Solubility of Adipic Acid in Acetone, Chloroform, and Toluene

Dongwei Wei* and Wenjiang Cao

Research and Development Center for Petrochemical Technology, Tianjin University, Tianjin 300072, P. R. China

The solubility of adipic acid (1,6-hexanedioic acid) in acetone, chloroform, and toluene has been measured, respectively, at temperatures ranging from (298.15 to 323.15) K and (298.15 to 333.15) K by a static analytical method. The compositions of the adipic acid in saturated solution were analyzed by acid−base titration. A modified Apelblat equation was used to correlate the experimental data.

Introduction

Adipic acid (1,6-hexanedioic acid, $C_6H_{10}O_4$, CASRN. 124-04-9) is of great importance commercially in that it is centrally implicated in the manufacture of nylon-6,6, which, in turn, is extensively used in many products such as tire reinforcements, adhesives, upholstery, specialty foams, carpet fibers, and several items of clothing.[1] The solubility of solids in liquids is one of the most important process parameters and is of scientific interest for the development of the solution theory. However, limited solubility data have been reported in the literature. As far as we know, the solubility available is only in water and several organic solvents such as alcohols, acetone, acetic acid, benzene, cyclohexanol, and cyclohexanone with few data.[2−4] In this paper, the solubility of adipic acid in acetone, chloroform, and toluene was systematically measured by a static analytical method.

Experimental Section

Chemicals. Analytically pure grade acetone, chloroform, and toluene were purchased from Tianjin Kewei Chemical Reagent Co. All the above solvents were refluxed over freshly activated CaO for 2 h and then fractionally distilled. Liquids were stored over freshly activated molecular sieves of type 4A. Analysis, using the Karl Fischer technique, showed that the water mass fraction in each of the solvents was less than 0.02 %. The mass fraction of purities of the solvents were determined in our laboratory by gas chromatography, for acetone, 99.95 %, for chloroform, 99.93 %, and for toluene, 99.96 %. Adipic acid, obtained from Beijing Qingshengda Chemical Technology Co., Ltd., was purified by recrystallization twice from water and dried at 120 °C. The sample was kept in a desiccator with dry silica gel. Its melting temperature was 152.1 °C, which agrees with the most reliable published data.[5,6]

Apparatus and Procedures. The experimental solubility of adipic acid in solvents in the temperature range from (298.15 to 333.15) K was measured by a static analytical method described in our previous work[7,8] and explained briefly here. The experimental saturated solutions were prepared by excess solute, adipic acid, in glass vessels containing the solvent. Solubilities were determined by equilibrating the solute with solvent in a water jacketed vessel with magnetic stirring in a constant-temperature water bath (± 0.05 K) for at least 3 days. Attainment of equilibrium was verified both by repetitive measurement after a minimum of 3 additional days and by approaching equilibrium from supersaturation by pre-equilibrating the solutions at a higher temperature. The actual temperature in the glass vessel was monitored by a mercury thermometer with an uncertainty of 0.05 K. The fluid between the internal and external glass tube can be exchanged by pressing or relaxing the gas-bag at the top of glass tube. Portions of adipic acid saturated solutions were transferred from the internal glass tube to the volumetric flasks to determine the amounts of samples diluted quantitatively with solvent mixtures by acid−base titration.[9]

Table 1. Solubility of Adipic Acid in Acetone, Chloroform, and Toluene

	x_1		
T/K	acetone	chloroform	toluene
298.15	0.01687	0.002095	0.0000453
300.15	0.01801	0.002205	0.0000564
302.15	0.01929	0.002308	0.0000716
304.15	0.02069	0.002450	0.0000941
306.15	0.02213	0.002590	0.000114
308.15	0.02354	0.002725	0.000146
310.15	0.02527	0.002858	0.000182
312.15	0.02715	0.002994	0.000223
314.15	0.02945	0.003141	0.000268
316.15	0.03172	0.003262	0.000324
318.15	0.03453	0.003402	0.000391
323.15	0.04302	0.003767	0.000614
328.15			0.000959
333.15			0.00146

Table 2. Regression Curve Coefficients in Equation 1 for Adipic Acid Solubility in Acetone, Chloroform, and Toluene

solvent	a	b	c	10^4rmsd
acetone	−511.59	20363.15	77.09	1.33
chloroform	152.69	−9246.01	−22.44	0.09
toluene	496.38	−31906.37	−70.10	0.08

Results and Discussion

Acid−base titration was chosen to determine the compositions of a saturated solution of adipic acid in the organic solvent. To check the reliability of the experimental method, known masses of adipic acid were completely dissolved in water and the concentrations of solution were titrated and measured by aqueous sodium hydroxide. The average relative uncertainty was 1.2 % ($N = 5$).

The solubilities of adipic acid in acetone, chloroform, and toluene reported in Table 1 represent an average of three

* Corresponding author. E-mail: weidwei@tju.edu.cn.

