

Estimation of Permeation Rate of Chemicals Through Elastometric Materials

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ABSTRACT: Elastometric materials are used as barriers to protect workers against exposure to chemicals. The effectiveness of a polymer as a chemical protective material therefore depends on the rate of the permeation of chemicals through it. The permeation rate depends on the solubility and the diffusion coefficient of chemicals in the materials. The diffusion coefficient itself is a strong function of concentration of the chemicals in the polymeric material. Permeation rates can be measured directly using a permeation cell or they can be calculated from the solubility and the diffusion coefficient data. Sorption/desorption experiments can be used to determine solubility and an expression for the diffusion coefficient in terms of concentration. Experiments were conducted for the sorption and desorption of ethyl acetate in three glove (one butyl and two neoprene materials) and two garment (neoprene and chlorinated polyethylene) materials. The data collected were used to estimate the steady-state permeation rates of ethyl acetate through the materials. The results of the experiments show that the solubility of ethyl acetate in butyl rubber is 0.795 g/cm^3 , and the steady-state permeation rate is $0.32 \mu\text{g cm}^{-2} \text{ s}^{-1}$. The solubility of the chemical through the three neoprene materials is in the range of $2.25\text{--}5.31 \text{ g/cm}^3$, and the steady-state permeation rates vary from 27 to $43 \mu\text{g cm}^{-2} \text{ s}^{-1}$. The solubility of ethyl acetate in the chlorinated polyethylene is 7.14 g/cm^3 , and the steady-state permeation rate is $62.43 \mu\text{g cm}^{-2} \text{ s}^{-1}$. The experimental method is very simple to use and it requires a small sample of the material (less than 1 cm^2) and only a few milliliters of the chemical. Sorption/desorption experiments can also provide information on the amount of additives extracted from an elastomeric material during contact with a chemical. © 2000 John Wiley & Sons, Inc. *J Appl Polym Sci* 79: 1265–1272, 2001

Key words: elastometric material; permeation rate; diffusion coefficient; solubility; sorption; desorption

INTRODUCTION

The majority of workers with a potential for skin contact by hazardous materials are protected by utilizing elastomers in a form of chemical protective clothing (CPC). Permeation of chemicals through elastomers has been studied extensively

during the last 20 years. In many of these investigations, an ASTM cell¹ or an equivalent permeation cell^{2,3} is used to determine the breakthrough time and the steady-state permeation rate of chemicals through elastometric materials. In a typical permeation experiment, a sample of an elastomer is exposed to the challenge chemical, and the chemical that is permeated through the material is collected in a collection medium. The rate of permeation is determined by the analysis of the collection medium. The time at which

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the chemical is first detected in the collection medium is called the breakthrough time. Due to the dependence of breakthrough time on the sensitivity of the analytical method, in the latest version of ASTM F739¹ (Standard Test Method for Resistance of Protective Clothing Materials to Permeation by Liquids or Gases Under Conditions of Continuous Contact), it is recommended that a normalized breakthrough time (time at which the permeation rate reaches $0.1 \mu\text{g cm}^{-2} \text{min}^{-1}$ for open-loop systems or the permeation mass reaches a value of $0.25 \mu\text{g/cm}^2$ for closed-loop systems) be reported.

The effectiveness of a polymeric material as a chemical protective clothing material depends on its physical properties (such as tensile strength, tear resistance, burst strength) and the rate of permeation of chemicals through it. An ideal CPC material for a specific chemical is a material that is impermeable to that compound, has desirable physical properties, with low cost, and can be easily fabricated into comfortable clothing. If the permeation rate is low enough, the material could still be considered to be a good barrier against that chemical. The steady-state permeation rate and breakthrough time are the main indicators of chemical resistance of elastomeric materials.

The permeation rate of a chemical through a polymer depends on the solubility and diffusion coefficient of the chemical in the membrane. The permeation process is usually described by Fick's law. If it is assumed that no bulk flow exists, the permeation flux is given by

$$J = -D \frac{dC}{dX} \quad (1)$$

where J is the rate of mass transfer per unit area; D , diffusion coefficient; C , concentration; and X , the distance.

