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Solubility of Phenanthrene in Binary Mixtures of C_1-C_4 Alcohols + 2-Propanol and Ethanol + Methanol at 298.2 K

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Experimental solubilities are reported for phenanthrene in binary solvent mixtures of ethanol + methanol, methanol + 2-propanol, ethanol + 2-propanol, 1-propanol + 2-propanol, and 1-butanol + 2-propanol at 298.2 K. Results of these measurements were used to evaluate the prediction capability of previously developed quantitative structure−property relationships employing the solubility data in monosolvents, and the mean deviations (MDs) of the models varied between (1.1 and 24.2) %. The overall MDs (OMDs) for these methods were 3.5 (± 3.1) % and 8.6 (± 9.4) %, respectively, for water-to-solvent and gas-to-solvent coefficients. Using ab initio prediction methods the MDs varied between (7.0 and 114.7) %, and the OMDs were 9.1 (± 1.9) % and 84.9 (± 22.9) %.

Introduction

Solubility data are important in various fields of industry and academia including separation, decontamination, and biological areas. In many instances, the solubility in a neat solvent is not sufficient for the desired application. Different methods have been developed for modification of solubility such as micellization, complexation, and cosolvency.[1−6] Choosing an appropriate solvent system for solubilizing a desired amount of a solute required experimental databases or reasonably accurate predictive models.

Phenanthrene is a polycyclic aromatic hydrocarbon (PAH) which is a byproduct in gas plants and can be used in dye, explosive, and pharmaceutical industries. Experimental solubility data of phenanthrene in binary mixtures of water + cosolvents including methanol, ethanol, 1-propanol, acetone, *n*-butylamine, and tetrahydrofuran have been reported in the literature.[3−8] Its solubility data in nonaqueous mixtures of toluene + heptane and toluene + 2,2,4-trimethylpentane have also been reported.[9] However, there is a lack of data in binary mixtures of commonly used organic solvents such as low molecular weight alcohols in the literature. The aim of this work is to report the experimental solubility data of phenanthrene in a number of nonaqueous binary solvent mixtures at 298.2 K. The mixtures include ethanol + methanol, methanol + 2-propanol, ethanol + 2-propanol, 1-propanol + 2-propanol, and 1-butanol + 2-propanol. Another attempt in the present work is to use the Jouyban−Acree model and its combined form with Abraham's solvation parameters for predicting the solubility in mixed solvents.[10] The Jouyban−Acree model is shown as

$$\ln C_{\mathrm{m}}^{\mathrm{Sat}} = x_1 \cdot \ln C_1^{\mathrm{Sat}} + x_2 \cdot \ln C_2^{\mathrm{Sat}} + x_1 \cdot x_2 \cdot \sum_{i=0}^{2} J_i \cdot (x_1 - x_2)^i \tag{1}$$

where $C_{\mathrm{m}}^{\mathrm{Sat}}$ is the solute mole per liter solubility in the binary solvent mixtures; x_1 and x_2 are the mole fractions of solvents 1 and 2 in the absence of the solute; C_1^{Sat} and C_2^{Sat} denote the mole per liter solubility of the solute in neat solvents 1 and 2; and J_i is the solvent−solvent and solute−solvent interaction terms. In a previous work, quantitative structure−property relationships (QSPR) based on the Jouyban−Acree model and Abraham's solvation parameters were proposed where J_i terms of the Jouyban−Acree model have been calculated using Abraham solvation parameters of the solute and solvents as follows for water-to-solvent coefficients of the solvents

$$J_0 = 0.028 + 2.123(c_1 - c_2)^2 - 0.160E(e_1 - e_2)^2 + 0.282S(s_1 - s_2)^2 + 1.713B(b_1 - b_2)^2 + 2.006V \cdot (v_1 - v_2)^2 \tag{2}$$

$$J_1 = 0.033 + 0.670(c_1 - c_2)^2 - 0.447E(e_1 - e_2)^2 + 0.051S(s_1 - s_2)^2 + 0.476B(b_1 - b_2)^2 - 0.234V(v_1 - v_2)^2 \tag{3}$$

