J. Chem. Eng. Data **2007,** *52,* 553−554

Solubility of Anthracene in *N*,*N*-Dimethyformamide, *N*,*N*-Dimethylacetamide, and *N*-Methyl-2-pyrrolidone

Wen-Lin Xu,* Feng Mao, Hong-Kun Zhao, Ya-Qiong Wang, and Jin Wang

College of Chemistry and Chemical Engineering, Yangzhou University, Yangzhou 225002, People's Republic of China

The solubility of anthracene in *N*,*N*-dimethyformamide, *N*,*N*-dimethylacetamide, and *N*-methyl-2-pyrrolidone was determined respectively at temperatures ranging from (293.25 to 358.45) K, (293.15 to 359.25) K, and (293.45 to 357.55) K at atmosphere pressure. The experimental solubility was correlated by the equation $\ln x = A + B/T + C \ln T$.

Introduction

Liquid−solid phase equilibrium data are important in many chemical engineering processes such as extraction and crystallization operation. Many researchers have studied liquid−solid phase equilibrium for a variety of chemicals in solvents.

Anthracene is the main component of coal tar and the important raw material of some fine chemicals.[1−3] During the process of the separation and extraction of anthracene from coal tar by solvent extraction, the optimization of process conditions and the rational choice of solvents are closely related to the solubility of anthracene in solvents. It is very important to measure the solubility of anthracene in different solvents for improving the extraction process. *N*,*N*-Dimethyformamide, *N*,*N*-dimethylacetamide, and *N*-methyl-2-pyrrolidone are very important solvents in the process of the separation and extraction of anthracene from coal tar. However, the solubility of anthracene in *N*,*N*-dimethyformamide, *N*,*N*-dimethylacetamide, and *N*-methyl-2-pyrrolidone was only scarcely reported in the literature.[4−8] In this paper, the solubility of anthracene in *N*,*N*-dimethyformamide, *N*,*N*-dimethylacetamide, and *N*-methyl-2-pyrrolidone was systematically determined. The results were also compared with the data reported in the literature.[4−8]

Experimental Section

Materials. Anthracene obtained from Farco is pure. *N*,*N*-Dimethyformamide, *N*,*N*-dimethylacetamide, and *N*-methyl-2-pyrrolidone of analytical grade were supplied by Shang hai Chemistry and Medicine Corporation and were used without further purification.

Apparatus and Procedure. Excess solute and solvent were placed in amber glass vessels and allowed to equilibrate in a constant temperature water bath at a given temperature for at least 3 days. Attainment of equilibrium was verified both by repetitive measurements after a minimum of 3 additional days and by approaching equilibrium from supersaturation by pre-equilibrating the solutions at a higher temperature. The temperature of the circulating water was controlled by a thermostat within ± 0.1 K. A condenser was connected to the vessel to prevent the solvents from evaporating. A mercury-in-glass thermometer was inserted into the inner chamber of the vessel with an uncertainty of 0.01 K. An analytical balance with an uncertainty of 0.0001 g was used.

Table 1. Mole Fraction Solubility of Anthracene (*x*) in DMF, DMA, and NMP

DMF		DMA		NMP	
T/K	*x*	*T*/K	*x*	*T*/K	*x*
293.35	0.0042	293.15	0.0083	293.45	0.0152
298.45	0.0054	298.85	0.0113	299.15	0.0186
303.00	0.0057[7]				
303.15	0.0068	303.85	0.0142	303.45	0.0217
308.00	0.0072 [7]				
308.45	0.0086	308.75	0.0159	308.55	0.0255
312.80	0.0101 [7]				
313.21	0.0113[6]				
313.65	0.0104	313.95	0.0185	314.05	0.0325
317.40	0.0110 [7]			317.84	0.0430[6]
318.15	0.0127[4]				
318.85	0.0131	319.75	0.0215	318.15	0.0409
322.80	0.0139 [7]				
323.25	0.0150	324.25	0.0250	324.75	0.0455
326.28	0.0165[6]				
				331.21	0.0546[6]
329.15	0.0180	328.05	0.0269	331.25	0.0539
333.15	0.0191[5]	332.20	0.0301 [8]		
334.55	0.0211	333.15	0.0290	335.25	0.0591
334.90	0.0221[6]				
339.75	0.0252	339.35	0.0338	339.75	0.0653
345.05	0.0292	344.95	0.0393	344.55	0.0738
348.29	0.0320[6]				
349.85	0.0336	348.45	0.0432	349.85	0.0804
		350.30	0.0502 [8]	350.45	0.0824[6]
353.55	0.0384	354.55	0.0485	353.55	0.0884
				357.51	
358.85	0.0448	359.25	0.0564	357.55	0.0930
359.48	0.0452[c]				0.0152

Aliquots of saturated anthracene solutions were transferred through a coarse filter into a tarred volumetric flask to determine the amount of sample and diluted quantitatively with DMF, DMA, and NMP for spectrophotometric analysis at 329.2, 329.1, and 318.2 nm on a Bausch and Lomb Spectronic spectrophotometer. Concentrations of the dilute solutions were determined from a Beer−Lambert law absorbance versus concentration working curve derived from measured absorbencies of standard solutions of known molar concentration.

Results and Discussion

The solubility of anthracene in DMF, DMA, and NMP is listed in Table 1. From the table, we found out that the experimental solubility data of anthracene in DMF, DMA, and NMP show good agreement with the data reported in the literature.[4−8]

---

* Corresponding author. E-mail: wlxu@mail.yzu.edu.cn.

