes through commercial TFE elastomer (FA 150L) has been studied in the temperature range 30–50 °C using sorption-gravimetric method. The Fickian diffusion equation was used to calculate the diffusion coefficients, which were dependent on the size of the alkanes and temperature. The diffusion coefficients at 30°C varied from 4.53×10^{-8} cm²/s (*n*-heptane) to 0.18×10^{-8} cm²/s (*n*-hexadecane). The liquid concentration profiles have also been computed using analytical solution of Fick's equation with the appropriate initial and boundary conditions and these were presented as a function of penetration depth of molecular migration and time of immersion. These results have been discussed in terms of molecular size of alkanes as well as temperature. In all the liquid penetrants, the transport phenomenon was found to follow the anomalous behavior. From the temperature dependence of diffusion and permeation coefficients, the Arrhenius activation parameters have been estimated. These parameters do not exhibit any systematic variation with the size of the penetrants. The resulting low diffusion coefficients, contribute to the superior barrier performance of the membrane, is due, in part, to the high glass transition temperature of Aflas™ TFE elastomer. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 101: 2228–2235, 2006

Key words: diffusion; barrier; swelling; activation energy

INTRODUCTION

The diffusion of small molecular liquids into polymers is a subject of intense study. This type of diffusion process plays an important role in several important areas of engineering and industry.[1,2] Membrane separation of liquids in the liquid and oil industry has become as wide spread as more traditional methods are based on absorption, pressure-swing adsorption, or cryogenics. The membrane process has certain benefits compared to the cryogenics process, for example lower investment cost and easier operation. Very important applications in the food industry involve impending the diffusion of liquids through thin polymer films, commonly used as packing food wrappers or protective coatings.[3,4] More applications are anticipated in the burgeoning field of biotechnology[5,6] such as biosensors, controlled release, and bioreactors. Moreover, the effects of interaction between rubbery polymers and small molecules are of practical interest to chemical engineers, because of the inherent sorption and transport of liquid penetrants present in most processes they encounter.

An extensive research has taken place in the field of diffusion and permeation of gases, vapors, and liquids through polymers.[7–10] Several reports are available on rubber–solvent interaction.[11–15] Recently in our laboratory, Kariduraganavar et al.[16] and Kulkarni et al.[17] investigated the sorption and diffusion behavior of esters, aldehydes, ketones, and aromatic liquids through fluoroelastomeric membranes and found that transport behavior is not merely on the size of the penetrants but also on the nature of liquid molecules and membrane. These studies are extremely important for the design of new polymer materials, which would greatly benefit for the development of high performance membranes.

In continuation of our ongoing program in this area,[16–19] we report here the experimental results of sorption kinetics, diffusion, and permeation of *n*-alkanes through Aflas™ TFE elastomer (FA 150L) at different temperatures. The purpose of the study was to examine the effects of size of the penetrants on the sorption and diffusion behavior of elastomer. Thus, the liquid penetrants were chosen of varying molecular size and keeping in view of their wide diversity and relevance in chemical engineering and other related areas. The concentration-independent diffusion

coefficients have been calculated from the Fick's diffusion equation. In addition, the analytical solution of Fick's equation has been used to calculate the liquid concentration profiles at different times and depth of liquids inside the membrane material. The temperature-dependent transport coefficients have been used to estimate the Arrhenius activation energies.

EXPERIMENTAL

Reagents and materials

The organic penetrants such as *n*-nonane, *n*-decane, *n*-dodecane, *n*-tetradecane, and *n*-hexadecane were obtained from s. d. fine Chemicals, Mumbai, India. *n*-Heptane, *n*-pentadecane, and *n*-octane were purchased from Riedel, Germany. All the chemicals are of analytical grade and were used without further purification.

Aflas TFE elastomer (FA 150L) is a low viscosity grade copolymer of tetrafluoroethylene/propylene, typically designed for chemical tank and wall-lining applications, or as a viscosity modifier in blends with other Aflas TFE elastomer grades. Generally, it is not used for molding applications, because of its relatively poor mechanical property. One part of sodium stearate was added to aid in mill release and cure rheology. The typical properties of the fluoroelastomer are as following: specific gravity, 1.55; color, dark brown; solubility, Freon™ TFE or tetrahydrofuran; and mooney viscosity, ~35 MLI + 10 at 100°C. The press-cured sample for 10 min at 177 °C possesses the following mechanical properties: tensile strength, 1610 psi; elongation at break, 350%; and hardness (shore A), 70. The sample was compounded with standard fillers and ingredients utilized in typical TFE elastomer formulations.

