Diffusion and Sorption of Sulfur Mustard and Bis(2-Chloroethyl)ether in Elastomers: A Comparative Study

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ABSTRACT: Bis(2-chloroethyl)ether, a nonvesicant and easy to handle chemical, was proposed as a model compound for the vesicant sulfur mustard (SM) in routine permeation testing of protective devices. The proposition was based on detailed studies on sorption of these chemicals in elastomers. From the sorption plots and permeation parameters, it was found that the model compound diffuses faster than SM, and the diffusion follows Fickian kinetics. Free volume models, such as those developed by Lee and Salame, together with solubility and thermodynamic interaction parameters, validated the observed sorption phenomenon and afforded a criterion for predicting the barrier properties of elastomers. © 2000 John Wiley & Sons, Inc. J Appl Polym Sci 77: 2472–2479, 2000

Key words: sulfur mustard; permeation; free volume; bis(2-chloroethyl)ether, elastomer

INTRODUCTION

The blistering agent sulfur mustard (SM), chemically known as bis(2-chloroethyl)sulfide, alkylates body cells even on short exposure and penetrates most materials.[1] It is therefore called the "King of Chemical Warfare (CW) Agents,"[1] and there were reports of its use in regional conflicts, such as the Iran–Iraq war. Protection against SM in the event of chemical contamination or chemical warfare is generally afforded by elastomeric barrier materials in the form of protective ensemble such as respirators, suits, gloves, and overboots.[1] Performance evaluation of these devices involves testing of breakthrough time (BTT) against SM, on a routine basis. Handling of SM poses practical difficulties due to its vesicant nature, and necessitates a search for a suitable non-blistering model compound.

In the present study, an oxygen analogue (OA) of SM, viz., bis (2-chloroethyl)ether, was investigated as a model compound for SM sorption studies in elastomers. OA is structurally similar to SM in molecular dimensions and has no perceptible blistering effect.[2] The sorption behavior of SM and OA in elastomers such as polyisoprene (PI), nitrile rubber (NBR), polyisobutylene (PIB), butyl rubber (IIR), polybutadiene (PB), styrene-butadiene rubber (SBR), and ethylene propylene diene methylene (EPDM) rubber, was studied. The sorption data was analyzed in terms of Fickian models of diffusion. Further, in order to interpret the dependence of permeation on cohesive energy density, free volume, and molecular structure of the polymer, the results were analyzed in terms of solubility parameters[3] of the polymers and permeants, Lee's[4] model (based on specific free volume), and Salame's[5] model (based on per-

machor number). These models also afford a criteria for theoretical estimation of extent of sorption in elastomers.

EXPERIMENTAL

Materials

The details of the rubbers and chemicals used are as reported[6,7] earlier. SM (GC purity 99.5%) and OA (E. Merck, Germany) were distilled prior to use.

Formulation and Preparation of Membranes

Membranes of PI, IIR, PIB, PB, SBR, NBR, and EPDM rubbers were made by casting from 10% w/v solution of the rubber in toluene using the reported[6,7] recipe. The thickness of the membranes was maintained at 0.3 ± 0.05 cm. The mechanical properties and crosslink densities of the cured membranes have been reported[8] earlier.

Determination of Permeation Parameters

The membranes were dried in a vacuum oven for 24 h at 100°C to remove surface absorbed moisture and then evaluated for SM diffusivity and OA sorption using the spot-disk breakthrough time test (SD BTT) and gravimetric method, respectively.

BTT and Diffusivity of SM

The BTT of SM was determined using a standard color reaction test (SD BTT)[6–9] based on oxidation of thioethers with active halogen.[10] The time taken for the first appearance of blue color on specially prepared detector paper (Congo red paper dotted with 2,4-dichlorophenylbenzoyl chloroimide) from the time of initial application of SM was termed the BTT. An average of five readings was reported: minimum and maximum readings varied $\pm 15\%$ from the average. For a given thickness (h) of the sample and temperature, the higher the BTT value, the lower is the diffusivity of the chemical. The diffusion coefficient D was calculated[6–8,11] from the relation

