Resistance of barrier elastomers to hazardous organic liquids*

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Abstract

   Resistance of barrier elastomers such as ethylene–propylene–diene terpolymer, styrene–butadiene rubber, nitrile–butadiene rubber, neoprene and natural rubber to several hazardous organic liquids has been studied from immersion/dimensional change measurements. Penetration of liquids into these membranes depends on the nature of the barrier material and the liquid molecules. Diffusion coefficients have been calculated for each of the elastomer–solvent systems and their dependencies on concentration have been investigated. Implications concerning the resistivity of elastomer membranes to the presence of hazardous liquids are discussed.

1. Introduction

   Recently, the environment and everything connected with it has become a major public concern all over the world. Increased attention is being paid to the proper management and safe handling of hazardous chemicals. Although many organic solvents find applications in producing a variety of useful products, there is a great need to insure protection of public health and the environment from such chemicals. In recent years we have witnessed a tremendous growth in polymer industries in producing several useful products. It would be of interest to know how these products will react to the presence of such aggressive media as hazardous organic chemicals.

   A number of polymer membranes such as natural rubber (NR), styrene–butadiene rubber (SBR), neoprene (CR), nitrile–butadiene rubber (NBR) and ethylene–propylene–diene terpolymer (EPDM) find a variety of applica-

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tions in several areas such as those including garments, gloves and other protective clothing materials; these may be used under situations consisting of hazardous liquids. Possibilities are that the liquids might penetrate through such barrier materials.

In this study, several organic liquids which are known to be hazardous have been selected in order to investigate their transport characteristics through polymer membranes. Among the liquids used are chlorinated compounds such as dichlorobenzene, methylene chloride and chloroform. Other solvents tested include bromoform, dioxane and tetrahydrofuran (THF). Almost all these solvents find useful applications in the production of synthetic rubbers, resins and paints. However, when such liquids come into contact with the finished rubber products, they may penetrate into these materials inducing swelling and thus impair their mechanical properties.

Liquid penetration into polymeric materials can be studied in terms of sorption and diffusion phenomena [1–5]. Swelling of polymer membranes caused by aggressive solvents is a phenomenon which is directly related to the sorption and diffusion of chemicals into barrier materials. The authors in their earlier studies [6–8] have developed methods to study diffusion properties of polymer–solvent systems from a measurement of the dynamic response of barrier materials to the presence of liquids. The present paper is aimed at assessing the suitability of some industrially important polymer membranes to be used as barrier materials in the presence of hazardous liquids.

2. Experimental procedures

Molded sheets of SBR, NBR, NR, CR and EPDM in dimensions of 1/16 in. × 6 in. × 6 in. (1.59 mm × 15.24 cm × 15.24 cm) were fabricated by UTEX Industries, Weimer, Texas. A 12 in. (30.48 cm) laboratory mill was used to mix and prepare the rubber compounds for molding. The rubber sheets were cured at 160°C for 20 min. The compositions and some representative engineering properties of the rubbers have been given earlier [9].

Reagent grade solvents, namely, dichlorobenzene, methylene chloride, THF, chloroform, bromoform and p-dioxane were double distilled before use. Some relevant solvent properties and toxicity data are given in Table 1. The rubber samples were cut circularly (diameter = 1.97 cm) by using a sharp edged steel die. The sample thicknesses were measured at several points on the membranes within an accuracy of ± 0.001 cm.

Swelling experiments have been performed on circularly cut polymer samples by immersing them in test bottles containing about 15–20 mL of the respective solvent maintained at 25°C in an electric oven (Memmert, Germany). At regular intervals, these samples were removed from the test bottles to measure the changes in thickness and diameter by means of micrometer screw gauge and vernier calipers, respectively, with an accuracy of ± 0.001 cm.