Sorption-gravimetric experiments

The circularly cut disc-shaped membrane samples of ≈2 cm in diameter were dried in a vacuum desiccator for 45 h over anhydrous $CaCl_2$ before performing the sorption experiments. These samples were then immersed in about 15–20 mL of liquids taken in airtight bottles and were maintained at 30, 40, and 50°C in an electronically controlled oven (WTB Binder, Germany) within an accuracy of ±0.5°C. Mass measurements were made by removing the samples at definite time intervals after wiping the surface-adhered solvent using smooth tissue paper. These samples were then immediately weighed on a top loading digital Mettler balance (Model AE 240, Switzerland) with an accuracy of ±0.01 mg. The samples were taken out no longer than 30–40 s outside the temperature-controlled oven. The procedure was continued until no more liquid uptake by the polymer was observed (equilibrium

sorption). Three independent readings were taken, and the average value was used in all the calculations.

Sorption coefficients were expressed as mol % and are calculated using the equation:

$$M_t = \left(\frac{W_t - W_0}{W_0} \right) \times \frac{100}{M} \tag{1}$$

where W_0 is the initial mass of the sample; W_t is the mass at time t, that is the immersion period; and M is the molar mass of the liquid.

Diffusion coefficients and concentration profiles

Diffusive migration of liquids depends on the concentration gradient and temperature, together with the type of polymer barrier and the nature of migrating liquids. The dynamic swelling properties of a polymer film include the solvent sorption rate, the rate of approach to equilibrium swelling, the solvent front velocity, and the transport mechanism controlling solvent sorption. For the Fickian transport, the rate of equilibrium approach can be characterized by the diffusion coefficient. For a plane geometry of the polymer sheet, the diffusion coefficient D can be calculated from:[20]

$$\frac{M_t}{M_\infty} = 1 - \sum_{n=0}^{\infty} \left[\frac{8}{(2n + 1)^2 \pi^2} \right] \exp \left[-(2n + 1)^2 \pi^2 \left(\frac{Dt^2}{h^2} \right) \right] \tag{2}$$

Here M_t and M_∞ are the cumulative masses sorbed from the polymer sample at time t and ∞, respectively, and h is the initial thickness of the polymer sample. Although this equation can be solved readily, it is suitable to consider a short-time limiting expression as well:[20]

$$\frac{M_t}{M_\infty} = \left[\frac{4}{\pi^{1/2}} \right] \left[\frac{Dt}{h^2} \right]^{1/2} \tag{3}$$

A single curve is obtained from the plot of M_t/M_∞ versus square root of time, which is linear initially, and thus D can be calculated from the rearrangement of eq. (3) as:[21–23]

$$D = \pi \left(\frac{h\theta}{4} \right)^2 \tag{4}$$

where θ is the slope of initial linear portion of sorption curves. Similarly, the permeability coefficients can be calculated using the relation: $P = D \times S$, which follows generally the same pattern as those of diffusivities. This simple relation holds for the permeation

TABLE I
Physical Properties and Sorption Coefficients of *n*-Alkanes at Different Temperatures through TFE Elastomeric Membrane

Liquids	Molar volume (cm^3/mol)	Sorption coefficients (M_∞) (mol %)		
		30°C	40°C	50°C
n-Heptane	147.5	0.176 ± 0.002	0.149 ± 0.002	0.100 ± 0.004
n-Octane	163.5	0.169 ± 0.002	0.136 ± 0.002	0.079 ± 0.005
n-Nonane	179.7	0.107 ± 0.003	0.095 ± 0.002	0.052 ± 0.002
n-Decane	195.9	0.077 ± 0.002	0.058 ± 0.002	0.048 ± 0.004
n-Dodecane	228.6	0.063 ± 0.002	0.056 ± 0.002	0.042 ± 0.004
n-Tetradecane	260.1	0.045 ± 0.003	0.042 ± 0.003	0.029 ± 0.003
n-Pentadecane	276.5	0.035 ± 0.003	0.030 ± 0.002	0.022 ± 0.004
n-Hexadecane	292.8	0.019 ± 0.003	0.015 ± 0.003	0.012 ± 0.005

process when D obeys the Fick's diffusion law and S obeys the Henry's law.[24] For the penetrant-polymer systems used in this study, it is not certain to what degree one or both laws are obeyed. Thus, the P values are considered as estimates of the permeability coefficients.

Liquid ingression into a polymeric material is a phenomenon of great technological importance. In many instances, it is necessary to know the penetration depth of liquids into polymer. In most application areas, the liquid penetration rates are calculated in terms of liquid concentration profiles. These are extremely useful to predict the self-life of the polymer while in contact with the liquids.

