Solubility of Phenanthrene in Binary Mixtures of C_1-C_4 Alcohols at 298.2 K

Mohammad A. A. Fakhree,[†] William E. Acree, Jr.,[‡] and Abolghasem Jouyban*[,§]

Biotechnology Research Center, Faculty of Pharmacy and Drug Applied Research Center, Tabriz University of Medical Sciences, Tabriz 51664, Iran, and Department of Chemistry, University of North Texas, Denton, Texas 76203-5070

Experimental solubilities are reported for phenanthrene in binary solvent mixtures of methanol + 1-propanol, methanol + 1-butanol, ethanol + 1-propanol, ethanol + 1-butanol, and 1-propanol + 1-butanol 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' predicted values from the measured data varied between (1.7 and 42.5) %. The overall MDs (OMDs) for the prediction methods were 2.8 (± 2.7) % and 14.3 (± 23.3) %, respectively, for water-to-solvent and gas-to-solvent coefficients. Using ab initio prediction methods, the MDs varied between (6.2 and 155.8) %, and the OMDs were 11.4 (± 6.5) % and 92.1 (± 48.0) %.

Introduction

One of the critical physicochemical properties of a compound is its solubility. Solubility in a given solvent is important because of its role in providing suitable media for a substrate to be dissolved, adsorbed, and precipitated. These subjects have attracted considerable attention in the chemical and pharmaceutical industries, and researchers try to find appropriate solvent systems for these purposes. However, a single solvent may not possess sufficient solubilization power, hence different kinds of solubilization methods have been employed such as cosolvency, complexation, and micellization.[1−6] Experimental determination is the most common method to find a suitable solubilization system; however, as an alternative, one can use accurate predictive models for this purpose.

Phenanthrene, a polycyclic aromatic hydrocarbon (PAH), can be used in solubility studies as a model compound because of its simple structure. Its solubility has been reported in binary aqueous mixtures of methanol, ethanol, 1-propanol, acetone, *n*-butylamine, and tetrahydrofuran[3−8] and in nonaqueous mixtures of toluene + heptane and toluene + 2,2,4-trimethylpentane.[9] In a recent paper, solubility data in binary mixtures of methanol + ethanol, methanol + 2-propanol, ethanol + 2-propanol, 1-propanol + 2-propanol, and 1-butanol + 2-propanol have been reported.[10] The aim of this work is to report the experimental solubility data of phenanthrene in primary alcohol mixtures including methanol + 1-propanol, methanol + 1-butanol, ethanol + 1-propanol, ethanol + 1-butanol, and 1-propanol + 1-butanol at 298.2 K. A second objective of 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.[11] The Jouyban−Acree model is shown as

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

where C_m^{Sat} is the solute mol·L^{-1} 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 mol·L^{-1} 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,[11] 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

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

$$J_1 = 0.033 + 0.670(c_1 - c_2)^2 - 0.477E(e_1 - e_2)^2 + 0.051S(s_1 - s_2)^2 + 0.476B(b_1 - b_2)^2 - 0.234V(v_1 - v_2)^2 \tag{3}$$

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

and for gas-to-solvent coefficients of the solvents

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

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

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* Author to whom correspondence should be addressed. E-mail: ajouyban@hotmail.com. Fax: +98 411 3363231.
[†] Biotechnology Research Center.
[‡] University of North Texas.
[§] Faculty of Pharmacy and Drug Applied Research Center.

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

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 McGowan volume of the solute; and L is the logarithm of the solute's gas−hexadecane partition coefficient at 298.15 K.[11] The numerical values of c, e, s, b, v, and l were taken from the literature[12] and are listed in Table 1.

Experimental Method

Materials. Phenanthrene (purity > 98 % in mass fraction) was purchased from Merck and recrystallized several times using acetone. Its purity was checked by thin-layer chromatography,[13] and also its melting temperature (372.15 K) was determined. Methanol (99.5 % in mass fraction), ethanol (99.9 % in mass fraction), 1-propanol (99.5 % in mass fraction), and 1-butanol (99.5 % in mass fraction) were 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 %, and 0.30 %, respectively, for methanol, ethanol, 1-propanol, and 1-butanol.

