

# Molecular Transport of n-Alkanes into PU/PBMA Interpenetrating Polymer Network Systems

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**ABSTRACT:** The methylene diisocyanate (MDI) and toluene diisocyanate (TDI) based polyurethane/polybutyl methacrylate (PU/PBMA-50/50) interpenetrating polymer network (IPN) membranes have been prepared. The molecular migration of n-alkane penetrants such as hexane, heptane, octane, nonane, and decane through PU/PBMA (50/50) membranes has been studied at 25, 40, and 60°C using a weight gain method. From the sorption results, diffusion ( $D$ ) and permeation ( $P$ ) coefficients of n-alkane penetrants have been calculated. Molecular migration depends on membrane-solvent interactions, size of the penetrants, temperature, and availability of free volume within the membrane matrix. Attempts have been made to estimate the parame-

ters of an empirical equation and these data suggest that molecular transport follows Fickian mode. From a study of temperature dependence of transport parameters, activation energy for diffusion ( $E_D$ ) and permeation ( $E_P$ ) have been estimated from the Arrhenius relation. Furthermore, sorption results have been interpreted in terms of enthalpy ( $\Delta H$ ) and entropy ( $\Delta S$ ) of sorption. The liquid concentration profiles have been computed using Fick's equation with appropriate initial and boundary conditions. © 2003 Wiley Periodicals, Inc. *J Appl Polym Sci* 90: 739–746, 2003

**Key words:** concentration profile; molecular transport; n-alkanes; PU/PBMA

## INTRODUCTION

Molecular transport of organic liquids through polymer membranes has been the subject of investigations over the past several years.<sup>1–12</sup> Such studies are necessary due to the production of innumerable polymer membranes of commercial importance.<sup>13</sup> Solvent resistivity of these membranes is an important factor in such applications, including protective coating, separation of liquid mixtures, and chemical waste pond-linings. An understanding of membrane transport properties such as sorption, diffusion, and permeation of organic liquids is important before seeking their applications. Molecular transport phenomenon is influenced greatly by physical and chemical nature, morphology, crosslink density of polymers, as well as temperature, shape, and size of the probe penetrant molecules.

With increasing importance of polymer blends in engineering areas,<sup>14–16</sup> studies have been directed to develop interpenetrating polymer network (IPN) polymers. In IPNs, the transport behavior depends on

the composition of components and their chemical interaction, as well as morphology.<sup>14</sup> Hence, IPNs have been used<sup>17</sup> to understand the structure-property relationships between polymer membranes and penetrant molecules. In this article, we present the experimental data on sorption and diffusion of aliphatic hydrocarbons through castor oil-based polyurethane/polybutyl methacrylate (PU/PBMA) IPNs. A computer simulation technique was employed to estimate the concentration profiles of liquids at different temperatures. Temperature dependant data, activation parameters, and concentration profiles have been discussed in terms of membrane-solvent interactions.

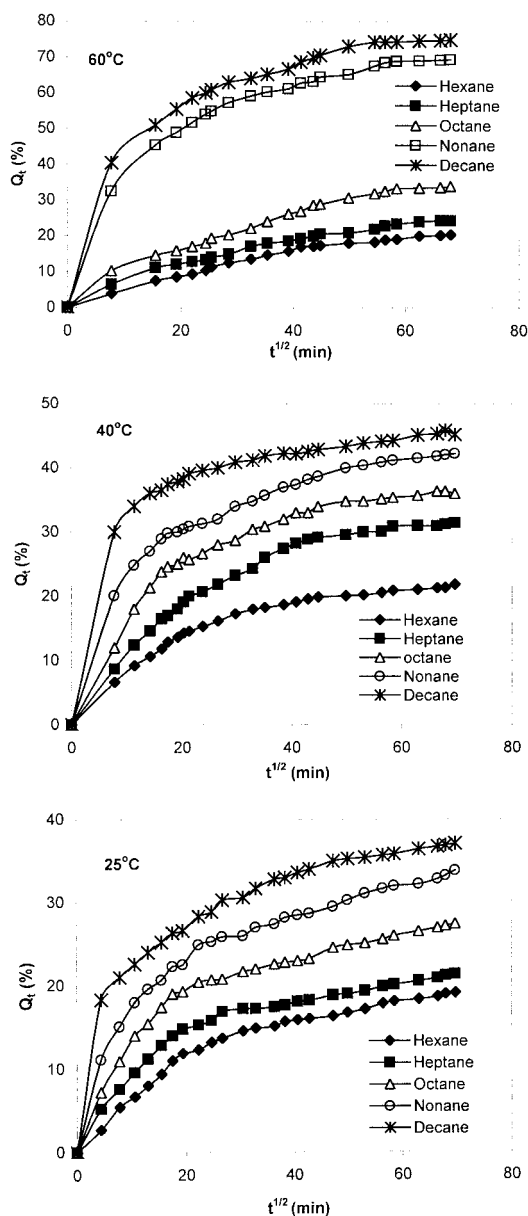
## EXPERIMENTAL

### Specimen preparation

The castor oil based PU/PBMA IPNs with methylene diisocyanate (MDI) and toluene diisocyanate (TDI) have been prepared as per the procedure reported elsewhere.<sup>18</sup> The obtained IPNs were tough and transparent films. Tensile strength and percentage elongation at break for TDI and MDI are, respectively, 3.0, 2.5 MPa and 50.0, 71.2 MPa. The prepared IPN sheets were cut into uniform circular pieces by specially designed sharp edge die (15 mm diameter). The cut membranes were kept in a vacuum oven at 25°C for 48 h before starting sorption experiments. Initial thickness of the specimens were measured at several points