The diffusion coefficient of chemicals through membranes depends on variables such as concentration, temperature, and chemical size and shape.⁴ Theoretical approaches to estimate the diffusion coefficients are generally based on the free-volume theory.⁵ In these models, it is assumed that the mobility of penetrant molecules in a polymer matrix, as well as that of polymer segments, depends on the amount of free volume present in the system. More specifically, the rate of diffusion of a small molecule in a polymer depends primarily upon the ease with which polymer chains can exchange positions with the pen-

etrant molecules. These models have been used primarily for polymer-gas systems, and their application for solute-rubbery polymer systems has produced less than accurate results.^{6,7} Because of the limitations of theoretical models for predicting the diffusion coefficient for rubbery polymer-liquid systems, empirical correlations have been developed. Southern and Thomas⁸ studied the diffusion of various liquids in natural rubber, at 25°C, and concluded that the diffusion coefficient depends mainly on the liquid viscosity. Schwöpe and Reid⁹ investigated the correlation of the diffusion coefficient with properties representative of the solute size and shape. Their best correlations were with molecular weight. Vahdat¹⁰ studied several organic liquid-elastomer systems and showed that diffusion coefficients depend mainly on the kinematic viscosity.

In all these studies, the effect of concentration on the diffusion coefficient was not considered and, in fact, an average diffusion coefficient (for the range of concentration in the polymer) was found. The purpose of this study was to use the results of sorption/desorption experiments to develop expressions for the diffusion coefficient as a function of concentration. The expressions are then used to predict the steady-state permeation rate.

THEORY

When a liquid comes into contact with a polymeric material, the molecules of the chemical first enter the material (this is known as the solution); next, they move through the solid by molecular diffusion. In the final step, the molecules desorb at the exit face of the material. The permeation rate of chemicals through polymers therefore depends on the solubility and diffusion coefficients of the permeating species in the material. The one-dimensional diffusion process in a membrane is usually described by the following expression:

$$\frac{\partial C}{\partial t} = \frac{\partial}{\partial x} \left(D \frac{\partial C}{\partial x} \right) \quad (2)$$

where t is the time. In general, the diffusion coefficient, D , is a function of concentration.⁵ The presence of diffusing molecules in a polymer increases the free volume and therefore has a direct effect on the diffusion coefficient. There is also a possibility of specific interaction between the dif-

fusing molecules and polymer segments, which affect the component mobility. At low concentrations, D is independent of concentration, and eq. (2) is reduced to

$$\frac{\partial C}{\partial t} = D \frac{\partial^2 C}{\partial x^2} \tag{3}$$

Equation (3) can be solved for the desorption of a chemical from a plane sheet of thickness, l , initially at a uniform concentration, whose surface concentrations are suddenly brought to zero at time equal to zero⁵:

$$\frac{M_t}{M_{\text{inf}}} = 4 \left(\frac{Dt}{l^2} \right)^{1/2} \left[\frac{1}{\pi^{1/2}} + 2 \sum_{n=0}^{\infty} (-1)^n \text{ierfc} \frac{nl}{2(Dt)^{1/2}} \right] \tag{4}$$

where M_t is the total mass desorbed from the sheet in a time t , and M_{inf} is the total mass desorbed after infinite time. For small values of t , eq. (4) is reduced to

$$\frac{M_t}{M_{\text{inf}}} = \left[\frac{16D}{l^2\pi} \right]^{1/2} t^{1/2} \tag{5}$$

In studies of the molecular transport of liquids into polymers, diffusion has been classified as Fickian and non-Fickian.¹¹ The diffusion processes that follow eq. (5) are called Fickian diffusion. [In the non-Fickian diffusion, the mass desorbed (or uptake) is not proportional to the square root of time; instead, it is proportional to t^n , where the value of n can be less than or greater than 0.5.¹² Equation (5) shows that for systems in which the diffusion coefficient is constant a plot of M_t/M_{inf} versus $t^{1/2}$ gives a straight line. The diffusion coefficient can therefore be calculated from the slope of the line. Most polymer-chemical systems, however, have diffusion coefficients that are concentration-dependent.⁵ For these cases, the initial gradient of the desorption curve gives some mean value for the diffusion coefficient:

$$\frac{M_t}{M_{\text{inf}}} = \left[\frac{16\bar{D}}{l^2\pi} \right]^{1/2} t^{1/2} \tag{6}$$

where \bar{D} is an average diffusion coefficient over the range of concentration from zero to C_0 (equilibrium concentration) that can be represented by

$$\bar{D} = \frac{1}{C_0} \int_0^{C_0} D dC \tag{7}$$

The dependence of the diffusion coefficient on the concentration of the permeating species is usually represented by the following equation⁵:

$$D = D_0 e^{\beta C} \tag{8}$$

where β is a constant, and D_0 , the diffusion coefficient as concentration approaches zero. Substitution of eq. (8) into (7) and integration yields

$$\bar{D} = \frac{D_0}{C_0\beta} [e^{\beta C_0} - 1] \tag{9}$$

Equation (4) can also be written in the following form⁵:

$$\frac{M_t}{M_{\text{inf}}} = 1 - \frac{8}{\pi^2} \sum_{m=0}^{\infty} \frac{1}{(2m+1)^2} \times \exp \left[- \frac{D(2m+1)^2\pi^2 t}{l^2} \right] \tag{10}$$

For large values of t (concentration of permeating species in the polymer approaches zero), all the exponential terms except the first one can be neglected:

$$\frac{M_t}{M_{\text{inf}}} = 1 - \frac{8}{\pi^2} \exp \left(- \frac{D_0\pi^2 t}{l^2} \right) \tag{11}$$

where D_0 is the diffusion coefficient as the concentration approaches zero. This equation can be rearranged to

$$\ln(M_{\text{inf}} - M_t) = \ln \left(\frac{8M_{\text{inf}}}{\pi^2} \right) - \frac{D_0\pi^2 t}{l^2} \tag{12}$$

Equation (12) shows that for large values of t a plot of $\ln(M_{\text{inf}} - M_t)$ versus t gives a straight line with a slope of θ :

$$\theta = - \frac{D_0\pi^2}{l^2} \tag{13}$$

The diffusion coefficient as concentration approaches zero, D_0 , therefore can be calculated from eq. (13).

Table I Materials Tested

Material	Manufacturer	Nominal Thickness (mm)
Butyl rubber (glove)	North (Charleston, SC)	0.81
Neoprene (glove)	Standard Safety (Palatine, IL)	0.71
Neoprene (glove)	Guardian (Willard, OH)	0.81
Neoprene (garment)	Respirex (Surrey, England)	0.41
Chlorinated polyethylene (garment)	Standard Safety (Palatine, IL)	0.58

The diffusion coefficient of polymer–chemical systems as a function of concentration can be determined if D_0 and β are known. These parameters can be calculated from desorption experiments as follows:

1. \bar{D} is calculated from the slope of the linear part of the graph of M_t/M_{inf} versus $t^{1/2}$.
2. D_0 is calculated from the slope of the graph of $\ln(M_{\text{inf}} - M_t)$ versus t (for large values of t).
3. β is calculated from eq. (9).

The steady-state rate of permeation of a chemical through a polymeric membrane can be found by the substitution of eq. (8) into (1) and the integration of the resulting expression:

$$J = \frac{D_0}{\beta l} [e^{\beta C_0} - 1] \quad (14)$$

EXPERIMENTAL

Materials

The protective clothing materials, manufacturers, and nominal thicknesses are shown in Table I. Three glove materials and two garment materials were tested against ethyl acetate. For the garment materials, samples were cut from a sheet of material provided by the manufacturers. For the glove materials, samples were cut from the palm of a glove. All the samples were cut using a cutter with a diameter of $\frac{3}{8}$ in. (9.52 mm). Ethyl acetate from Fisher Scientific (Norcross GA) was used without additional purification.

Procedure

To conduct an experiment, a specimen was cut from a material and washed with Ivory soap and dried. The diameter and thickness of the speci-

men were measured at several locations using a micrometer. The specimen was then weighed and placed in a 10-mL container. Approximately 2 mL of the test liquid was added to the container and covered and stored in a fume hood for at least 72 h, at a temperature of $25 \pm 2^\circ\text{C}$.

