Solubility of Xylitol in Ethanol, Acetone, *N*,*N*-Dimethylformamide, 1-Butanol, 1-Pentanol, Toluene, 2-Propanol, and Water

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The solubility of xylitol in ethanol, acetone, *N*,*N*-dimethylformamide (DMF), 1-butanol, 1-pentanol, toluene, 2-propanol, and water was measured using a laser technique with the temperature range from (293 to 343) K and at atmospheric pressure. The results were correlated with a semiempirical equation. The experimental solubility and correlation equation in this work can be used as essential data and models in the purification process of xylitol.

Introduction

Xylitol, a white crystal, is an important sweetener that is one kind of sugar substitute. The main branches of xylitol use are food production, perfumery, pharmaceutics, and chemistry.[1−3] In industrial manufacture, xylitol is crystallized from solution as the final step. So, crystallization is a key step since, in many respects, it determines the yield and quality of the target product. To determine proper solvents and to design an optimized production process, it is necessary to know the solubility of xylitol in different solvents. However, from a review of the literature, it was found that no experimental solubility data were available. In this paper, the solubility of xylitol in ethanol, acetone, *N*,*N*-dimethylformamide (DMF), 1-butanol, 1-pentanol, toluene, 2-propanol, and water were experimentally determined in the temperature range from (293 to 343) K. The method employed in this work was classified as a synthetic method, which was much faster and more readily available than the analytical method.[4]

Experimental Sections

Materials. A crystal of xylitol, with a melting/decomposition point of 367.65 (± 0.5) K, was prepared by recrystallization from aqueous solution. Its purity is higher than 99.9 mass %. It was dried in vacuo at 50 °C for 48 h and stored in a desiccatior. All solvents used for experiments were of analytical reagent grade. The crystal was dehydrated with molecular sieves, and its purity was higher than 99.8 mass % checked by gas chromatography. Distilled, deionized water of HPLC grade was used.

Apparatus and Procedures. The solubility of xylitol was measured using an apparatus similar to that described as literature[5] and described briefly here. A 100 mL or 350 mL jacked vessel was used to determine the solubility; the temperature was controlled to be constant (fluctuates with 0.05 K) through a thermostat water bath. The dissolution of the solute was examined by the laser beam penetrating the vessel. To prevent the evaporation of the solvent, a condenser vessel was introduced. The masses of the samples and solvents were weighted using an analytical balance (Sartorius CP124S, Germany) with an uncertainty of ± 0.0001 g.

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The solubility of xylitol was determined by using a laser technique.[4−9] During experiments, the fluid in the glass vessel was monitored by a laser beam. Predetermined excess amounts of solvent and xylitol of known mass were placed in the inner chamber of the vessel. The contents of the vessel were stirred continuously at a required temperature. In the early stage of the experiment, the laser beam was blocked by the undissolved particles of xylitol in the solution, so the intensity of laser beam penetrating the vessel was lower. Along with the dissolution of the particles of the solute, the intensity of the laser beam increased gradually. When the solute dissolved completely, the solution was clear and transparent, and the laser intensity reached maximum. Then additional solute of known mass [about (1 to 5) mg] was introduced into the vessel. This procedure was repeated until the penetrated laser intensity could not return maximum, or in other words, the last addition of solute could not dissolve completely. The interval of addition was 30 min. The total amount of the solute consumed was recorded. The same solubility experiment was conducted three times, and the mean values were used to calculate the mole fraction solubility (x_1) based on

$$x_1 = \frac{m_1/M_1}{m_1/M_1 + m_2/M_2} \tag{1}$$

where m_1 and m_2 represent the mass of the solute and solvent, and M_1 and M_2 are the molecular weight of the solute and the solvent, respectively.

Results and Discussion

The solubilities of xylitol in different pure solvents at different temperatures are shown in Table 1. The solubility of a solid in a liquid may be expressed in a very general manner by

$$\ln x_1 = -\frac{\Delta H_{f,1}}{RT_{f,1}}\left(\frac{T_{f,1}}{T} - 1\right) - \frac{\Delta C_{pf,1}}{R}\left(\frac{T_{f,1}}{T} - 1\right) + \frac{\Delta C_{pf,1}}{R}\ln\frac{T_{f,1}}{T} - \ln\gamma_1 \tag{2}$$

where x_1, γ_1, $\Delta H_{f,1}$, $\Delta C_{pf,1}$, $T_{f,1}$, R, and T stand for the mole fraction of the solute, activity coefficient, enthalpy of fusion, difference in the solute heat capacity between the solid and liquid at the melting temperature, melting temperature of the

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Table 1. Mole Fraction Solubility (x_1) of Xylitol in Selected Solvents with the Temperature Range from (293 to 343) K

