

Solubility of Alkanes in a Polystyrene Matrix

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ABSTRACT: The solubilities of seven alkanes in a matrix of uncrosslinked polystyrene were measured at several different temperatures. A gravimetric method reported earlier was used to monitor the sorption of the solvents. The experimental measurements showed unambiguously that the solubility of the alkanes diffusing into a polystyrene matrix reached a constant and reproducible value typical for each system and temperature. These values could be interpreted very well with the Flory–Huggins solubility parameter (χ

= $a(\text{entropic contribution to } x) + b(\text{enthalpic contribution to } x)/T[\text{absolute temperature in Kelvin (K)}]$) or with the van't Hoff equation ($\ln K_S = \Delta S/R(\text{gas constant}) - \Delta H/RT$, where K_S is the equilibrium constant, ΔS is the entropy change, and ΔH is the enthalpy change). © 2008 Wiley Periodicals, Inc. *J Appl Polym Sci* 110: 2393–2398, 2008

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INTRODUCTION

Most materials and particularly polymers can absorb large quantities of some liquids or gases. Polymers can swell, and this can significantly change their properties. For this reason, the diffusion and solubility of compounds in polymers have been extensively studied, and to explain their behavior, many theories have been suggested. To test these theories, reliable, systematic, and accurate data are needed. Recent work^{1–10} has shown that the diffusion front is followed by a constant concentration, which is controlled by a thermodynamic equilibrium. Unlike the diffusion rate, this equilibrium is independent of the thermal history of the polymer.¹

In this work, we have applied the gravimetric method reported earlier^{1,2} to measure the solubility diagrams of a series of alkanes in a polystyrene (PS) matrix.

In the past, numerous experimental studies on the transport kinetics and swelling of alkanes in PS have been performed.^{11–22} However, in those studies, the authors' main concerns were the type of diffusion kinetics (Fickian, case II, or anomalous) and how that type varied with the experimental conditions. In fact, in the late 1960s and in the 1970s, the diffusion of alkanes in PS attracted much interest, particularly

because PS–alkane systems show, according to the authors at that time, all the different types of kinetic behavior (Fickian, case II, and anomalous diffusion), depending on the experimental conditions. The diffusion experiments then reported in the literature were performed in one of two ways: (1) the diffusion of vapors (activity between 0 and 0.85),^{11–15,18–21} in which the maximum activity that can be achieved is $\cong 0.85$ because at higher activities condensation starts to occur, leading to considerable experimental errors, and (2) the diffusion of pure liquids (activity = 1).^{12,14–17,20,22} In those studies, it was concluded that the sorption of alkanes, either as pure liquids (activity = 1) or as vapors at high activities (activity $\cong 0.85$) and at temperatures well below the glass-transition temperature (T_g) of PS, causes microvoiding of PS samples. Because of microvoiding, the samples become white and opaque. Microvoiding is observed only at high activities ($0.85 \leq \text{activity} \leq 1$), corresponding to penetrant concentrations sufficient to produce osmotic stresses that are large enough to cause local fracture of the material. The different types of behavior observed in the diffusion of alkanes in PS were fully discussed by Hopfenberg and Frisch.²³ Despite all these studies, as far as we know, no systematic study on the variation of the equilibrium swelling, as a function of temperature, for alkanes in PS has ever been performed.

Because alkane molecules possess no functional groups (apart from $-\text{CH}_2-$ and $-\text{CH}_3-$ units), their interactions occur mainly through dispersion forces. Therefore, they represent an ideal system of reference against which to compare the solubility of other homologous series containing polar functional

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TABLE I
Melting and Boiling Points of the Alkanes

Alkane	mp (°C)	bp (°C)
Hexane	– 95.3	68.7
Octane	– 56.8	125.7
Iso-octane	– 107.4	99.2
Decane	– 29.6	174.2
Dodecane	– 9.6	216.3
Tetradecane	5.9	253.6
Hexadecane	18.2	286.9

The values were taken from Daubert et al.²⁷ All the alkanes were used in their liquid states, that is, at temperatures between their melting and boiling points.

groups. This fact, as well as the lack of data in the literature on the swelling of PS with alkanes as a function of temperature, was the main motivation for this study.

