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Short communication

New empirical expressions to correlate solubilities of solids in supercritical carbon dioxide

Chandrasekhar Garlapati, Giridhar Madras*

Department of Chemical Engineering, Indian Institute of Science, Bangalore 560012, India

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ABSTRACT

Supercritical processes are gaining importance in the last few years in the food, environmental and pharmaceutical product processing. The design of any supercritical process needs accurate experimental data on solubilities of solids in the supercritical fluids (SCFs). The empirical equations are quite successful in correlating the solubilities of solid compounds in SCF both in the presence and absence of cosolvents. In this work, existing solvate complex models are discussed and a new set of empirical equations is proposed. These equations correlate the solubilities of solids in supercritical carbon dioxide (both in the presence and absence of cosolvents) as a function of temperature, density of supercritical carbon dioxide and the mole fraction of cosolvent. The accuracy of the proposed models was evaluated by correlating 15 binary and 18 ternary systems. The proposed models provided the best overall correlations.

1. Introduction

Supercritical fluid technology (SFT) finds applications in chemical, biochemical, pharmaceutical and food processing industries. Supercritical fluids (SCFs) have diffusivities between that of gases and liquids, compressibilities comparable to gases, densities comparable to liquids and negligible surface tension. These properties make them attractive solvents for many industrial applications [1]. Carbon dioxide (CO_2) is commonly used as a supercritical fluid because it is nontoxic and nonflammable. Polar substances are poorly soluble in $SCCO_2$ because of its lack of polarity. The solubilities can be enhanced by adding cosolvents [2,3]. Solubility data in SCFs are important for the successful implementation of SFT. An exhaustive account of experimental data on the solubilities of various organics in SCFs is available [4]. The experimental determination of solubilities of organic solids in SCFs at various temperatures and pressures is expensive. Therefore, modeling and prediction of solubilities is essential [5].

Models used for correlating solubilities can be broadly classified as equation of state (EOS) based models and empirical models. EOS based models require parameters such as critical constants and the sublimation pressure of the solids. These are normally not available for many high molecular weight compounds and are calculated using group contribution methods, which could lead to error. Therefore, empirical models are used in correlating solubilities of solids in $SCCO_2$ [6–10].

In this study, solvate complex models published in the literature are discussed. A new set of empirical equations is proposed to correlate solubilities of high molecular weight solids in $SCCO_2$. The accuracy of the proposed models was evaluated by correlating solubilities of various solids in supercritical fluids both in the presence and absence of cosolvents for several systems.

2. Theoretical section

2.1. Solubilities of organic solids in SCF

Chrastil [11] correlated the solubility of a solute (y_2) in SCFs to the density (ρ_1, g mL^{-1}) and temperature (T, K) as

$$\ln(y_2) = A_0 \ln(\rho_1) + \frac{A_1}{T} + A_2 \tag{1}$$

where A_0–A_2 are the model constants that can be estimated from experimental solubility data in SCF.

The solubility of a solute in SCFs can be correlated to the density and temperature by the modified Adachi-Lu equation [12]

$$\ln(y_2) = (B_0 + B_1\rho_1) \ln(\rho_1) + \frac{B_2}{T} + B_3 \tag{2}$$

where B_0–B_3 are the model constants.

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* Corresponding author. Tel.: +91 80 22932321; fax: +91 80 23600683.
E-mail addresses: giridharmadras@gmail.com, giridhar@chemeng.iisc.ernet.in (G. Madras).

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Table 1a
Range and source of binary system solubility data of various solutes in $SCCO_2$.