Desorption experiments were conducted using a thermogravimetric analyzer (Model 2050, TA Instruments, Inc.). The instrument was calibrated according to the manufacturer's instruction. The atmosphere around the specimen holder was purged with nitrogen. The instrument's furnace was preheated to 30°C . The specimen was then removed from the container and patted dry on both sides with lint-free wipes. The specimen was then placed onto the specimen holder, the balance assembly was closed, and the automated recording of weight loss versus time was started. The experiments were continued until no change in the mass of the specimen was noticed. Desorption time for all the five materials are given in Table II. At least two desorption experiments were conducted for each elastometric material.

RESULTS AND DISCUSSION

The solubility of ethyl acetate in the five elastometric materials is given in Table III. Solubility is based on the volume of the dry sample (weight

Table II Desorption Time

Material	Desorption Time (h)
Butyl rubber (North)	30
Neoprene (Standard Safety)	13
Neoprene (Guardian)	11
Neoprene (Respirex)	13
Chlorinated polyethylene (Standard Safety)	13

Table III Solubility of Ethyl Acetate in CPC Materials

Material	Solubility (g/cm ³)	% Solubility (g/g)
Butyl rubber (North)	0.795	11.2
Neoprene (Standard Safety)	5.31	57.5
Neoprene (Guardian)	3.59	39.9
Neoprene (Respirex)	2.25	21.8
Chlorinated polyethylene (Standard Safety)	7.14	81.5

uptake/volume of the dry sample), and percent solubility is defined as [100(weight uptake/weight of the dry sample)]. The solubility of ethyl acetate in chlorinated polyethylene was greater than that of all the other materials (81.5% increase in weight). Butyl had the lowest solubility with a 11.2% weight increase.

The results of the desorption experiments are presented in Figures 1–5. Percent weight loss is defined as

$$\% \text{ Weight loss} = \frac{W_t}{W_0} (100) \quad (15)$$

where W_0 is the weight of sample plus the chemical before the start of the desorption experiment (time zero), and W_t , the weight at time t . The weight of the samples was measured automatically every few seconds. Each weight-loss curve in these figures therefore represents thousands of actual weight measurements. It is clear that de-

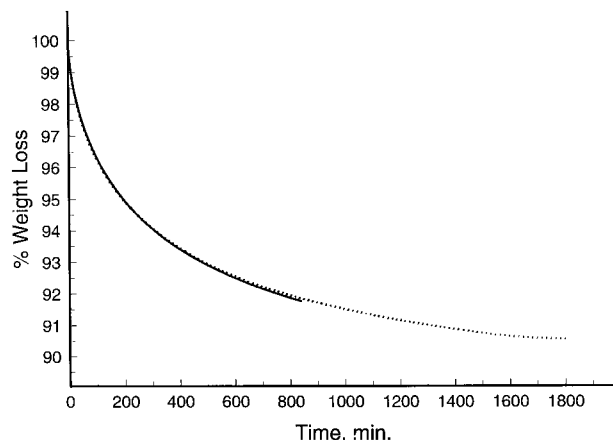


Figure 1 Two replicates of desorption curves for ethyl acetate in butyl (North).

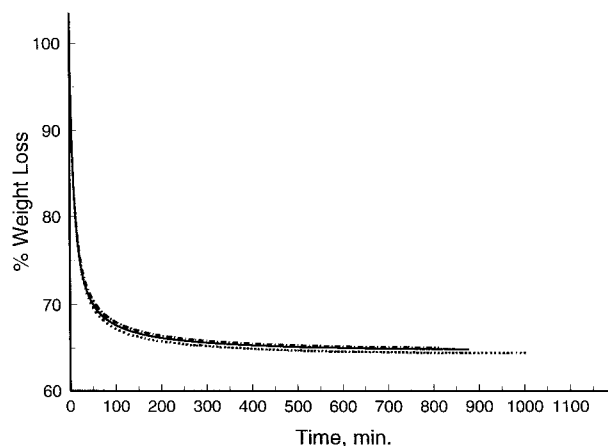


Figure 2 Three replicates of desorption curves for ethyl acetate in neoprene (Standard Safety).

sorption proceeds very quickly in the first few minutes, and then the rate of desorption decreases as the concentration of the chemical in the material decreases. This trend continues until most of the molecules of the chemical have been desorbed from the polymer.

