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Measurement and correlation solubility and mixing properties of dimethyl succinylsuccinate in pure and mixture organic solvents from (278.15 to 333.15) K

Yonghong Hu[a,*], Yumei Kai[a], Zheng Cao[a], Jiaojiao Li[a], Wenge Yang[b]

[a] College of Biotechnology and Pharmaceutical Engineering, Nanjing University of Technology, No.200, North Zhongshan Road, Nanjing 210009, China
[b] Jiangsu Engineering Technology Research Center of Polypeptide Pharmaceutical, Nanjing 210038, China

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ABSTRACT

The solubilities of dimethyl succinylsuccinate in tetrahydrofuran, acetic ether, acetone, acetonitrile, 1-propanol, ethanol and methanol pure solvents and tetrahydrofuran + methanol mixed solvents were measured within the temperature range 278.15 K to 333.15 K at atmospheric pressure. The solubility of dimethyl succinylsuccinate in those selected solvents increased with increasing temperature, and the dissolution in tetrahydrofuran is best. The semi-empirical Buchowski–Ksiazczak λh equation, the modified Apelblat equation and CNIBS/R-K equation were used to correlate with the solubility. In pure solvents the values of the sum of deviation ($\sum(\%Dev)$) for Apelblat and Buchowski–Ksiazczak λh equations are 1.1115 and 1.0383, respectively. In the tetrahydrofuran and methanol mixture, the sums of deviation ($\Sigma(\%Dev)$) for Apelblat equation, Buchowski–Ksiazczak λh equation and CNIBS/R-K equation are 0.7587, 0.7059 and 1.4090, respectively. By comparing models, the solubilities predicted by Buchowski–Ksiazczak λh equation and Apelblat equation is reasonable in mixture solvent and pure solvent, respectively. The thermodynamic properties of the solution process, including the enthalpy, and entropy were calculated by the van't Hoff analysis. The value of enthalpy and entropy were positive.

1. Introduction

Dimethyl succinylsuccinate (Fig. 1), a white or pale yellow crystalline powder, is an important symmetrical multifunctional intermediate used as the core structure of many natural products. Dimethyl succinylsuccinate has been employed widely for constructing various polycyclic molecular scaffolds, such as 4,8-Diacetoxy-1,5-dimethyl-1,2,3,5,6,7-hexahydro-s-indacene [1] and Poly amine or esters [2]. More recently, dimethyl succinylsuccinate has been used as a building block in the synthesis of novel organic pigments, displaying excellent color, light fastness, weather fastness, thermal stability and resistance to interference, such as 2,9-di(methylsulphonyl) quinacridone [3]. Furthermore, due to the symmetrical structure of dimethyl succinylsuccinate, it can construct various polymers and oligomers such as oligo(phenylenevinylene)s [4] and poly[*p*-(2,5-dihydroxy)-phenylenebenzobisoxazole] fiber [5].

In order to get the pure dimethyl succinylsuccinate, some chemical techniques for separation such as flocculation, absorption, and re-crystallization were employed in the industry. Crystallization process is the key step that determined the quality of the final product. The solubility of a compound in different solvents played a crucial role in the determination of proper solvents and the crystallization process. Therefore, it is necessary to determine the solubility of dimethyl succinylsuccinate in different solvents to optimize the operating conditions in the processes for purification. Unfortunately, the solubility of dimethyl succinylsuccinate in common solvents was rarely reported.

In this work, the solubility of dimethyl succinylsuccinate in pure and mixed organic solvent was measured over the temperature range from 278.15 K to 333.15 K at atmospheric pressure and correlate the measured solubilities by Combined Nearly Ideal Binary Solvent/Redlich (CNIBS/R-K) equation, modified Apelblat equation and the semi-empirical Buchowski–Ksiazczak λh equation. The solution enthalpy and entropy of dimethyl succinylsuccinate were estimated based on regression of solubility data by utilizing the van't Hoff equation.