SN	Solute	T (K)	P (MPa)	Mole fraction ($\times 10^5$)	N_i	Reference
1	2-Chlorobiphenyl	313–333	10.0–30.0	40.1–561.0	48	[18]
2	4-Chlorobiphenyl	313–333	10.0–30.0	47.0–1740.0	48	[18]
3	2,2′-Dichlorobiphenyl	313–333	10.0–30.0	27.2–1210.0	48	[18]
4	4,4′-Dichlorobiphenyl	313–333	10.0–30.0	14.3–175.0	48	[18]
5	2,4′,5-Trichlorobiphenyl	313–333	10.0–30.0	31.4–393.0	48	[18]
6	3,3′,4,4′-Tetrachlorobiphenyl	313–333	10.0–30.0	0.08–9.71	48	[18]
7	2,2′,4,5,5′-Pentachlorobiphenyl	313–333	10.0–30.0	2.3–157.0	48	[18]
8	2,2′,4,4′,5,5′-Hexachlorobiphenyl	313–333	10.0–30.0	1.5–67.7	48	[18]
9	2,2′,3,4,4′,5,5′-Heptachlorobiphenyl	313–333	10.0–30.0	0.8–52.4	48	[18]
10	Benzoic acid	308–343	11.0–36.4	22.0–1280.0	39	[19]
11	Anthracene	313–333	11.8–35.59	2.2–18.3	90	[20]
12	Pyrene	313–333	11.4–35.0	2.2–55.0	96	[20]
13	Perylene	323–333	17.2–35.0	0.062–0.22	19	[20]
14	Flurbiprofen	303–323	8.9–24.5	1.672–19.63	27	[21]
15	Aspirin	308–328	12.0–25.0	6.3–34.7	24	[22]

González et al. [13] extended the Chrastil equation to model the solubilities in presence of a cosolvent as

$$\ln(y_2') = C_0 \ln(\rho_1) + C_1 \ln(y_3) + \frac{C_2}{T} + C_3 \tag{3}$$

where y_2' is solubility in presence of cosolvents and y_3 is the mole fraction of cosolvent and C_0–C_3 are model constants.

The solubility of solute in SCFs can be correlated to the density and temperature as [14,15]

$$\ln(y_2) = E_0 \ln(\rho_1 T) + E_1 \rho_1 + \frac{E_2}{T} + E_3 \tag{4}$$

where E_0–E_3 are the model constants.

Li et al. [14] correlated the solubility of a solute in SCFs in presence of cosolvent as

$$\ln(y_2') = F_0 \ln(y_3 \rho_1 T) + F_1 \rho_1 + \frac{F_2}{T} + F_3 \tag{5}$$

where F_0–F_3 are the model constants.

Garlapati and Madras [16] correlated the solubility of a solute in SCFs to the density and temperature as

$$\ln(y_2) = G_0 \ln(\rho_1 T) + \frac{G_1}{T} + G_2 \tag{6}$$

where G_0–G_2 are the model constants

Garlapati and Madras [16] correlated the solubilities of a solute in SCFs in presence of cosolvents as

$$\ln(y_2') = H_0 \ln(\rho_1 T) + H_1 \ln(y_3 T) + H_2 \ln(T) + \frac{H_3}{T} + H_4 \tag{7}$$

where H_0–H_4 are the model constants.

The density-based models (Eqs. (1)–(7)) indicate the existence of:

a. non-linear relationship between $\ln(y_2)$ and density (ρ_1) when temperature is constant (Isothermal condition).
b. non-linear relationship between $\ln(y_2)$ and temperature (T) when density is constant (Isopycnic condition).
c. linear relation between $\ln(y_2)$ and $\ln(\rho_{1T})$ in a certain range of density and temperature.
d. non-linear relationship between $\ln(y_2')$ and density (ρ_1) when cosolvent mole fraction and temperature are constant.
e. non-linear relationship between $\ln(y_2')$ and temperature (T) when cosolvent mole fraction and density are constant.
f. linear relation between $\ln(y_2')$ and $\ln(y_3)$ when temperature and density are constant.
g. linear relation between $\ln(y_2')$ and $\ln(y_3 \rho_{1T})$ in a certain range of density, temperature and cosolvent mole fraction.