Figure 6 represents plots of M_t/M_{inf} versus $t^{1/2}$ for neoprene (Standard Safety). Similar plots were prepared for the other four materials. As noted in the Theory section, the linear initial curve indicates that diffusion followed a Fickian process. The curves become concave for M_t/M_{inf} values ranging from 0.6 to 1, which is typical for Fickian sorption/desorption and is due to the alterations in the value of the diffusion coefficient beyond this limit. From the slope of the linear portion of the curves, the diffusion coefficient of ethyl acetate through each elastometric material

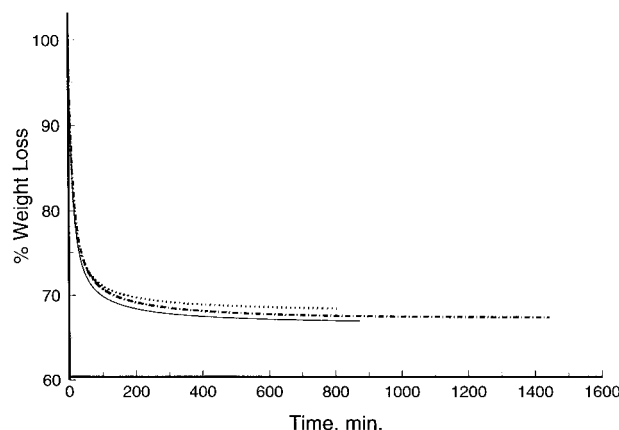


Figure 3 Three replicates of desorption curves for ethyl acetate in neoprene (Guardian).

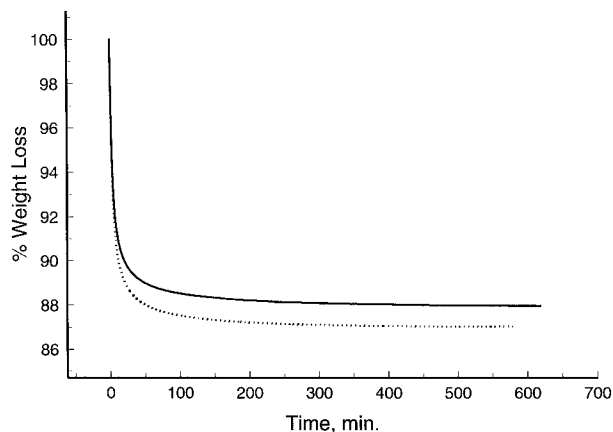


Figure 4 Two replicates of desorption curves for ethyl acetate in neoprene (Respirex).

(\bar{D}) was calculated and the results are presented in Table IV. The diffusion coefficients given in this table are the average values from two or three desorption experiments. Diffusion coefficients range from 3.3×10^{-8} cm²/s (butyl) to 6.2×10^{-7} cm²/s (neoprene, Guardian). Ranking of the materials in terms of the diffusion coefficient does not match the order of solubility. The highest solubility is observed for chlorinated polyethylene, whereas the highest diffusion coefficient is exhibited by neoprene (Guardian). This type of behavior has been reported for other polymeric material/chemical systems.¹³ The diffusion coefficients calculated from M_t/M_{inf} versus $t^{1/2}$ curves are actually average values over a concentration range of 0 to C_0 (solubility of chemical in the polymer) as indicated by eq. (7).

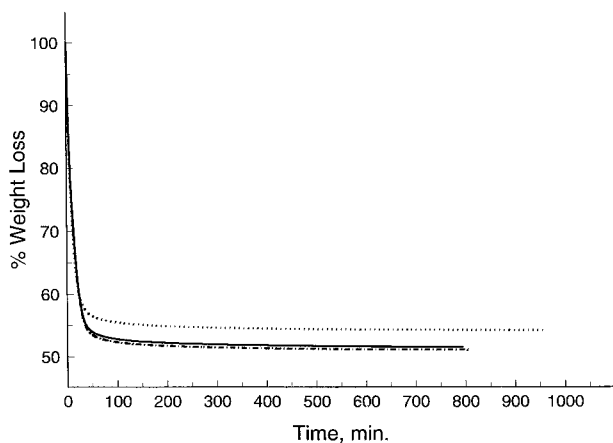


Figure 5 Three replicates of desorption curves for ethyl acetate in chlorinate polyethylene (Standard Safety).