TABLE 1

Some properties of hazardous liquids and their toxicity data

Liquids	Molar volume	Boiling point (°C)	Density at 25°C (g/cm³)	TLV[a] (ppm)
Methylene chloride	64.50	139.60	1.3168	500
Chloroform	80.68	61.20	1.4797	50
Tetrahydrofuran	82.12	65.97	0.8781	200
p-Dioxane	85.71	101.32	1.0280	100[b]
Bromoform	87.82	149.21	2.8779	0.5[b]
o-Dichlorobenzene	113.05	180.50	1.3003	50

[a]Threshold Limit Value toxicity.
[b]For skin.

The total amount of time spent by the samples outside the test bottles was within 30–40 s and this introduced negligible errors in the measurements of dimensional changes of the samples. This procedure was repeated and the experiments were continued until attainment of equilibrium saturation, as evidenced by no further swelling of the membranes.

3. Calculation of diffusion coefficient

Diffusion coefficients, D_Q, of polymer–solvent systems have been obtained from a consideration of a relation based on weight uptake of soaked polymer samples [10,11]. According to this method, the percent weight uptake, Q_t, of the polymer is given as:

$$Q_t = (M_t/M_0) = (4M_m/h_0 M_0)(D_Q/\pi)^{1/2} t^{1/2} \tag{1}$$

where M_t is the mass uptake by the polymer at time t, M_0 is the initial weight of the dry membrane, M_m is the weight gain at equilibrium saturation and h_0 represents the initial thickness of the polymer sample. Assuming that the increase in volume of the membrane at any given instant of time is proportional to the weight of the liquid taken up to that time then, $M_t \propto$ (volume of polymer at time t − initial volume of dry polymer).

$$M_t = k\pi(r_t^2 h_t - r_0^2 h_0) \tag{2}$$

$$M_m = k\pi(r_m^2 h_m - r_0^2 h_0) \tag{3}$$

where k's in eqs. (2) and (3) are the proportionality constants; r_0, r_t and h_0, h_t are radii and thicknesses of the polymer samples at time $t=0$ and at longer time t, respectively. The quantities, r_m and h_m, represent the equilibrium values, i.e. when $t=t_m$. Substituting for M_t and M_m from eqs. (2) and (3) into eq. (1) we get,

$$r_t^2 h_t - r_0^2 h_0 = [4(r_m^2 h_m - r_0^2 h_0)/h_0][D_V/\pi]^{1/2} t^{1/2} \qquad (4)$$

The left hand side of eq. (4), i.e. $(r_t^2 h_t - r_0^2 h_0)$ is the change in volume, ΔV_t, during polymer swelling. The term $(r_m^2 h_m - r_0^2 h_0)$ is equal to maximum change in volume, ΔV_m. Rearrangement of eq. (4) yields the following relation for diffusivity:

$$D_V = (\pi/t)(\Delta V_t h_0/4\Delta V_m)^2 \qquad (5)$$
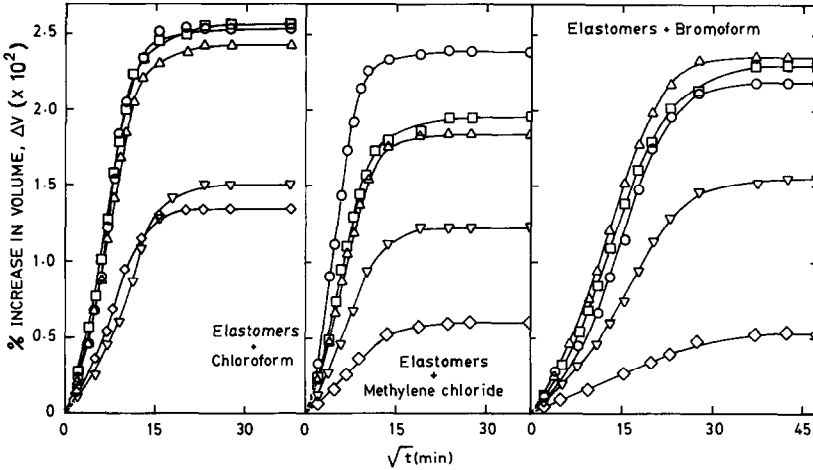

