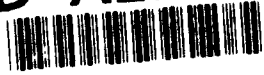


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# Diffusion and Sorption of 1,4-Dioxane Into Polychloroprene

Geralda Severe

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February 1994



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## Introduction

Insufficient agreement between the droplet permeation model (1) and the experimental data for the permeation of di-isopropylmethylphosphonate (DIMP) in Neoprene (2) prompted inquiry about the diffusion behavior for the mentioned polymer/solvent system. However, there is an absence of information in the literature to verify the assumption that the diffusion coefficient of DIMP in the DIMP/Neoprene system was independent of concentration. As such, the difficulty justifying the discrepancy between theory and experiment prompted the experimental study of the DIMP/Neoprene system.

Among the methods available to obtain the diffusion coefficient, vapor sorption is the best for investigating concentration dependence. Unfortunately, the vapor sorption apparatus at the U.S. Army Research Laboratory (ARL) in Watertown, MA was not able to provide the ballast volume needed to maintain constant vapor pressure of DIMP during the sorption or desorption runs because of the extremely low vapor pressure of DIMP. To obtain the necessary information without going through the redesigning of the apparatus, the same study was done with 1,4-dioxane which has a high vapor pressure than DIMP. The magnitudes in the weight uptake by the extracted Neoprene with both solvents were similar enough to make it a plausible substitution.

## Experimental

Experiments (immersion and vapor sorption) were carried on Neoprene samples with a nominal thickness of 0.0305 and 0.0356 cm (12 and 14 mils) that were compounded by Smithers Scientific Services of Akron, OH. To extract the plasticizer (Diester of Triethylene Glycol), which constituted about 9% weight of the total amount of ingredients present (see Table 1) (2), a sample of Neoprene was immersed in toluene that was constantly stirred for one week. The Neoprene was then dried in a vacuum oven at 50°C until it reached an equilibrium weight. An average weight loss of 12.86% was recorded at the end of the extraction process. Two specimens were made from this extracted neoprene; one was used for the vapor sorption studies and the other to obtain the equilibrium weight gain by immersing it into 1,4-dioxane at 30°C.

### Liquid Immersion

The weight changes of Neoprene during liquid immersion experiments were measured by microbalance after removing the sample from the solvent and blotting it dry as quickly as possible. The weight changes were recorded on the second, tenth, and the thirty-fourth day for the extracted sample for an overall period of thirty-four days. The unextracted (as received) sample was weighed after thirteen, twenty-six, and forty-eight days. The duration of the immersion experiments was forty-eight days for the unextracted (as received) sample. At the end, the equilibrium weight gain for the unextracted sample was 79% while the extracted sample was 69%.

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Table 1. Smithers No. L0831 (Neoprene)

Ingredient	PHR	Grams
Neoprene W	100	760
Scorchguard O	4	30.4
Stearic Acid	0.5	3.8
Wingstay 100 AZ	2	15.2
Age Rite Stalite S	1	7.6
N-774 Carbon Black	65	494.4
Van Wax H	1	7.6
Plasticizer	18	136.8
Darvan ME	1	7.6
Polyethylene AC617	1	7.6
Zinc Oxide	5	38.0
Ackrochem ETU-22 PM	0.67	5.1
Methyl Tuads	0.5	3.8
Totals	199.67	1517.5

### Vapor Sorption

The weight uptake of 1,4-dioxane into the unextracted (as received) and extracted neoprene was measured using the incremental vapor sorption method. The sample was suspended from a helical quartz spring in a temperature controlled glass vacuum apparatus. The solvent uptake was measured as a function of time by measuring the extension of the spring with a cathetometer. The spring extension was followed until there was no further change in its length. This procedure was repeated successfully to an activity of 0.7 ( $a = \text{partial pressure/saturation pressure}$ ). At higher activities ( $a = 0.8$  and  $0.9$ ) the experiments were interrupted because it was difficult for the spring to reach an equilibrium deflection (see Figures 1, 2, and 3). For the purpose of data analysis, the equilibrium deflection in those cases was taken as the height reached by the suspended sample after 20 to 31 minutes from the start of the experiment before occurrence of the fluctuation (see Figures 4, 5, and 6). Once the sorption process was completed it was immediately followed by a desorption process over the same range of pressure. As during the sorption process, the displacement and the pressure were measured as a function of time until an equilibrium length of the spring was achieved. Again, the changes in length were converted into weight loss. The results for the desorption process are not reported in this paper.