10.1021/je060454x CCC: $37.00

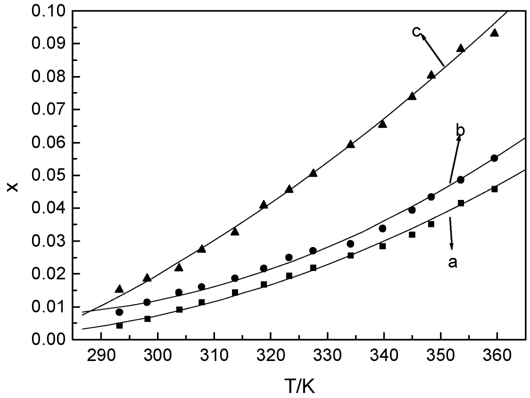

Figure 1. Solubility of anthracene in the three solvents: (a) *N*,*N*-dimethylformamide, (b) *N*,*N*-dimethylacetamide, (c) *N*-methyl-2-pyrrolidone. ■, ●, ▲, experimental data; —, calculated by the empirical equation.

Table 2. Parameters of Equation 1 for Anthracene in DMF, DMA, and NMP

solvent	*A*	*B*	*C*	rmsd
DMF	321.0	-1.861×10^4	−46.00	0.55×10^{-3}
DMA	98.20	-7.288×10^3	−13.70	0.81×10^{-3}
NMP	193.1	-1.193×10^3	−28.00	5.27×10^{-3}

The temperature dependence of the solubility of anthracene in pure solvents was described by the empirical equation:[9−11]

$$\ln x = A + B/(T/\mathrm{K}) + C \ln(T/\mathrm{K}) \qquad (1)$$

where x is the mole fraction solubility of anthracene; T is the absolute temperature; and A, B, and C are parameters. The regressed values of parameters A, B, C and the root-mean-square deviations (rmsd) are listed in Table 2. The rmsd is defined as the following:

$$\mathrm{rmsd} = 4\left\{\frac{\sum_{i=1}^{N}[(x_i^{\mathrm{calcd}} - x_i)]^2}{N}\right\}^{1/2} \qquad (2)$$

where N is the number of experimental points; x_i^{calcd} represents the solubility calculated from eq 1; and x_i represents the experimental values. In Figure 1, curves a to c represent the system of anthracene + *N*,*N*-dimethylformamide, anthracene + *N*,*N*-dimethylacetamide, and anthracene + *N*-methyl-2-pyrrolidone, respectively.

Conclusions

From the data listed in Tables 1 and 2 and in Figure 1, we can draw the following conclusions: (i) The solubility of anthracene in DMF, DMA, and NMP increases with the increasing temperature. (ii) The solubility of anthracene in NMP is much higher than that in DMA and DMF. The solubility of anthracene in DMF is the lowest. (iii) These experimental data were able to be regressed by eq 1 for each solvent.

Literature Cited

(1) Ihara, T.; Fujii, T.; Mukae, M.; Kitamura, Y.; Jyo, A. Photochemical ligation of DNA conjugates through anthracene cyclodimer formation and its fidelity to the template sequences. *J. Am. Chem. Soc.* **2004**, *126* (29), 8880−8881.
(2) Wang, Z.; Zhang, D. Q.; Zhu, D. B. A new saccharide sensor based on a tetrathiafulvalene-anthracene dyad with a boronic acid group. *J. Org. Chem.* **2005**, *70*, 5729−5732.
(3) Kesavan, S.; Su, Q.; Shao, J.; James, S. P. Enantioselective synthesis of linear polypropionate arrays using anthracene-tagged organosilanes. *J. Org. Chem.* **2004**, *48*, 3654−3657.
(4) Lu, J.; Song, Y. X.; Zhang, D. X.; Gao, J. S. Study on the liquid−solid phase equilibrium of anthracene and carbazole in some organic solvents. *J. East China Univ. Sci. Technol. (Social Sci. Ed.)* **2000**, *26*, 70−73.
(5) Zhao, Z. B.; Wang, Z. Z. Study on solubility of anthracene and carbazole in organic solvents. *Coal Convers.* **2002**, *25*, 92−94.
(6) Song, Y. X.; Hu, Z. Q.; Gao, J. S. Study on the liquid−solid phase equilibrium of anthracene in some organic solvents. *Coal Convers.* **1999**, *22*, 94−96.
(7) Emilio, A. C.; Beatriz, G.; Mario, D. Solubility of anthracene and anthraquinone in some pure and mixed solvents. *J. Chem. Eng. Data* **1989**, *34* (3), 273−275.
(8) Zygmunt, L.; Malgorzata, E. J. (Solid + liquid) equilibria in (polynuclear aromatic + tertiary amide) systems. *J. Chem. Thermodyn* **2000**, *32* (10), 1335−1353.
(9) Apelblat, A.; Manzurola, E. Solubilities of *o*-acetylsalicylic, 4-aminosalic, 3,5-dinitrosalicylic, and *p*-toluic acid, and magnesium-DL-aspartate in water from $T = (278 \text{ to } 348)$ K. *J. Chem. Thermodyn* **1999**, *31*, 85−91.
(10) Wang, L. C.; Wang, F. A. Solubility of niacin in 3-picoline + water from (287.65 to 359.15) K. *J. Chem. Eng. Data* **2004**, *49* (1), 155−156.
(11) Hao, H. X.; Wang, J. K.; Wang, Y. L. Solubility of dexamethasone sodium phosphate in different solvents. *J. Chem. Eng. Data* **2004**, *49* (6), 1697−1698.

Received for review October 12, 2006. Accepted December 1, 2006. We are very grateful to be supported by the National Natural Science Foundation of China (Grants 20176047 and 20576111) and supported by the Natural Science Foundation of the Education Department of Jiangsu Province (Grant 03KJB530164).

JE060454X