Liquid concentration profiles can be calculated from eq. (5) under suitable initial and boundary conditions to yield an equation for solvent uptake $C_{(x,t)}$ inside the membrane thickness h, at time t and distance x as:[25,26]

$$\frac{C_{(x,t)}}{C_\infty} = 1 - \frac{4}{\pi} \sum_{m=0}^{\infty} \frac{1}{(2m+1)} \exp$$

$$\left[-\frac{D(2m+1)^2 \pi^2 t}{h^2} \right] \sin\left[\frac{(2m+1)\pi x}{h} \right] \quad (5)$$

where m is an integer. These data are useful to study the liquid migration as a function of time and penetration depth of liquids from the face to the middle of the elastomer along the thickness direction.

RESULTS AND DISCUSSION

Sorption kinetics

Sorption coefficient is a thermodynamic parameter, which depends on the strength of the interaction in polymer/penetrant mixture and describes the initial penetration and dispersal of permeant molecules into the polymer matrix. The dynamic sorption data generated at three different temperatures are presented in
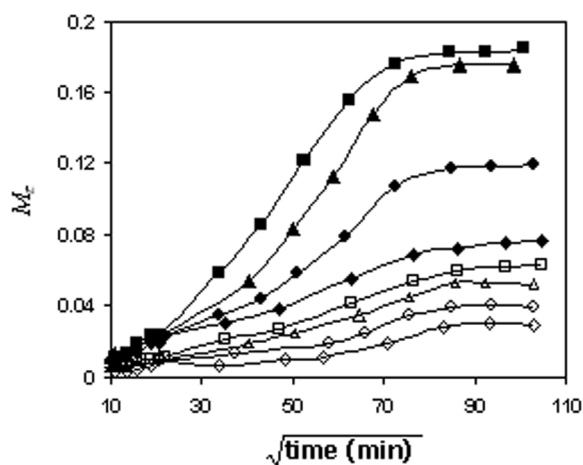

Figure 1 Plots of M_t vs. square root of time for TFE elastomer at 30°C with (■) *n*-heptane, (▲) *n*-octane, (•) *n*-nonane, (♦) *n*-decane, (□) *n*-dodecane, (△) *n*-tetradecane, (○) *n*-pentadecane, and (◇) *n*-hexadecane.
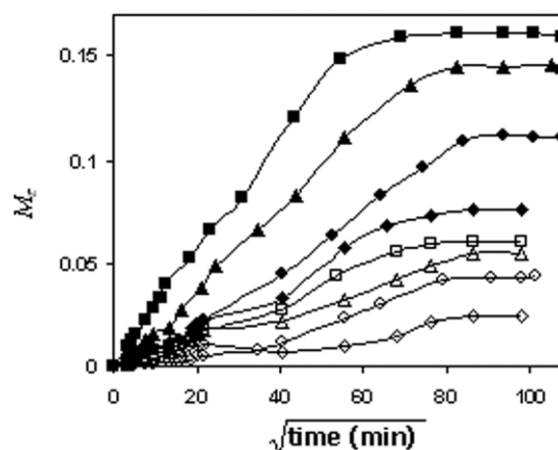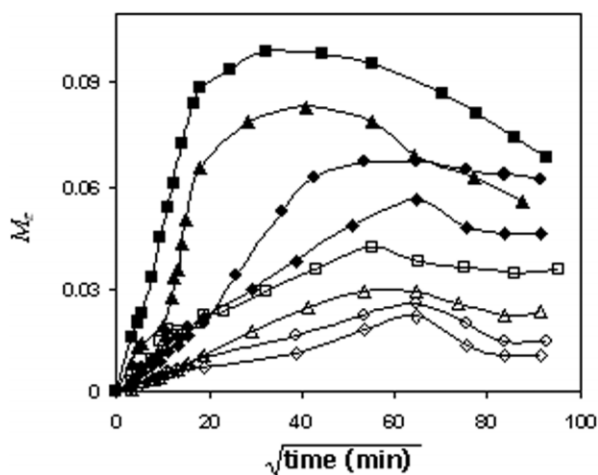

Figure 2 Plots of M_t vs. square root of time for TFE elastomer at 40°C with (■) *n*-heptane, (▲) *n*-octane, (•) *n*-nonane, (♦) *n*-decane, (□) *n*-dodecane, (△) *n*-tetradecane, (○) *n*-pentadecane, and (◇) *n*-hexadecane.

Figure 3 Plots of M_t vs. square root of time for TFE elastomer at 50°C with (■) n-heptane, (▲) n-octane, (•) n-nonane, (◆) n-decane, (□) n-dodecane, (△) n-tetradecane, (○) n-pentadecane, and (◇) n-hexadecane.