Apparatus and Procedures. The binary mixtures of the solvents were prepared as volume fractions from 0 to 1 at 0.10 intervals, and the mole fractions of the solvents were computed using the densities of the solvents. The solvent composition could be calculated with the uncertainty of 0.001 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 10000 rpm for 10 min (MSE Micro Center MSB010.CX2.5, SANYO, Japan) and then were diluted using 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 $\varepsilon/(\mathrm{L \cdot mol^{-1} \cdot cm^{-1}}) = 261$ to $\varepsilon/(\mathrm{L \cdot mol^{-1} \cdot cm^{-1}}) = 267$ for phenanthrene compositions ranging from $(2.2 \cdot 10^{-3}$ to $4.5 \cdot 10^{-3})$ mol·L^{-1}. Each experimental data point is an average of at least three experimental measurements with the measured mol·L^{-1} solubilities being reproducible to within ± 4.2 %. Calculated standard deviations ranged from $(\sigma_{n-1} = 0.001$ to $\sigma_{n-1} = 0.076)$ mol·L^{-1}. Densities of the saturated solutions were determined using a 5 mL pycnometer with the uncertainty of $(\sigma_{n-1} = 0.001$ to $\sigma_{n-1} = 0.014)$ g·cm^{-3}.

Table 1. Abraham Solvent Coefficients of the Solvents[12]

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
ethanol	0.208	0.409	−0.959	0.186	−3.645	3.928
methanol	0.329	0.299	−0.671	0.080	−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
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

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·L^{-1}				
x_1	ρ/g·cm^{-3}	exptl	method I[a]	method II[a]	method III[a]	method IV[a]
		Methanol (1) + 1-Propanol (2)				
1.000	0.796	0.133	0.133	0.133	0.108	0.241
0.943	0.798	0.156	0.146	0.146	0.119	0.262
0.881	0.801	0.173	0.158	0.160	0.130	0.282
0.812	0.803	0.183	0.169	0.174	0.140	0.301
0.735	0.805	0.193	0.179	0.187	0.150	0.318
0.649	0.807	0.200	0.188	0.201	0.159	0.335
0.552	0.808	0.208	0.196	0.215	0.168	0.350
0.442	0.809	0.202	0.202	0.228	0.175	0.361
0.316	0.810	0.196	0.204	0.235	0.179	0.361
0.170	0.811	0.187	0.199	0.224	0.178	0.333
0.000	0.811	0.174	0.174	0.174	0.158	0.247
		Methanol (1) + 1-Butanol (2)				
1.000	0.796	0.133	0.133	0.133	0.108	0.241
0.953	0.800	0.149	0.147	0.158	0.119	0.285
0.900	0.803	0.163	0.161	0.186	0.131	0.336
0.841	0.806	0.181	0.176	0.218	0.144	0.393
0.772	0.808	0.198	0.190	0.254	0.156	0.458
0.693	0.811	0.207	0.204	0.297	0.168	0.534
0.601	0.815	0.217	0.217	0.348	0.180	0.624
0.492	0.815	0.219	0.228	0.406	0.190	0.727
0.361	0.818	0.216	0.236	0.455	0.198	0.813
0.201	0.819	0.213	0.233	0.422	0.197	0.752
0.000	0.826	0.194	0.194	0.194	0.166	0.344
		Ethanol (1) + 1-Propanol (2)				
1.000	0.801	0.182	0.182	0.182	0.198	0.336
0.920	0.802	0.183	0.183	0.185	0.197	0.334
0.837	0.803	0.186	0.184	0.186	0.195	0.330
0.749	0.805	0.187	0.184	0.187	0.192	0.324
0.658	0.806	0.189	0.184	0.187	0.189	0.316
0.562	0.807	0.190	0.183	0.186	0.185	0.306
0.461	0.809	0.190	0.182	0.184	0.180	0.295
0.354	0.810	0.188	0.180	0.181	0.175	0.283
0.243	0.812	0.187	0.179	0.179	0.170	0.271
0.125	0.814	0.184	0.177	0.176	0.164	0.259
0.000	0.811	0.174	0.174	0.174	0.158	0.247
		Ethanol (1) + 1-Butanol (2)				
1.000	0.801	0.182	0.182	0.182	0.198	0.336
0.934	0.802	0.186	0.186	0.194	0.199	0.357
0.862	0.804	0.197	0.189	0.204	0.200	0.375
0.785	0.807	0.203	0.192	0.214	0.199	0.392
0.702	0.808	0.205	0.194	0.222	0.197	0.406
0.610	0.811	0.204	0.196	0.230	0.194	0.419
0.511	0.813	0.203	0.197	0.237	0.191	0.430
0.402	0.815	0.198	0.198	0.242	0.187	0.437
0.282	0.817	0.195	0.198	0.242	0.181	0.434
0.148	0.818	0.193	0.197	0.229	0.175	0.408
0.000	0.826	0.194	0.194	0.194	0.166	0.344
		1-Propanol (1) + 1-Butanol (2)				
1.000	0.811	0.174	0.174	0.174	0.158	0.247
0.917	0.808	0.175	0.176	0.183	0.160	0.265
0.830	0.810	0.171	0.179	0.190	0.161	0.281
0.741	0.811	0.172	0.181	0.197	0.162	0.297
0.647	0.813	0.177	0.183	0.203	0.163	0.313
0.550	0.814	0.182	0.185	0.209	0.164	0.328
0.449	0.814	0.188	0.187	0.214	0.164	0.343
0.344	0.817	0.190	0.189	0.217	0.165	0.356
0.234	0.818	0.193	0.190	0.217	0.165	0.364
0.120	0.822	0.194	0.192	0.210	0.166	0.362
0.000	0.826	0.194	0.194	0.194	0.166	0.344