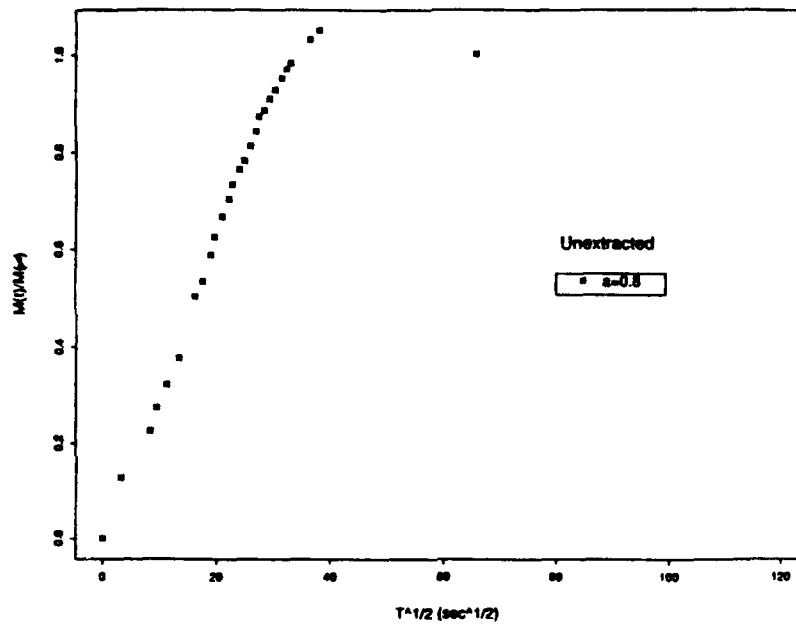


Figure 1. Sorption curves for Neoprene (unextracted) at  $a = 0.8$ .

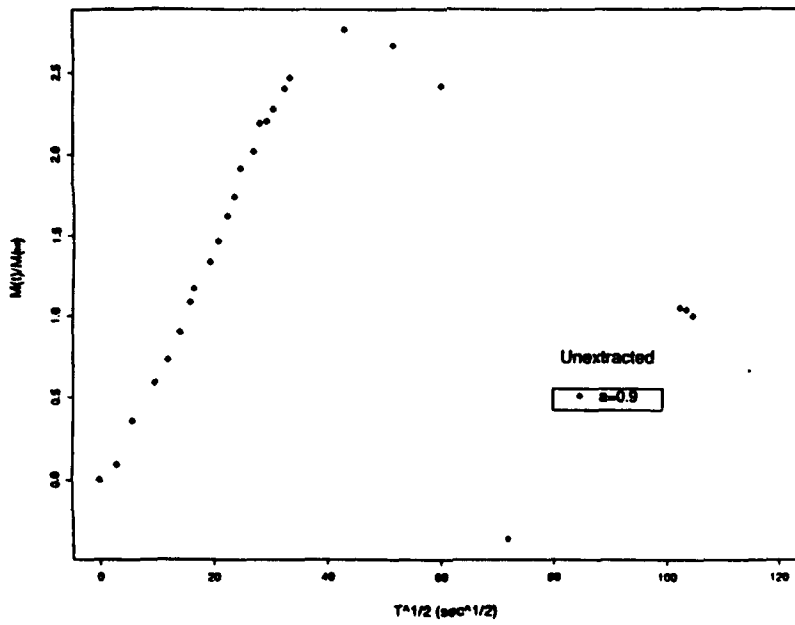


Figure 2. Sorption curves for Neoprene (unextracted) at  $a = 0.9$ .



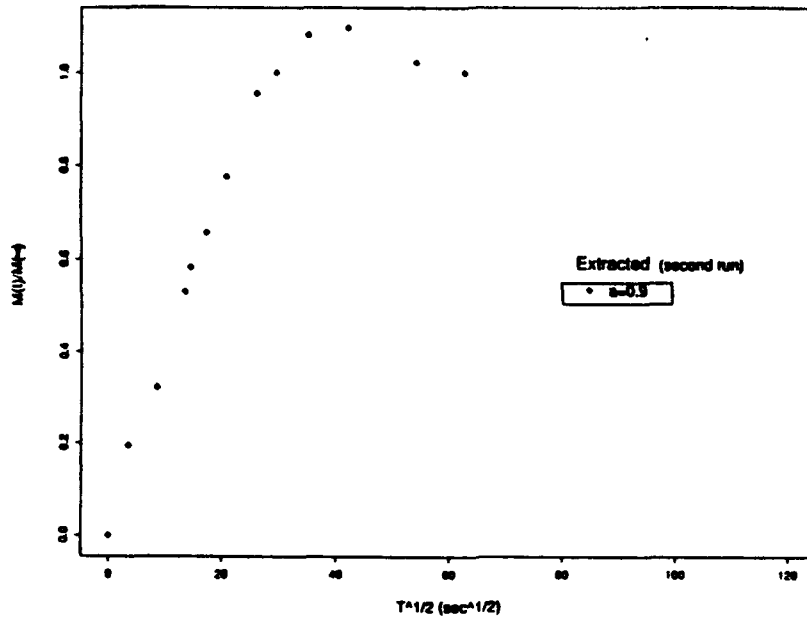


Figure 3. Sorption curves for Neoprene (extracted) at  $a = 0.9$  (second run).