On comparison of Figures 1–3, it is clearly noticed that the overall sorption curves decreased in all the cases with increasing temperature. This is in contrast to the general tendency, wherein sorption increases with increasing temperature due to increased segmental motion of the polymer chains, which creates extra free-volume within the polymer matrix. This unusual tendency may be attributed to higher volatility of liquid penetrants, which leads to significant evaporation with increasing temperature. This tendency is generally expected due to increased thermal energy at higher temperature provided the interaction exists between the membrane and polar liquid penetrants. However, this can be ruled out in the present systems, since the liquids used are basically nonpolar in nature. Further, it is heartening to note that the equilibrium sorption curves decreased remarkably after reaching a maxima particularly at 50°C for all the liquids (see Fig. 3). However, the effect is more prominent particularly in case of n-heptane and n-octane. Such decrease in sorption may be interpreted either as (i) solvent loss due to desorption/evaporation, or as (ii) mass loss due to membrane leaching effects. But in the present case, the former appears to be more likely because both the liquids are low boiling as compared to other n-alkanes. Similar observations were noticed by Aminabhavi and Naik[27] and in our earlier work.[28]

Table I, while the sorption plots of n-alkanes measured at 30, 40, and 50°C are displayed in Figures 1–3, respectively. From the plots, it is observed that sorption attains equilibrium more quickly for smaller liquid penetrants as compared with bigger penetrants. Further sorption decreases systematically as expected with increasing the size of the penetrats and in accordance, the sorption coefficients decreased with increasing the size of the liquid molecules as seen from Table I. For instance, n-heptane with a lower molar volume (147.5 cm³/mol) exhibits the highest equilibrium sorption; on the contrary, n-hexadecane with a higher molar volume (292.8 cm³/mol) exhibits the lowest equilibrium sorption among the liquids studied. This is indicative of slow down molecular transport with increasing the penetrant size, giving the slight sigmoidal shape of the sorption curves as clearly seen from Figures 1–3. A more sigmoidal behavior was observed for larger molecules, viz., C_{10} to C_{16} alkanes as compared to smaller molecules (C_7 to C_9).

Diffusion coefficients and concentration profiles

Diffusivity is a kinetic parameter, which depends on the polymer segmental mobility. The estimated values of diffusion coefficients (D) at different temperatures are given in Table II. It is observed that the values of D decreased from n-heptane to n-hexadecane as the size of the penetrant increased at all temperatures, signifying the dependence of D on the molecular size of penetrant. On the other hand, D values increased significantly with increasing temperature. This is due to the expansion of free volume within the polymer matrix. The largest D values of 4.53×10^{-8} cm²/s is

TABLE II
Diffusion (D) and Permeation (P) Coefficients of n-Alkanes at Different Temperatures through TFE Elastomeric Membrane

Liquids	D (10^8) cm²/s			P (10^9) cm²/s		
	30°C	40°C	50°C	30°C	40°C	50°C
n-Heptane	4.53 ± 0.01	6.69 ± 0.02	7.36 ± 0.04	7.97 ± 0.08	10.0 ± 0.1	7.4 ± 0.3
n-Octane	3.37 ± 0.01	4.33 ± 0.02	6.28 ± 0.05	5.70 ± 0.07	5.89 ± 0.09	5.0 ± 0.3
n-Nonane	2.69 ± 0.02	3.93 ± 0.02	5.42 ± 0.05	2.88 ± 0.08	3.73 ± 0.07	2.8 ± 0.1
n-Decane	1.00 ± 0.02	2.02 ± 0.02	4.10 ± 0.05	0.77 ± 0.02	1.17 ± 0.04	2.0 ± 0.1
n-Dodecane	0.66 ± 0.02	0.75 ± 0.02	0.85 ± 0.05	0.42 ± 0.02	0.42 ± 0.02	0.36 ± 0.04
n-Tetradecane	0.54 ± 0.02	0.70 ± 0.03	0.82 ± 0.04	0.24 ± 0.02	0.29 ± 0.02	0.24 ± 0.02
n-Pentadecane	0.32 ± 0.03	0.52 ± 0.03	0.74 ± 0.04	0.11 ± 0.01	0.16 ± 0.01	0.16 ± 0.03
n-Hexadecane	0.18 ± 0.03	0.47 ± 0.03	0.70 ± 0.04	0.03 ± 0.01	0.07 ± 0.01	0.08 ± 0.03

observed for n-heptane at 30°C, whereas the lowest of 0.18×10^{-8} cm^2/s is observed for n-hexadecane among the alkanes studied. Permeation is a collective process of diffusion and sorption, and consequently the permeability of liquid molecules into polymer membrane depends upon both diffusivity and sorptivity. The calculated permeability coefficients (P) are also included in Table II. It is observed that permeation coefficient values are also found to decrease with increasing penetrant size as those of diffusivity coefficients in the investigated temperature range.

Concentration profiles have been calculated from eq. (5) with the procedure suggested earlier.[25,29–31] These profiles have been calculated at different membrane thickness, i.e. penetration depth of liquids and at different time intervals during the sorption experiments. A few representative plots are displayed in Figures 4–6. In all the cases, a noticeable effect of the temperature was prevalent on the shapes of the concentration profiles. For instance, the concentration profiles of all the liquids exhibited higher value at 50°C than those at 30 and 40°C. This indicates the clear-cut dependence of D values on the temperature.