EXPERIMENTAL

Polymer

We used the same PS previously used to study the solubility of alcohols¹ and carboxylic acids,² that is, PS (in the form of granules) purchased from Sigma–Aldrich [Gillingham, Dorset, U.K.] (catalogue number 43,010-2) with the following specifications: weight-average molecular weight = 230,000, number-average molecular weight = 140,000, melt flow index = 7.5 g/10 min, and $T_g = 94^\circ\text{C}$.

Solvents

As solvents, we tested the following seven alkanes of different molecular weights purchased from Sigma–Aldrich (purity $\geq 99\%$): hexane, octane, iso-octane, decane, dodecane, tetradecane, and hexadecane. Their corresponding melting and boiling points are all listed in Table I.

Calculation of the solubilities

In this work, we used three different ways of expressing solubility: (1) the mass percentage of the solvent in the total mass, (2) the volume fraction of the solvent in the total volume, and (3) the millimoles of the solvent per gram of the polymer. These were all used and fully described in our previous work.^{1,2}

As has been explained before,^{1,2} in the calculation of the volume fractions, it is assumed that the volumes of the polymer and solvent are additive; that is, no volume changes occur on mixing. This assumption is common in studies of polymer swelling (e.g., refs. 1,2, and 24–26). Our attempts to evaluate the volume changes experimentally were unsuccessful, and therefore we consider this assumption to be reasonable and the volume changes to be well within the experimental error.

Temperature dependence of the density

To calculate the volume fraction from the mass fraction (obtained by weighing), the densities of both the polymer and the solvent must be accurately known for each temperature used.

The densities (g/cm^3) for the alkanes used in this work are listed in Table II (the data were taken from Daubert et al.²⁷).

The densities of PS (g/cm^3) used in this work are listed in Table III. These were calculated with the same arguments that we have used before,^{1,2} taking into account that our PS had a T_g value of 94°C as indicated in the Aldrich catalogue.

Experimental procedure

For experimental convenience, the PS granules, purchased from Sigma–Aldrich, were transformed into flat PS discs 30 mm in diameter with average thicknesses of 0.45, 0.85, and 1.70 mm, as measured with a micrometer. This was accomplished through compression molding on a laboratory hot press at an approximate pressure of 3 tons and at an approximate

TABLE II
Densities of Alkanes (g/cm^3) at Different Temperatures

	Temperature (°C)										
	65	75	85	95	105	115	125	135	145	155	165
Hexane	0.6183	0.6083	—	—	—	—	—	—	—	—	—
Octane	0.6666	0.6580	0.6493	0.6404	0.6314	0.6221	0.6126	0.6028	—	—	—
Iso-octane	0.6561	0.6471	0.6379	0.6285	0.6188	—	—	—	—	—	—
Decane	0.6963	0.6885	0.6806	0.6725	0.6643	0.6559	0.6474	0.6388	0.6299	—	—
Dodecane	0.7167	0.7094	0.7020	0.6944	0.6868	0.6791	0.6713	0.6633	0.6553	—	—
Tetradecane	0.7317	0.7246	0.7175	0.7102	0.7029	0.6955	0.6880	0.6804	0.6728	0.6650	0.6571
Hexadecane	0.7429	0.7361	0.7292	0.7223	0.7153	0.7082	0.7010	0.6938	0.6864	0.6790	0.6715

The values were taken from Daubert et al.²⁷

TABLE III
Density of PS at Different Temperatures

Temperature (°C)	Density (g/cm ³)
65	1.0372
75	1.0345
85	1.0319
95	1.0289
105	1.0228
115	1.0168
125	1.0107
135	1.0047
145	0.9986
155	0.9926
165	0.9865

temperature of 200°C. After compression, the samples were allowed to cool slowly at normal atmospheric pressure to remove possible stresses that could remain if the samples had been cooled quickly. As previously shown, the extruded (granule) samples and compression-molded samples had identical swelling behavior. However, being much thinner, the flat (compression-molded) samples had the great advantage of allowing the attainment of saturation to occur much more quickly.