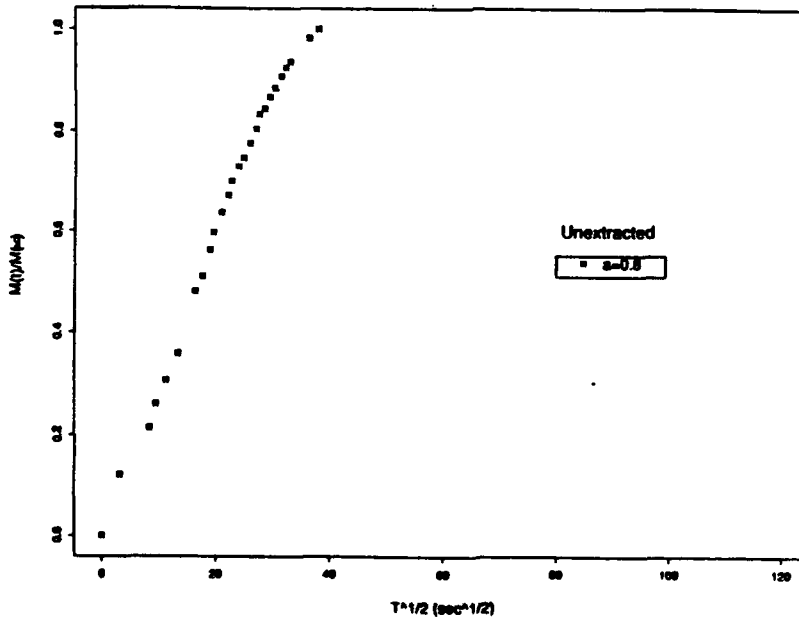


Figure 4. Sorption curves for Neoprene (unextracted) at  $a = 0.8$ .

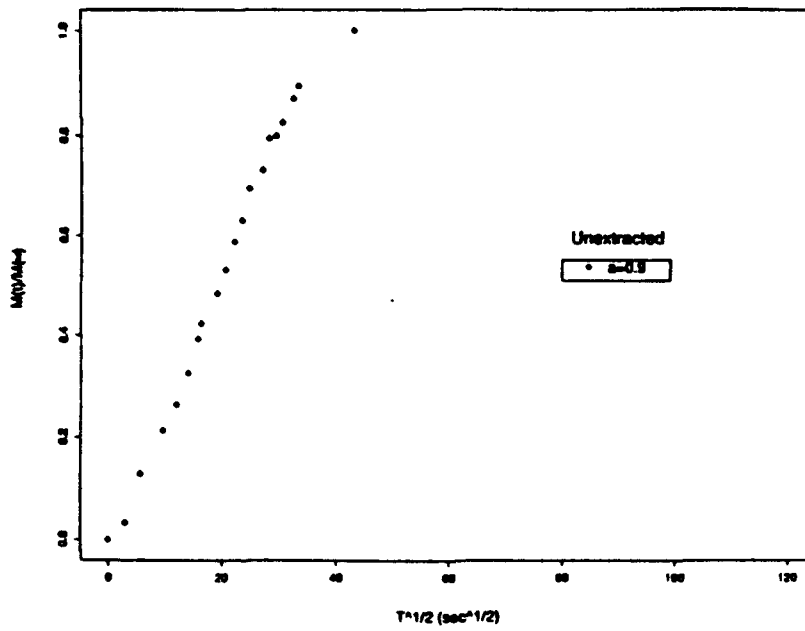


Figure 5. Sorption curves for Neoprene (unextracted) at  $a = 0.9$ .

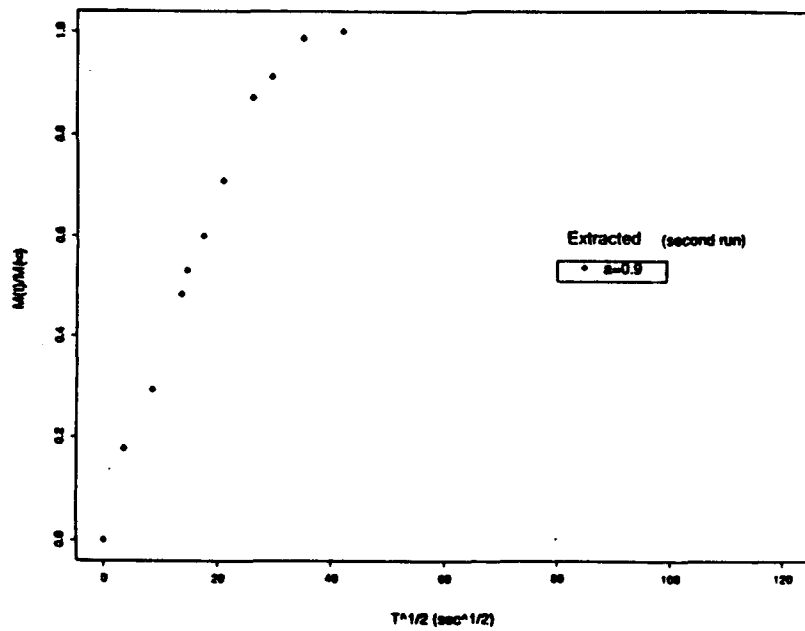


Figure 6. Sorption curves for Neoprene (extracted) at  $a = 0.9$  (second run).

## Results and Discussion

### Sorption Curve Analysis

Data obtained from the sorption experiments were plotted as  $M(t)/M(\infty)$  (fractional weight uptake) versus square root of time (see Figures 7, 8, and 9), also known as sorption curves, where  $M(t)$  represents the weight absorbed by the sample at time  $t$ , and  $M(\infty)$  represents the equilibrium vapor absorbed. The sorption curves were analyzed to determine whether they exhibited Fickian behavior, which would be indicated when the sorption curves are linear in the initial stage (up to 60% or more) and concave to the time axis at higher sorption amounts. Furthermore, the results for different thickness must overlap when plotted as  $M(t)/M(\infty)$  versus  $t^{1/2}/L$  (3).