Fig. 1. Percent increase in volume of the elastomer samples vs square root of time for chloroform, methylene chloride and bromoform at 25°C. Symbols for elastomers: ($\diamond$) EPDM; ($\triangledown$) CR; ($\square$) NR; ($\bigcirc$) NBR; ($\triangle$) SBR.
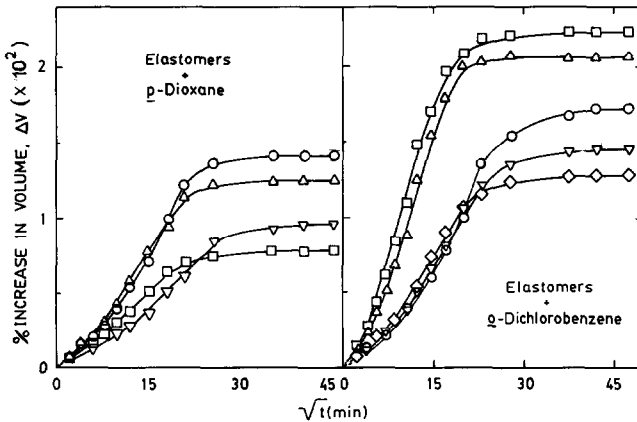

Fig. 2. Percent increase in volume of the elastomer samples vs square root of time for p-dioxane and o-dichlorobenzene at 25°C. Symbols for elastomers: ($\diamond$) EPDM; ($\triangledown$) CR; ($\square$) NR; ($\bigcirc$) NBR; ($\triangle$) SBR.
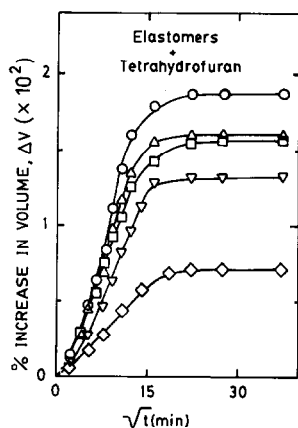
Fig. 3. Percentage increase in volume of the elastomer samples vs square root of time for tetrahydrofuran at 25 °C. Symbols for elastomers: ($\Diamond$) EPDM; (∇) CR; ($\Box$) NR; ($\bigcirc$) NBR; ($\triangle$) SBR.

alternatively,

$$D_V = \pi (h_0 \theta / 4 \Delta V_m)^2 \tag{6}$$

where θ is the slope of the initial linear portion of the plot of ΔV_t versus $t^{1/2}$ before the completion of 50% of equilibrium. The symbol, D_V, designates diffusion coefficients calculated on the basis of volume changes of polymer samples. The plots of the percent increase in volume, ΔV, defined as $(\Delta V_t / V_0) \times 100$ (i.e. increase in volume per unit volume of the polymer membrane) versus $t^{1/2}$ are presented in Figs. 1–3 for all the polymer–solvent systems.

4. Results and discussion

The results of maximum percent increase in thickness, Δh_m, and diameter, Δd_m, of the polymer samples with respect to various liquids are included in Table 2. It is found that for methylene chloride, chloroform, THF and p-dioxane, Δh_m and Δd_m values are quite high for NBR whereas, for bromoform, SBR shows high values for both Δh_m and Δd_m. Similarly, for dichlorobenzene, SBR shows high swelling in the thickness direction whereas, NR exhibits high values in Δd_m. Compared to all the membranes, EPDM appears to exhibit high resistivity to solvent penetration as it has the smallest values of Δh_m and Δd_m as compared to other rubbers.