T/K	$10^3 x_1$	$10^3(x_1 - x_1^{calc})$	T/K	$10^3 x_1$	$10^3(x_1 - x_1^{calc})$
		Ethanol			
293.23	1.728	−0.060	318.29	7.090	−0.105
298.28	2.419	0.090	323.24	9.657	−0.014
303.17	3.091	0.057	328.19	13.15	0.081
308.21	3.974	−0.043	333.22	17.85	0.002
313.19	5.224	−0.115			
		Acetone			
293.29	0.2026	−0.0012	313.20	0.5433	0.0006
298.18	0.2401	−0.0083	318.23	0.7428	0.0003
303.26	0.3040	−0.0107	323.20	1.0053	−0.030
308.22	0.4011	−0.0066			
		DMF			
293.21	41.11	−0.15	318.32	109.9	−0.17
298.29	50.42	0.43	323.20	134.4	0.15
303.26	60.88	0.34	328.24	165.6	0.45
308.24	73.59	0.02	333.27	203.7	0.20
313.22	89.46	−0.20			
		1-Butanol			
293.16	1.128	−0.056	323.23	5.883	−0.193
298.21	1.542	0.008	328.32	7.983	−0.128
303.26	2.117	0.109	333.24	10.86	0.00
308.27	2.682	0.052	338.22	14.56	0.09
313.22	3.422	−0.069	343.76	19.42	−0.02
318.16	4.425	−0.166			
		1-Pentanol			
293.20	0.2980	−0.0210	323.29	2.629	−0.089
298.18	0.4830	0.0129	328.23	3.636	−0.071
303.26	0.7339	0.0458	333.22	4.998	−0.021
308.29	1.009	0.019	338.33	6.817	0.043
313.19	1.364	−0.030	343.24	9.034	0.082
318.24	1.885	−0.073			
		Toluene			
293.26	0.0399	0.0009	323.21	0.0731	0.0008
298.28	0.0418	−0.0004	328.22	0.0837	0.0013
303.19	0.0452	−0.0009	333.30	0.0960	0.0011
308.17	0.0500	−0.0009	338.16	0.1091	−0.0001
313.23	0.0563	−0.0005	343.19	0.1241	−0.0029
318.33	0.0642	0.0002			
		2-Propanol			
293.28	1.128	−0.056	323.16	5.883	−0.193
298.20	1.542	0.008	328.21	7.983	−0.128
303.23	2.117	0.109	333.27	10.86	0.00
308.19	2.682	0.052	338.19	14.56	0.09
313.32	3.422	−0.069	343.23	19.42	−0.02
318.21	4.425	−0.166			
		Water			
293.28	161.9	2.5	313.29	266.1	2.8
298.30	179.7	−1.3	318.26	301.7	4.1
303.17	203.1	−1.5	323.25	339.1	2.9
308.21	232.4	0.3	328.21	376.6	−2.5

Table 2. Parameters of Equation 5 for Xylitol in Selected Solvents with the Temperature Range from (293 to 343) K

	a	b	c	10^5 msd
ethanol	−409.66	13942	62.628	7.73
acetone	−891.14	36012	133.75	1.37
DMF	−226.87	7080	35.124	27.69
2-propanol	−319.82	9937	49.145	10.59
toluene	−316.75	12371	46.543	0.12
1-butanol	−68.92	−2701	12.391	4.22
1-pentanol	47.018	−8215	−4.7616	5.60
water	−73.963	1306	11.912	266.16

solute, gas constant, and equilibrium temperature in the saturated solution, respectively. For regular solutions, the activity coefficient is given by

$$\ln \gamma_1 = A + \frac{B}{T/\mathrm{K}} \tag{3}$$

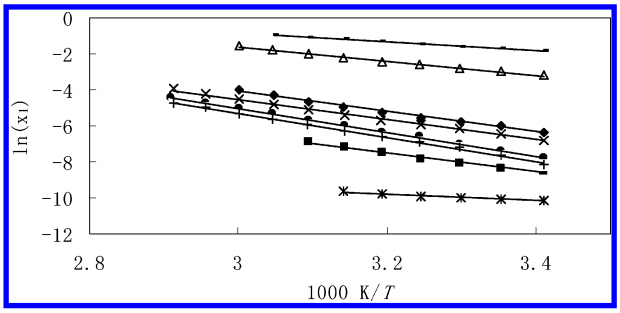

Figure 1. Mole fraction solubility of xylitol in eight different pure solvents between (293 and 343) K: —, water; △, DMF; ◆, ethanol; ×, 2-propanol; ●, 1-butanol; +, 1-pentanol; ■, acetone; ∗, toluene. Full lines were calculated using eq 5 with the parameters listed in Table 2.

where A and B stand for empirical constants. Introducing γ_1 from eq 3 into eq 2 and subsequent rearrangements results in

$$\ln x_1 = \left[\frac{\Delta H_{f,1}}{RT_{f,1}} + \frac{\Delta C_{pf,1}}{R}(1 + \ln T_{f,1}) - A\right] - \left[B + \left(\frac{\Delta H_{f,1}}{RT_{f,1}} + \frac{\Delta C_{pf,1}}{R}\right)T_{f,1}\right]\frac{1}{T} - \frac{\Delta C_{pf,1}}{R}\ln T \tag{4}$$

Equation 4 can be written as

$$\ln x_1 = a + \frac{b}{T/\mathrm{K}} + c \ln T/\mathrm{K} \tag{5}$$

where T is the absolute temperature, and a, b, and c are empirical constants.

The solubility data are correlated with eq 5. The difference between experimental and calculated results is presented in Table 1. The values of the three parameters a, b, and c together with the root-mean-square deviations (rmsd) are listed in Table 2. The rmsd is defined as the following:

$$\mathrm{rmsd} = \left[\frac{\sum_{j=1}^{N}(x_{1,j} - x_{1,j}^{calc})^2}{N-1}\right]^{1/2} \tag{6}$$

where N is the number of experimental points; $x_{1,j}^{calc}$ is the solubility calculated from eq 5; and $x_{1,j}$ is the experimental value of solubility.

Conclusions

From Tables 1 and 2, the following conclusions can be drawn: (1) For all selected pure solvent systems, solubility is a function of temperature, and solubility increases with increasing temperature. (2) The solubility of xylitol in eight solvents decrease in the order water > DMF > ethanol > 2-propanol > 1-butanol > 1-pentanol > acetone > toluene. (3) The calculated solubility shows good agreement with the experimental values (see Figure 1). The experimental solubility and correlation equation presented can be used as essential data and models in the process of the resolution of xylitol.

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