Generalization of the Effects of Solvent–Polymer Interactions with Linear Multiparameter Equations

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ABSTRACT: A quantitative generalization of the indices of swelling process of polymers, containing the nonhydrocarbon groups (polyurethanes, fluoropolymers) in organic solvents, as well as of the process of dissolution of poly(methy1 methacrylate) was successfully realized by means of multiparametric equations, which take into account the solvents capacity for specific and nonspecific solvation of polymers and solvents' molar volumes. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 106: 3417–3422, 2007

Key words: interaction; solvent-polymer; multiparameters; generalization

Polymer/organic-solvent interaction processes, particularly the ability of solvents to penetrate polymer structures (i.e., their swelling behavior), are at present usually considered from the standpoint of both the Hildebrand theory of regular solutions[1] and the Flory–Huggins model.[2] They are based on the assumption that the degree of solvent adsorption is determined by the solvent cohesion energy density (δ) or, even better, by the difference between the solvent and polymer solubility parameters ($\delta_1 - \delta_2$). However, in reality, these dependences give only a semiquantitative bell-shaped picture, with maxima for the solvents characterized by δ values that are the same or nearly the same as the δ values of the polymers. At the same time, there are frequently numerous deviations from these dependences, even when the examined solvents are divided into separate groups (see ref. 3); thus, to obtain acceptable outcomes, it is often necessary to introduce empirical corrective coefficients.[4]

Recent reviews have been published of Aminabhavi et al.'s investigations of solvent penetration into the structure of diverse polymer membranes.[5] These authors proposed the molar volume (V_m) of the solvent as the parameter that determines the degree of polymer swelling (Q). This supposition should be considered logical because, undoubtedly, the larger the molecular dimensions are of a solvent, the more difficult it will be for the solvent to penetrate the polymer structure. However, we can observe a linear dependence between log Q in various solvents and their V_m values (even conceding that there are a number of exceptions) when considering three distinct solvent groups: the aliphatic hydrocarbons, monocyclic aromatics, and esters.

Obviously, such discrepancies in the proposed models (particularly by Aminabhavi et al.[5]) are caused because only physical factors are taken into account, and the consideration of possible chemical solvation processes is set aside.[6] At the same time, however, attempts to describe the swelling process by chemical characteristics alone (e.g., the Reichardt electrophilicity parameter) are successful only for distinct groups of solvents.[7] Thus, it seems presumptuous to maintain that the processes of polymer/organic-solvent interactions are significantly more complicated and furthermore that their generalization by means of a single parameter is impossible.

Therefore, we suggest that the swelling process be considered as an equilibrant intersolution of components and experimental results to be generalized on the basis of the linear free energy concept, which takes into account various possible aspects of interactions. Such an approach was developed in chemical kinetics by Koppel and Palm[8] and by Kamlet et al.[9] Thus, it may be expedient to generalize the data from Aminabhavi et al.[5] on polymer/organic-solvent interactions by means of the Koppel–Palm

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equation supplemented with terms that take into account the influence of V_m (mol/mL) and the cohesion energy (expressed by the square of the Hildebrand solubility parameter):

$$\log Q = a_0 + a_1 \frac{n^2 - 1}{n^2 + 2} + a_2 \frac{\varepsilon - 1}{2\varepsilon + 1} + a_3 B + a_4 E_T + a_5 \delta^2 + a_6 V_m \quad (1)$$

where Q is expressed as the moles of the absorbed liquid; n and ε are the refractive index and permittivity of the solvent, respectively, which determine the solvent polarizability and polarity responsible for nonspecific solvation; and B and E_T are the Palm basicity and Reichardt electrophilicity, respectively, which determine the specific solvation.

Not only has this approach been found to be effective for the generalization of data on the swelling of such low-polarity polymers as polyethylene,[10] butyl rubber,[11] polybutadiene,[12] and natural and synthetic rubber[13] in different solvents, but it also reveals the influence of certain solvation factors on the chemical nature of the process and can predict Q in investigated solvents with sufficient reliability. However, the applicability of eq. (1) was verified in the cited articles in general for hydrocarbon polymers with relatively low polarity. That is why it seemed to be reasonable to verify its applicability to polymers that contain nonhydrocarbon polar groups.