The plots of percent increase in volume, ΔV, versus $t^{1/2}$ are presented in Figs. 1–3. The results for EPDM + dioxane system are not included in Table 2 and also in Fig. 2 because sorption of dioxane by EPDM membrane was extremely small even after a week of continuous immersion. It may be noted that the least swelling was observed in case of EPDM with all the solvents. The swelling of CR membrane is found to be higher than EPDM but smaller than

TABLE 2

Sorption and diffusion data of hazardous liquids into polymer membranes at 25°C

Liquids	Elastomers	Δh_m (%)	Δd_m (%)	Diffusion coefficients (cm^2/s)		
				$\bar{D}$	D_v	D_Q
Methylene chloride	EPDM	19.0	16.0	7.16	7.72	9.57
	CR	40.3	25.9	12.31	10.96	11.94
	NR	50.2	40.1	12.88	11.83	13.38
	NBR	58.2	40.4	26.57	18.95	19.61
	SBR	49.1	37.9	20.43	14.01	16.05
Chloroform	EPDM	36.2	31.0	6.70	5.47	8.59
	CR	42.7	32.3	8.17	6.94	8.83
	NR	60.2	49.0	7.58	8.41	10.61
	NBR	58.4	49.3	10.20	8.95	11.45
	SBR	59.5	46.7	11.36	9.27	11.23
Tetrahydrofuran	EPDM	21.8	18.0	6.29	4.92	5.73
	CR	38.5	29.4	7.69	6.72	7.83
	NR	43.1	34.0	7.09	7.25	9.19
	NBR	48.4	38.8	13.02	6.10	5.57
	SBR	44.9	34.0	10.57	7.57	9.42
p-Dioxane	EPDM	–	–	–	–	0.76
	CR	30.4	22.4	2.60	1.50	1.60
	NR	24.9	19.7	2.33	2.23	2.45
	NBR	37.9	32.2	2.73	1.60	1.53
	SBR	35.0	28.9	3.99	2.22	2.16
Bromoform	EPDM	16.4	14.7	2.65	1.53	1.60
	CR	43.6	33.2	2.90	1.96	2.24
	NR	54.3	46.2	3.71	2.46	2.42
	NBR	53.9	44.1	2.31	1.47	1.68
	SBR	56.9	46.4	3.57	2.41	2.71
o-Dichlorobenzene	EPDM	32.8	31.0	3.08	2.56	2.92
	CR	44.8	29.9	3.33	2.49	2.62
	NR	52.3	45.7	3.97	3.52	3.24
	NBR	43.5	37.8	1.88	1.22	1.60
	SBR	52.6	44.2	4.65	2.83	3.56

those of NR, NBR and SBR membranes. From the swelling results of chloroform presented in Fig. 1, it is found that NBR, NR and SBR membranes behave almost identically in the initial stages but near equilibrium, SBR exhibits lower swelling than NR and NBR membranes. The swelling curve for CR with chloroform is initially more sigmoidal and its maximum swelling value is slightly higher than that of EPDM membrane.

With methylene chloride, initially, there is a linear variation of ΔV versus $t^{1/2}$ suggesting a strict Fickian behavior (see Fig. 1). Moreover, with methylene

chloride, NBR shows higher increase in ΔV than SBR and NR membranes; even the values for CR and EPDM membranes are quite different as compared to chloroform. On the other hand, with bromoform, the dependencies of ΔV on $t^{1/2}$ for NR, SBR and NBR are almost identical; these systems also exhibit the sigmoidal shapes during early stages of swelling. The time required to attain equilibrium saturation for bromoform + EPDM is longer than all the other bromoform-containing systems. Similarly, with dioxane and dichlorobenzene, the elastomers behaved quite differently (see Fig. 2). In general, ΔV results are smaller for dioxane than dichlorobenzene. However, with the latter solvent, NBR exhibits a non-Fickian behavior whereas, NR and SBR exhibit Fickian diffusion patterns; the ΔV values for these systems are higher than that of NBR membrane. Particularly, the membranes such as EPDM, CR and NBR behave identically (i.e. ΔV vs $t^{1/2}$ curves vary identically) in the initial stages and near equilibrium, these systems exhibit quite different values of ΔV_m. The swelling results for THF solvent with elastomers (see Fig. 3) are also indicative of the Fickian solvent transport. In this case, NBR shows the highest swelling and the swelling characteristics of SBR and NR are almost identical.