$$J_2 = 0.022 + 2.024(c_1 - c_2)^2 - 0.204E(e_1 - e_2)^2 + 0.034S(s_1 - s_2)^2 + 0.243B(b_1 - b_2)^2 + 0.848V(v_1 - v_2)^2 \tag{4}$$

and for gas-to-solvent coefficients of the solvents

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$$J_0 = 0.062 + 0.118(c_1 - c_2)^2 - 0.332E(e_1 - e_2)^2 + 0.410S(s_1 - s_2)^2 + 2.399B(b_1 - b_2)^2 + 15.715L(l_1 - l_2)^2 \quad (5)$$

$$J_1 = 0.103 - 1.864(c_1 - c_2)^2 - 1.590E(e_1 - e_2)^2 + 0.119S(s_1 - s_2)^2 + 1.010B(b_1 - b_2)^2 - 9.493L(l_1 - l_2)^2 \quad (6)$$

$$J_2 = -0.008 + 1.075(c_1 - c_2)^2 + 0.053E(e_1 - e_2)^2 + 0.084S(s_1 - s_2)^2 + 0.414B(b_1 - b_2)^2 + 7.727L(l_1 - l_2)^2 \quad (7)$$

where c, e, s, b, v, and l are the Abraham solvation coefficients of the solvents; subscripts 1 and 2 denote solvents 1 and 2; E is the excess molar refraction of the solute; S is the dipolarity/polarizability of the solute; B stands for the solute's hydrogen-bond basicity; V is the McGowan volume of the solute; and L is the logarithm of the solute's gas−hexadecane partition coefficient at 298.15 K.[10] The numerical values of c, e, s, b, v, and l were taken from the published literature[11] and are listed in Table 1.

Experimental Method

Materials. Phenanthrene (purity > 98 %) was purchased from Merck and used as received. Its purity was checked by thin layer chromatography,[12] and also its melting temperature (374.1 K) was determined using a differential scanning calorimeter (Shimadzu, Japan). The measured melting temperature was slightly higher than the reported values in the literature (372.4 K,[13] 372.3 K[14]). Methanol (99.5 %), ethanol (99.9 %), 1-propanol (99.5 %), 2-propanol (99.5 %), and 1-butanol (99.5 %) were also purchased from Merck. The water contents of the solvents were checked using Karl Fischer titration, and the found water contents were 0.04 %, 0.13 %, 0.07 %, 0.03 %, and 0.30 %, respectively, for methanol, ethanol, 1-propanol, 2-propanol, and 1-butanol.

Apparatus and Procedures. The binary mixtures of solvents were prepared as volume fractions from 0 to 1 at 0.10 intervals, and the mole fractions of the solvents were computed from the volume fractions employing the densities of the pure solvents taken from the literature. The solvent composition could be calculated with the uncertainty of 0.002 in mole fraction. The solubility of phenanthrene was determined by equilibrating an excess amount of the solid with the binary solvent mixtures using a shaker (Behdad, Tehran, Iran) placed in an incubator equipped with a temperature controlling system at (298.2 ± 0.2) K (Nabziran, Tabriz, Iran). For assurance of equilibrium, samples were incubated for 3 days. Samples of the saturated solutions were centrifuged in 10 000 rpm for 10 min (MSE Micro Center MSB010.CX2.5, SANYO, Muriguchi City, Japan), and then 0.250 mL of the saturated solutions was diluted in a 10 mL flask by adding acetone for spectrophotometric analysis. Absorbances of the diluted solutions were recorded at 345 nm using a UV−vis spectrophotometer (Beckman DU-650, Fullerton, USA). Molar concentrations of the dilute solutions were determined from a UV absorbance calibration graph with the molar absorptivities of phenanthrene ranging from $\varepsilon/(\mathrm{L \cdot mol^{-1} \cdot cm^{-1}}) = 213$ to $\varepsilon/(\mathrm{L \cdot mol^{-1} \cdot cm^{-1}}) = 203$ for phenanthrene compositions ranging from $(3.4 \cdot 10^{-3}$ to $5.6 \cdot 10^{-3})$ $\mathrm{mol \cdot L^{-1}}$. Each experimental data point is an average of at least three experimental measurements with the measured $\mathrm{mol \cdot L^{-1}}$ solubilities being reproducible to within ± 3.5 %. Calculated standard deviations ranged from $(\sigma_{n-1} = 0.001$ to $\sigma_{n-1} = 0.016)$ $\mathrm{mol \cdot L^{-1}}$. Densities of the saturated solutions were determined using a 5 mL pycnometer with the uncertainty of $(\sigma_{n-1} = 0.001$ to $\sigma_{n-1} = 0.013)$ $\mathrm{g \cdot cm^{-3}}$.