The diffusion coefficients of n-heptane is higher than those of other liquids and hence, its concentration profiles are higher at all temperatures (see Fig. 4). In case of hexadecane, diffusivity is quite low and so are its concentration profiles (see Fig. 6). However, n-decane exhibits intermediate diffusivity values among the alkanes studied and in accordance, its concentration profiles fall in between n-heptane and n-hexadecane. The same trend is also observed for other liquids, but these plots are not displayed to reduce the number of plots. However, the observed diffusion coefficients and concentration profiles of Aflas TFE membrane appear to be small in comparison with Fluorel membranes.[16,17] The high glass transition temperature of Aflas TFE membrane (-7°C) compared with Fluorel membranes (-21°C) may account for the difference.[32,33]

Transport mechanism

The results of the sorption data were analyzed to know the type of diffusion mechanism using the empirical relation:[34–36]

$$\log\frac{M_t}{M_\infty} = \log K + n\log t \qquad (6)$$

where M_t and M_∞ are the mol % sorption at t and t_∞, respectively. The parameters n and K have been estimated by the method of least squares at 95% confidence limit. These data are presented in Table III. The value of K depends upon structural features of polymer in addition to its interaction with the solvent, and n indicates the mechanism of sorption. According to
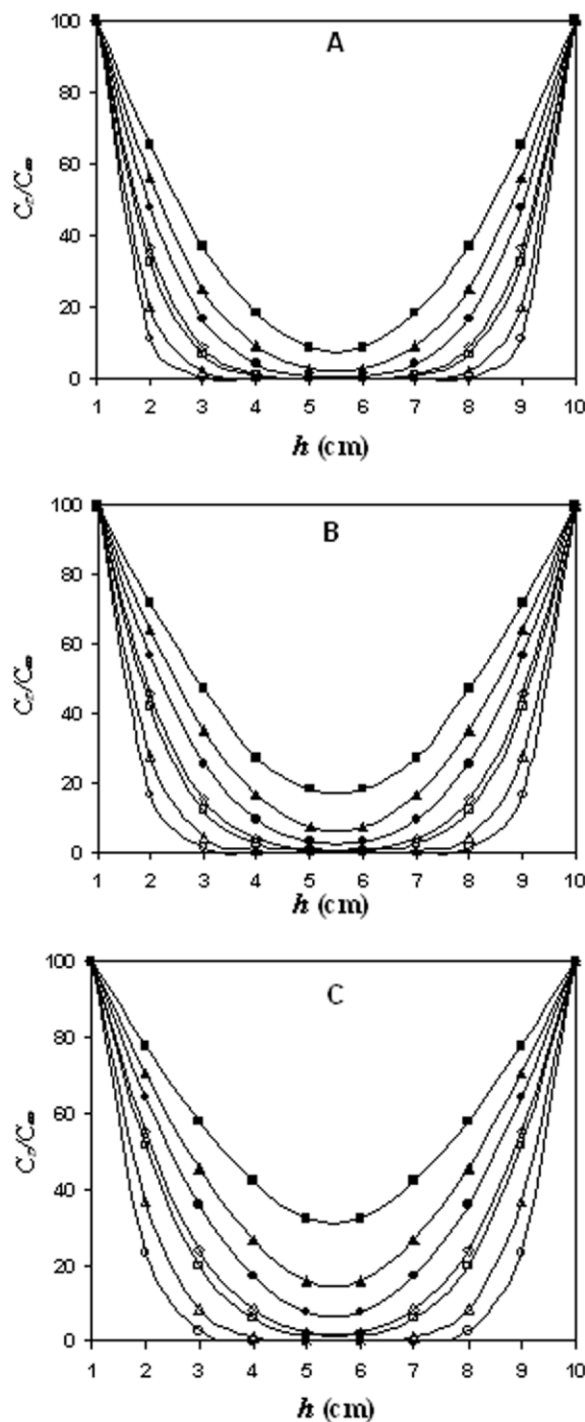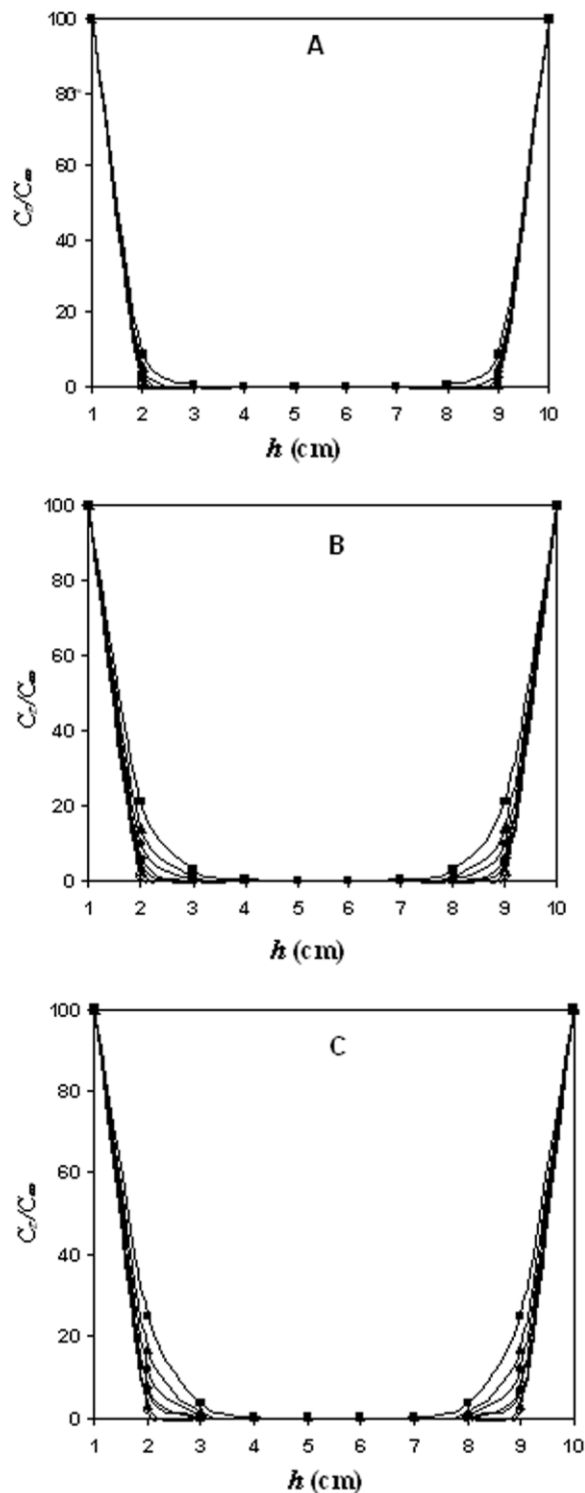