The observed sigmoidal shapes of the swelling curves for the majority of the systems suggest that such solvents are highly aggressive and might induce a slow relaxation of the polymer chain segments. The diffusion coefficients, D_V, have been calculated from the slopes, θ, of the curves of ΔV versus $t^{1/2}$ (before completion of 50% equilibrium volume) by using eq. (6). These data are included in Table 2.

Attempts have also been made to calculate diffusion coefficients, D_h, from a measurement of changes in thicknesses of the polymer samples during the immersion experiments. Thus, in an analogous manner it is possible to calculate the percent increase in thickness, Δh, defined as $(\Delta h_t/h_0) \times 100$. Here, Δh_t is the change in material thickness at time t. The plots of percent increase in thickness versus $t^{1/2}$ are given in Figs. 4 and 5 for all the systems except bromoform. The Δh dependence for NBR, SBR and NR membranes with chloroform are almost identical as shown in Fig. 4. However, with methylene chloride only SBR and NR behaved identically, while NBR showed higher values of Δh_m. On the other hand, with THF each of the polymer membranes behaved differently as evidenced by different values of Δh_m. For chloroform, methylene chloride and THF the plots of Δh vs $t^{1/2}$ (Fig. 4) exhibit Fickian transport mode, whereas for dioxane and dichlorobenzene (Fig. 5), the curves are somewhat more sigmoidal than those presented in Fig. 4, and thus these systems exhibit a non-Fickian transport phenomenon.

By analogy to eq. (6), the diffusion coefficients, D_h, in the thickness direction of the polymer samples have been calculated as [10]:

$$D_h = \pi (h_0 G/4\Delta h_m)^2 \tag{7}$$

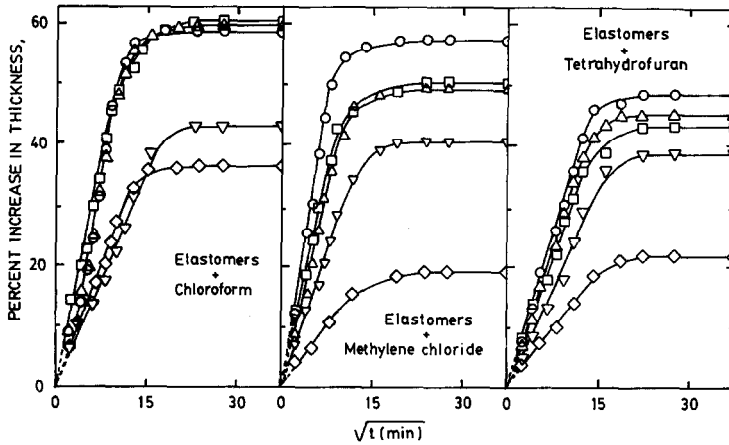where Δh_m represents the maximum percent increase in thickness; G is the

Fig. 4. Percent increase in thickness of the elastomer samples vs square root of time for chloroform, methylene chloride and tetrahydrofuran at 25°C. Symbols for elastomers: ($\diamond$) EPDM; ($\triangledown$) CR; ($\square$) NR; ($\bigcirc$) NBR; ($\triangle$) SBR.
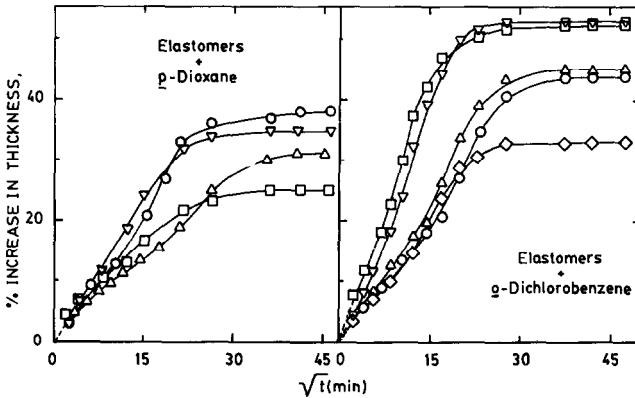