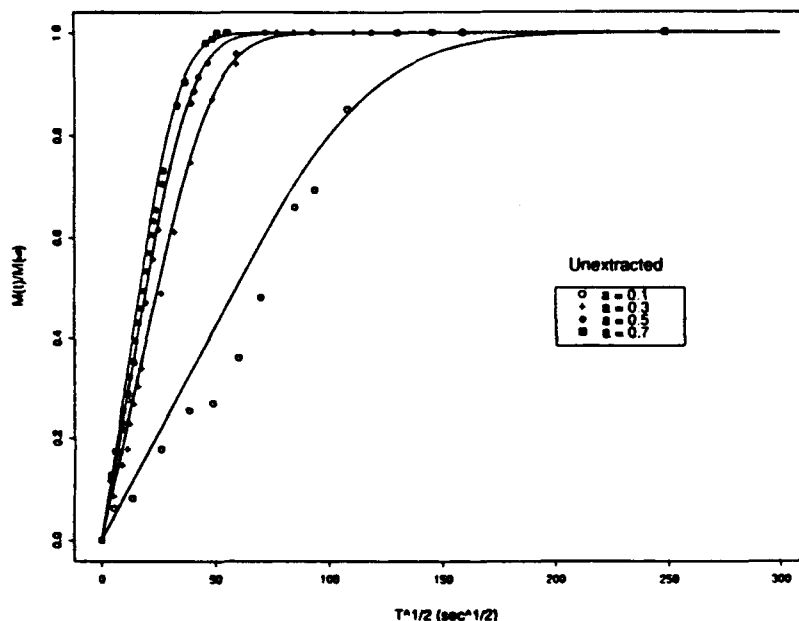


Figure 7. Sorption curves for the unextracted Neoprene at different activity.

The vapor sorption curves for the extracted and the unextracted Neoprene samples with 1,4-dioxane had shown the first two characteristics of a Fickian behavior. They were linear in the early stage and were concave to the time axis beyond the linear portions (see Figures 7, 8, and 9). Since the experiments were not performed at different thicknesses, a conclusion on Fickian behavior cannot be made. Comparison of the sorption curves for the unextracted (as received) sample with the sorption curves obtained by Khinnavar and Aminabhavi (4) had shown a different behavior. Their reported results for the immersion tests of 1,4-dioxane at 25°C demonstrate an initial sigmoidal shape which could result from the fact that their test samples were thicker (thickness range of 0.190 and 0.245 cm). The response of the polymer to stresses induced by swelling due to the sorption of solvent may be slower for a thicker material than for a thinner material.

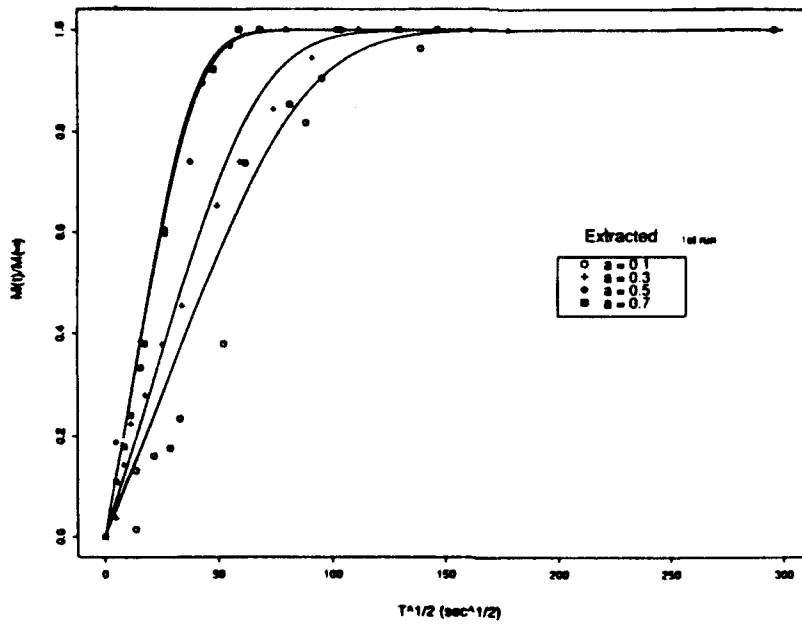


Figure 8. Sorption curves for the unextracted Neoprene at different activity (first run).

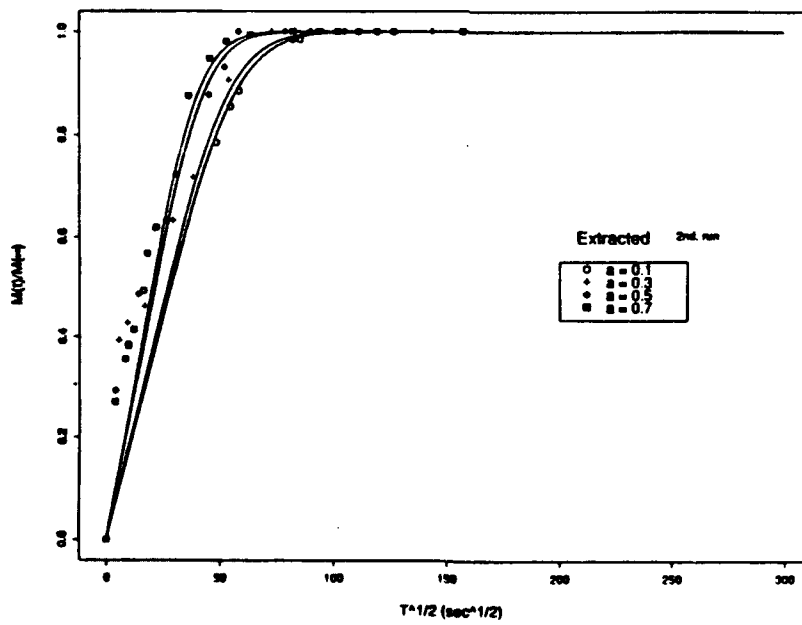


Figure 9. Sorption curves for the unextracted Neoprene at different activities (second run).