Taking into account all the above observations for a better correlation, the following empirical equation was proposed for the solubilities of organic solids in $SCCO_2$ by Jouyban et al. [8,9]:

$$\ln(y_2) = m_0 + m_1 \rho_1 + m_2 P^2 + m_3 PT + \frac{m_4 T}{P} + m_5 \ln(\rho_1) \tag{8}$$

where m_0–m_5 are model constants. We propose a new model as

$$\ln(y_2) = M_0 + (M_1 + M_2 \rho_1) \ln(\rho_1) + \frac{M_3}{T} + M_4 \ln(\rho_1 T) \tag{9}$$

Table 1b
Range and source of ternary system solubility data of various solutes in presence of different cosolvents in $SCCO_2$.

SN	Solute–cosolvent	T (K)	P (MPa)	Cosolvent (mol %)	Mole fraction ($\times 10^5$)	N_i	Reference
1	2-Chlorobiphenyl–methanol	313–333	10.0–30.0	0.05	34.6–541.0	48	[18]
2	4-Chlorobiphenyl–methanol	313–333	10.0–30.0	0.05	43.5–1690.0	48	[18]
3	2,2′-Dichlorobiphenyl–methanol	313–333	10.0–30.0	0.05	25.8–1160.0	48	[18]
4	4,4′-Dichlorobiphenyl–methanol	313–333	10.0–30.0	0.05	13.2–171.0	48	[18]
5	2,4′,5-Trichlorobiphenyl–methanol	313–333	10.0–30.0	0.05	30.2–386.0	48	[18]
6	3,3′,4,4′-Tetrachlorobiphenyl–methanol	313–333	10.0–30.0	0.05	0.07–9.42	48	[18]
7	2,2′,4,5,5′-Pentachlorobiphenyl–methanol	313–333	10.0–30.0	0.05	2.1–152.	48	[18]
8	2,2′,4,4′,5,5′-Hexachlorobiphenyl–methanol	313–333	10.0–30.0	0.05	1.8–65.8	48	[18]
9	2,2′,3,4,4′,5,5′-Heptachlorobiphenyl–methanol	313–333	10.0–30.0	0.05	0.8–49.1	48	[18]
10	2-Chlorobiphenyl–n-butane	313–333	10.0–30.0	0.05	40.1–561.0	48	[18]
11	4-Chlorobiphenyl–n-butane	313–333	10.0–30.0	0.05	47.0–1740.0	48	[18]
12	2,2′-Dichlorobiphenyl–n-butane	313–333	10.0–30.0	0.05	27.2–1210.0	48	[18]
13	4,4′-Dichlorobiphenyl–n-butane	313–333	10.0–30.0	0.05	14.3–175.0	48	[18]
14	2,4′,5-Trichlorobiphenyl–n-butane	313–333	10.0–30.0	0.05	31.4–393.0	48	[18]
15	3,3′,4,4′-Tetrachlorobiphenyl–n-butane	313–333	10.0–30.0	0.05	0.08–9.71	48	[18]
16	2,2′,4,5,5′-Pentachlorobiphenyl–n-butane	313–333	10.0–30.0	0.05	2.3–157.0	48	[18]
17	2,2′,4,4′,5,5′-Hexachlorobiphenyl–n-butane	313–333	10.0–30.0	0.05	1.5–67.7	48	[18]
18	2,2′,3,4,4′,5,5′-Heptachlorobiphenyl–n-butane	313–333	10.0–30.0	0.05	0.8–52.4	48	[18]

Table 2a
Correlation results based on the proposed model (Eq. (9)) for various systems.

SN[a]	M_0	M_1	M_2 ($\times 10^3$)	M_3	M_4	AARD %	R_{adj}	F value
1	−54.59	−4.56	0.30	−572	6.37	3.74	0.9962	5030.25
2	−80.37	−9.12	0.53	−1492	11.10	4.22	0.9970	155.59
3	−77.20	−9.33	0.59	−2313	11.10	2.79	0.9987	20350.30
4	−78.50	−9.10	0.20	−2311	11.10	4.01	0.9940	9796.58
5	−82.12	−9.48	0.31	−196	11.10	3.65	0.9951	6297.23
6	−77.57	−9.19	0.83	−4558	11.10	5.03	0.9975	10374.77
7	−82.20	−9.53	0.78	−1259	11.10	4.02	0.9982	6917.29
8	−83.75	−9.16	0.50	−1332	11.10	4.36	0.9966	6249.93
9	−75.93	−9.64	0.77	−3460	11.10	3.44	0.9983	15546.6
10	−29.51	−1.10	0.43	−4794	3.51	0.30	0.9898	1994.91
11	101.99	24.18	−0.20	−12070	−18.82	6.03	0.9827	3201.78
12	−99.99	−11.09	0.491	−678	13.27	4.81	0.9936	5062.29
13	−48.47	−6.71	0.79	−575	6.20	1.16	0.9984	4980.43
14	21.25	−2.01	1.38	−8401	0.18	6.07	0.9848	1301.16
15	−5.62	1.99	0.36	−6160	0.09	5.41	0.9744	976.84