For this goal, we have treated data borrowed from studies performed by Aminabhavi and coworkers[5,14–16] on the swelling of polyurethane rubber. The object of their investigation was Vibrathane B600, that is, the product of the reaction of poly(propylene oxide) and toluylene diisocyanate stabilized by 4,4′-methylene bis(*o*-chloroaniline), which was used as an additive. The molecular mass of the poly(ester diol) segments was 300–6000, and that of the aromatic moiety was 500–3000. Plate-shaped samples were subjected to swelling. The degree of swelling was monitored gravimetrically. The data were presented in millimoles of the absorbed solvent per gram of the polymer at 25°C (Table I).

The treatment of the data on swelling with eq. (1) yields an expression characterized by an insufficiently high multiple correlation coefficient ($R = 0.943$), although the equation per se is adequate on the basis of the Fischer criterion for the corresponding number of degrees of freedom at a confidence level of $\alpha = 0.95$. However, the exclusion of the most deviating data for only two solvents (1,1′,2,2′-dichloroethane and cyclohexanone) yields an expression with an acceptable value of $R = 0.965$:[17]

$$\log Q = -0.002 + (6.93 \pm 0.93) f(n) + (1.30 \pm 0.69) f(\varepsilon) + (1.00 \pm 0.55) \times 10^{-3} B - (3.13 \pm 17.1) \times 10^{-3} E_T - (1.04 \pm 0.57) \times 10^{-3} \delta^2 - (10.8 \pm 1.09) \times 10^{-3}\, V_m \quad (2)$$

The number of embraced solvents is $N = 36$, R is 0.965, and the standard deviation is $S = 0.128$.

An analysis of the signs of individual terms of the equation demonstrates that according to the aforementioned considerations, a rise in the size of the molecules (V_m) reduces their ability to penetrate the polymer, as confirmed by the minus sign at the coefficient before V_m. A similar negative effect is caused by association in the medium (obviously because of a rise in the energy required for the separation of individual molecules from associates of the liquid) as well as the capacity for electrophilic solvation (E_T), presumably because high E_T values characterize solvents (e.g., alcohols) that are capable of self-association due to the formation of intermolecular bonds.

At the same time, the terms $f(n)$, $f(\varepsilon)$, and B have plus signs, thus suggesting nonspecific solvation of some polymer domains by the absorbed solvent and hydrogen bonding between hydrogen atoms of NH groups of polyurethane and basic solvents, a phenomenon that facilitates swelling.

According to ref. 17, the significance of the individual terms of eq. (2) was determined by their one-by-one exclusion followed by the determination of R for derived equations with a smaller numbers of terms. When R was reduced insubstantially, the role of the excluded parameter was regarded as inessential. In this way, it was established that parameter E_T was insignificant ($R = 0.965$) and B had only a low significance ($R = 0.961$); as a result, the following equation was derived:

$$\log Q = -0.14 + (6.39 \pm 0.66) f(n) + (1.97 \pm 0.35) f(\varepsilon) + (1.06 \pm 0.31) \times 10^{-3} \delta^2 - (10.4 \pm 1.08) \times 10^{-3}\, V_m \quad (3)$$

where R is 0.961 and S is 0.131.

The values of log Q calculated by eq. (3) and their deviations (Δ log Q) from the experimental values are listed in Table I. The deviations are mainly within the corridor of errors ($S = 0.131$) or beyond it only slightly (*p*-xylene, dibromoethane, and some alcohols and esters), except for 1,1′,2,2′-tetrachloroethane and cyclohexanone, which were not taken into account in the calculations.