Beakers with a solvent were placed inside an oven at a constant temperature, as measured with a mercury thermometer with a precision of 0.5°C. After stabilization of the temperature, one PS sample was dipped into each beaker. Then, at periodic time intervals dependent on the diffusion rates and sample thicknesses, the samples were removed from the solvent (after quenching with a cold solvent in the case of solvents with melting points well below room temperature), cleaned with absorbent paper, weighed on an electronic balance, and finally returned to the oven. To guarantee that full saturation was attained, the samples were allowed to stay inside the solvent for twice the time needed for the attainment of the concentration plateau. For each system and temperature, at least five samples were tested; the scatter of data between the individual samples was less than 4% at full saturation, and for average data and repeated testing, it was less than 1% (see Fig. 1).

RESULTS AND DISCUSSION

The results for the mass percentage of solvent uptake as a function of time are shown in Figure 1 for the typical situation of tetradecane diffusing into PS at 95°C. Results for six different polymer samples with very similar thicknesses are plotted. The results show that after saturation, the concentration plateau remains constant even after the saturation time has been doubled.

One problem that we have encountered, especially with hexane, is that in experiments of diffusion at

temperatures lower than or equal to 65°C (the boiling point of hexane is 68.7°C) and therefore at temperatures far below T_g of PS, the concentration plateau that is reached at saturation does not remain very stable and the concentration continues increasing, although very slowly, with time. When this phenomenon has occurred, we have observed that the swollen polymer samples become slightly whitish and opaque at the temperature of diffusion. We have also observed this phenomenon with some other alkanes at the temperature of 75°C, but in such cases, the variation has been almost negligible. Because of this problem, which happens mainly with hexane, we have decided not to include the results obtained for hexane. As we have mentioned in the introduction, similar phenomena have already been observed previously and reported to be a result of the occurrence of microvoiding. According to previous authors, the conditions necessary for the occurrence of microvoiding in PS-alkane systems are well known: temperatures well below T_g of PS and high activities of the solvent.

Table IV shows all the obtained results expressed as the mass percentage of alkane, the alkane volume fraction, and the milimoles of alkane per gram of PS. Figure 2 shows the results for the mass percentage of alkane plotted in the form of solubility diagrams.

For iso-octane and octane, the maximum temperatures used (95 and 125°C, respectively) in the solubility diagrams correspond to the maximum temperatures (multiples of 5°C) immediately below their respective boiling points (99.2 and 125.7°C, respectively). For the other systems, the maximum temperatures correspond to the maximum temperatures at which we could detect no dissolution of PS. The possible dissolution of PS in alkanes was

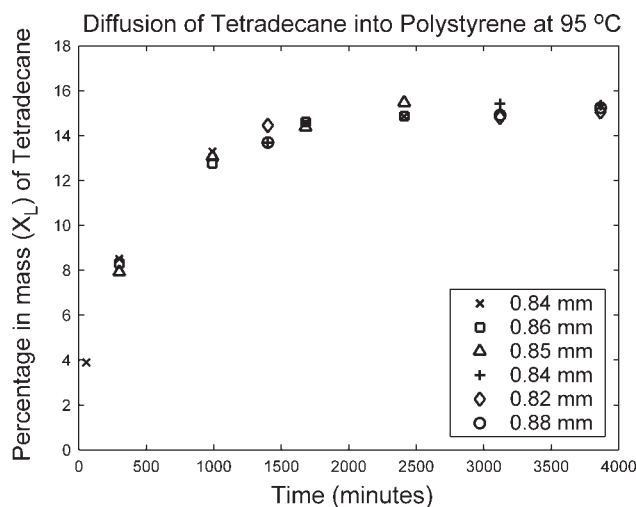


Figure 1 Mass increase (%) versus the time for the PS-tetradecane system at 95°C. The thickness of the samples is shown.