$$D = h^2/\bar{\Lambda}^2 \text{ (BTT)} \qquad (1)$$

Diffusivity of OA

The SD test does not respond to OA due to the absence of thio moiety in the molecule. Therefore, the gravimetric method was used to measure the sorption of OA. The membranes were exposed to the chemical in a closed chamber at 298 K. The specimen was taken out periodically, dried between folds of filter paper, and weighed. The mole percent sorption Q_t was obtained from the gain in weight of the specimen, and related to equilibrium sorption uptake, Q_∞ through an empirical relation[12,13]:

$$Q_t/Q_\infty = Kt^n \qquad (2)$$

where K is a constant characteristic of the polymer–permeant interaction, n is the transport coefficient that describes the nature of sorption. For $n \leq 0.5$, diffusion is Fickian while for $n > 0.5$, it is non-Fickian. K and n were obtained from the intercept and slope respectively of the plot of log Q_t/Q_∞ vs log t (for $Q_t/Q_\infty \leq 0.6$).

For a plane geometry of the polymer sheet, the total mass uptake of penetrant can also be described by either of the following equations[14]:

$$\frac{Q_t}{Q_\infty} = 1$$

$$- (8/\pi)^2 \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp \frac{-[(2n+1)^2 \pi^2 Dt]}{h^2} \qquad (3)$$

$$\frac{Q_t}{Q_\infty} = (4/h)(Dt/\pi)^{0.5} + (8/h)$$

$$\times (Dt)^{0.5} \sum_{n=1}^{\infty} (-1)^n \text{ierfc}[nh/2\sqrt{(Dt)}] \qquad (4)$$

Equation (3) converges rapidly at long times, whereas eq. (4) converges rapidly at short times. Methods for obtaining a diffusion coefficient from sorption data are based on one of these two equations. From the measurement of the initial slope (θ) of a plot of Q_t/Q_∞ vs $t^{0.5}$, and using only the first term of eq. (4), at sufficiently short times, D was obtained from[15]

$$D = \pi \left(\frac{h\theta}{4Q_\infty} \right)^2 \qquad (5)$$

where h is the thickness of the membrane.

Solubility of OA

For low concentrations of permeants in the membranes, solubility S is described by Henry's law,[11] which permits the determination of the concen-

Table I   Physicochemical Properties of SM and OA

Property	SM	OA
Molecular formula	$C_4H_8Cl_2S$	$C_4H_8Cl_2O$
Chemical name	Bis(2-chloroethyl)sulphide	Bis(2-chloroethyl)ether
Trivial name	Sulfur mustard	Chlorex
CAS registry number	505-60-2	111-44-4
Physical state	Oily liquid	liquid
Appearance	Colorless	Colorless
Odour	Mustard-like	Odorless
Molecular weight	159	143
Boiling point (°C)	228	178
Freezing point (°C)	-14.4	-46.8
Specific gravity (g/cc, 298 K)	1.27	1.21
Vapor pressure (mm Hg, 298 K)	0.20	1.55
Molecular diameter (Å)	6.98	6.83
Cross-sectional area (m^2)	-38.33×10^{-20}	-36.70×10^{-20}
Solubility in water	0.8 g/L	1.02%
Chemical stability	Stable	Stable
pH	Neutral, slowly turns acidic on keeping	Neutral
Refractive index (298 K)	—	1.4533
Vesicant action	Blister agent	Nonblister agent

tration of the permeant (C_a) at the exposed surface of the membrane by a linear equation:

$$C_a = Sp_a \qquad (6)$$

where p_a is the partial pressure of the organic vapor to which the membrane is exposed.