In summary, multiparameter equations are applicable to the quantitative description of data on the swelling of polyurethane rubbers. Moreover, the significant factors appear to be the same as those for

TABLE I
Experimental Swelling Ratios for Polyurethane at 25°C from Refs. 5 and 14–16 and Those Calculated with Eq. (3)

Solvent	Q (mmol/g)	log Q_{exp}	log Q_{cal}	Δlog Q	V_m (cm^3/mol)
n-Hexane	0.950	−0.0223	0.0870	−0.109	131.61
n-Heptane	0.687	−0.1630	−0.0305	−0.133	147.46
n-Octane	0.542	−0.2660	−0.1576	−0.108	162.60
Isooctane	0.510	−0.2924	−0.1626	−0.130	165.10
n-Nonane	0.473	−0.3251	−0.2904	−0.0348	178.64
n-Decane	0.354	−0.4510	−0.4434	−0.0076	194.89
Cyclohexane	2.080	0.3181	0.4758	−0.158	108.09
Benzene	9.160	0.9619	0.8864	0.0755	89.41
Toluene	6.780	0.8312	0.7406	0.0906	106.85
p-Xylene	5.430	0.7348	0.5645	0.170	123.30
Mesitylene	3.530	0.5478	0.3992	0.149	139.11
Tetraline	4.590	0.6618	0.5828	0.0790	136.27
Tetrachloromethane	6.880	0.8376	0.7425	0.0951	96.50
1,2-Dichloroethane	12.86	1.109	1.172	−0.0633	78.74
1,1,2,2-Tetrachloroethane[a]	31.06	1.492	—	—	104.74
Trichloroethylene	12.60	1.100	0.9642	0.136	89.74
Tetrachloroethylene	5.020	0.7007	0.7662	−0.0655	102.42
Chlorobenzene	10.15	1.006	1.101	−0.0950	101.79
Bromoform	22.09	1.344	1.315	0.0296	87.42
1,2-Dibromoethane	9.870	0.9943	1.196	−0.202	86.21
Bromobenzene	9.950	0.9978	1.121	−0.123	105.03
Methanol	7.780	0.8910	0.7400	0.151	40.41
Ethanol	7.250	0.8603	0.8760	−0.0156	58.37
n-Propanol	6.320	0.8007	0.8687	−0.0680	74.70
Isopropyl alcohol	4.180	0.6212	0.8479	−0.227	76.75
n-Butanol	6.380	0.8048	0.7809	0.0239	91.53
Isobutyl alcohol	5.25	0.7202	0.7688	−0.0487	92.34
2-Butanol	4.490	0.6522	0.7797	−0.127	91.93
Isoamyl alcohol	4.700	0.6721	0.6609	0.0112	108.56
Anisole	8.030	0.9047	0.9409	−0.0362	109.31
Cyclohexanone[a]	37.07	1.569	—	—	103.56
Methyl acetate	7.790	0.8915	0.8178	0.0738	80.14
Ethyl acetate	5.870	0.7686	0.6935	0.0751	97.78
n-Propyl acetate	5.370	0.7300	0.5735	0.156	115.15
n-Butyl acetate	4.280	0.6314	0.4170	0.214	131.63
Isoamyl acetate	3.320	0.5211	0.2449	0.276	149.32
Methyl benzoate	8.190	0.9133	0.8478	0.0655	125.05
Diethyl malonate	2.200	0.3424	0.4635	−0.121	151.78

[a] The data were omitted from the calculation with eq. (2).

polymers free of polar groups: V_m of the solvents and their capacity for nonspecific solvation.

In the same way, the use of the aforementioned relationship allowed us to treat the data on the interactions between the solvents and fluoropolymers containing carbon black. In refs. 18–20, the experimental results for swelling in different organic solvents of DuPont's Viton A-201C series with the designated sample numbers 2093, 2094, and 2095, which contained 10, 20, and 30 wt % carbon black at 25, 40, and 60°C, respectively, are presented. The authors do not relate the obtained data to the properties of the solvents.

We have generalized experimental data for the swelling of polymer Viton A-201C N2094 in 18 solvents at 25°C with eq. (1).[21] R was found to be unsatisfactorily small ($R = 0.907$). However, when the data on only one solvent [dimethylformamide (DMF)] were excluded, a satisfactory correlation was achieved with $R = 0.967$:

$$\begin{aligned}\log Q = 2.048 - (8.77 \pm 1.56)f(n) + (1.90 \pm 0.83)f(\varepsilon) \\ + (2.11 \pm 0.40) \times 10^{-3}B + (0.03 \pm 0.03)E_T \\ + (0.011 \pm 0.903) \times 10^{-3}\delta^2 \\ + (1.957 \pm 1.609) \times 10^{-3}\, V_m \quad (4)\end{aligned}$$

where S is 0.203. This equation is adequate according to the Fischer criterion.