[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.

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

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

	C_1^{Sat} or C_2^{Sat}								
	$\mathrm{mol \cdot L^{-1}}$					100·MD			
solvent	this work	ref 10	ref 12	eq 8	eq 9	ref 10	ref 12	eq 8	eq 9
1-butanol	0.194	0.188	0.190	0.166	0.344	3.1	2.1	14.4	77.3
1-propanol	0.174	0.174	0.178	0.158	0.247	0.0	2.3	9.2	42.0
ethanol	0.182	0.187	0.186	0.198	0.336	2.7	2.2	8.8	84.6
methanol	0.133	0.133	0.142	0.108	0.241	0.0	6.8	18.8	81.2

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, solubilities of phenanthrene in neat solvents were predicted using eqs 8 and 9

$$\log\left(\frac{C_S}{C_W}\right) = c + e \cdot E + s \cdot S + a \cdot A + b \cdot B + v \cdot V \tag{8}$$

$$\log\left(\frac{C_S}{C_G}\right) = c + e \cdot E + s \cdot S + a \cdot A + b \cdot B + l \cdot L \tag{9}$$

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 moles per liter, 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.[12] 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 $L = 7.632$.[12]

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

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

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

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

		100·MD			
solvent 1	solvent 2	method I	method II	method III	method IV
methanol	1-propanol	4.8	7.1	17.2	68.9
methanol	1-butanol	3.1	42.5	16.2	155.8
ethanol	1-propanol	2.3	1.8	6.2	61.9
ethanol	1-butanol	2.3	10.5	6.6	100.5
1-propanol	1-butanol	1.7	9.8	10.6	73.6
	overall MD %	2.8	14.3	11.4	92.1