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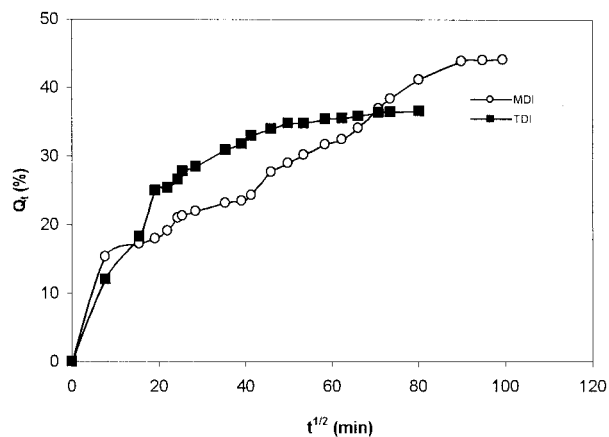


**Figure 1** Percentage mass uptake for TDI-based PU/PBMA IPN at different temperatures.

and ranged from 0.089 to 0.150 cm (Mitutoyo, Japan with a precision of  $\pm 0.001$  cm). Dry weights of PU/PBMA membranes were taken before immersion into penetrants. The penetrants used in this research are n-alkanes (n-hexane, n-heptane, n-octane, n-nonane, and n-decane). These were of analytical grade solvents supplied by S.D.F. Chemicals, Boisar, India. These are used after distillation.

### Sorption experiments

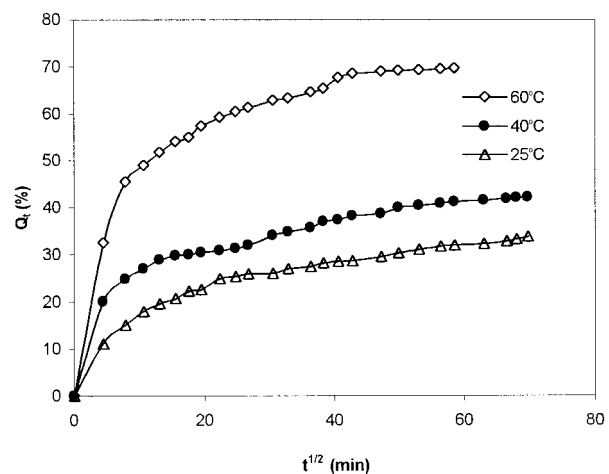
Sorption experiments at 25, 40, and 60°C were performed by immersing the cut specimens in test solvents taken in screw-tight test bottles maintained at the desired constant temperature ( $\pm 0.5^\circ\text{C}$ ) in a thermostatically controlled oven (M/s Tempo Instru-



**Figure 2** Percentage mass uptake for TDI- and MDI-based PU/PBMA IPNs for octane at 40°C

ments, Mumbai, India). At specified intervals of time, PU/PBMA membranes were removed from airtight container, surfaces were dried in between smooth filter paper wraps and were weighed immediately to the nearest  $\pm 0.1$  mg by placing them on a watch glass with a cover, using analytical balance (Mettler Toledo, Switzerland). The samples were then placed back immediately to the test liquid and transferred to the temperature-controlled oven. The total time spent by the membrane outside the solvent was kept within 30–40 s so as to minimize the experimental error due to evaporation losses. This error was negligible considering the small amount of time spent by the membrane outside the solvent. The procedure was repeated until maximum (saturation) was reached. The time taken for the attainment of equilibrium sorption for different liquids varied from 70 to 90 h. The percentage weight gain  $Q(t)$  of the soaked membrane was calculated as:

$$Q(t) = [(M_t - M_i) / M_i] \times 100 \quad (1)$$



**Figure 3** Percentage mass uptake for TDI-based PU/PBMA IPN for nonane at different temperatures.

**TABLE I**  
**Sorption Data for PU/PBMA IPN + *n*-Alkane Systems<sup>a</sup>**

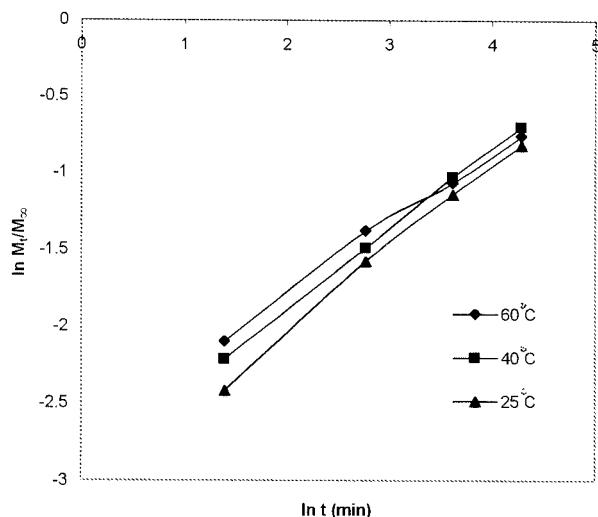
Penetrants	Temp. (°C)	$n \pm 0.009$		$K \times 10^2 \pm 0.04$ (g/g min <sup>n</sup> )	
		IPN1	IPN2	IPN1	IPN2
Hexane	25	0.577	0.538	4.50	4.08
	40	0.500	0.520	9.07	7.07
	60	0.571	0.518	5.50	6.08
Heptane	25	0.550	0.555	4.50	4.98
	40	0.550	0.480	5.50	7.43
	60	0.524	0.455	6.08	7.43
Octane	25	0.500	0.461	4.08	4.50
	40	0.461	0.476	4.48	6.08
	60	0.444	0.480	4.50	5.50
Nonane	25	0.538	0.480	3.69	4.50
	40	0.476	0.520	3.69	3.34
	60	0.451	0.524	4.08	3.02
Decane	25	0.600	0.524	2.02	2.47
	40	0.545	0.550	3.34	2.02
	60	0.452	0.500	3.02	2.24

<sup>a</sup> IPN1, TDI based PU/PBMA; IPN2, MDI based PU/PBMA.

where,  $M_i$  is initial weight of the membrane and  $M_t$  is the weight at time,  $t$ .

## RESULTS AND DISCUSSION

Sorption results of *n*-alkanes at 25, 40, and 60°C for the TDI-based PU/PBMA IPN membranes are presented in Figure 1. At all the temperatures, transport follows a systematic trend with the size of *n*-alkanes. For instance, sorption values for *n*-hexane are much smaller than those observed for *n*-decane. Sorption behavior at 25 and 40°C is almost complete, but at 60°C sorption tendencies for nonane and decane are widely different than those observed for lower *n*-alkanes viz., hexane, heptane, and octane. It is observed that sorption values of all the alkanes increase system-



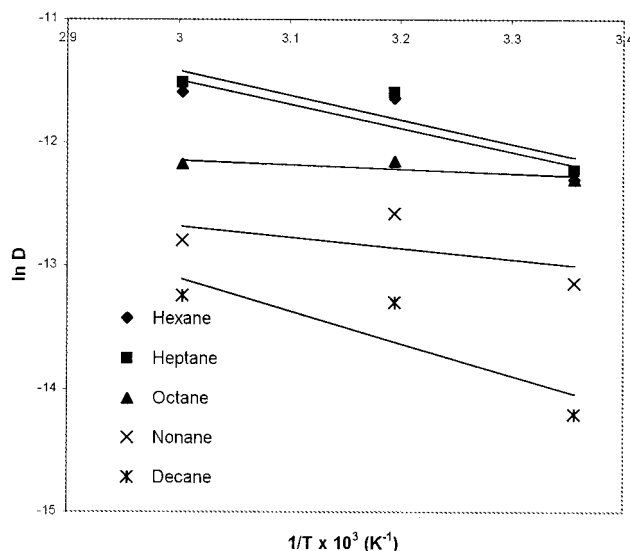
**Figure 4**  $\ln M_t/M_\infty$  versus  $\ln t$  for TDI-based PU/PBMA IPN for nonane at different temperatures.

**TABLE II**  
**Sorption (*S*) and Diffusion (*D*) Coefficients of PU/PBMA IPN + *n*-Alkane Systems<sup>a</sup>**

Penetrants	Temp. (°C)	$S \times 10^2$ (mol %)		$D \times 10^7 \pm 0.04$ (cm <sup>2</sup> /s)	
		IPN1	IPN2	IPN1	IPN2
Hexane	25	37.11	38.72	71.84	45.41
	40	45.20	57.11	79.41	87.78
	60	75.12	61.28	98.24	92.23
Heptane	25	33.88	34.00	47.01	50.67
	40	42.25	48.90	93.13	92.46
	60	69.65	56.99	97.30	100.33
Octane	25	27.44	29.88	45.75	45.60
	40	36.00	42.22	56.79	53.08
	60	34.02	47.02	66.89	51.91
Nonane	25	21.36	24.35	11.46	19.58
	40	31.45	32.35	33.89	36.40
	60	24.55	35.90	60.94	27.60
Decane	25	19.09	19.94	9.17	6.81
	40	21.86	27.10	53.20	16.80
	60	20.58	30.06	57.32	17.70

<sup>a</sup> IPN1, TDI based PU/PBMA; IPN2, MDI based PU/PBMA.

atically with increasing temperature.<sup>19</sup> Similar effects can be seen with the MDI-based IPNs, but these data are not presented here in order to avoid redundancy. However, the sorption plots of both MDI- and TDI-based IPNs in octane at 40°C are compared in Figure 2. It was observed that TDI-based IPNs attained equilibrium quicker than the MDI-based IPNs. This is due to higher amounts hard segments (two phenyl group) present in the MDI-based IPN membrane. The sorption plots of TDI-based PU/PBMA IPNs with nonane at different temperatures are shown in Figure 3. It is observed that for both the IPNs in nonane, equilibrium sorption increases with an increase in temperature.