When Henry's law is obeyed, the solubility of the permeant in the polymer is constant at a given temperature and can be calculated based on penetrant concentration in the membrane at equilibrium with the permeant at partial pressure p_a, i.e.,

$$S = C_a/p_a \qquad (7)$$

C_a was determined by the weight-gain method by computing the number of moles of the chemical sorbed per unit volume (area $\times$ thickness) of the membrane at atmospheric pressure. The p_a values were taken from the literature.[1]

Permeability of OA

Permeation of chemicals through a nonporous rubbery membrane occurs via a solution-diffusion[15] mechanism. Permeability P is therefore a function of both solubility S and diffusivity D of the permeant in the polymer material, i.e.,

$$P = D \cdot S \qquad (8)$$

RESULTS AND DISCUSSION

Few important physicochemical properties of the two challenge chemicals—SM and its OA—are collated in Table I for comparison. The cross-sectional area (σ) of the permeants was theoretically calculated using[2]

$$\sigma = 4(0.866)[M/4 \sqrt{2Al}]^{2/3} \qquad (9)$$

where M is the molecular weight of the permeant, l is its density, and A the Avogadro's number. Knowing σ, the molecular diameter was computed using the relation[2]

$$\pi r^2 = \sigma \qquad (10)$$

where r is the radius of the molecule. It is observed that SM and OA have similar cross-sectional areas and molecular diameters besides being structurally similar.

Diffusivity of SM

The breakthrough time of SM in the cured elastomers evaluated by the SD method and shown in

Table II   Crosslink Densities and SM Diffusivities of Elastomers at 298 K

Sample	Crosslink Densities[a]		SM Diffusivity	
	M_c	N	BTT (h)	D (m²/s)
PIB	—	—	18.00	2.60×10^{-13}
IIR	400	2.30	16.00	2.60×10^{-13}
NBR	369	2.66	4.00	1.13×10^{-13}
EPDM	2050	0.48	0.30	1.35×10^{-11}
SBR	485	2.0	0.16	1.52×10^{-11}
PI	1227	0.74	0.10	1.85×10^{-11}
PB	167	5.40	0.03	3.38×10^{-11}

[a] Obtained using Flory's equation.[8,15] M_c is molecular weight between the crosslinks and N is the moles of crosslink per dm³ of polymer.

Table II, varied in the order PB < PI < SBR < EPDM < NBR < IIR < PIB. In spite of a high crosslink density of 5.4 moles/dm³, diffusion coefficient D of SM in PB is higher as compared to other elastomers where the crosslink densities are much lower (0.48–2.6 moles/dm³). This disparity in crosslink densities and diffusivities is due to the reason that polymer permeability is a complex function of various factors, such as chain flexibility, intermolecular interaction, phase and aggregation state of a polymer, density, and macromolecular packing besides extent of cross-linking.[11,15]

Within the same physical state, the packing of macromolecules assumes greater importance in determining permeability, which explains why the closely packed isobutylene chains in IIR show lower permeability in contrast to polybutadiene and nitrile rubbers, in spite of higher crosslink densities in the latter. In nitrile rubber, increased interchain interaction due to the presence of polar C≡N groups increases activation energy of diffusion, making the rubber relatively more impermeable[16] than EPDM and PI. A higher diffusivity of SM in PB, PI, and SBR also may be attributed to their irregular structure, loose packing, and contribution to some extent from the unsaturated structure.

Sorption of OA

The extent to which permeant molecules are sorbed and their mode of sorption in a polymer can be classified[15,16] on the basis of the relative strengths of the interactions between the permeant molecules and the polymer or between the polymer molecules themselves within the matrix. The degree of swelling of the polymer in the permeant can give a fair idea of the polymer–permeant interactions. It is observed from Table III that the degree of swelling in SBR, PB, and NBR upon sorption of OA is greater than 100%, indicating a relatively higher interaction of OA with these elastomers.

The extent of sorption also depends on solubility (δ) and thermodynamic interaction (χ) parameters. The solubility parameter of the polymer is defined[3] as the square root of the cohesive energy density (ced) in the amorphous state at room temperature (298 K), i.e.,

$$\delta = (ced)^{0.5} = \sqrt{E_{\text{coh}}/V} \qquad (11)$$

where E_{coh} is the cohesive energy and V is the molar volume. V was obtained from the group contribution values proposed by Fedors[17] and

Table III   Experimental and Theoretical Parameters for Sorption of Liquid OA in Elastomers at 298 ± 1 K