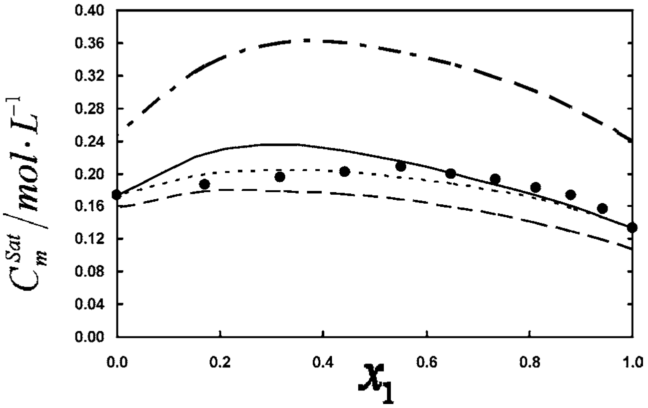

Figure 1. Experimental and predicted moles per liter solubility of phenanthrene at various mole fractions of methanol in methanol (1) + 1-propanol (2) mixtures: ●, experimental. The computed solubilities using: - - - -, method I; —, method II; — —, method III; and — - — -, method IV.

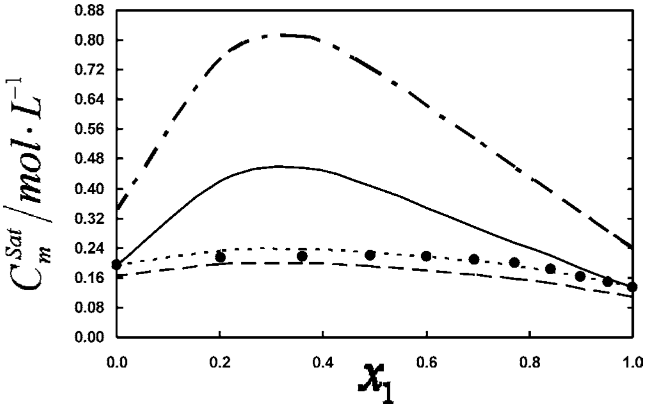

Figure 2. Experimental and predicted moles per liter solubility of phenanthrene at various mole fractions of methanol in methanol (1) + 1-butanol (2) mixtures: ●, experimental. The computed solubilities using: - - - -, method I; —, method II; — —, method III; — - — -, method IV.

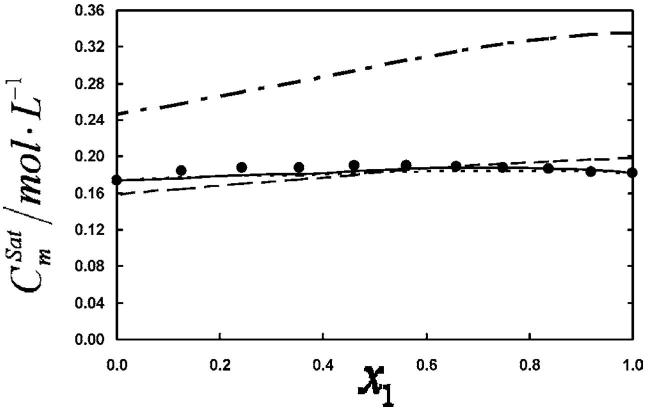

Figure 3. Experimental and predicted moles per liter solubility of phenanthrene at various mole fractions of ethanol in ethanol (1) + 1-propanol (2) mixtures: ●, experimental. The computed solubilities using: - - - -, method I; —, method II; — —, method III; — - — -, method IV.