TABLE IV
Solubility of Alkanes in PS

Liquid	Temperature (°C)	Mass percentage of alkane	Alkane volume fraction	mmol L/g of PS
Octane	75	19.7	0.278	2.14
	95	22.1	0.313	2.49
	125	26.5	0.373	3.16
Decane	95	20.5	0.283	1.81
	125	25.6	0.349	2.42
	145	30.0	0.405	3.02
Dodecane	95	18.0	0.245	1.29
	125	23.5	0.316	1.80
	145	28.5	0.378	2.34
Tetradecane	95	15.2	0.206	0.90
	125	21.3	0.284	1.37
	145	26.4	0.347	1.81
	165	30.1	0.393	2.17
Hexadecane	95	12.7	0.172	0.64
	125	18.6	0.248	1.01
	145	23.2	0.305	1.33
	165	27.8	0.361	1.70
Iso-octane	65	15.6	0.226	1.62
	75	16.5	0.240	1.73
	95	17.5	0.258	1.86

monitored by Fourier transform infrared spectroscopy of the solvent after the sample was fully saturated and also (whenever possible) by the residue after the solvent evaporated. In all cases (solvent and temperature) mentioned in Table IV, no dissolution of PS has been detected. However, PS dissolution has been observed, in some cases, at temperatures higher than those mentioned in Table IV.

The lower temperature limits of the solubility diagrams of Figure 2 were dictated by the very long times needed for full saturation (due to the low rate of diffusion).

As shown in Figure 2, the solubility diagrams of alkanes in PS are all approximately parallel to one another and show very low sensitivity to the molecular weight of the solvent (i.e., the solubility lines are all very close to one another). Furthermore, the equilibrium sorption values of alkanes in PS do not exhibit a very strong temperature dependence (i.e., the solubility lines in Figure 2 are very steep), and this is indicative of low heats of solution. This results from the fact that because of the aliphatic nature of

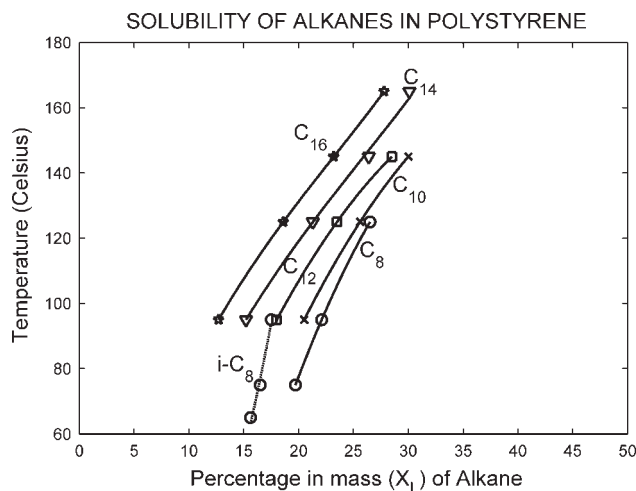


Figure 2 Solubility diagrams of alkanes in PS: (○) octane (C_8) and iso-octane ($i-C_8$), (×) decane (C_{10}), (□) dodecane (C_{12}), (▽) tetradecane (C_{14}), and (☆) hexadecane (C_{16}). Linear alkanes are represented by solid lines; branched alkanes (iso-octane) are represented by dotted lines.

alkanes, only van der Waals and dispersion forces are involved in the guest–host intermolecular interactions, and this limits any enthalpic contributions.

Discussion based on the Flory–Huggins theory

The theory of Flory²⁸ and Huggins²⁹ states that

$$\ln a_1 = \ln \phi_1 + \left(1 - \frac{1}{r}\right)(1 - \phi_1) + \chi(1 - \phi_1)^2 \quad (1)$$

where a_1 , ϕ_1 , r , and χ are the solvent activity, the volume fraction of the solvent, the ratio of the molar volumes of the polymer and solvent molecules, and the dimensionless Flory–Huggins interaction parameter between the polymer and solvent, respectively.

In the case of the mixing of low-molecular-weight solvents with high-molecular-weight polymers, the term $1/r$ in eq. (1) is usually negligibly small. For the same reason mentioned in our previous work^{1,2} $1/r$ from eq. (1) can also be neglected in this work.