Fig. 5. Percent increase in thickness of the elastomer samples vs square root of time for p-dioxane and o-dichlorobenzene at 25°C. Symbols for the elastomers; ($\diamond$) EPDM; ($\triangle$) CR; ($\square$) NR; ($\bigcirc$) NBR; ($\triangledown$) SBR.

slope of the linear portions of the Δh versus $t^{1/2}$ plots (Figs. 4 and 5) and this was obtained by the least squares procedure. The values of D_h as calculated from eq. (7) are not to be regarded as the true diffusion coefficients because it is likely that when the polymer samples are immersed in aggressive solvents they might penetrate into the available free volume of the membranes. Thus, a correction to include diffusivity in the radial and the angular directions of the disk-shaped polymer samples should be considered. In this pursuit, mathematical relations have been developed earlier [7] wherein the relation for the average diffusion coefficient, $\bar{D}$, has been given as:

$$\bar{D}=D_h(1+\bar{h}/\bar{r}+\bar{h}/2\pi\bar{r})^2 \tag{8}$$

where $\bar{h}$ and $\bar{r}$ represent the average values of thickness and radius, respectively, before and after swelling. Equation (8) takes into account the solvent diffusivities in the angular and radial directions in addition to thickness direction. The results of $\bar{D}$ are also included in Table 2 along with the values of diffusion coefficients, D_Q, obtained previously [12] from the weight gain assay method. A comparison of the diffusivities (D_V, $\bar{D}$ and D_Q) given in Table 2 suggests that the correction to include three-dimensional diffusivity is necessary because the D_h values without such corrections (as given in eq. (8)) were smaller than the D_V or D_Q data. In any case, the values of D_V and D_Q agree better than those of $\bar{D}$ and D_Q for all the polymer–solvent systems (Table 2). A perusal of the data given in Table 2 further suggests that there is a direct relationship between the diffusivity and the parameters related to dimensional changes (i.e. Δh_m and Δd_m) of the systems. For example, diffusivities are smaller if the dimensional changes are small.

In order to confirm the validity of the diffusion and sorption data of the systems studied here, attempts have been made to perform two different sets of sorption experiments. In the first set, the polymer samples were directly soaked in the liquids and the swelling data were obtained; later, after reaching complete saturation, the desorption runs were made on these samples. In the second set, the completely desorbed and perfectly dried samples were again soaked into the liquids to ascertain the loss of any additives present in the rubber systems. A slight decrease in the original weights of the samples were observed in the second set of experiments thereby indicating the loss of indigenous materials of the rubbers. However, the shapes of the sorption curves at the initial stages remained the same and almost identical slopes were obtained in both sets of experiments. These data are given in Fig. 6 typically, for methylene chloride with all the polymer membranes; here, the mole percent increase, Q_x, calculated as the percent weight uptake per mole of the solvent is plotted. Although for each polymer–solvent system the maximum sorption was higher during the second set of swelling experiments than the first one, the calculated diffusivities (from both sets of experiments) remained almost the same (say within 1%).