Parameter	PIB	IIR	NBR	EPDM	SBR	PI	PB
ΔW^a (%)	3	8	422	6	102	45	124
ΔV^b (%)	5	9	—	12	46	—	—
S (mol · m⁻³ · Pa⁻¹)	3.51	2.98	187.70	2.08	53.40	16.47	43.09
E_{coh} (KJ · mol⁻¹)	17.89	17.989	21.889	10.551	26.24	22.88	18.58
V (CC · mol⁻¹)	63.90	63.81	53.17	38.30	71.49	73.70	59.20
δ (J¹ᐟ² · cm⁻³ᐟ²)	16.73	16.79	20.28	16.59	19.15	17.61	17.71
$\Delta\delta$	4.93	4.77	1.28	5.97	2.41	3.95	3.85
χ	1.17	1.12	0.40	1.55	0.54	0.87	0.50

[a] Degree of swelling $\Delta W = (W_s - W_0)100/W_0$, where W_0 is the initial weight of the elastomer and W_s is the weight after swelling in OA.
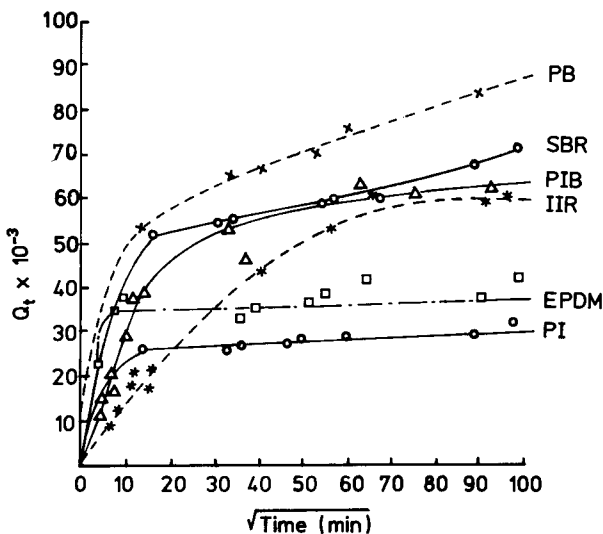[b] Volume swell.

Figure 1 Sorption of OA in elastomers at 298 K.

E_{coh} was calculated using the updated values given by Hoftyzer and Van Krevelen[3]. For low molecular substances, the values given by Bunn[18] have proved useful to give by far the best prediction of E_{coh}. For OA, E_{coh} calculated using Bunn's values is 43.66 KJ/mol; molar volume is 84 cc/mol and δ calculated according to eq. (11) is 21.56 J½ cm$^{-3/2}$.

As a general rule, chemical and structural similarities favor solubility. In terms of the abovementioned quantities, this means that the solubility of a permeant in a polymer is enhanced if the difference in their solubility parameters (Δδ) is small. As observed from Table III, there is a good agreement between Δδ and the solubility of OA in them. Nonpolar elastomers with saturated hydrocarbon backbone exhibiting higher Δδ values have a lower solubility of OA in them. The value is lowest in nitrile rubber; OA solubility is therefore maximum in this rubber. The solubility was determined by the weight-gain method as described earlier.

Within the limits of experimental error, an acceptable description of the sorption capacity of the polymers can also be afforded by the thermodynamic interaction parameter χ. The χ is a measure of thermodynamic affinity[3] of a polymer for a permeant, expressed as

$$\chi = (V_s/RT)(\delta p - \delta s)^2 + \chi_s \qquad (12)$$

where V_s is molar volume of permeant, R is the gas constant, T the absolute temperature, δ_p and δ_s are solubility parameters of the polymer and permeant respectively. The χ_s is the entropic contribution ranging between 0.1 and 0.5. It is often assumed constant with value around 0.35. The smaller the value of $(\delta_p - \delta_s)^2$, the smaller will be χ and greater will be the solubility of the permeant in the polymer. In other words, this implies that extent of sorption decreases as χ increases. As seen from Table III, for isobutylene-based polymers (IIR and PIB) χ is greater than one. This signifies a lower thermodynamic affinity of these polymers for the permeants and hence a lower sorption.

