Solubility of Sodium Dimethyl Isophthalate-5-sulfonate in Water and in Water + Methanol Containing Sodium Sulfate

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The solubility of sodium dimethyl isophthalate-5-sulfonate in water and in a mixed solvent (water + methanol) containing sodium sulfate was determined from 292.7 K to 360.0 K. Sodium sulfate has a salting out effect on the solubility of sodium dimethyl isophthalate-5-sulfonate in water. The results were correlated by a polynomial equation.

Introduction

Sodium dimethyl isophthalate-5-sulfonate, $C_{10}H_9O_7SNa$ (SDIS), is increasingly used in the dyeing industry. Despite the appearance of new applications of SDIS, there have been few physical property data reported, and for solubility no experimental data are available. This scarcity of basic data hinders progress in the design of production flow processes or expanding production capacity. In this work, the solubilities of SDIS in water and in a mixed solvent (water + methanol) containing sodium sulfate were experimentally determined from 292.7 K to 360.0 K to provide essential data for process design.

Methods of measuring solubility of a solid in liquid mixtures can be classified as analytical and synthetic (Nývlt, 1977; Vold and Vold, 1949; Kertes et al., 1968). The advantages of the analytical method lie in the possibility of measuring a large number of samples simultaneously with a reliable method, even for solid-phase compositions. Analytical methods have been used to measure the solubility of lobenzarit disodium salt in ethanol−water mixtures (Pino-García and Rasmuson, 1998), the solubility of trioxane in water (Brandani et al., 1994), and the solubility of potassium dichromate in dilute aqueous ethanol solutions (Korin and Soifer, 1997). In the work of Korin and Soifer (1997), the solid phase was identified by X-ray analysis. The analytical method is disadvantageous in that it is tedious and time consuming. Solubility data can be obtained much faster and more easily by means of a synthetic method. Synthetic methods involve on weighing or measuring the individual components to obtain a system with a known composition; the state in which the solid phase just disappears is then determined for this system. Disappearance of the solid phase can be achieved either by a change in the temperature or by the addition of a known amount of solvent (Nývlt, 1977). According to the properties of the solid studied in this work and the requirement of the industry, we chose the last crystal disappearance method (Křlvánková et al., 1992), one of the synthetic methods, to determine the solubility of SDIS in water and in water + methanol containing sodium sulfate.

Experimental Section

Chemicals. SDIS was obtained from Takehon Co., Japan; its business name is Delion-MS. It is a white powder of small particle size and contains <0.4 wt % water and <300 ppm SO_4^{2-}. The methanol used for experiments was obtained from the Beijing Chemical Plant. Distilled water of HPLC grade and analytical reagent grade sodium sulfate were used directly in this work. All the chemicals used in the experiment have a minimum purity of 99.5 mol %. Densities and refractive indices of the solvents are reported in Table 1 in comparison with the literature data of Riddick et al. (1986) at 298.15 K and atmospheric pressure.

Table 1. Refractive Index n_D and Density ρ at 298.15 K

solvent	n_D exptl	n_D lit.[a]	ρ/(kg·m^{-3}) exptl	ρ/(kg·m^{-3}) lit.[a]
water	1.3325	1.332 502 9	997.05	997.0474
methanol	1.3264	1.326 52	786.51	786.37

[a] Riddick et al. (1986).

Procedure. Solubility was determined by the last crystal disappearance method. This method is based on sequentially adding known masses of a solid compound to a stirred solution kept at a fixed temperature. The initial mass of solvent is known precisely (in this case, about 100 g). When a solid increment had completely dissolved, the next portion of solid was introduced. This procedure was repeated until the last increment remained partially undissolved. The mass of the increment was gradually decreased when the solution saturation was approached, which is indicated by the lengthening time necessary for each increment to dissolve completely. This process needs more than about 4 h, and the last increment added is approximately 0.01 g. The solution in which the last increment remained undissolved was regarded as saturated. The presence of solid in the solution was indicated visually as a cloud point which appears when light is passed through the solution.

The equilibrium cell is a cylindrical glass vessel with a diameter of 70 mm and a height of 150 mm. The cell used in this experiment is a double-jacketed vessel. Heated water from a water bath can be circulated through the jacket. The water was thermostated and controlled with a thermoelectric controller (type CS501, China) to the desired temperature (±0.1 K). The cell has a perforated rubber cover plate to prevent the solvent from evaporating, through which a mercury thermometer with an uncertainty of ±0.05 K was inserted. The mixtures of solute and solvent in the vessel were stirred with a magnetic stirrer. The masses of the solute and solvent were measured with an analytical balance (type AE200, Switzerland) with an accuracy of ±0.0001 g. Some of the solubility experiments

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Table 2. Experimental Solubility Data of SDIS in 100 g of Water

T/K	SDIS/g	T/K	SDIS/g
292.7	0.1989	324.0	0.8608
297.2	0.2908	333.5	1.1509
303.2	0.3705	343.4	1.9142
308.2	0.4841	351.9	2.6424
313.2	0.5704	358.2	3.4935

Table 3. Experimental Solubility Data of SDIS in 100 g of Water with 3.6274 g of Sodium Sulfate

T/K	SDIS/g	T/K	SDIS/g
298.6	0.1634	333.3	0.6883
306.9	0.2796	342.9	0.9259
314.1	0.3631	351.6	1.1709
323.2	0.4581	360.0	1.4626

Table 4. Experimental Solubility Data of SDIS in 100 g of Mixed Solvent (1.0427 g of Methanol + 98.9573 g of Water) with 3.6280 g of Sodium Sulfate