2. Experimental

2.1. Materials

A pale yellow crystalline powder with the mass fraction of 0.99 was supplied by Aladdin chemistry Co. Ltd. and prepared by

* Corresponding author. Tel.: +86 25 83587108; fax: +86 25 83587108.
E-mail addresses: hyh@njut.edu.cn, yonghonghu11@126.com (Y. Hu).

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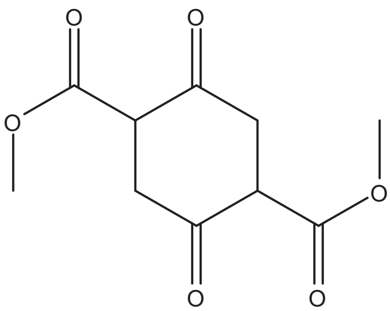

Fig. 1. Molecular structure of dimethyl succinylsuccinate.

recrystallizing from the solution of ethanol two times and its mass fraction is more than 0.9987 determined in our high performance liquid chromatography (Model: P680). The organic solvents were analytical purity grade obtained from Tianjin Chemical Industry. Table 1 summarizes the materials information.

2.2. Apparatus and procedure

The solubility was measured by a static equilibrium method that was described in our previous work [6,7], so only a brief presentation is made here. A slurry of dimethyl succinylsuccinate crystal was prepared by adding excess solute into solvent, and a mercury thermometer with a fluctuating temperature of $\pm$0.05 K was used to measuring the equilibrium temperature of solution. The dissolution of the solute $\pm$was carried out in a jacketed glass vessel and its constant temperature was maintained by circulating water through the outer jacket from a super thermostatic water-circulator bath (ScienTZ DC-2006). The mixture was stirred vigorously for 12 h to ensure the solution reaching equilibrium before stopped. Then, the solution was allowed to settle for at least 3 h to ensure the suspended solid phase precipitated to the bottom of the tube before sampling. Then 1 mL of the supernatant was transferred to a 5 mL beaker of given mass and the breaker was weighed again. Finally, the beaker was put into a dryer and weighed regularly until reaching the constant weight. The masses were measured by an analytical balance (Sartorius BSA224S) with an uncertainty of $\pm$ 0.0001 g. The same experiment was conducted in triplicate, and the mean values listed in Table 2 were considered as the measured results and were used to calculate the mole fraction solubility (x_1) based on the following:

$$x_1 = \frac{m_1/M_1}{m_1/M_1 + \sum m_i/M_i} \quad (1)$$

$$x_2 = \frac{m_2/M_2}{m_2/M_2 + m_3/M_3} \quad (2)$$

where m_1, m_2, and m_3 represent mass of the dimethyl succinylsuccinate, methanol and according solvent, respectively. M_1, M_2 and M_3 are molecular weight of the dimethyl succinylsuccinate, methanol and according solvent, respectively.

Table 1
Provenance and purity of the materials used.

Solvent	Provenance	Purity
Dimethyl succinylsuccinate	Aladdin Chemistry Co. Ltd.	99.87%
Methanol	Tianjin Chemical Industry	≥99.90%
Ethanol	Tianjin Chemical Industry	≥99.93%
1-Propanol	Tianjin Chemical Industry	≥99.93%
Acetic ether	Tianjin Chemical Industry	≥99.98%
Acetonitrile	Tianjin Chemical Industry	≥99.91%
Tetrahydrofuran	Tianjin Chemical Industry	≥99.95%
Acetone	Tianjin Chemical Industry	≥99.96%

Table 2
Solubilities of dimethyl succinylsuccinate in pure organic solvents from T = 278.15 K to 333.15 K at atmospheric pressure.