From the foregoing discussion, it is apparent that the solvent penetration consists of two processes: (a) diffusion of solvent molecules into the polymer matrix and (b) local relaxation of polymer segments (an intrinsic property of rubbery polymers at room temperature). The characteristic times for diffusion and relaxation (and the changes in these characteristic times as the polymer swells) determine whether normal Fickian transport is observed. Based on previous experimental studies with these polymer membranes, slight deviations from the regular Fickian behavior is expected. As can be seen from the data given in Table 2, the smaller molecules are kinetically more favorable and
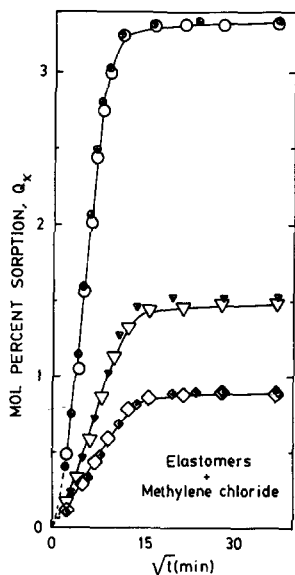
Fig. 6. Mole percent increase in sorption vs square root of time for methylene chloride at 25°C. First cycle of sorption for (◇) EPDM; (▽) CR; (○) NBR. The second cycle of sorption for (◈) EPDM; (▼) CR; (⊕) NBR.
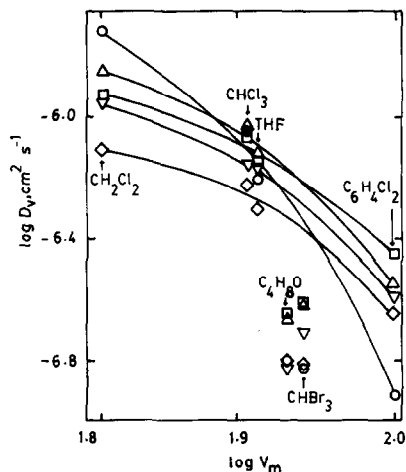

Fig. 7. Dependence of logarithm of diffusivity on logarithm of molar volume for rubber–solvent systems at 25°C. Symbols for elastomers are the same as in Fig. 1.

penetrate at significantly higher rates. An approximate measure of molecular size of solvent molecules is molar volume, V_m. Such a dependence is shown in Fig. 7 for all the polymer–solvent systems. While there is a systematic decrease in the dependence of $\log D_V$ on $\log V_m$ for methylene chloride, chloroform, THF and dichlorobenzene, this effect is not evident for p-dioxane and bromoform
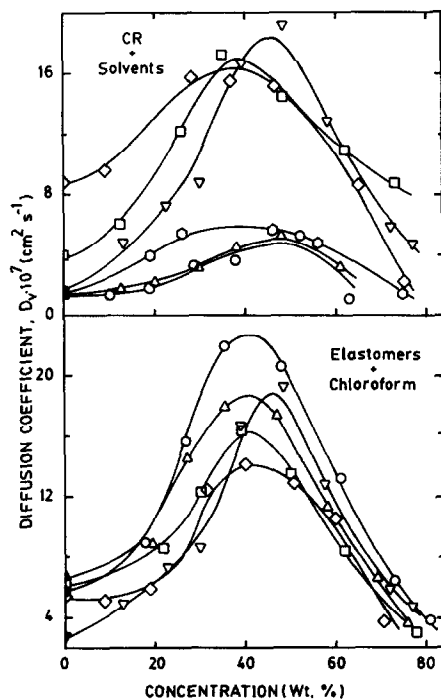
Fig. 8. Dependence of diffusion coefficient on concentration for rubber–solvent systems at 25 °C. Symbols for solvent: ($\Diamond$) methylene chloride; (∇) chloroform; ($\square$) THF; ($\bigcirc$) p-dioxane; ($\triangle$) bromoform; ($\circ$) dichlorobenzene. Symbols for elastomers: ($\Diamond$) EPDM; (∇) CR; ($\square$) NR; ($\bigcirc$) NBR; ($\triangle$) SBR.

solvents. This might be attributed to two different factors: (a) bromoform interacts somewhat strongly with all the membranes occupying all the available free volume within the rubber matrix thereby not maintaining a steady state equilibrium and (b) dioxane which shows extremely low levels of swelling may not enter into all of the available free volume. Thus, with both of these solvents, there is no linear relationship between log D_V and log V_m.