Table 1. Abraham Solvent Coefficients of the Solvents[11]

water to solvent	c	e	s	a	b	v
1-butanol	0.152	0.437	−1.175	0.098	−3.914	4.119
1-propanol	0.148	0.436	−1.098	0.389	−3.893	4.036
2-propanol	0.063	0.32	−1.024	0.445	−3.824	4.067
ethanol	0.208	0.409	−0.959	0.186	−3.645	3.928
methanol	0.329	0.299	−0.671	0.08	−3.389	3.512
gas to solvent	c	e	s	a	b	l
1-butanol	−0.039	−0.276	0.539	3.781	0.995	0.934
1-propanol	−0.028	−0.185	0.648	4.022	1.043	0.869
2-propanol	−0.06	−0.335	0.702	4.017	1.04	0.893
ethanol	0.012	−0.206	0.789	3.635	1.311	0.853
methanol	−0.004	−0.215	1.173	3.701	1.432	0.769

Computational Methods. Using the measured phenanthrene solubilities in solvents 1 and 2, and the calculated coefficients of the Jouyban−Acree model based on Abraham's solvation parameters, the solubility of phenanthrene was predicted for the five binary solvent mixtures at each composition studied. In numerical method I, the J_i terms of eq 1 were computed employing eqs 2 to 4, whereas in numerical method II, eqs 5 to 7 were used to calculate the J_i terms. For checking the full predictive capability of the model, solubility amounts in neat solvents were predicted using eqs 8 and 9

$$\log\left(\frac{C_S}{C_W}\right) = c + eE + sS + aA + bB + vV \quad (8)$$

$$\log\left(\frac{C_S}{C_G}\right) = c + eE + sS + aA + bB + lL \quad (9)$$

The predicted solubilities of phenanthrene in both neat solvents were used in eq 1 along with the J_i terms computed from eqs 2 to 4 and 5 to 7. These were called numerical methods III and IV, respectively. In eqs 8 and 9, C_S and C_W represent the molar solubility of the solute in the neat organic solvent and in water in units of $\mathrm{mol \cdot L^{-1}}$, respectively; a is the Abraham solvent coefficient; A denotes the solute's hydrogen-bond acidity; and C_G is the gas phase concentration of the solute.[11] The numerical values of Abraham's solute parameters for phenanthrene are: $\log C_W = -5.17$, $\log C_G = -7.97$, $E = 2.055$, $S = 1.29$, $A = 0.00$, $B = 0.29$, $V = 1.4544$, and $L = 7.632$.[11]

For all predicted solubilities in mixed solvents (C_m^{Sat}), the mean deviations (MD) were calculated as a criterion of error by

$$\mathrm{MD} = \frac{\sum \left[\frac{|(C_m^{Sat})_{pred} - (C_m^{Sat})_{exp}|}{(C_m^{Sat})_{exp}} \right]}{N} \quad (10)$$

where N is the number of data points in each set.

Results and Discussion

Mole fraction compositions of the binary solvent mixtures, densities of the saturated solutions, and the experimental phenanthrene solubility data are reported in Table 2. Also

Table 2. Solvent Composition, Density ρ of Saturated Solutions, Experimental Solubility of Phenanthrene C_m^{Sat} in Binary Mixtures at 298.2 K, and the Predicted Values Using Numerical Methods I to IV