To ascertain the results obtained for the extracted analysis, repeated sorption experiments were done with the same sample. Repeated sorption cycling did not affect the sorption behavior. However, the rate of weight uptake was not identical for the different runs. The rate was slower for the first exposure and faster with each new cycle (see Figure 10). This behavior was not anticipated. Both Neoprene extracted and as received had also shown that the rate uptake increases with activity which is an indication that the diffusion constant is a function of concentration.

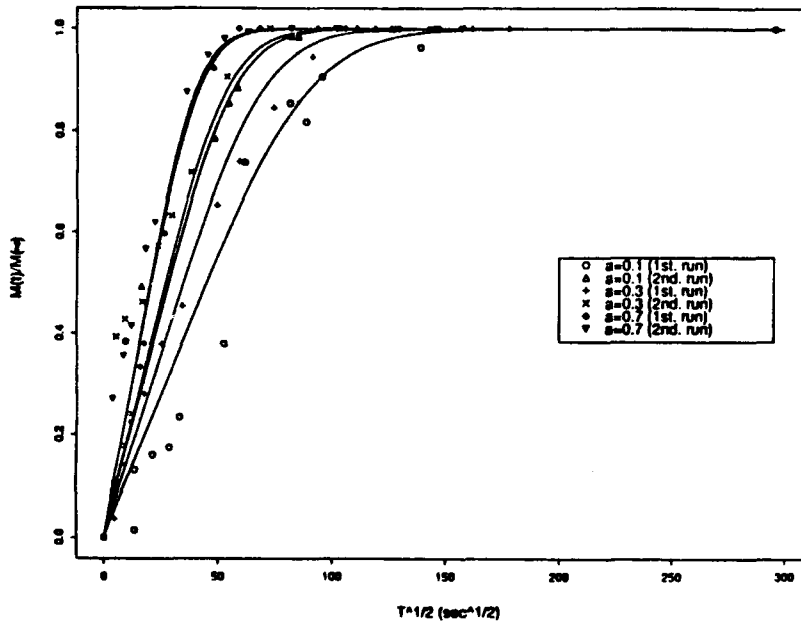


Figure 10. Comparison of the two sets of data obtained at different activities for the same extracted Neoprene sample.

Comparison of the sorption rate uptake for the extracted with the unextracted (as received) samples demonstrated mixed behavior. At lower activity; i.e., at  $a = 0.1$  and  $0.2$ , the uptake for the extracted sample was faster and at  $a = 0.3$  and higher the opposite occurred (see Figure 11). The expectations were that the presence of plasticizer would facilitate the sorption thereby speed up the uptake. No plausible explanation had been reached on this particular behavior.

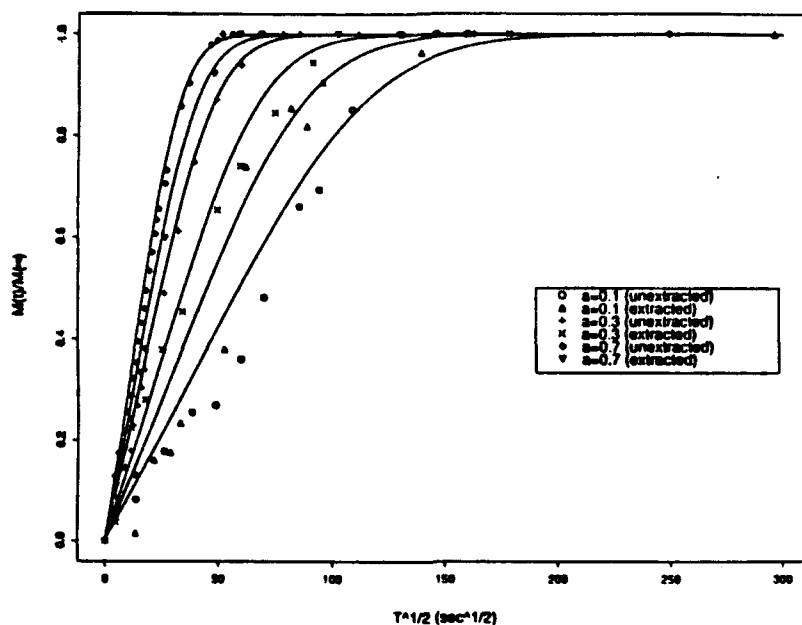


Figure 11. Investigation of the influence of plasticizer on the sorption curves at different activities.