The sorption kinetics of liquid OA in elastomer membranes at 298 K, obtained using the weight-gain measurement, are plotted in Figures 1 and 2. These depict, in general, a steady state after an initial breakthrough and finally an equilibrium at longer times. The sorption plots for PI and EPDM indicate a significant level of sorption at relatively shorter times. This may probably be due to initial sorption in some kind of sorption sites[11,16] akin to Langmuir-type sorption. When nearly all of the sites are occupied, a very small amount of the permeant may randomly dissolve in the polymer. Swelling of the elastomers SBR and PB probably causes an increase in free volume and facilitates transport of the permeant.[11,16] This explains the similar nature of the sorption plots for these elastomers. Due to a higher interaction, equilibrium is attained at relatively much shorter time in NBR.
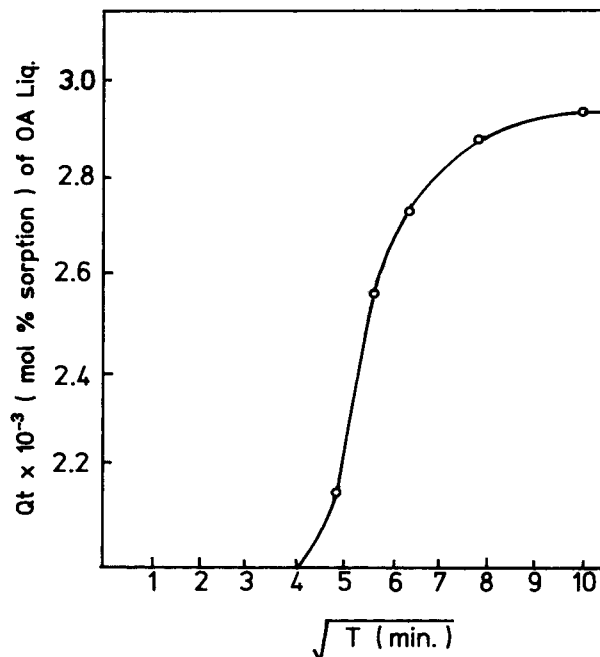

Figure 2 Sorption of OA in nitrile rubber at 298 K.

Table IV   Permeation Parameters for Sorption of OA in Elastomers at 298 K

Sample Code	$Q_m \times 10^2$ (mole %)	K (g/g · min^n)	n	S (moles · m^{-3} · Pa^{-1})	D (m^2/s)	P (moles · m^{-1} s^{-1} · Pa^{-1})
PIB	6.0	0.053	0.50	3.51	3.19×10^{-15}	1.2×10^{-14}
IIR	6.0	0.035	0.34	2.98	8.59×10^{-13}	2.5×10^{-12}
NBR	295.0	—	—	187.79	4.15×10^{-11}	7.8×10^{-9}
EPDM	3.8	0.446	0.15	2.07	1.78×10^{-5}	5.7×10^{-11}
SBR	7.0	0.158	0.28	53.44	2.80×10^{-12}	1.5×10^{-10}
PI	30.0	—	—	16.47	6.77×10^{-12}	1.1×10^{-10}
PB	87.0	0.178	0.23	43.09	3.93×10^{-13}	1.7×10^{-11}

The permeation parameters derived from these sorption plots are tabulated in Table IV. It is observed that n is less than or equal to 0.5 in all elastomers, implying Fickian kinetics. It is therefore possible to calculate D from the initial slope (θ) of the rectilinear portion of the sorption plots, according to eq. (5). PI and NBR became brittle in OA while PB and SBR showed tackiness; so the values of S, D, and P in these cases are only approximate. The sorption coefficient S of OA was the lowest in EPDM while the diffusion coefficient was of the same order (10^{-11} m^2/s) as in PB. The permeability coefficient P is of the order of 10^{-14} m^2/s in NBR and 10^{-14} m^2/s in PIB, implying a higher transport of OA through NBR than PIB. The lower P values of isobutylene rubbers implies that these rubbers are the most resistant to permeation of OA among the elastomers studied.