		$C_m^{Sat}/(mol \cdot L^{-1})$				
x_1	$\rho/g \cdot cm^{-3}$	experimental	method I[a]	method II[a]	method III[a]	method IV[a]
		ethanol (1) + methanol (2)				
1.000	0.798	0.187	0.187	0.187	0.198	0.336
0.862	0.799	0.192	0.195	0.199	0.199	0.359
0.735	0.799	0.180	0.195	0.204	0.192	0.368
0.618	0.798	0.177	0.190	0.204	0.183	0.369
0.510	0.799	0.178	0.185	0.202	0.172	0.366
0.409	0.798	0.170	0.178	0.198	0.161	0.358
0.316	0.797	0.165	0.171	0.192	0.151	0.346
0.229	0.798	0.152	0.163	0.182	0.141	0.329
0.148	0.797	0.147	0.154	0.168	0.130	0.305
0.071	0.796	0.138	0.144	0.152	0.119	0.275
0.000	0.797	0.133	0.133	0.133	0.108	0.241
		methanol (1) + 2-propanol (2)				
1.000	0.797	0.133	0.133	0.133	0.108	0.241
0.944	0.798	0.139	0.145	0.147	0.118	0.265
0.883	0.798	0.148	0.155	0.161	0.127	0.288
0.815	0.799	0.154	0.163	0.174	0.134	0.309
0.739	0.798	0.153	0.169	0.187	0.140	0.329
0.654	0.797	0.160	0.173	0.200	0.144	0.349
0.557	0.796	0.157	0.174	0.212	0.146	0.366
0.447	0.795	0.147	0.173	0.222	0.146	0.379
0.321	0.794	0.142	0.167	0.223	0.143	0.375
0.174	0.794	0.135	0.154	0.197	0.134	0.327
0.000	0.791	0.124	0.124	0.124	0.109	0.201
		ethanol (1) + 2-propanol (2)				
1.000	0.798	0.187	0.187	0.187	0.198	0.336
0.922	0.799	0.184	0.184	0.185	0.192	0.331
0.840	0.798	0.176	0.180	0.182	0.185	0.323
0.753	0.797	0.170	0.174	0.178	0.177	0.313
0.663	0.797	0.170	0.168	0.173	0.168	0.302
0.567	0.796	0.169	0.162	0.168	0.159	0.289
0.466	0.795	0.159	0.155	0.161	0.149	0.276
0.359	0.794	0.152	0.148	0.154	0.139	0.261
0.247	0.793	0.143	0.141	0.146	0.129	0.244
0.127	0.792	0.138	0.133	0.136	0.119	0.225
0.000	0.791	0.124	0.124	0.124	0.109	0.201
		1-propanol (1) + 2-propanol (2)				
1.000	0.812	0.174	0.174	0.174	0.158	0.247
0.902	0.810	0.165	0.169	0.170	0.153	0.245
0.803	0.806	0.161	0.164	0.166	0.148	0.242
0.705	0.804	0.157	0.159	0.161	0.143	0.238
0.605	0.802	0.153	0.154	0.156	0.138	0.234
0.505	0.800	0.148	0.149	0.151	0.133	0.230
0.405	0.798	0.141	0.143	0.146	0.128	0.225
0.305	0.796	0.134	0.138	0.141	0.123	0.220
0.204	0.793	0.130	0.133	0.135	0.118	0.215
0.102	0.790	0.122	0.129	0.130	0.113	0.208
0.000	0.791	0.124	0.124	0.124	0.109	0.201
		1-butanol (1) + 2-propanol (2)				
1.000	0.821	0.188	0.188	0.188	0.166	0.344
0.883	0.818	0.183	0.181	0.185	0.160	0.333
0.770	0.815	0.174	0.173	0.180	0.153	0.320
0.661	0.812	0.168	0.166	0.173	0.146	0.305
0.556	0.808	0.159	0.158	0.167	0.140	0.290
0.455	0.807	0.156	0.152	0.160	0.134	0.275
0.358	0.804	0.146	0.145	0.153	0.128	0.261
0.264	0.799	0.138	0.140	0.147	0.123	0.246
0.173	0.796	0.135	0.134	0.139	0.118	0.232
0.085	0.794	0.124	0.129	0.132	0.113	0.217
0.000	0.791	0.124	0.124	0.124	0.109	0.201

[a] Numerical methods I and III, the J_i terms of eq 1 were computed employing eqs 2 to 4; methods II and IV, eqs 5 to 7 were used to calculate the J_i terms. In methods I and II, the experimental values of C_1^{Sat} and C_2^{Sat} were used in eq 1, whereas in methods III and IV, the predicted C_1^{Sat} and C_2^{Sat}, respectively, by eqs 8 and 9 were used in the predictions.