### Diffusion Coefficients

Although the diffusion coefficient ( $D$ ) during each sorption step was considered to be constant, taken together it was a function of concentration. To obtain a numerical value for the diffusion coefficient, since the sorption curves were linear up to 60% of concentration, the half-time method was used to obtain a value for the diffusion coefficient to substitute into:

$$\frac{M(t)}{M(\infty)} = 1 - \frac{8}{\pi^2} \sum_{m=0}^{\infty} \frac{1}{(2m+1)^2} \exp\left(\frac{-Dt(2m+1)^2 \pi^2}{l^2}\right) \quad (1)$$

where  $M(t)$  and  $M(\infty)$  were as defined earlier,  $D$ , the diffusion coefficient and  $l$ , the thickness of the dry sample (5). The fractional weight of uptake acquired from Equation 1 was plotted versus square root of time (see Figures 9, 10, and 11). The solid lines on the sorption curves, as shown in Figures 9, 10, and 11, represent the results of the above equation. However, the diffusion coefficients obtained from the above equation were an approximation of a mean diffusion coefficient in the sorption interval.

Duda, et al. (6), had shown that the actual  $D(c)$  can be set equal to the mean diffusion coefficient ( $\bar{D}(c)$ ) with minor error at a prescribed concentration interval, with  $\bar{D}$  depending upon whether  $D$  increased or decreased with concentration. In a sorption experiment, if  $D$  was an increasing function of concentration,  $k$  in Equation 2 was set to 0.7 (0.56 otherwise). As seen from Tables 2 through 4, the diffusion coefficient increased with concentration; therefore,  $k = 0.7$  was used to calculate the prescribed concentration ( $c$ ) which was sometimes referred to as the adjusted concentration:

$$c = c_i + k(c_f - c_i). \quad (2)$$

Table 2. Unextracted Neoprene sample

Activity coefficient	Concentration (mg solvent/mg polymer) x 100	Adjusted concentration (defined in Equation 2)	Diffusion coefficient $D \times 10^8$ (cm <sup>2</sup> /sec)
0.1	1.34	0.94	1.00
0.2	2.21	1.95	3.47
0.3	3.91	3.40	8.92
0.4	5.91	5.31	11.7
0.5	8.46	7.70	15.5
0.6	11.69	10.72	20.8
0.7	16.18	14.83	19.2
0.8	27.32	23.98	25.0
0.9	34.96	32.67	18.3

Table 3. Extracted Neoprene sample (first run)

Activity coefficient	Concentration (mg solvent/mg polymer) x 100	Adjusted concentration (defined in Equation 2)	Diffusion coefficient $D \times 10^8$ (cm <sup>2</sup> /sec)
0.1	1.125	0.788	2.65
0.2	1.73	1.55	4.25
0.3	3.05	2.65	5.05
0.4	4.54	4.09	5.87
0.5	5.33	5.09	13.2
0.6	6.30	6.01	10.8
0.7	7.36	7.04	12.5
0.8	8.37	8.067	33.3
0.9	11.53	10.582	16.5

Table 4. Extracted Neoprene sample (second run)

Activity coefficient	Concentration (mg solvent/mg polymer) x 100	Adjusted concentration (defined in Equation 2)	Diffusion coefficient $D \times 10^8$ (cm <sup>2</sup> /sec)
0.1	0.821	0.57	6.67
0.2	1.27	1.14	7.17
0.3	1.99	1.77	7.42
0.4	2.75	2.52	10.5
0.5	3.57	3.32	11.7
0.6	4.65	4.33	11.7
0.7	5.65	5.35	13.3
0.8	10.28	9.41	18.3
0.9	11.73	11.30	26.7

The plot of  $D$  versus concentration for the unextracted sample (see Figure 12) increased rapidly at low concentration then tended to level off. Extrapolation for the zero concentration diffusion coefficient was found to be about  $4.0E-9$  cm<sup>2</sup>/sec. This value is lower than the diffusion coefficient at  $c = 0$  ( $D_0$ ) for natural rubber in toluene which was  $7.8E-8$  cm<sup>2</sup>/sec (7) and higher than the diffusion coefficient for butyl rubber in toluene ( $1.2E-9$  cm<sup>2</sup>/sec) (8). The values obtained for the diffusion coefficient in the analysis of the extracted sample display a rapid increase of diffusion coefficient as activity increases. However, these data seem to have larger errors than the results for the unextracted sample. The values obtained for the two sets of data for the extracted Neoprene were difficult to interpret; they did not overlap with each other nor did they show a clear trend (see Figure 12). Literature data for comparison purposes have not been found.

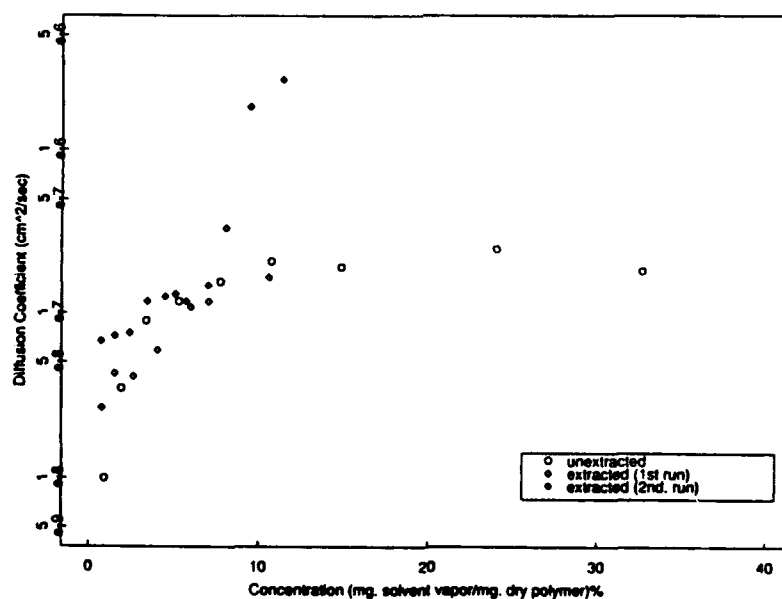


Figure 12. Investigation of the influence of plasticizer on the diffusion coefficient.