Dependence of Permeation on Free Volume

The free volume available per unit mass in a polymer controls the rate of diffusion, and hence the rate of permeation. If the diffusant is smaller than the statistical average free volume (at a specified diffusion temperature), it will diffuse through the material with ease resulting in a large diffusion coefficient.[11–16] Conversely, when a diffusant is comparable in size with the available free volume in a material, its diffusion is restricted. Since free volume embodies effects (interrelating or otherwise) from the various molecular structure factors, it can be used for correlating polymer structure with permeability. Correlation of the permeation behavior of elastomers to the challenge chemicals was attempted through two approaches: the Lee's theory based on specific free volume[4] and Salame's method based on permachor numbers.[5]

Lee's Approach:

According to Lee,[4] the controlling physical parameter in permeation is the specific free volume (SFV) defined on a unit weight basis as

$$\text{SFV} = V_F / M \qquad (13)$$

where M is the molecular weight of repeating structural unit and V_F is the molar free volume of an amorphous polymer (in cc/mol). The V_F is defined by[3]

$$V_F = V_T - V_0 \qquad (14)$$

where V_T is the molar volume of an amorphous polymer at T K and V_0 is the zero point molar volume. A good approximation to V_0 is after Bondi,[19]

$$V_0 = 1.3 V_w, \qquad (15)$$

The van der Waal's volume (V_w) can be obtained through a group contribution method. V_T can be replaced by V_s or the specific volume, which in turn is taken as the reciprocal of the density of the polymer[3] (ρ_r), i.e.,

$$V_s = 1/\rho_r \qquad (16)$$

These quantities were computed for the unfilled elastomers studied, as given in Table V.

The SFV is related to a mobility term M_p defined as the mobility of the penetrant relative to the polymer in its free volume space and expressed as[3]

$$M_p = A \cdot \exp(-B/\text{SFV}) \qquad (17)$$

Table V   Theoretical Estimation of Free Volumes in Elastomers

Sample	V_w (cc · mol^{-1})	V_0 (cc · mol^{-1})	ρ_r^* (g/cc)	V_s (cc · mol^{-1})	V_F (cc · mol^{-1})	SFV (cc · mol^{-1})
PIB	40.93	53.02	0.920	60.87	7.66	0.136
IIR	41.06	53.37	0.918	62.48	02.87	0.158
NBR	35.67	46.37	0.980	54.79	08.43	0.156
EPDM	24.24	31.51	0.980	33.86	2.35	0.070
SBR	48.82	63.46	0.965	79.25	15.79	0.206
PI	47.66	61.95	0.910	74.73	12.78	0.187
PB	37.36	68.56	0.900	60.05	11.49	0.212

[a] Taken from product catalogue.

where A and B are constants independent of penetrant concentration and temperature. The permeability P is related to M_p through the expression[3]

$$P = (D) \cdot (S) = (S) \cdot RTM_p$$
$$= (SRT)A \cdot \exp(-B/\text{SFV}) \quad (18)$$

At a given temperature, log P is proportional to $(-1/\text{SFV})$, provided the solubility is not strongly dependent on SFV. The 1/SFV term has the unit of density and can be considered as a measure of the "tightness" of the polymer structure. The larger its value, the tighter the structure becomes, thus restricting the permeation. In terms of the free volume available per unit mass for permeant, the smaller the SFV is, the more difficult is the permeation process. Thus, a good barrier material should have a smaller SFV. The theoretical linear relationship between log P and 1/SFV should serve as a useful tool for the selection of barrier materials for protective purposes. However, Figure 3 depicts that for the elastomers studied, the linearity between log P and 1/SFV is not precise; thereby necessitating corrections for dependence of solubility on SFV.