T/K	SDIS/g	T/K	SDIS/g
299.6	0.2561	338.2	1.0462
307.2	0.3959	344.5	1.5299
317.7	0.5211	353.6	2.0852
327.3	0.6975	359.2	2.5703

Table 5. Experimental Solubility Data of SDIS in 100 g of Mixed Solvent (15.9050 g Methanol + 84.0950 g of Water) with 11.1490 g of Sodium Sulfate

T/K	SDIS/g	T/K	SDIS/g
298.2	0.1596	312.6	0.3802
302.4	0.2431	317.4	0.4352
308.0	0.2998		

Table 6. Experimental Solubility Data of SDIS in 100 g of Mixed Solvent (9.5088 g of Methanol + 90.4912 g of Water) with 8.6971 g of Sodium Sulfate

T/K	SDIS/g	T/K	SDIS/g
298.9	0.2037	312.9	0.4595
303.0	0.2923	317.4	0.5511
308.4	0.3937		

Table 7. Experimental Solubility Data of SDIS in 100 g of Mixed Solvent (0.1015 g of Methanol + 99.8985 g of Water) with 0.3014 g of Sodium Sulfate

T/K	SDIS/g	T/K	SDIS/g
298.2	0.2542	335.6	0.9573
308.2	0.4135	348.4	1.7762
318.1	0.5363	353.3	2.3683
328.2	0.6864	358.5	2.9825

were conducted three times to check the reproducibility. The accuracy of the experimental solubilities was <0.01 g of SDIS/100 g of solvent. To verify the accuracy of this experiment, another experiment was done in which the solubility of thiourea in 1-butanol was determined. Compared with the literature data (Kim et al., 1994), the deviation of the solubility was <2%.

Results and Discussion

The saturated solubilities of SDIS in water and in water + methanol were measured over the temperature range

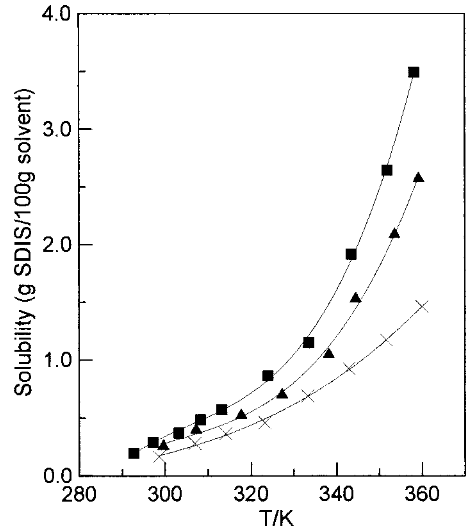

Figure 1. Solubility curves of SDIS in 100 g of water (■), in 1.0427 g of methanol + 98.9573 g of water (▲), and in 100 g of water with 3.6274 g of sodium sulfate (×).

from 292.7 K to 360.0 K. The solubilities of SDIS in various solutions which were calculated from the total solid mass introduced and the initial solvent mass, are listed in Tables 2−7.

The solubility data were correlated with the equation

$$S = a + bT + cT^2 + dT^3 \quad (1)$$

where S represents the solubility of SDIS in units of g of solute/100 g of solvent, T is the absolute temperature, and a, b, c, and d are constants. The values of these constants along with the root-mean-square deviations (RMSDs) are listed in Table 8. The RMSD is defined as

$$\mathrm{RMSD} = [\sum_{i=1}^{N}(S_i^{\mathrm{calc}} - S_i^{\mathrm{exptl}})^2/(N-1)]^{1/2} \quad (2)$$

where S is the solubility of SDIS in water and in water + methanol, N is the number of experimental points, and the superscripts calc and exptl refer to the values calculated from eq 1 and to the data for experimental solubilities, respectively.

It is clear from Tables 2−7 and Figure 1 that the solubility of SDIS increases with temperature. From Tables 2 and 3 and Figure 1, one can see that when sodium sulfate was added to water, the solubility of SDIS decreased. This shows that sodium sulfate has a salting out effect on the solubility of SDIS. From Tables 3 and 4, one can conclude that the presence of methanol in the solution increases the solubility of SDIS. With increasing concentrations of sodium sulfate, the mass of SDIS dissolved in the mixed solvent (methanol + water) decreases. Sodium sulfate has a salting out effect on the solubility of SDIS in water in the sodium sulfate concentration range 0.007 to 0.785 mol/kg of solvent, as one can see from Tables 2−7.

Table 8. Parameters for Correlation Equation of Various Solvents

system	a	b	10^2c	10^5d	10^2RMSD
100 g of water	−445.126	4.336 12	−1.411 83	1.538 06	3.40
100 g of water + 3.6274 g of Na_2SO_4	−35.159	0.362 86	−0.127 40	0.152 41	1.60
1.0427 g of methanol + 98.9573 g of water + 3.6280 g of Na_2SO_4	−252.460	2.485 47	−0.818 24	0.901 91	4.59
15.9050 g of methanol + 84.095 g of water + 11.1490 g of Na_2SO_4	−480.869	4.631 00	−1.489 60	1.601 51	0.99
9.5088 g of methanol +90.4912 g of water + 8.6971 g of Na_2SO_4	−923.466	8.928 12	−2.881 13	3.104 72	0.46
0.1015 g of methanol + 99.8985 g of water + 0.3014 g of Na_2SO_4	−664.475	6.359 81	−2.030 78	2.164 90	2.52

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