The slight sigmoidal shapes of the curves given in Figs. 1–5, at least in some cases, indicate the strong concentration dependence of diffusion coefficients. This was studied by using the Joshi and Astarita procedure [13], wherein the model parameters were varied incrementally over a prescribed range and a fit to the experimental data was subjected to the least squares test to select the best set of values. The diffusion coefficients so obtained over a range of concentrations are plotted as a function of concentration (Fig. 8). The upper part of Fig. 8 presents such data for CR + solvents while the lower part presents similar data for various elastomers in the presence of chloroform. It is observed that for those liquids which exhibit high swelling, the D_V versus concentration curves pass through maxima. However, the maxima decrease considerably for

TABLE 3

Coefficients k and n of eq. (9)

Liquids	Coefficients	EPDM	CR	NR	NBR	SBR
Methylene chloride	k	4.01	3.03	3.83	4.26	3.84
	n	0.60	0.70	0.71	0.75	0.70
Chloroform	k	3.24	2.14	3.60	2.48	2.45
	n	0.65	0.66	0.67	0.75	0.75
Tetrahydrofuran	k	2.84	2.29	2.66	2.11	2.72
	n	0.65	0.66	0.69	0.74	0.69
p-Dioxane	k	–	1.84	3.25	1.84	1.91
	n	–	0.56	0.53	0.59	0.63
Bromoform	k	2.97	1.70	1.67	1.53	1.76
	n	0.51	0.60	0.64	0.62	0.64
o-Dichlorobenzene	k	1.99	1.35	2.09	1.61	2.13
	n	0.60	0.64	0.67	0.57	0.63

the low swelling membranes. Particularly for CR with dichlorobenzene, brom-oform and p-dioxane, no sharp maxima are observed. On the other hand, chloroform shows high maxima for all the rubbers. Similar findings have also been reported earlier in the literature [14].

In order to establish the type of transport mechanism for rubber–solvent systems, attempts have been made to analyze the swelling data with an empirical relation of the type [11]:

$$\log (\Delta V_t / \Delta V_m) = \log k + n \log t \tag{9}$$

Here, k is a constant characteristic of the polymer–solvent system and the numerical value of n indicates the degree of deviation of the transport mechanism from a Fickian value of $n=0.5$. Some aspects of these problems have been discussed in an earlier review on the subject [15]. The estimated values of k and n are given in Table 3. The values of n for almost all the polymer–solvent systems are higher than 0.5 and in some cases, as high as 0.75. This suggests the existence of non-Fickian transport mechanisms operating in the polymer–solvent systems. This further confirms the presence of slight sigmoidal shapes of the curves given in Figs. 1–5. However, at this stage, there is no intention to attach any great significance to the values of k as these are strictly empirical values; moreover, these values vary from one system to the other.

5. Conclusions

The focal point of the research program is to probe the solvent transport through engineering polymer membranes to extract information about their

resistivity in field applications when in contact with the hazardous media. The research results of this study indicate that the dimensional response of polymer membranes when exposed to toxic chemicals depends not only on the nature of the interacting probe molecules but also on the polymer membrane system. The factors such as solvent type and chemical nature of the barrier material exert tremendous effect on their transport characteristics.

EPDM and CR are well-known engineering materials which seem to exhibit high resistivity to the presence of liquids studied in this research. Many of the solvents (save methylene chloride) used here have shown significant swelling characteristics as evidenced by the sigmoidal shapes of the swelling curves. The Fickian model has been used to estimate the diffusion coefficients of polymer–solvent systems and these values are corrected to include three-dimensional swelling of the membranes.

Concentration dependencies of diffusion coefficients have been studied and used in the discussion of the swelling results. The overall objective of this research program is the development of suitable liner membrane materials for the destruction of hazardous wastes (especially chlorinated hydrocarbons) in the complex environment of hazardous chemical ponds. With such a predictive tool, it would be possible to assist rubber industries in the design and use of suitable lining materials.

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