[a] SN, the system name and number is same as in Table 1a.

Table 2b
Correlation results based on the Jouyban et al. model (Eq. (8)) for various systems.

SN[a]	m_0	m_1 ($\times 10^3$)	m_2 ($\times 10^3$)	m_3 ($\times 10^3$)	m_4	m_5	AARD %	R_{adj}	F value
1	−24.86	1.19	−4.63	0.80	0.065	2.07	4.77	0.9932	2905.20
2	−33.12	−0.07	−7.43	1.30	0.088	3.21	7.97	0.9896	1919.86
3	−35.83	−1.94	−7.34	1.29	0.070	3.81	8.09	0.9899	44.76
4	−36.88	−3.96	−8.29	1.45	0.092	3.80	8.98	0.9735	1589.01
5	−27.97	−0.32	−5.92	1.04	0.080	2.46	6.24	0.9869	2253.98
6	−48.14	−1.50	−11.01	1.93	0.118	4.33	11.62	0.9872	1386.25
7	−33.60	2.18	−7.12	1.26	0.090	2.66	7.07	0.9941	2486.98
8	−35.85	0.12	−7.48	1.32	0.097	3.08	7.53	0.9902	2140.17
9	−41.54	−1.13	−9.38	1.68	0.109	3.73	9.08	0.9983	2197.88
10	−32.35	−1.68	−6.19	1.17	0.057	3.30	21.54	0.9703	446.22
11	−51.49	−9.64	−4.11	0.84	0.014	6.84	8.61	0.9607	2840.89
12	−52.37	−8.91	−2.70	0.55	−0.020	7.33	5.48	0.9936	9086.47
13	33.06	37.40	−2.20	0.41	0.349	−12.39	2.47	0.9987	2982.05
14	−55.29	−7.51	−10.99	1.82	0.071	6.56	6.12	0.9802	1089.09
15	−42.91	−2.38	−11.1	1.88	0.143	3.97	5.8	0.9686	636.42

[a] SN, the system name and number same as Table 1a.

where M_0–M_4 are the model constants and ρ is the density of the $SCCO_2$ at different pressures and temperatures.

For the solubilities of organic solids in $SCCO_2$ in presence of cosolvent, Jouyban et al. [10] proposed:

$$\ln(y_2') = n_0 + n_1 y_3 + n_2 \rho_1 + n_3 P^2 + n_4 PT + \frac{n_5 T}{P} + n_6 \ln(\rho_1) \qquad (10)$$

where n_0–n_6 are model constants. We propose a new model as

$$\ln(y_2') = N_0 + (N_1)\ln(\rho_1) + N_2\rho_1 + \frac{N_3}{T} + N_4 \ln(T) + N_5 \ln(y_3) + N_6 \ln(y_3\rho_1 T) \qquad (11)$$

where N_0–N_6 are the model constants.

Table 3a
Correlation results based on the proposed model (Eq. (11)) for various systems.