**Figure 5** Arrhenius plots of diffusivity for MDI-based PU/PBMA IPN for *n*-alkanes.

**TABLE III**  
**Equilibrium Sorption Constant ( $K_s$ ) and Permeation**  
**Coefficient ( $P$ ) of PU/PBMA IPN + n-Alkane Systems**

Penetrants	Temp. (°C)	$K_s \times 10^3 \pm$ 0.01 (m mol/g)		$P \times 10^7$ (cm <sup>2</sup> /s)	
		IPN1	IPN2	IPN1	IPN2
Hexane	25	4.32	4.50	26.66	17.58
	40	5.26	6.64	35.61	50.12
	60	8.74	7.13	73.80	56.52
Heptane	25	3.39	3.40	15.93	17.22
	40	4.23	4.89	39.35	45.21
	60	6.97	5.67	67.77	57.17
Octane	25	2.41	2.62	12.55	13.63
	40	3.16	3.70	20.44	22.41
	60	2.98	4.13	22.76	24.41
Nonane	25	1.67	1.90	2.45	4.77
	40	2.76	2.53	10.66	11.78
	60	1.92	2.81	14.96	9.80
Decane	25	1.34	1.40	1.75	1.36
	40	1.54	1.91	11.63	4.55
	60	1.45	2.12	11.80	5.32

Sorption results have been analyzed by using an empirical relationship of the type:

$$M_t/M_\infty = Kt^n \quad (2)$$

where  $M_t$  and  $M_\infty$  are mass uptake values at time  $t$  and at equilibrium, respectively;  $K$  and  $n$  are empirical parameters, of which  $K$  is related to polymer-solvent interaction, and  $n$  is the type of transport mechanism. From the least square analysis of  $\ln(M_t/M_\infty)$  versus  $\ln t$ , the values of  $K$  and  $n$  have been estimated and these data presented in Table I. Typical plots of  $\ln(M_t/M_\infty)$  versus  $\ln t$  for TDI-based PU/PBMA IPNs in nonane are given in Figure 4. The average uncertainty in the estimation of  $n$  value is  $\pm 0.007$  units. It is found that

the values of  $n$  for both the PU/PBMA IPNs vary from 0.44 to 0.60, suggesting the transport to follow Fickian trend. The results of  $K$  decrease with increasing size of the penetrant molecules. However, there is no systematic effect of temperature either on  $n$  or  $K$  values.

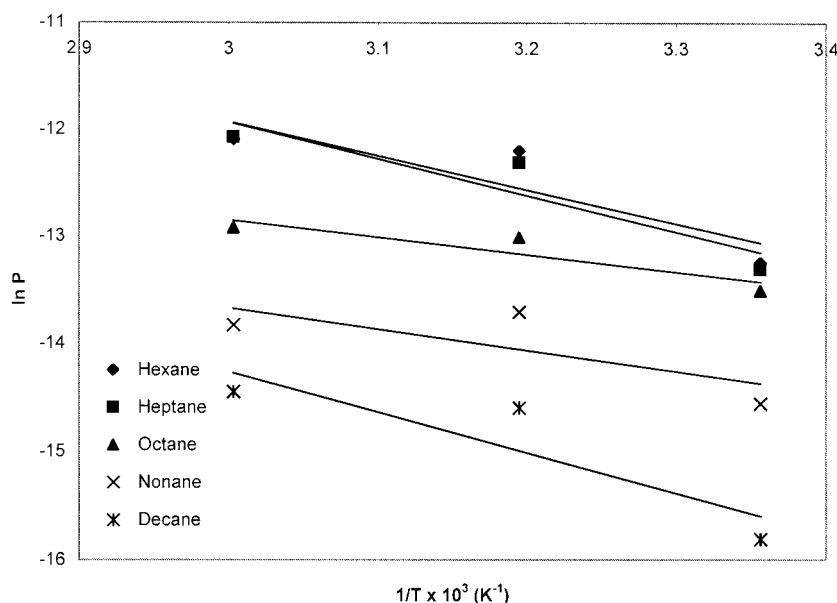
The diffusion values for each IPN-penetrant system calculated from the initial slope method are presented in Table II. A triplicate evaluation of  $D$  from the sorption curves gave us  $D$  values with an error of  $\pm 0.003$  units at 25°C,  $\pm 0.004$  units at 40°C and  $\pm 0.005$  units at 60°C for all the IPN-penetrant systems. The variation of  $D$  depends upon the nature of the penetrant molecules in addition to the composition of PU/PBMA and morphology of IPNs. From Table II, it can be seen that the  $D$  values decrease with an increase in the molecular weight of the penetrants. The sequence of variation of  $D$  with respect to penetrants is: hexane > heptane > octane > nonane > decane.  $D$  values also increase with an increase in temperature. Sorption results calculated from the equilibrium sorption curves also follow the same trend. The variation of  $D$  with temperature for MDI-based system is shown in Figure 5.

The permeability coefficient ( $P$ ) was calculated by the relation,<sup>20</sup>

$$P = D \times S \quad (3)$$

The calculated  $P$  values are reported in Table III. The results of  $P$  also follow the same trends as those of  $D$  with respect to temperature and size of the penetrant molecules. The Arrhenius plots of  $\ln P$  versus  $1/T$  are shown in Figure 6.