Because at equilibrium swelling $\ln a_1 = 0$ and by approximation $1/r = 0$, eq. (1) may then be written as follows:

TABLE V
 χ Values of Alkanes with PS at Different Temperatures

Organic liquid	χ			
Octane	1.0707 (75°C)	1.0055 (95°C)	0.9136 (125°C)	—
Iso-octane	1.1905 (65°C)	1.1550 (75°C)	1.1130 (95°C)	—
Decane	1.0607 (95°C)	0.9478 (125°C)	0.8724 (145°C)	—
Dodecane	1.1429 (95°C)	1.0003 (125°C)	0.9069 (145°C)	—
Tetradecane	1.2466 (95°C)	1.0588 (125°C)	0.9508 (145°C)	0.8874 (165°C)
Hexadecane	1.3598 (95°C)	1.1358 (125°C)	1.0195 (145°C)	0.9303 (165°C)

$$\ln \phi_1 + (1 - \phi_1) + \chi(1 - \phi_1)^2 = 0 \quad (2)$$

Therefore, at equilibrium, the interaction parameter can be calculated as follows:

$$\chi = -\left(\frac{\ln \phi_1 + (1 - \phi_1)}{(1 - \phi_1)^2}\right) \quad (3)$$

The polymer–solvent interaction parameter χ is temperature-dependent, and its values, calculated with eq. (3), are listed in Table V together with the corresponding temperatures ($^{\circ}\text{C}$) and plotted in Figure 3 for each solvent on the $1/T$ scale. As shown in Figure 3, the values of χ depend linearly on $1/T$ and therefore can be expressed by the simple expression suggested in the classical form of the Flory–Huggins theory:

$$\chi = a + \frac{b}{T} \quad (4)$$

We have used the least squares method to fit the values of χ to straight lines, and from the intercept on the origin and the slope, we have determined the values of a and b , respectively, for eq. (4). The obtained values are listed in Table VI together with the corresponding correlation coefficients.

Our results show that the enthalpic contribution of b to χ is always positive, reflecting a net repulsion between the polymer and solvent, and thus is unfavorable to mixing. The entropic contribution a is always negative and thus favors mixing, except in the case of the branched alkane (iso-octane). The positive value of a obtained for iso-octane is probably due to the fact that branched molecules require

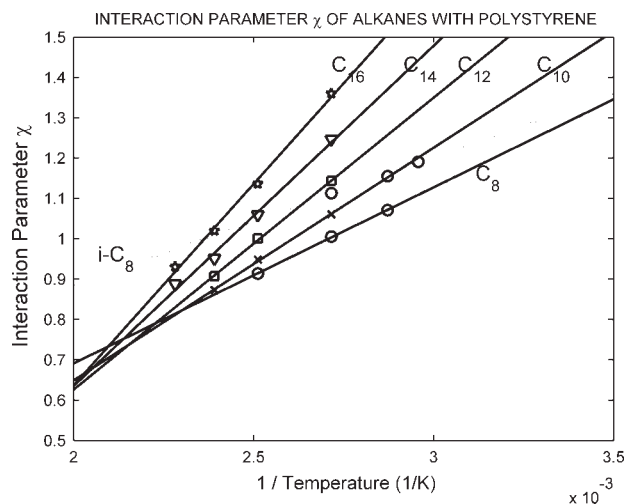


Figure 3 χ values of alkanes with PS: (○) octane (C_8) and iso-octane ($i\text{-C}_8$), (×) decane (C_{10}), (□) dodecane (C_{12}), (▽) tetradecane (C_{14}), (☆) hexadecane (C_{16}). Linear alkanes are represented by solid lines; branched alkanes (iso-octane) are represented by dotted lines.

TABLE VI
Values of a and b in eq. (4) for All the Systems and Corresponding Correlation Coefficients

	Entropic and enthalpic contributions to χ		
	a	b	Correlation coefficient
Octane	− 0.1812	436.2	0.9998
Iso-octane	+ 0.2553	315.1	0.9928
Decane	− 0.5045	576.7	0.9994
Dodecane	− 0.8206	723.4	0.9996
Tetradecane	− 1.051	843.1	0.9964
Hexadecane	− 1.358	997.5	0.9981

greater space, so their solubility is entropically less favorable.