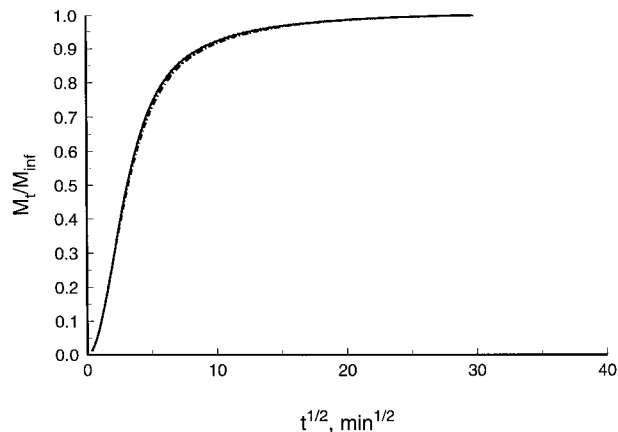


Figure 6 Three replicates of M_t/M_{inf} versus $t^{1/2}$ for ethyl acetate in neoprene (Standard Safety).

Figure 7 represents plots of $\ln(M_{inf} - M_t)$ versus t for neoprene (Standard Safety). Similar plots were prepared for the other four elastomers. The curves become linear for large values of t , which corresponds to low concentrations of the chemical in the polymer. As indicated in the Theory section, the slope of the linear portion of the curves can be used to determine the diffusion coefficient as concentration approaches zero (D_0). The results of calculations for all the five elastomers are presented in Table IV. The values of D_0 for the three neoprene materials and chlorinated polyethylene are more than one order of magnitude less than are the corresponding values of \bar{D} . This shows the strong dependence of the diffusion coefficient on the concentration. The value of D_0 for butyl is only about 40% less than its value for \bar{D} . This should be expected because the equilibrium concentration of ethyl acetate in butyl (solubility) is small compared to the other four materials. Consequently, the difference between the diffusion coefficient at the equilibrium concentration and at low concentration should also be small.

The diffusion coefficient data collected for the desorption of ethyl acetate from the five elastomers were used to compute the constant β . For each elastomer, values of D_0 and \bar{D} were used in eq. (9) to calculate β . This equation is nonlinear and should be solved by computer. MathCAD (Mathsoft, Inc.) software was used to solve the equation, and the results are given in the last column of Table IV.

The steady-state permeation rate of ethyl acetate through the materials was then calculated from eq. (14), and the results are presented in

Table IV Diffusion Coefficient of Ethyl Acetate in CPC Materials

Material	\bar{D} (cm ² /s)	D_0 (cm ² /s)	β
Butyl rubber (North)	3.3×10^{-8}	2.2×10^{-8}	0.96
Neoprene (Standard Safety)	5.5×10^{-7}	1.9×10^{-8}	0.94
Neoprene (Guardian)	6.2×10^{-7}	2.0×10^{-8}	1.4
Neoprene (Respirex)	4.8×10^{-7}	9.3×10^{-9}	2.5
Chlorinated polyethylene (Standard Safety)	5.3×10^{-7}	1.1×10^{-8}	0.78

Table V. The permeation rates range from 0.32 to 75 $\mu\text{g cm}^{-2} \text{s}^{-1}$. As expected, butyl exhibits the lowest permeation rate (0.32 $\mu\text{g cm}^{-2} \text{s}^{-1}$). No experimental data for the permeation of ethyl acetate through the 32-mil butyl (North) is available in the literature. However for a 17-mil butyl glove by the same manufacturer, the measured steady-state permeation rate of ethyl acetate is 0.34 $\mu\text{g cm}^{-2} \text{s}^{-1}$.¹⁴ Using eq. (14), the steady-state permeation rate of ethyl acetate through a 17-mil (0.043-cm) butyl is calculated to be 0.61 $\mu\text{g cm}^{-2} \text{s}^{-1}$, which is a reasonable estimation of the measured permeation rate. The steady-state permeation rate of ethyl acetate through neoprene (Guardian) was reported to be greater than 8.3 $\mu\text{g cm}^{-2} \text{s}^{-1}$.¹⁵ The result of this study gives a value of 27 $\mu\text{g cm}^{-2} \text{s}^{-1}$.