The Arrhenius apparent activation parameters for diffusion ( $E_d$ ) and permeation ( $E_p$ ) have been calculated using the relationship:



**Figure 6**  $\ln P$  versus  $1/T$  for MDI-based PU/PBMA IPN for n-alkanes.

**TABLE IV**  
**Activation Energy for Diffusion ( $E_D$ , J/mol), Permeation ( $E_P$ , J/mol), Enthalpy of Sorption ( $\Delta H$ , J/mol), and Entropy of Sorption ( $\Delta S$ , J/mol K  $\pm$  1) for PU/PBMA IPN + *n*-Alkane Systems<sup>a</sup>**

Penetrants	Parameter	PU/PBMA IPN	
		IPN1	IPN2
Hexane	$E_D$	7.42	16.21
	$E_P$	24.26	26.60
	$-\Delta S$	10.73	9.00
	$\Delta H$	16.80	10.54
Heptane	$E_D$	16.63	15.69
	$E_P$	33.82	27.70
	$-\Delta S$	9.8	6.60
	$\Delta H$	17.11	12.00
Octane	$E_D$	8.89	2.91
	$E_P$	13.83	13.59
	$-\Delta S$	33.41	13.84
	$\Delta H$	4.83	10.48
Nonane	$E_D$	38.91	7.45
	$E_P$	41.74	16.16
	$-\Delta S$	42.63	22.91
	$\Delta H$	2.85	8.56
Decane	$E_D$	41.90	21.87
	$E_P$	43.65	31.22
	$-\Delta S$	49.21	22.31
	$\Delta H$	1.63	9.52

<sup>a</sup> IPN1, TDI based PU/PBMA; IPN2, MDI based PU/PBMA.

$$\ln X = \ln X_0 - (E_x/RT) \quad (4)$$

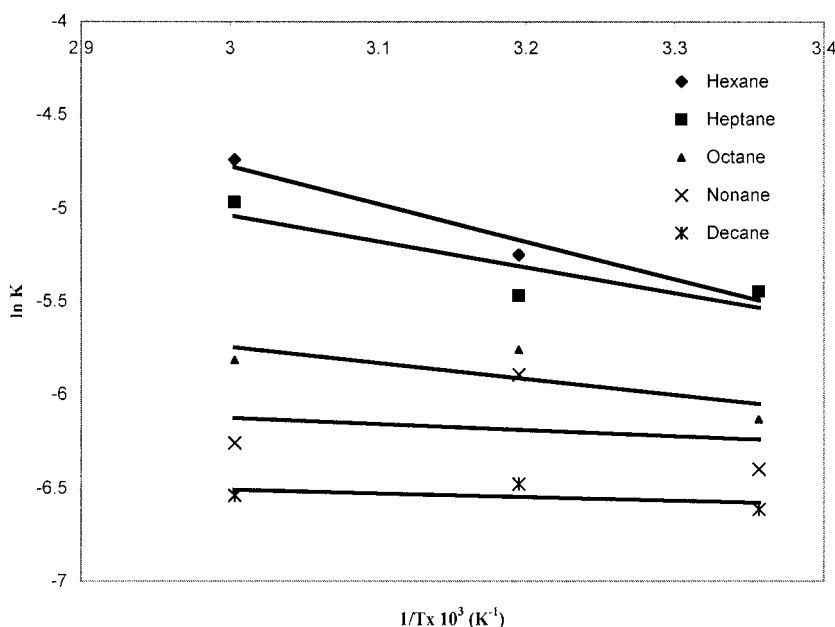
where  $X_0 = D_0$  or  $P_0$  is the pre-exponential factor,  $R$  is the molar gas constant,  $T$  is absolute temperature,  $E_X$  is  $E_D$  or  $E_P$ . Using least-squares method to the slopes of the straight lines, the values of  $E_D$  and  $E_P$  have been calculated and these results are presented in Table IV.

In general, higher  $E_D$  and  $E_P$  values are observed for higher alkanes in case of both MDI- and TDI-based IPNs.

In a similar fashion, the temperature dependent equilibrium sorption constants,  $K_s$  (Table III) have been fitted to vant Hoff's relation,<sup>21</sup> to estimate the enthalpy ( $\Delta H$ ) and entropy ( $\Delta S$ ) of sorption as:

$$\log K_s = [(\Delta S/2.303R) - (\Delta H/2.303R)(1/T)] \quad (5)$$

The vant Hoff's plots for TDI-based PU/PBMA in *n*-alkane penetrants are given in Figure 7. The  $\Delta H$  and  $\Delta S$  values have been calculated from the slopes and intercepts, respectively, from vant Hoff's plot. These data are given in Table IV. The average estimated error in  $\Delta H$  is about  $\pm 4$  J/mol whereas for  $\Delta S$  it is about  $\pm 1$  J/mol K. From Table IV, it can be observed that  $\Delta S$  values are negative for both the IPNs (except hexane and heptane with TDI-based IPN), suggesting that the solvent structure is retained in the sorbed state. From Table IV, it is observed that  $\Delta S$  values are in the range from  $-10.73$  to  $49.21$  kJ/mol K and from  $6.62$  to  $22.91$  kJ/mol K, respectively, for the TDI- and MDI-based systems. On the other hand,  $\Delta H$  values for all liquids are positive, which suggests that sorption follows endothermic process as dominated by Henry's law mode, in other words, sorption proceeds through the creation of new sites or pores in the polymer. The  $\Delta H$  values for TDI- and MDI-based IPNs are in the range from  $1.63$  to  $17.11$  kJ/mol and from  $8.56$  to  $12.0$  kJ/mol, respectively. From the data given in Table IV there appears to be no systematic variation in  $\Delta H$  as the nature of the polymer or solvent molecule.