SN[a]	N_0	N_1	N_2 ($\times 10^3$)	N_3	N_4	N_5	N_6	AARD %	R_{adj}	F value
1	−0.99	0.243	4.88	−3055	0.070	−0.66	−0.325	5.38	0.9896	2143.99
2	−7.29	0.736	4.40	−4155	1.098	1.467	0.5319	4.67	0.9957	4686.58
3	−7.67	1.159	3.60	−5120	1.001	1.595	0.715	3.51	0.9973	18971.23
4	−5.18	1.887	14.65	−5551	−0.080	0.764	0.574	3.71	0.9944	9834.88
5	−10.39	1.093	1.30	−3099	1.001	2.504	0.776	3.53	0.9939	8016.04
6	−7.29	1.156	5.79	−7534	1.001	1.467	0.700	4.80	0.9974	14155.31
7	−8.71	0.775	5.51	−4306	1.001	1.942	0.562	3.62	0.9981	11761.56
8	−9.46	0.877	3.86	−4180	1.001	2.192	0.638	5.36	0.9943	3625.70
9	2.51	1.507	5.48	−7392	−1.001	−1.807	−0.230	3.93	0.997	15326.54
10	−10.33	0.729	2.35	−2232	1.098	2.482	0.632	3.36	0.9955	5379.77
11	−6.04	0.543	4.90	−3966	1.001	1.051	0.359	3.83	0.9973	4945.54
12	−7.77	1.10	3.74	−4875	1.001	1.638	0.690	3.55	0.9974	15768.46
13	−10.12	1.51	0.02	−4741	1.001	2.413	0.973	3.97	0.993	8446.11
14	−10.60	1.03	1.46	−2780	1.001	2.572	0.752	3.52	0.9941	7916.72
15	−6.79	0.956	6.22	−7153	1.001	1.302	0.585	5.17	0.9971	8310.66
16	−9.30	0.813	5.22	−4023	1.001	2.138	0.600	4.33	0.9976	8869.72
17	−10.89	1.278	2.98	−3618	0.694	2.671	0.767	5.80	0.9946	2863.92
18	−6.01	0.642	5.91	−5777	1.001	1.041	0.403	5.7 0	0.9961	6148.30

[a] SN, the system name and number same as Table 1b.

Table 3b
Correlation results based on the Jouyban et al. model (Eq. (10)) with $n_0 = -6.049$, $n_1 = -101.99$.

SN[a]	n_2 ($\times 10^3$)	n_3 ($\times 10^3$)	n_4 ($\times 10^3$)	n_5	n_6	AARD %	R_{adj}	F value
1	3.35	−4.02	0.61	0.0181	0.0417	9.67	0.9694	623.16
2	3.75	−5.28	0.81	0.0059	0.0356	15.00	0.9628	358.02
3	2.88	−5.97	0.88	−0.0151	0.1053	19.20	0.9449	298.22
4	0.54	−6.38	0.93	−0.0123	0.0826	20.22	0.8634	196.66
5	2.50	−4.73	0.72	0.0141	0.0339	13.95	0.9337	347.25
6	2.09	−5.00	0.62	−0.1220	−0.1154	31.88	0.9138	109.55
7	4.21	−3.77	0.51	−0.0440	−0.0546	18.5	0.9607	296.21
8	3.18	−3.86	0.54	−0.0368	−0.1037	18.70	0.9393	217.62
9	2.22	−5.09	0.71	−0.0698	−0.0776	25.29	0.9254	173.16
10	3.35	−0.40	0.61	0.0182	0.0417	10.20	0.9694	537.16
11	3.79	−4.95	0.78	0.0103	0.0364	12.46	0.9729	476.24
12	3.08	−5.76	0.85	−0.0132	0.1015	18.23	0.9504	317.71
13	7.73	−6.17	0.90	−0.0081	0.0690	19.52	0.8662	194.38
14	2.74	−4.52	0.68	0.0152	0.0317	12.51	0.9461	419.33
15	2.32	−4.74	0.59	−0.1173	−0.1253	29.50	0.9238	128.17
16	4.15	−3.73	0.51	−0.0466	−0.0568	17.26	0.9639	320.07
17	3.52	−3.45	0.46	−0.0377	−0.0818	17.93	0.9427	232.32
18	2.82	−4.53	0.62	−0.0669	−0.0947	22.26	0.9414	208.07

[a] SN, the system name and number same as Table 1b.