Salame's Approach

Besides Lee's model, another commonly used empirical technique for estimating permeability, is the permachor method proposed by Salame[5] in which the polymer structure and morphology are correlated to gas permeability. Salame suggested a scale of numerical values, termed permachor number (denoted as Π) and based on polymer ced and fractional free volume (FFV) to predict permeability. FFV is the ratio of free volume to the specific volume,[3] i.e.,

$$FFV = V_f/V_s \quad (19)$$

As seen from Table V, the variation in FFV in elastomers is similar to the trend in SFV. PB and SBR exhibit a relatively higher FFV and SFV. These elastomers, therefore, facilitate the permeation of the challenge chemical. The permachor values were determined by the group contribution method:

$$\pi = \sum \pi_i/n \quad (20)$$

where Π is the polymer permachor, Π_i are the individual segmental values of the backbone and side groups, and n is the number of individual units in the backbone repeat unit. The Π values are plotted against log P_{OA} in Figure 4.
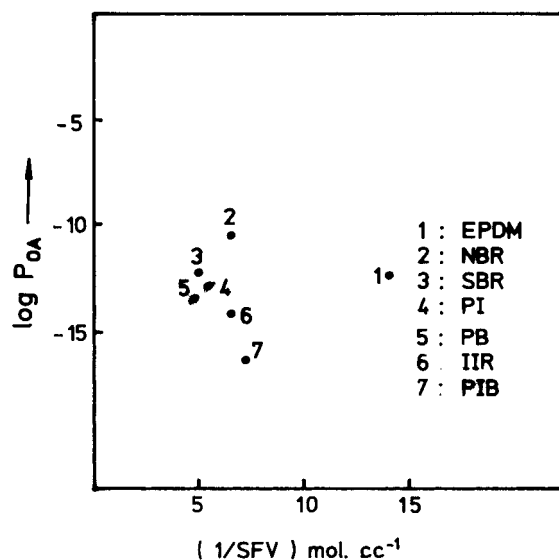

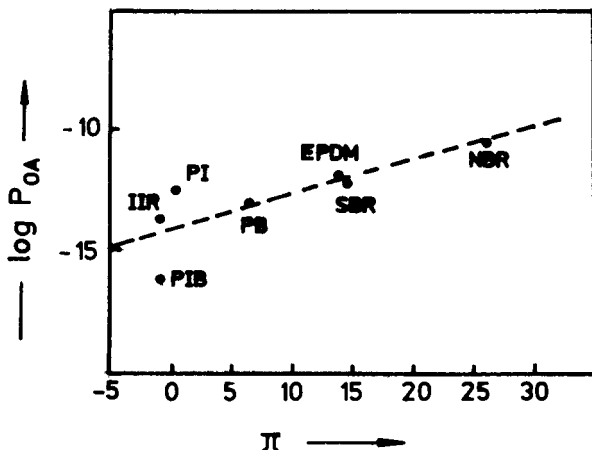**Figure 3**   Relation between log P and 1/SFV.

Figure 4   Relation between log P and Π.

The relation between Π and log P_{OA} is seen to be almost linear, as Π increases, permeability decreases. The higher the value of Π, the stronger are the forces that hold the chains together.[3] It follows therefore that a good barrier material should have a larger Π value. Knowing the molecular structure of an elastomer, it would be possible to calculate its Π value and predict the permeability of OA in it, using Figure 4.

CONCLUSION

A comparison of the diffusivities of SM and its OA indicates that OA diffuses faster in most elastomers. The sorption of SM and OA in butyl rubber was compared in our earlier work,[7] and it was found that while the BTT of OA is lower, its diffusibility and permeability is greater in comparison to SM. Hence, the formulation providing resistance to permeation of OA would also afford protection against SM. Bis(2-chloroethyl)ether or OA can therefore be safely chosen as a suitable model compound of SM for routine testing of protective devices.

The permeation behavior of a polymer–permeant system is a sum total of various factors, such as free volume, cohesive energy density, thermodynamic interaction, and solubility parameters. The theoretical models simplify greatly the selection of barrier materials. For example, for a given barrier application, a critical specific volume and permachor number can be first defined from its barrier requirements. The polymer structures having SFV smaller than the critical values and the required permachor number can

then be identified. These are the polymers that would have the necessary barrier performance. An attempt can also be made to further refine the models through a combination of free volume concepts and molecular theories of diffusion.

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