Figure 4 Concentration profiles calculated from eq. (5) for n-heptane through TFE elastomer at (A) 30°C, (B) 40°C, and (C) 50°C for (o) 25 min, (△) 50 min, (□) 100 min, (◇) 120 min, (•) 200 min, (▲) 300 min, and (■) 500 min.

Alfrey et al.,[37] liquid transport into polymer material follows the Fickian mechanism when the solvent concentration is low and swelling is not so important. At higher concentrations, swelling becomes important and relieves mechanical stresses so that transport deviates from the regular Fickian mechanism and is

referred as non-Fickian or Case II transport. When the Case I transport mechanism dominates the process, the amount of the liquid sorbed is proportional to the square root of the sample immersion time, i.e. $t^{1/2}$. If both the mechanisms are operative, then the time ex-
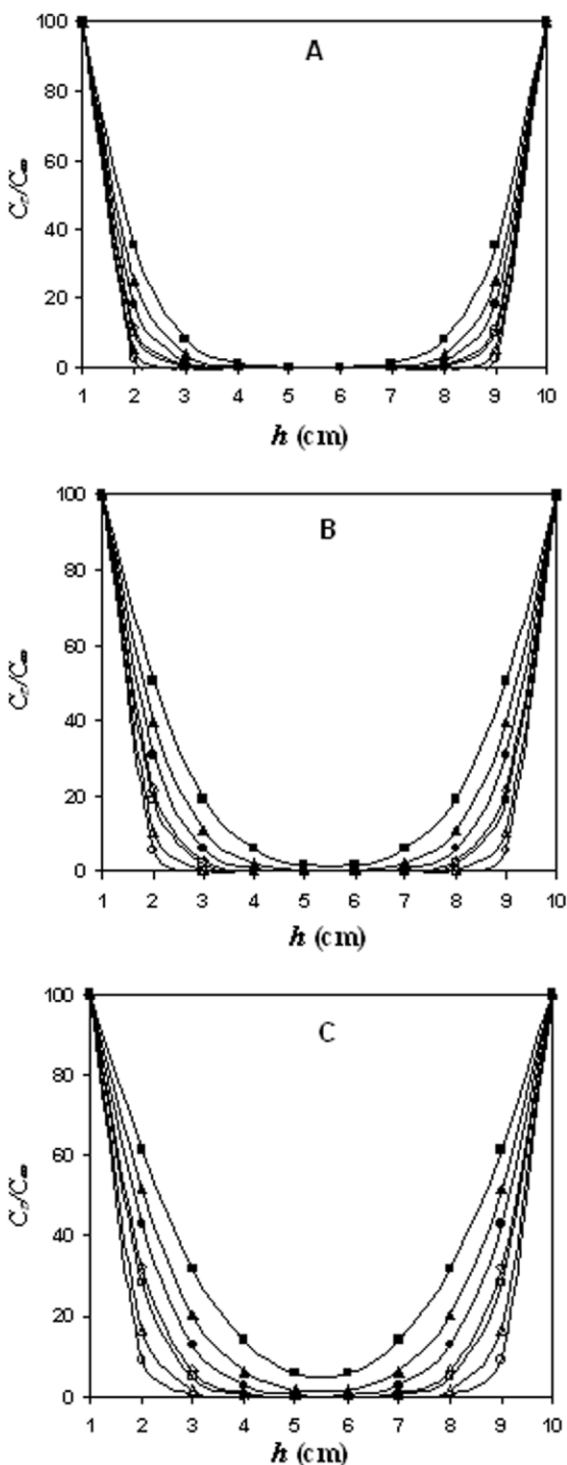
Figure 5 Concentration profiles calculated from eq. (5) for *n*-decane through TFE elastomer at (A) 30°C, (B) 40°C, and (C) 50°C for (o) 25 min, (△) 50 min, (□) 100 min, (◇) 120 min, (•) 200 min, (▲) 300 min, and (■) 500 min.