As in the previous case, low values of the coefficients of pair correlation between log Q and individual terms of eq. (4) made it impossible to estimate the extent to which each parameter affected the magnitude under consideration. Therefore, the significance of individual terms of eq. (4) was determined by their successive exclusion from calculation. Thus, we revealed the insignificance of parameters δ^2 and E_T. The presence of fluorine atoms with negative charges in the polymer structure made the electrophilic solvation of the polymer impossible.

Hence, the following four-parameter equation is adequate:

$$\begin{aligned}\log Q = 1.168 - (8.86 \pm 0.99)f(n) \\ + (0.91 \pm 0.42)f(\varepsilon) + (2.07 \pm 0.41) \times 10^{-3}B \\ + (2.86 \pm 1.10) \times 10^{-3}\ V_m \end{aligned} \quad (5)$$

where R is 0.962 and S is 0.105.

When the polarity, basicity, and V_m of solvents increase, their ability to penetrate the polymer structure increases as well; on the contrary, a rise in the polarizability of the solvents reduces this ability. This is probably caused by the high polarizability of halogen atoms. The plus sign for coefficient B allows us to admit the existence of specific solvation between negatively charged fluorine atoms and electron-donor molecules, presumably on the types of complexes with charge transfer.

The values of log Q calculated by eq. (5) and Δ log Q from the experimental data are listed in Table II. The deviations are within the corridor of errors ($S = 0.105$), except for DMF, which was excluded from the calculations.

TABLE II
Swelling Ratios for Viton A201-C N2094 Polymers at 25°C According to Refs. 18–20 and Those Calculated with Eq. (5)

Solvent	log Q_{exp}	log Q_{cal}	Δ log Q
DMSO	−0.1090	0.0581	0.1672
DMF[a]	−0.6253	0.1432	−0.7685
Tetrahydrofuran	−0.2586	0.2171	−0.0415
1,4-Dioxane	−0.0110	−0.1435	−0.1325
Dimethyl acetamide	0.2322	0.2569	0.0247
Acetone	0.2916	0.2517	−0.0399
Methyl ethyl ketone	0.2177	0.2304	0.0126
Cyclohexanone	0.1186	−0.0016	−0.1202
Acetonitrile	0.1611	0.2452	0.0842
Methyl acetate	0.2148	0.1559	−0.0590
Ethyl acetate	0.2000	0.1224	−0.0777
Propyl acetate	0.1539	0.1493	−0.0036
Butyl acetate	0.0565	0.0829	0.0264
Isoamyl acetate	−0.0114	0.1666	0.1781
Methyl benzoate	−0.3363	−0.4800	−0.1437
Ethyl benzoate	−0.3507	−0.4152	−0.0646
Benzene	−0.8633	−0.8658	−0.0025
Toluene	−0.9747	−0.7825	0.1925

[a] The data were excluded from the calculations.

The same approach was found to be effective for the data obtained at the higher temperatures as well as the generalization of the data on the swelling of Viton A-201C N2093 and N2095 polymers.

Ueberreiter and Asmussen[22] showed that the dissolution of polymers was preceded by the formation of a rubberlike surface layer. Correspondingly, it was assumed that the rate of dissolution followed the second Fickian law. According to the Einstein–Stokes theory