10.1021/je8006887 CCC: $40.75
Published on Web 11/25/2008

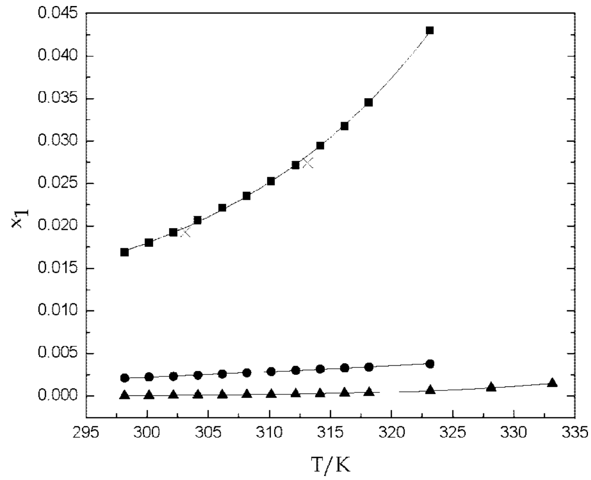

Figure 1. Solubility of adipic acid in various solvents: ■, acetone; ●, chloroform; ▲, toluene; ×, literature data.[2] The line is the best fit of the experimental data calculated with the modified Apelblat equation.

measurements with reproducibility better than 97 %. From the results, we can see that the solubilities of adipic acid in solvents increase as the temperature increases.

The relationship between temperature and solubility of adipic acid is correlated with the modified Apelblat equation[8,10,11]

$$\ln x_1 = a + \frac{b}{T/\mathrm{K}} + c \ln T/\mathrm{K} \quad (1)$$

where x_1 and T are the mole fraction of the solute and absolute temperature, respectively, and a, b, and c are empirical constants.

The experimental data of mole fraction solubility in Table 1 were correlated with eq 1 and plotted as shown in Figure 1, whereas the parameter values of a, b, and c and the root-mean-square deviation (rmsd) are given in Table 2. The rmsd is defined as

$$\mathrm{rmsd} = \left[\frac{1}{N-1}\sum_j^N \left(x_{1,j} - x_{1,j}^{\mathrm{calcd}}\right)^2\right]^{1/2} \quad (2)$$

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

Conclusion

The solubilities of adipic acid in acetone, chloroform, and toluene have been measured, respectively, at temperatures ranging from (298.15 to 323.15) K and (298.15 to 333.15) K by a static analytical method. The solubilities of adipic acid in solvents increase as the temperature increases and decrease in the order acetone > chloroform > toluene. According to the theory of "similarity and intermiscibility", it is reasonable that the higher polar adipic acid is more easily dissolved in the higher polar solvents. The modified Apelblat equation was employed to correlate the experimental data with good agreement.

Literature Cited

(1) Thomas, J. M.; Raja, R. The materials chemistry of inorganic catalysts. *Aust. J. Chem.* **2001**, *54*, 551–560.

(2) Gaivoronskii, A. N.; Granzhan, V. A. Solubility of adipic acid in organic solvents and water. *Russ. J. Appl. Chem.* **2005**, *78*, 404–408.

(3) Davies, M.; Griffiths, D. M. L. The soubilities of dicarboxylic acids in benzene and aqueous solutions. *Trans. Faraday Soc.* **1953**, *49*, 1405–1410.

(4) Fan, L. H.; Ma, P. S.; Xiang, Z. L. Study on crystallization thermodynamics of adipic acid. *Shiyou Huagong/Petrochem. Technol.* **2006**, *35*, 245–249, in Chinese.

(5) Gokel, G. W. *Dean's Handbook of Organic Chemistry*, 2nd ed.; McGraw-Hill: New York, 2004.

(6) Lide, D. R. *CRC Handbook of Chemistry and Physics: A Ready-reference Book of Chemical and Physical Data*, 86th ed.; CRC Press: Boca Raton, 2005.

(7) Wei, D.; Jiang, H.; Jing, X.; Yuan, J. Measurement and correlation of solubilities of 4-hydroxybenzaldehyde and its bromo-derivatives in chloroform. *J. Chem. Ind. Eng. (China)* **2004**, *55*, 1192–1195, in Chinese.

(8) Wei, D.; Pei Y. Solubility of diphenyl carbonate in pure alcohols from (283 to 333) K. *J. Chem. Eng. Data*, in press.

(9) Azab, H. A.; Ahmed, I. T.; Mahmoud, M. R. Potentiometric determination of the apparent dissociation constants of some dicarboxylic acids in various hydroorganic media. *J. Chem. Eng. Data* **1997**, *42*, 593–596.

(10) Liu, B. S.; Gong, J. B.; Wang, J. K.; Jia, C. Y. Solubility of potassium calvulanate in ethanol, 1-propanol, 1-butanol, 2-propanol, and 2-methyl-1-propanol between 273 and 305 K. *J. Chem. Eng. Data* **2005**, *50*, 1684–1686.

(11) Yang, Z. J.; Hu, H. B.; Zhang, X. H.; Xu, Y. Q. Solubility of phenazine-1-carboxylic acid in water, methanol, ethanol from (278.2 to 328.2) K. *J. Chem. Eng. Data* **2007**, *52*, 184–185.

Received for review September 16, 2008. Accepted November 5, 2008.

JE8006887