Figure 6 Concentration profiles calculated from eq. (5) for *n*-hexadecane through TFE elastomer at (A) 30°C, (B) 40°C, and (C) 50°C for (o) 25 min, (△) 50 min, (□) 100 min, (◇) 120 min, (•) 200 min, (▲) 300 min, and (■) 500 min.

ponent varies between 0.5 and 1, and the mechanism of sorption follows an anomalous trend. In the present polymer–liquid systems, the values of *n* vary from 0.50 to 0.59, confirming the transport mechanism to be

TABLE III
Estimated Parameters for *n*-Alkanes Calculated from Eq. (6) at Different Temperatures for TFE Elastomeric Membrane

	n			*K*		
Liquids	30°C	40°C	50°C	30°C	40°C	50°C
n-Heptane	0.58	0.60	0.50	1.58	2.61	3.27
n-Octane	0.59	0.57	0.55	2.21	3.16	4.87
n-Nonane	0.58	0.58	0.51	1.34	2.94	3.42
n-Decane	0.60	0.59	0.55	1.06	2.02	2.64
n-Dodecane	0.50	0.56	0.58	0.91	1.81	2.02
n-Tetradecane	0.58	0.61	0.52	0.87	1.64	2.26
n-Pentadecane	0.50	0.57	0.60	0.51	1.07	2.31
n-Hexadecane	0.52	0.54	0.56	0.31	0.96	1.56

of an anomalous type. Such observations were also made earlier in the literature.[16–18] It may further be noted that *n* does not depend on temperature, whereas the values of *K* depend systematically on temperature, as they tend to increase with a rise in temperature. This suggests an increase in polymer–liquid interactions with temperature. Although the observed interaction is not contributed by the membrane and nonpolar liquids in real sense, it may be attributed to the increased of free-volume within the polymer matrix upon increasing temperature.

Activation parameters

Temperature plays a very important role in the diffusion of penetrants through polymeric membranes. As observed elsewhere,[7] both the diffusion and permeation coefficients are found to increase with increasing temperature. This is mainly attributed to the creation of additional free volume because of increased segmental motion of the polymer chain upon increasing temperature. This effect prompted us to calculate the Arrhenius activation energy for diffusion (E_D) and permeation (E_p) according to the relation:[23]

$$X = X_0 \exp\left(\frac{-E_x}{RT}\right) \tag{7}$$

where X represents D or P; X_0 is a constant representing D_0 or P_0; and E_x represents E_D or E_p, depending upon the transport process under consideration. RT is the usual energy term. Figures 7 and 8 display the Arrhenius plots for all the liquids. The activation energies for diffusivity and permeability were estimated from the least square fitting of the linear plots of log X versus $1/T$ and the values so obtained are presented in Table IV. It is noticed that E_D and E_p values do not show any regular trend with the size of the liquid penetrants. The E_D values range from 10.29 to 57.44 kJ/mol and E_p values range from 0.63 to 129.57 kJ/mol. Using the results of E_D and E_p, we have calculated the heat of sorption as $\Delta H_S = E_P - E_D$. The resulting values are included in Table IV. The ΔH_S gives the additional information about the transport of molecules through the polymer matrix. The ΔH_S is a composite parameter involving contributions from
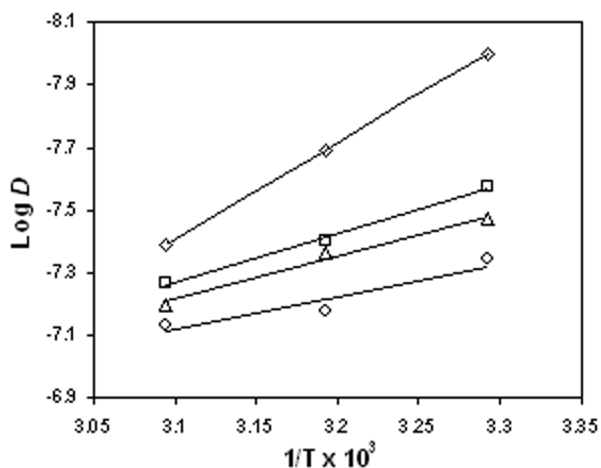