**Figure 7** vant Hoff plots of  $\ln K_s$  versus  $1/T$  for TDI-based PU/PBMA IPN.

### Concentration profiles

It is important to consider the concentration profiles of liquid molecules before implementing membrane applications in a hostile environment. Therefore, we have calculated the concentration profiles of liquids through PU/PBMA IPN membranes using the computer-simulation method.<sup>22–24</sup> To calculate the concentration profiles, we have used Fick's second order differential equation given in its most general form:

$$\partial c / \partial t = D(\partial^2 c / \partial x^2) \quad (6)$$

where  $D$  is concentration-independent diffusion coefficient,  $t$  is the sorption time,  $C$  is liquid concentration within the membrane materials and  $\partial c / \partial x$  is concentration gradient along the  $x$  direction. Equation (6) was solved under the following assumptions: (i) liquid diffusion into the membrane takes place in one direction; (ii) sorption takes place under transient conditions with a constant diffusivity; (iii) during sorption, when the membrane is exposed to solvent, its concentration on the membrane surface reaches equilibrium immediately; (iv) the time required for the membrane to establish thermal equilibrium is negligible when compared to the time of sorption; and (v) changes in the membrane dimensions are negligible during the liquid exposure under the initial and boundary conditions,

$$t = 0 \quad 0 \leq x < h \quad C = 0 \quad (7)$$

$$t \geq 0 \quad x = 0, \quad x = h \quad C = C_\infty \quad (8)$$

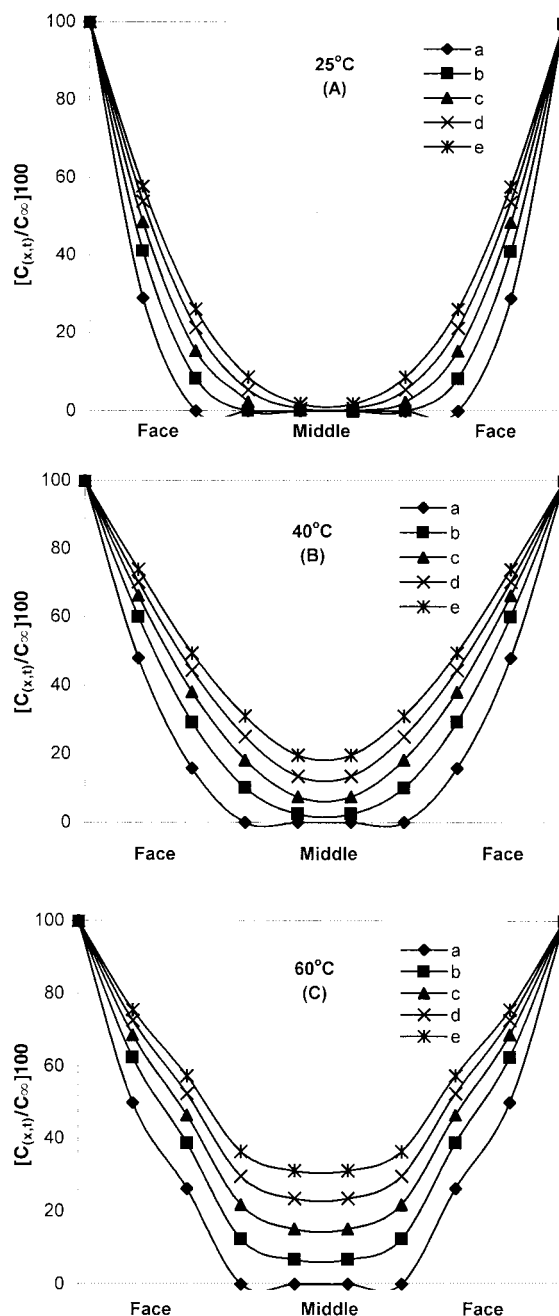
$$\partial C / \partial x = 0, \quad x = 0, \quad t > 0 \quad (9)$$

to give the concentration profile  $C_{(t,x)}/C_\infty$  of the liquids within the polymer membrane as a function of the penetration depth (i.e., distance  $x$ ), that is, the thickness of the membrane and time,  $t$ :

$$C(x,t)/C_\infty = 1 - \frac{4}{\pi} \sum_{m=0}^{\infty} \frac{1}{(2m+1)} \exp[-D(2m+1)^2 \pi^2 t / h^2] \sin[(2m+1)\pi x / h] \quad (10)$$

where  $m$  is an integer. Solving Eq. (10),<sup>22–24</sup> we obtained concentration profiles of the migrating liquids developed within the membrane. These data are useful to study the liquid migration as a function of time and penetration of the liquid from face to the middle of the membranes along the thickness direction.