Discussion of results based on the van't Hoff equation

The van't Hoff equation is derived from chemical equilibrium thermodynamics and correlates the constant of thermodynamic equilibrium of a reaction (K_S) with the entropy change (ΔS) and enthalpy change (ΔH) of that reaction as follows:

$$\ln K_S = \left(\frac{\Delta S}{R}\right) - \left(\frac{\Delta H_S}{R}\right)\left(\frac{1}{T}\right) \quad (5)$$

This equation has been successfully used by several authors,^{2,30–35} in studies of solvent sorption by polymer matrices to correlate the amount of solvent uptake with the temperature. The idea underlying the use of eq. (5) consists of considering that the sorption of an organic solvent (OS) into a polymer

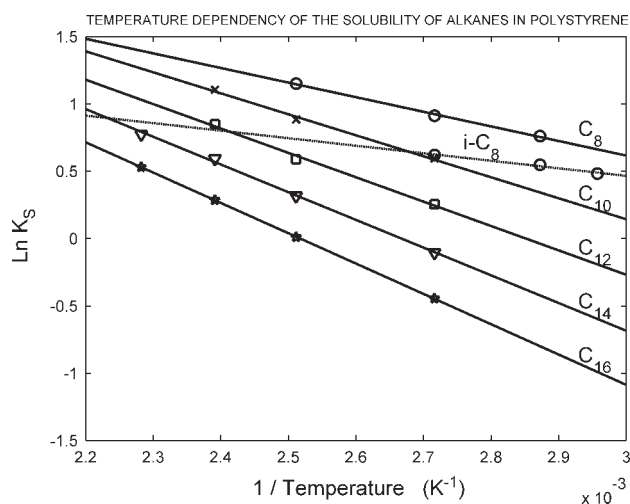


Figure 4 van't Hoff plots for alkanes in PS: (○) octane (C_8) and iso-octane ($i\text{-C}_8$), (×) decane (C_{10}), (□) dodecane (C_{12}), (▽) tetradecane (C_{14}), and (☆) hexadecane (C_{16}). Linear alkanes are represented by solid lines; branched alkanes (iso-octane) are represented by dotted lines.

TABLE VII
Values of $\Delta S/R$ and $\Delta H/R$ from the van't Hoff Equation
leq. (5) and Corresponding Correlation Coefficients

	$\Delta S/R$	$\Delta H/R$	Correlation coefficient
Octane	3.8703	1084.8	0.9987
Iso-octane	2.145	559.8	0.9901
Decane	4.8212	1559.4	0.9974
Dodecane	5.165	1811.5	0.9968
Tetradecane	5.4904	2058.6	0.9988
Hexadecane	5.6719	2253.0	0.9999

matrix (PM) can be considered a chemical equilibrium given by



where OSPM represents the absorption complex that results from the adsorption of one organic solvent molecule into one polymer adsorption site.

In agreement with Hung's suggestion,³⁰ K_S is expressed as the number of millimoles of the solvent sorbed per gram of the polymer. Through the plotting of eq. (5), straight lines are usually obtained, the entropy of mixing and the enthalpy of mixing corresponding to the intercept on the origin and to the slope of the resulting curves, respectively.

We show in Figure 4 a plot of $\ln K_S$ versus $1/T$ for alkanes in PS, where K_S is the equilibrium sorption constant expressed in millimoles of alkanes sorbed per gram of PS. As shown, in the temperature range considered (65–165°C), the linear relationship in eq. (5) applies perfectly well to the equilibrium sorption of alkanes in PS, in analogy with the behavior reported earlier for carboxylic acids.²

From the intercept on the origin and from the slope of the resulting curves, we have calculated, using the least squares method, the values of $\Delta S/R$ and $\Delta H/R$, and these are indicated in Table VII together with the corresponding correlation coefficients. As Figure 4 and Table VII show, all the alkanes have positive enthalpies of sorption and positive entropies of sorption. Therefore, the sorption of alkanes into PS occurs because the positive ΔS value (favorable to mixing) outweighs the also positive ΔH value (unfavorable to mixing).

CONCLUSIONS

In this work, we have measured the solubility of alkanes in a matrix of high-molecular-weight PS, using a gravimetric method described previously.¹ In the case of diffusion at temperatures far below T_g of PS, some microvoiding has been observed, especially with hexane. The obtained solubility diagrams are very steep (low sensitivity to temperature) and show very low sensitivity to the chain length of the alkane molecules.

The classical Flory–Huggins theory has been successfully used to fit the experimental data with a temperature-dependent interaction parameter, $\chi = a + (b/T)$, with different constants a and b for each type of solvent.

A further analysis also shows that our results can be fitted perfectly well with the van't Hoff equation.

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