	Methanol			Ethanol		
T (K)	10^3x_1	$100(x_1-x^{cal})/x_1$		10^3x_1	$100(x_1-x^{cal})/x_1$	
		Eq. (4)	Eq. (3)		Eq. (4)	Eq. (3)
278.15	0.10	−7.677	7.797	0.13	−3.468	5.498
283.15	0.14	−5.190	5.95	0.19	2.238	8.52
288.15	0.18	−4.970	2.667	0.24	−1.443	3.097
293.15	0.24	−6.322	−1.584	0.32	−1.911	0.961
298.15	0.33	−1.460	0.728	0.42	−1.559	−0.078
303.15	0.42	−2.129	−1.733	0.56	−1.334	−0.928
308.15	0.55	−0.256	−1.096	0.71	−3.692	−4.057
313.15	0.70	0.358	−1.157	0.95	0.487	−0.306
318.15	0.90	1.455	−0.181	1.21	−0.398	−1.331
323.15	1.13	0.935	−0.319	1.59	2.733	2.009
328.15	1.44	2.640	2.304	1.98	1.158	0.923
333.15	1.71	−2.174	−1.062	2.43	−1.365	0.152

	1-Propanol			Acetic ether		
T (K)	10^3x_1	$100(x_1-x^{cal})/x_1$		10^3x_1	$100(x_1-x^{cal})/x_1$	
		Eq. (4)	Eq. (3)		Eq. (4)	Eq. (3)
278.15	0.23	−4.128	4.023	3.23	1.430	3.737
283.15	0.30	−3.221	2.649	3.94	−1.420	0.317
288.15	0.38	−4.153	−0.168	5.12	2.763	3.879
293.15	0.49	−2.795	−0.482	6.03	−2.158	−1.492
298.15	0.64	−1.316	−0.335	7.28	−4.029	−3.787
303.15	0.81	−1.320	−1.333	9.14	−1.260	−1.359
308.15	1.04	1.175	0.496	11.31	0.551	0.221
313.15	1.30	1.352	0.317	13.79	1.385	0.933
318.15	1.58	−0.704	−1.802	16.47	0.642	0.182
323.15	1.99	1.307	0.514	19.71	0.523	0.191
328.15	2.45	1.511	1.332	23.46	0.254	0.200
333.15	2.91	−1.458	−0.664	27.65	−0.651	−0.259

	Acetonitrile			Tetrahydrofuran		
T (K)	10^3x_1	$100(x_1-x^{cal})/x_1$		10^3x_1	$100(x_1-x^{cal})/x_1$	
		Eq. (4)	Eq. (3)		Eq. (4)	Eq. (3)
278.15	0.75	0.449	4.765	4.74	−2.27	−1.130
283.15	1.02	3.699	6.778	5.90	−3.218	−2.338
288.15	1.28	−0.208	1.996	7.69	1.259	1.850
293.15	1.61	−3.132	1.746	9.42	0.200	0.561
298.15	2.04	−4.510	3.848	11.68	1.002	1.152
303.15	2.65	−2.518	2.439	13.95	−1.320	−1.346
308.15	3.50	1.793	1.479	17.32	0.824	0.666
313.15	4.32	−0.002	0.551	20.77	0.031	−0.208
318.15	5.47	1.345	0.753	24.93	−0.173	−0.427
323.15	6.69	−0.193	0.657	29.98	0.275	0.085
328.15	8.39	1.279	1.155	35.73	0.250	0.212
333.15	10.09	−0.964	0.530	42.20	−0.290	−0.071

	Acetone		
T (K)	10^3x_1	$100(x_1-x^{cal})/x_1$	
		Eq. (4)	Eq. (3)
278.15	1.72	3.946	2.654
283.15	2.24	4.431	3.710
288.15	2.83	2.825	2.508
293.15	3.40	−3.068	−3.106
298.15	4.26	−4.029	−3.891
303.15	5.48	−1.545	−1.339
308.15	7.09	2.101	2.289
313.15	8.53	−0.874	−0.750
318.15	10.77	1.526	1.539
323.15	12.92	−0.645	−0.757

3. Results and discussion

3.1. In pure solvents

In this work, to more quantitatively describe the solid-liquid equilibrium, the modified Apelblat equation and the