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 tabulated in Table 2 are the predicted values from numerical methods I to IV. There is good agreement between measured solubility data in the monosolvents and data from the literature which are compared with each other in Table 3.[10,12] The difference between solubility data of previously reported data which used phenanthrene without recrystallization[10] and measured data using recrystallized phenanthrene is less than 5 % (see Table 3). The maximum phenanthrene solubilities were observed in (0.552, 0.601, 0.461, 0.402, and 0.000) mol fractions of the solvent 1, respectively, in the mixtures of methanol (1) + 1-propanol (2), methanol (1) + 1-butanol (2), ethanol (1) + 1-propanol (2), ethanol (1) + 1-butanol (2), and 1-propanol (1)

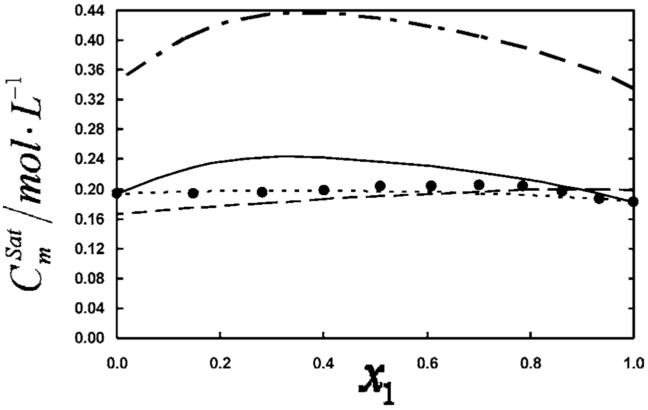

Figure 4. Experimental and predicted moles per liter solubility of phenanthrene at various mole fractions of ethanol in ethanol (1) + 1-butanol (2) mixtures: ●, experimental. The computed solubilities using: - - - -, method I; —, method II; — —, method III; — - — -, method IV.

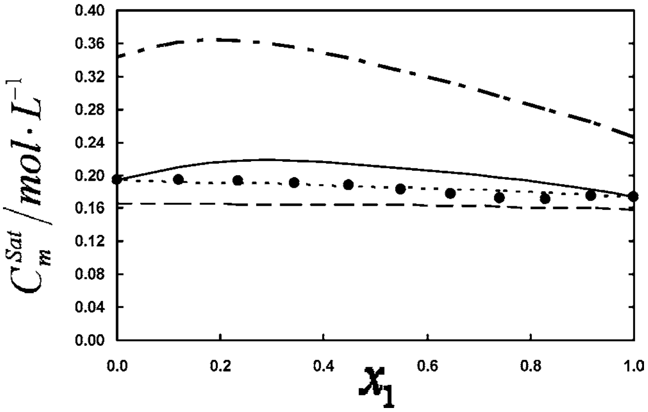

Figure 5. Experimental and predicted mole per liter solubility of phenanthrene at various mole fractions of 1-propanol in 1-propanol (1) + 1-butanol (2) mixtures: ●, experimental. The computed solubilities using: - - - -, method I; —, method II; — —, method III; — - — -, method IV.

+ 1-butanol (2). The predicted solubilities in the mixed solvents using numerical methods I to IV were 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 ethanol + 1-butanol (1.6 %), ethanol + 1-propanol (1.7 %), ethanol + 1-propanol (6.1 %), and ethanol + 1-propanol (62.1 %), respectively. The maximum MDs of methods I to IV are obtained for methanol + 1-propanol (3.6 %), methanol + 1-butanol (42.9 %), methanol + 1-propanol (16.1 %), and methanol + 1-butanol (156.5 %), and the overall MDs (± SD) were 2.6 (± 2.4) %, 15.3 (± 23.4) %, 10.8 (± 6.1) %, and 94.5 (± 48.3) %. Method IV gives a high prediction MD because of its higher uncertainty in predicting solubility in neat solvents. Figures 1 to 5 illustrated the experimental and predicted solubility of phenanthrene at various mole fractions of the solvent 1 in the mixtures. On the basis of these figures and the MDs of methods I to IV, it is obvious that method I provides better results which is in agreement with previous findings.[10,11,14] As an ab initio approach, method III gave acceptable results in comparison with the other method which is in agreement with other works.[10,11,14] It has been shown that the predictability of the proposed model is acceptable when compared against the experimental uncertainty. On the basis of previous[10] and recent reports, it is possible to suggest that method III can be used for prediction of the solubility of phenanthrene in different nonaqueous solvent mixtures.

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