$$D = \frac{kT}{6\pi}\frac{1}{\eta r} \quad (6)$$

where D is the diffusion coefficient, k is Boltzman constant, T is absolute temperature in kelvin, η is the viscosity of the solvent, and r is its molecular radius, which is approximately equal to the cube root of V_m. However, attempts to quantitatively relate the dissolution rate of polystyrene to the $1/(\eta r)$ value failed.[23] Asmussen and Ueberreiter[23] explained this negative result as the effect of steric factors and advanced the idea that there are two criteria of solvent quality: one is thermodynamic, being due to the interaction between the components in a system and related to the second virial coefficient (B) of the equation for osmotic pressure, and the other is kinetic, being determined by the ease of solvent penetration into the polymer structure, that is, by V_m of the solvent. Studying the rate of dissolution of polystyrene in several alkyl acetates, Asmussen and Ueberreiter[24] revealed that the polymer dissolution rate and the D values successively decreased from methyl to isoamyl acetate. However, the derived relations appeared to be nonlinear throughout a homologous ether series, and this was explained by a possible effect of thermodynamic factors, that is, of chemical interactions. In a study on the diffusion of solvents in membranes made of six synthetic rubbers and their swelling in these solvents,[5] it was also assumed that V_m had a determining effect on D; however, linear log D–V_m relationships were observed (with a number of exceptions) only when three groups of solvents (alkanes, cyclic compounds, and esters) were treated separately.

There are also other opinions on the solvent properties that determine the diffusion of solvents into a polymer and the rate of its possible dissolution. These are the permittivity, surface tension, solubility parameter, and so forth. However, all the derived relationships are only qualitative in character or at most fit into narrow homologous series of solvents only.

According to Van Krevelen and Hoftyzer,[25] the polymer dissolution process comprises two steps: the penetration of solvent molecules into the polymer (a quasi-induction step) and the time period during which the polymer and the solvent volumetric diffusion fluxes are equal (a steady-state step).

Because the swelling of polymers is the first stage of their dissolution, as well as the diffusion and penetration of solvents into their structure, it is reasonable to expect that the influence of the solvent properties on the characteristics of the processes may be generalized in terms of eq. (1) as well. Really, we found that the use of this equation makes it possible to describe the data on the diffusion of solvents through polyethylene, chloroprene, and butadiene–styrene rubber membranes, the penetration of liquids into polyurethane rubber, and the dissolution of poly(methyl methacrylate) in organic liquids.[26–28]

To reveal a possible relation between the solvent properties and the dissolution rate of polymers, we studied the dissolution of poly(methyl methacrylate) (weight-average molecular mass = 1.39×10^5) in 15 solvents at 15 and 30°C.[29] Polymer specimens in the form of plates were placed into stoppered, thermostated weighing bottles containing 25 cm^3 of a solvent. The change in the mass of the samples was measured gravimetrically with a precision of ±0.0002 g.

A relative coefficient of the polymer dissolution rate was selected to characterize the dissolving power of the solvents. This coefficient was determined from a decrease in the sample mass as the rate constant of a zero-order process in the linear region of the dissolution curve.

The data on the dissolution of poly(methyl methacrylate) at 15°C are shown in Table III. $R = 0.782$ for all 15 solvents examined is unsatisfactorily low (i.e., markedly lower than the recommended optimal value of $R \geq 0.95$),[17] and the coefficients of pair correlation between log Q and the individual terms of eq. (1) are equal to 0.141, 0.519, 0122, 0.557, 0.326, and 0.547, thus making it impossible to draw conclusions about the determining influence of any of these factors on the process. Therefore, according to the International Union of Pure and Applied Chemistry recommendations,[17] the most deviating data for tetrachloromethane, trichloroethylene, and chloroform were successively excluded from the calculations. As a result, the R value increased to 0.920, 0.931, and 0.979, respectively. Thus, an equation generalizing the data on the dissolution of poly(methyl methacrylate) in 12 solvents with a satisfactory correlation was derived:

$$\begin{aligned}\log Q = 3.30 &+ (0.359 \pm 1.78)f(n) + (7.02 \pm 1.45)f(\varepsilon) \\ &+ (1.07 \pm 0.99) \times 10^{-3}B - (0.164 \pm 0.059)E_T \\ &- (1.50 \pm 1.14) \times 10^{-3}\delta^2 - (17.3 \pm 2.6) \times 10^{-3}\ V_m \end{aligned} \tag{7}$$

where N is 12, R is 0.979, and S is 0.119.