The results of this study show that sorption/desorption experiments can be used to estimate permeation rates of chemicals through elastomeric materials. This is an estimation method, and, obviously, the results will not be as accurate as are measuring the permeation rate in a permeation cell. This method can be used for screening potential polymeric materials. Sorption/de-

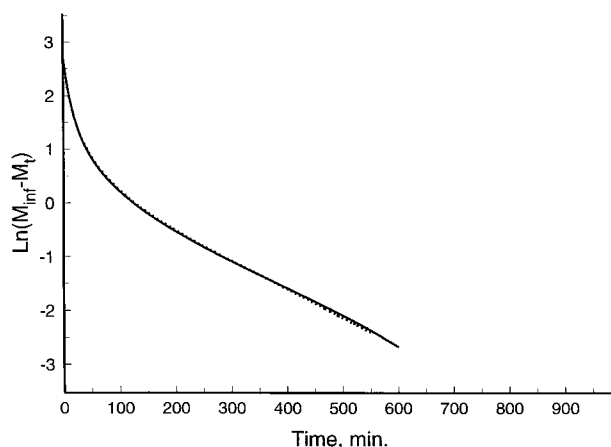


Figure 7 Three replicates of $\ln(M_{\text{inf}} - M_t)$ versus t for ethyl acetate in neoprene (Standard Safety).

sorption experiments are very simple, and each experiment requires only a small sample of the material (less than 1 cm²) and a few milliliters of the challenge chemical. This is an important factor. Chemical waste disposal is a major problem for industry. The Environmental Protection Agency (EPA) has suggested that source reduction should be the top priority for any waste-management program.¹⁶ Industrial hygienists and other professionals can reduce waste generation by using this method. The method also provides an expression for the diffusion coefficient as a function of concentration of the challenge chemical, which can be used to predict the steady-state permeation rate at any concentration of the chemical.

Sorption/desorption experiments can also provide information on the amount of additives extracted from a polymer during contact with a chemical. The weight of additives extracted is given by the following equation:

$$\text{Weight extracted} = W - W_f \quad (16)$$

where W is the weight of the dry specimen (before immersion in a chemical), and W_f , the weight of specimen at the end of the desorption experiment. The percent extraction for the five elastomers tested is given in Table V. Fillers are usually added to elastomeric materials to improve their physical properties. For example, carbon black is added to natural rubber and related materials in order to improve tear and abrasion resistance, resilience, and tensile strength.¹⁷⁻¹⁹ The amount of fillers extracted from an elastomer during exposure to a chemical is useful information that industrial hygienists and other professionals can use during the CPC material-selection process.

CONCLUSIONS

Diffusion and permeation characteristics of elastomeric materials to liquids were studied. Sorp-

Table V Steady-state Permeation Rate and Percent Extraction for Ethyl Acetate/Polymeric Material Pairs

Material	Permeation Rate ($\mu\text{g}^{-1} \text{cm}^{-2} \text{s}^{-1}$)	% Extraction
Butyl rubber (North)	0.32	4.9
Neoprene (Standard Safety)	43	7.3
Neoprene (Guardian)	27	14
Neoprene (Respirex)	35	3.4
Chlorinated polyethylene (Standard Safety)	62	25

tion/desorption experiments were used to determine solubility and an expression for the diffusion coefficient (as a function of concentration) in polymeric materials. Experiments were conducted for the sorption and desorption of ethyl acetate in five commercially available glove and garment materials. The calculated solubility and diffusion coefficients were then used to estimate the steady-state permeation rates. The calculated values of the permeation rates are in agreement with the experimental data.

Sorption/desorption experiments are easily conducted, and they require only a small sample of material (less than 1 cm^2) and a few milliliters of a chemical. Reduction of chemical waste should be an important incentive for the professionals to use this method for the selection of protective clothing.

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