Figure 7   Arrhenius plots of log *D* vs. 1/*T* for TFE elastomer for (○) *n*-heptane, (△) *n*octane, (□) *n*-nonane, and (◇) *n*-decane.
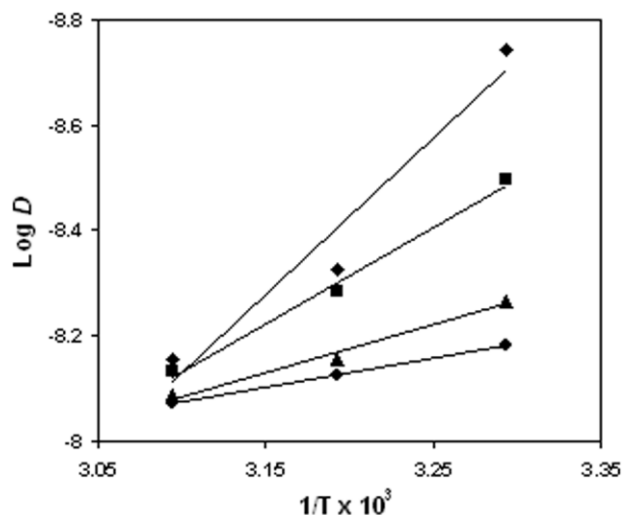
Figure 8   Arrhenius plots of log *D* vs. 1/*T* for TFE elastomer for (●) *n*-dodecane, (▲) *n*-tetradecane, (■) *n*-pentadecane, and (◆) *n*-hexadecane.

TABLE IV
Activation Energy for Diffusion, Permeation, and Heat of Sorption of n-Alkanes for TFE Elastomeric Membrane

Liquids	E_P (kJ/mol)	E_D (kJ/mol)	ΔH_S (kJ/mol)
n-Heptane	3.02	19.88	−16.86
n-Octane	5.52	25.28	−19.76
n-Nonane	0.63	28.54	−27.91
n-Decane	56.57	57.44	−0.87
n-Dodecane	6.13	10.29	−4.16
n-Tetradecane	0.70	17.05	−16.35
n-Pentadecane	15.34	34.19	−18.85
n-Hexadecane	129.57	55.53	74.04

Henry's and Langmuir's type of sorption. The results of the heat of sorption ΔH_S are positive in most of the cases, suggesting an endothermic contribution (Henry's mode of sorption), except for *n*-hexadecane, whose ΔH_S value is negative, suggesting an exothermic contribution (Langmuir's mode of sorption).

CONCLUSIONS

The transport of a series of *n*-alkanes through Aflas TFE elastomer (FA 150L) membrane has been studied using the sorption-gravimetric method. The sorption coefficients determined in the present study decreased with increasing the size of the penetrants. At 50°C the sorption decreased after reaching maxima, which is particularly prominent for *n*-heptane and *n*-octane. This is attributed to the loss of solvent because of evaporation at higher temperature. The *D* values increased systematically with increasing temperature because of the expansion of free-volume within the polymer matrix and show a systematic decrease with increasing the size of the penetrants. The *n*-heptane has the largest *D* value of 4.53×10^{-8} cm²/s, whereas *n*-hexadecane has the lowest *D* value of 0.18×10^{-8} cm²/s among the liquids studied at 30°C. The liquid concentration profiles showed dependence on diffusivity values and temperature. Both diffusivity coefficients and concentration profiles of the present Aflas TFE membrane are small as compared with Fluorel membranes, which contribute to the superior barrier performance of membrane in various situations. The permeability coefficients showed the same effect as those of diffusivity values. Molecular transport is found to follow the anomalous type of behavior in all the cases over the investigated temperature range. The E_D and E_p values range from 10.29 to 57.44 and 0.63 to 129.57 kJ/mol, respectively. The ΔH_S values are positive in most of the cases, suggesting the Henry's mode of sorption, except for *n*-hexadecane, suggesting the Langmuir's mode of sorption.

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