Table 4
Statistical summary of the models proposed for binary (Tables 2a and 2b) and ternary (Tables 3a and 3b) systems.

Binary system	Eq. (8)			Eq. (9)		
	AARD[a]	R_{adj}[b]	F value[c]	AARD[a]	R_{adj}[b]	F value[c]
Mean	0.0809[d]	0.985[e]	2267.01[f]	0.04522[d]	0.993[e]	6549.06[f]
Standard deviation	0.043	0.0116	2095.4	0.01177	0.00712	5576.7
Ternary system	**Eq. (10)**			**Eq. (11)**		
	AARD[a]	R_{adj}[b]	F value[c]	AARD[a]	R_{adj}[b]	F value[c]
Mean	0.1846[g]	0.9383[h]	302.96[i]	0.0430[g]	0.9955[h]	8731.72[i]
Standard deviation	0.0603	0.032	140.5	0.00818	0.00215	4798.99

[a] AARD calculated using Eq. (12).

[b] $R_{adj} = \sqrt{|R^2 - (Q(1-R^2)/(N-Q-1))|}$ where R is correlation coefficient, N is the number of experimental data points in each set, Q is number of independent variables in each equation.

[c] F value is the ratio of mean square regression to the mean square residual.

[d] The binary systems mean AARDs are significantly different from 4.52% (paired t-test, $P < 0.005$).

[e] The binary systems mean R_{adj} are significantly different from 0.993 (paired t-test, $P < 0.005$).

[f] The binary systems mean F values are significantly different from 6549.06 (paired t-test, $P < 0.005$).

[g] The binary systems mean AARDs are significantly different from 4.3% (paired t-test, $P < 0.0005$).

[h] The binary systems mean R_{adj} are significantly different from 0.9955 (paired t-test, $P < 0.0005$).

[i] The binary systems mean F values are significantly different from 8731.72 (paired t-test, $P < 0.0005$)

2.2. Methodology

The density of supercritical carbon dioxide required for the modeling was determined with Span and Wagner equation of state [17]. The optimization procedure reduces the percentage average absolute deviation, AARD % defined as

$$\text{AARD } \% = \frac{100}{N_i - Z} \sum_{i=1}^{N_i} \frac{|y_2^{\text{calc}} - y_2^{\text{exp}}|}{y_2^{\text{exp}}} \qquad (12)$$

where N_i is the number of data points, Z is the curve fitting parameters for each model, y_2 represents the molar solubility of the solute. The superscripts calc and exp denotes the calculated and experimental values, respectively. The Z values for Eqs. (8), (9), (10) and (11) are 6, 5, 7 and 7, respectively. The correlation of the experimental solubility data requires an optimization process where the constants for empirical models were determined by using the non-linear regression. Tables 1a and 1b show the systems studied both in the presence and absence of cosolvents.

3. Results

To illustrate the usefulness of the current models, the solubilities of solids in $SCCO_2$ and $SCCO_2$ along with cosolvent were compared with the existing solvate complex models, i.e., Eqs. (1)–(9) (see the supplementary information). Clearly, the equation proposed by Jouyban et al. (Eq. (8)) is superior to the existing models. However, the model proposed in this study (Eq. (9)) is even superior to this model, as shown in Tables 2a and 2b and Tables 3a and 3b.

From Tables 2a and 2b, it is clear that the proposed models correlate the solubility better. Tables 3a and 3b show that the newly proposed model correlates the solubilities of solids in supercritical fluid in presence of cosolvents better than existing models. The proposed models, Eqs. (9) and (11), and the previous models, Eqs. (8) and (10), are statistically significantly (paired t-test, $P < 0.005$) different. The adjusted correlation coefficient, R_{adj} and F values for binary and ternary systems are reported in Table 4.

4. Conclusions

New empirical models were developed for correlating the solubilities of solids in supercritical fluids with and without cosolvents.

The new models proposed in this work were successful in correlating solubility of solids in $SCCO_2$ with and without cosolvent and were found to correlate the systems better than existing models.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.tca.2009.12.004.

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