The concentration profiles generated for TDI-based IPN at 25, 40, and 60°C with different time intervals are given in Figure 8. It was observed that higher values of  $C_{(x,t)}/C_\infty$  at 60°C for PU/PBMA TDI system are prevalent. The concentration profiles for TDI- and



**Figure 8** Concentration profiles calculated from Eq. (10) for hexane at (a) 12 min, (b) 24 min, (c) 36 min, (d) 48 min, and (e) 60 min for TDI-based PU/PBMA IPN at different temperatures.

MDI-based IPNs at 25, 40, and 60°C for octane are given in Figures 9 and 10, respectively. It can be seen from these figures that with an increase in temperature the values of  $C_{(x,t)}/C_\infty$  also increase. The concentration profiles for TDI-based IPN at 25 and 60°C with hexane are shown in Figure 11. Figures 9 and 10 show higher  $C_{(x,t)}/C_\infty$  values for TDI system when compared with MDI-based IPN. This difference is due to higher hard segment present in MDI-based IPN than TDI. Figures 9 and 11 show that higher  $C_{(x,t)}/C_\infty$  values are found for hexane than octane. These higher values are

due to lower molar volume of hexane than octane. Thus, the simulation method appears to be a sensitive test tool to predict the concentration profiles. It may further be demonstrated that with an increasing immersion time the concentration profile also increases drastically.

CONCLUSIONS

In this investigation, *n*-alkane penetrants have been selected and their interactions have been studied with structurally different PU/PBMA IPN membranes.

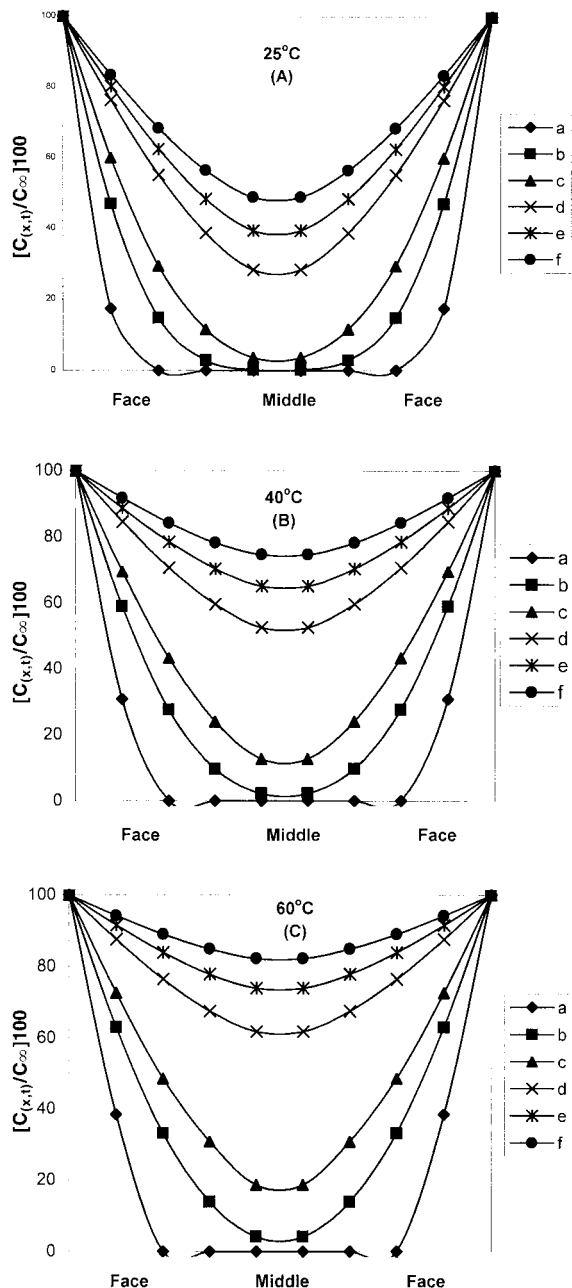


Figure 9 Concentration profiles calculated from Eq. (10) for octane at (a) 12 min, (b) 60 min, (c) 120 min, (d) 360 min, (e) 480 min, and (f) 600 min for TDI-based PU/PBMA IPN at different temperatures.