The diffusion coefficient obtained in the analysis of the unextracted and the extracted Neoprene samples were expressed in terms of the self-diffusion coefficient or solvent mobility  $D_1$ . The mean diffusion constant obtained in the sorption interval was first converted to the mutual diffusion coefficient  $D_{1,2}$  using the relation:

$$D_{1,2} = \frac{D}{\Phi_2^2} \quad (3)$$

where  $\Phi_2$  is the volume fraction of the polymer at the adjusted concentration (see Equation 2). Then, the self-diffusion coefficient or solvent mobility was obtained using the relation:

$$D_1 = \left( \frac{1}{\Phi_2} \right) \left( \frac{d \ln \Phi_1}{d \ln a_1} \right)_{T,P} D_{1,2} \quad (4)$$

where the thermodynamic factor,  $(d \ln \Phi_1 / d \ln a_1)_{T,P}$  was obtained from the slope of the sorption isotherm. The results for the solvent mobility and the mutual diffusion coefficient plotted in Figure 13 showed that the diffusion coefficient rapidly increased with concentration at low concentration then started to more or less level off at higher concentration. Interestingly, these curves show, within experimental error, that the mutual diffusion for the extracted Neoprene was about the same as the mutual diffusion for the unextracted sample. The self-diffusion coefficient (solvent mobility) for the extracted sample was not as clear. If the last three data points for the self-diffusion coefficient of the extracted sample were ignored, then it would be possible to say that the self-diffusion coefficient for the extracted and the unextracted were similar.

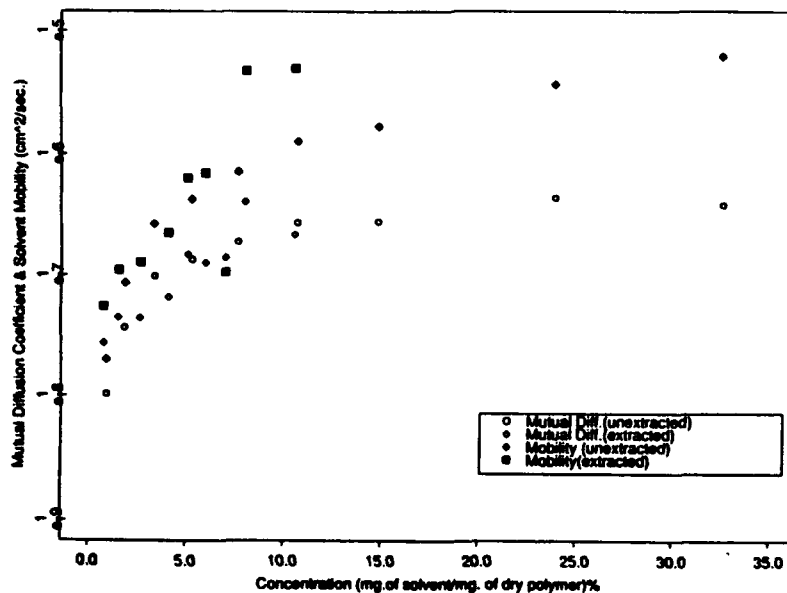


Figure 13. Investigation of the influence of plasticizer on the mutual and self-diffusion coefficient.

## Sorption Isotherms

The sorption isotherm which represents the sorbed concentration as weight percent (mg of solvent vapor/mg of dry polymer) versus activity for the unextracted and the extracted sample are shown in Figure 14. The sorption isotherms for the unextracted Neoprene behaved as that of a high penetrant uptake in a rubbery polymer. It displayed an initial linear increase then a sharp bend as activity approached unity. The sorption isotherms for the extracted, on the other hand, increased linearly with concentration. Comparison of the isotherm for the extracted and the unextracted sample demonstrated that at a given activity, the increase in concentration was higher for the unextracted than for the extracted sample indicating that the presence of plasticizer had reduced the activity of vapor mix. Therefore, in order to reach equilibrium the polymer swelled more where the plasticizer was present. To plot the data (sorption curves and sorption isotherms, etc.) for the second run of the extracted sample, the difference in weight (0.3 mg) which resulted from the fact that the sample did not completely desorb at the end of the desorption run was added to the results of the second sorption run. All the calculations were done with respect to the dry polymer. Interestingly, although the second sorption run for the extracted sample did not overlap with the first, their sorption isotherms more or less did.