The significance of the individual terms was verified by successive exclusion with the determination of the significance of R for an equation with a smaller number of terms. In this way, the actual insignificance of $f(n)$ was revealed: when it was excluded, the R value for the five-parameter equation obtained was not reduced. The effects of B and δ^2 also appeared to be of low significance (after their exclusion, R was 0.974 and 0.964, respectively). Finally, the rate of the dissolution of poly(methyl methacrylate) was correlated with the properties of the solvents with sufficient accuracy by the three-parameter equation:

$$\log Q = 2.94 + (7.07 \pm 1.34)f(\varepsilon) - (0.169 \pm 0.041)E_T - (15.4 \pm 2.2) \times 10^{-3}\ V_m \tag{8}$$

where N is 12, R is 0.964, and S is 0.123.

TABLE III
Experimentally Observed Dissolution Rates and Dissolution Rates Calculated with Eq. (8) for Poly(methyl methacrylate) at 15°C

Solvent	Q ($\times 10^3$ min^{-1})	$-\log Q_{\mathrm{exp}}$	$-\log Q_{\mathrm{cal}}$	$\Delta \log Q$
n-Hexane	0.855	3.068	3.010	−0.059
Benzene	2.24	2.65	2.602	−0.048
Toluene	1.48	2.830	2.738	−0.092
o-Xylene	1.24	2.907	2.904	−0.003
m-Xylene	1.71	2.767	2.889	0.122
Chloroform[a]	43.1	1.366	—	—
Tetrachloromethane[a]	0.144	3.842	—	—
Trichloroethylene[a]	30.35	1.518	—	—
1,4-Dioxane	1.71	2.767	2.818	0.051
Acetone	13.2	1.881	2.029	0.148
Cyclohexanone	9.40	2.027	2.126	0.099
Acetophenone	2.11	2.676	2.481	−0.195
Ethyl acetate	5.12	2.291	2.277	−0.014
n-Amyl acetate	0.711	3.148	3.241	0.092
DMF	4.39	2.358	2.255	−0.103

[a] The data were excluded from the calculations.

The pair correlation coefficients between log Q and significant parameters are larger than 0.7. The term comprising V_m appears in the equation with a minus sign, which is consistent with the idea[5,30] that an increase in the solvent molecular mass lowers the rate of polymer dissolution in the liquid. However, for an adequate description of the process, two other solvent parameters (polarity and electrophilicity) must be taken into account. The plus sign, indicating a higher dissolution rate in more polar media, suggests that nonspecific salvation of poly(methyl methacrylate) is possible because this polymer contains polar ester groups, which facilitate the dissolution. The minus sign at the coefficient for the E_T parameter, reflecting the solvent electrophilicity, indicates the slowdown of dissolution with an increase in this parameter. This fact is more difficult to explain. However, it must be taken into account that poly(methyl methacrylate) is insoluble in strong proton-donating solvents such as water and alcohols, which are characterized by high E_T values. Therefore, these solvents have not been studied. Moreover, even a weak proton donor, such as chloroform, does not fall into the general dependence, and this probably indicates an individual type of solvation interaction due to the specific hydrophobicity of the polymer. In addition, we should note the relatively low significance of E_T: when this parameter is excluded, R is 0.885 for the two-parameter equation log $Q = f(\varepsilon, V_m)$, whereas the decrease in R for the functions log $Q = f(E_T, V_m)$ and log $Q = f(\varepsilon, E_T)$ is markedly larger (0.826 and 0.708, respectively). The values of log Q calculated by eq. (8) and Δ log Q from the experimental data are presented in Table III. The deviations practically do not exceed $S = 0.123$.

The examination of the cited data leads to a conclusion about the complex nature of the interactions of organic solvents with polymers containing nonhydrocarbon groups (polyacrylates, polyurethanes, etc.): it cannot be adequately described by means of only one parameter. This process depends in a complicated manner on diverse solvent characteristics. A quantitative generalization of the indices of such processes as polymer swelling and the penetration and diffusion of solvents into polymer films and the rate and value of their solubility may be realized only by the use of multiparameter equations, which take into account a solvent's capacity for specific and nonspecific solvation of polymer diverse fragments, which favor the interactions and V_m of solvents: with their growth, the penetration of liquids into polymer structures as a rule is hampered.

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