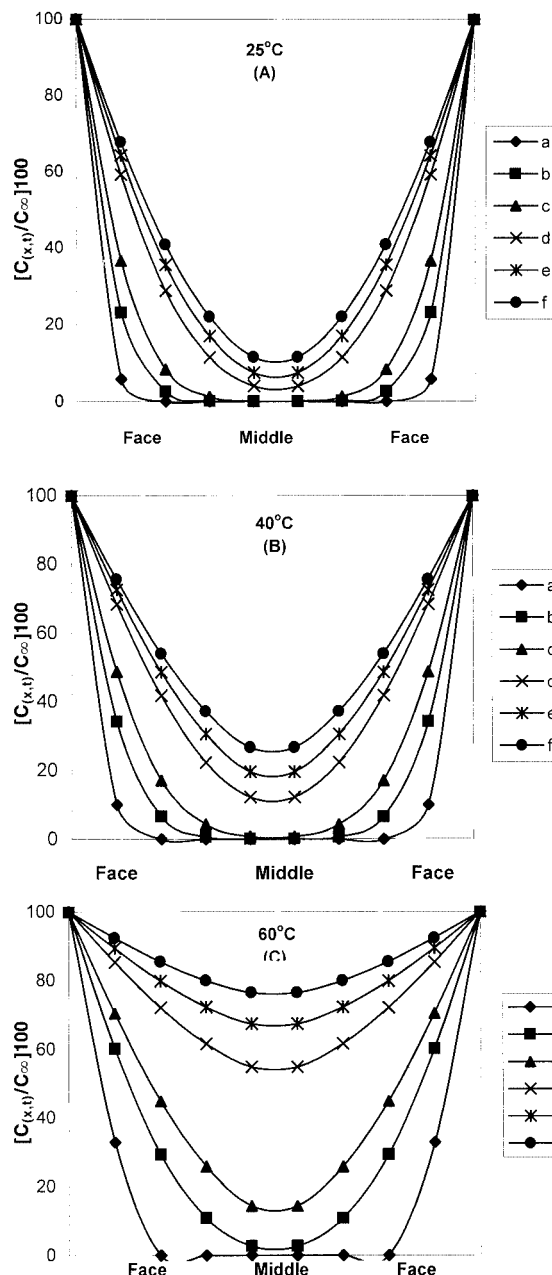
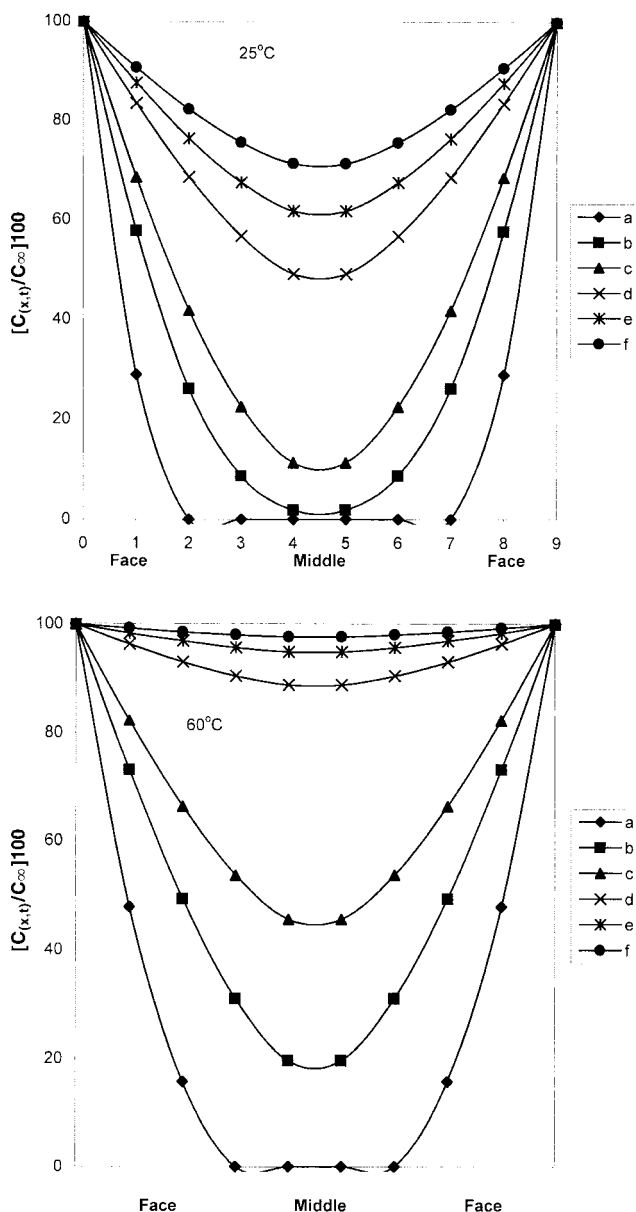


Figure 10 Concentration profiles calculated from Eq. (10) for octane at (a) 12 min, (b) 60 min, (c) 120 min, (d) 360 min, (e) 480 min, and (f) 600 min for MDI-based PU/PBMA IPN at different temperatures.

From this study, it would be possible to predict their transport behavior. An increase in polymer-solvents interactions leads to increased sorption and transport of liquids such that diffusion process often becomes concentration dependent. The Fickian model has been used to estimate the diffusion coefficient. The lower diffusion coefficients are observed for MDI-based PU/PBMA IPNs compared with TDI. Diffusion coefficients decrease with increasing size of the penetrant. The temperature dependence of transport coefficients has been used to compute the activation parameters from the Arrhenius plots. The  $\Delta H$  values are positive



**Figure 11** Concentration profiles calculated from Eq. (10) for hexane at (a) 12 min, (b) 60 min, (c) 120 min, (d) 360 min, (e) 480 min, and (f) 600 min for TDI-based PU/PBMA IPN at different temperatures.

whereas  $\Delta S$  values are negative for both TDI- and MDI-based PU/PBMA IPNs. The simulation method was helpful to predict the concentration profiles of liquids.

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