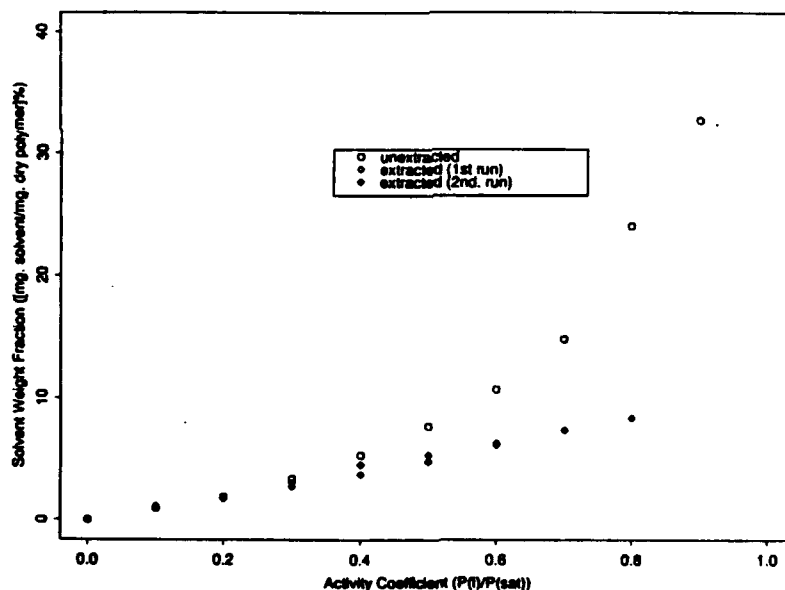


Figure 14. Investigation of the influence of plasticizer on the sorption isotherm.

An attempt was made to fit the Flory-Rehner equation:

$$\ln(a) = \ln(1 - \Phi_2) + \Phi_2 + \chi \Phi_2^2 + \frac{\rho V_1}{M_c} \left[ 1 - 2 \frac{M_c}{M} \right] \left[ \Phi_2^{1/3} - \frac{\Phi_2}{2} \right] \quad (5)$$

where  $a$  is the solvent activity,  $\Phi_2$  is the polymer volume fraction,  $\chi$  is the solvent-polymer interaction parameter,  $\rho$  is the polymer density,  $V_1$  is the solvent molar volume,  $M_c$  is the molecular weight between crosslinks, and  $M$  is the molecular weight of the polymer. For convenience in modeling,  $\rho V_1/M_c[1-2M_c/M]$  in the above equation was set equal to  $K$ . Then, using  $\Phi_2$  obtained from the immersion data at activity of one and  $K = 0.002$ , a value of  $\chi = 0.8$  was determined for the unextracted Neoprene using Equation 5. The Flory-Rhener equation fit well with the experimental sorption isotherm for the unextracted Neoprene (see Figure 15).

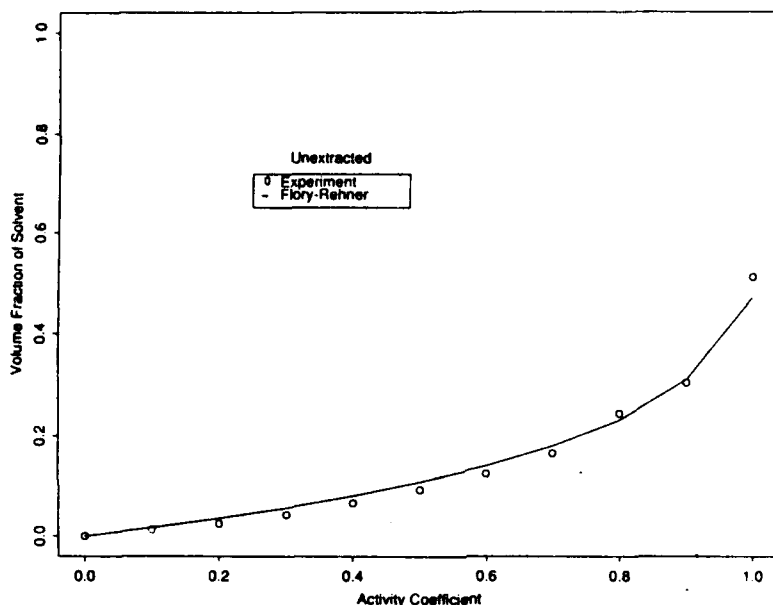


Figure 15. Comparison of Flory-Rhener isotherm with the experimental data for the unextracted sample.

### Conclusion

The vapor sorption experiments for 1,4-dioxane in Neoprene had shown that the isotherm for the unextracted sample behaved like that of a high penetrant uptake in a rubbery polymer. It exponentially increased as activity increased. Also, it clearly shows that at a given activity the increases in concentration were higher for the unextracted than for the extracted Neoprene. On the other hand, observation of the diffusion coefficients demonstrate that within the experimental error there is no significant difference between the diffusion coefficient for the unextracted and the extracted Neoprene. This conflicts with the expectation that the plasticizer would increase the diffusion coefficient by at least an order of magnitude as observed for the initial dependence on the solvent concentration. Therefore, for any given vapor pressure, the permeation rate of the extracted sample which depends on the product of  $D$  and  $C$  will be lower than that for the unextracted sample.

In the mathematical model for the droplet permeation (1), the assumptions were: the diffusion coefficient was independent of concentration; at the emerging surface of the membrane the concentration was zero or close to zero depending on the gas stream mass transfer resistance; and at the upstream surface the concentration was equal to the equilibrium concentration. This means that at the emerging surface the diffusion coefficient will be closer to  $D_0$  than to  $D$  which corresponds to the diffusion coefficient at the equilibrium concentration. This study clearly showed that  $D_0$  is about three orders of magnitude lower than the diffusion coefficient at the upper surface of the membrane which itself is equivalent to the diffusion coefficient at an activity of one. Based on the observations in this study, it can be seen that the dependence of the diffusion coefficient on concentration must be taken into consideration and further experimental study for those chemical agents is needed.

### Acknowledgment

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