Table 3. Experimental, Literature Reported, and the Predicted Solubility Values in Neat Solvents by Equations 8 and 9 and Their Mean Deviation (MD) from the Generated Data from This Work

	C_1^{Sat} or C_2^{Sat}						
	$mol \cdot L^{-1}$				$100 \cdot MD$		
solvent	this work	ref 11	eq 8	eq 9	ref 11	eq 8	eq 9
1-butanol	0.188	0.190	0.166	0.344	1.3	11.6	83.2
1-propanol	0.174	0.178	0.158	0.247	2.3	9.1	42.1
2-propanol	0.124	0.126	0.109	0.201	1.8	11.8	62.6
ethanol	0.187	0.186	0.198	0.336	0.1	6.2	80.2
methanol	0.133	0.142	0.108	0.241	6.9	18.8	81.1

Table 4. Values of the Mean Deviation (MD) for Prediction Methods I to IV

		$100 \cdot MD$			
solvent 1	solvent 2	method I	method II	method III	method IV
ethanol	methanol	4.1	11.2	8.1	100.9
methanol	2-propanol	8.6	24.1	9.2	114.7
ethanol	2-propanol	1.8	1.6	7.0	74.5
1-propanol	2-propanol	1.8	2.9	9.0	56.7
1-butanol	2-propanol	1.1	3.3	12.1	77.8
overall MD %		3.5	8.6	9.1	84.9

tabulated in Table 2 are the predicted values from methods I to IV. There is good agreement between our measured solubility data in the monosolvents and data from the literature which are compared with each other in Table 3.[11] The mole fraction solubilities of phenanthrene in methanol (0.00543[15] and 0.00589[16]), ethanol (0.01282[15] and 0.01114[16]), 1-propanol (0.01355[16]), 2-propanol (0.00977[16]), and 1-butanol (0.01771[16]) from the literature are in good agreement with our mole fraction data for methanol (0.00548), ethanol (0.01114), 1-propanol (0.01321), 2-propanol (0.00960), and 1-butanol (0.01739). The maximum phenanthrene solubilities were observed in (0.862, 0.654, 1.000, 1.000, and 1.000) mol fractions of solvent 1, respectively, in the mixtures of ethanol (1) + methanol (2), methanol (1) + 2-propanol (2), ethanol (1) + 2-propanol (2), 1-propanol (1) + 2-propanol (2), and 1-butanol (1) + 2-propanol (2). The predicted solubilities in the mixed solvents using numerical methods I to IV are compared with the experimental data, and the calculated MD values are summarized in Table 4. The minimum MDs for methods I to IV are observed for the solubility data of phenanthrene in 1-butanol + 2-propanol (1.1 %), ethanol + 2-propanol (1.6 %), ethanol + 2-propanol (7.0 %), and 1-propanol + 2-propanol (56.7 %), respectively. The maximum MDs of methods I to IV are obtained for methanol + 2-propanol (8.6 %), methanol + 2-propanol (24.1 %), 1-butanol + 2-propanol (12.1 %), and methanol + 2-propanol (114.7 %), and the overall MDs (± SD) for methods I to IV are 3.5 (± 3.1) %, 8.6 (± 9.4) %, 9.1 (± 1.9) %, and 84.9 (± 22.9) %, respectively. Methods III and IV could be considered as ab initio methods since no experimental data are required in the prediction processes. From these methods, method IV produced relatively high prediction error which is due to the high prediction errors of eq 9 for the solubility of phenanthrene in monosolvents as shown in Table 3. Of the four methods considered, method I provided the better predicted results, which is in agreement with previous findings.[10,17] As a full predictive approach, method III gave acceptable results in comparison with the other method. It has been shown that the predictability of the proposed model is acceptable when compared against the experimental errors. Our computational results suggest that method III can be used for prediction of the solubility of phenanthrene in different nonaqueous solvent mixtures.

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