Solubility of Fluorene in Benzene, Chloroform, Acetone, 1-Propanol, Isobutanol, and Methylbenzene from (283 to 323) K

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Solubility of fluorene in benzene, chloroform, acetone, 1-propanol, isobutanol, and methylbenzene was experimentally measured at temperatures ranging from (283.00 to 323.00) K. The laser monitoring observation technique was used to determine the disappearance of the solid phase in a solid + liquid mixture. The results of these measurements were correlated with the Apelblat equation, which can be used as a useful model in the purification process of fluorene.

Introduction

Fluorene is one of the polycyclic aromatic hydrocarbons, which is purified from coal tar.[1] In organic synthesis, fluorene is an important intermediate material, which is widely used for the synthesis of advanced pigments, dyes, polymers, and drugs. The entire usage requires purified fluorene products. Crystallization processes are the critical steps that determine the quality of the final product. The solubility of solid compounds in solvents plays a crucial role in the determination of proper solvents and the development and operation of crystallization processes. Therefore, knowing the solubility of the product is necessary. The published works relating to fluorene are mainly concerned with synthesis and degradation. Literature on the solubility of fluorene in common solvents is scarce.

In this paper, the solubility of fluorene in benzene, chloroform, acetone, 1-propanol, isobutanol, and methylbenzene was experimentally determined from (283.00 to 323.00) K using a laser-monitoring observation technique.[2,3]

Experimental Section

A white and powder-like crystal of fluorene was used, and its purity is higher than 99.0 % mass fraction. The solvents used including benzene, chloroform, acetone, 1-propanol, isobutanol, and methylbenzene (purchased from the Tianjin Kewei Co. of China) were of analytical reagent grade, and their mass fraction purity was higher than 99.8 %. The solubility was measured by the method which is described in the literature.[4−6]

The solubility of fluorene was measured by the last crystal disappearance method. The laser monitoring observation technique was used to determine the disappearance of the last crystal in the solid + liquid mixtures. The system consisted of a laser generator, a photoelectric transformer, and a light intensity display. The equilibrium cell is a cylindrical double-jacketed glass vessel. A constant desired temperature was maintained by circulating water through the outer jacket from a thermostat. The uncertainty in temperature was ± 0.05 K. Continuous stirring was achieved with a magnetic stir bar. A condenser was connected with the vessel to prevent the solvent from evaporating. The masses of the solvent and solute were weighed using an analytical balance (type TG 332A, China) with an accuracy of ± 0.0001 g.

During the measurement, predetermined excess amounts of solute and solvents of known masses were transferred to the equilibrium vessel. The contents of the vessel were stirred continuously at an invariable and required temperature for 30 min. Then, additional solvent of known mass was introduced into the cell. When the last portion of solute just disappeared, the intensity of the laser beam penetrating the vessel reached the maximum, and the intensity of the laser beam hardly changed for at least 30 min. The solvent mass consumed in the measurement would be recorded. Together with the mass of solute, the solubility would be obtained. The saturated mole fraction solubility of the solute (x_1) in different solvents can be obtained as follows

$$x_1 = \frac{m_A/M_A}{m_A/M_A + m_B/M_B} \quad (1)$$

in which m_A and m_B represented the masses of solute and solvents. M_A and M_B are the molecule weight of solute and solvents, respectively.

All the experiments were conducted three times, and the mean values were used to calculate the mole fraction solubility x_1 of fluorene in benzene, chloroform, acetone, 1-propanol, isobutanol, and methylbenzene. The relative uncertainty of the experimental solubility values is within 0.02.

Results and Discussion

The solubility of fluorene in benzene, chloroform, acetone, 1-propanol, isobutanol, and methylbenzene at different temperatures is listed in Table 1 and graphically plotted in Figure 1. To verify the uncertainty of the measurement, the solubility of fluorine in methylbenzene in this work was compared with the literature data, in which the solubility of fluorene was measured by a synthetic method.[7] Compared with the literature data, the relative deviation of the solubility was lower than 0.05.

The solubility of fluorene can be computed by the Apelblat equation[8] deduced from the solid−liquid phase equilibrium as follows

$$\ln x = A + \frac{B}{T/\mathrm{K}} + C \ln(T/\mathrm{K}) \quad (2)$$

The experimental solubility values were correlated with eq 2 using the least-squares method, and the difference between

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Table 1. Mole Fraction Solubility of Fluorene (x_1) in Benzene, Chloroform, Acetone, 1-Propanol, Isobutanol, and Methylbenzene from (283.00 to 323.00) K

T/K	x_1^{exptl}	$100\ (x_1^{exptl} - x_1^{calcd})/x_1^{exptl}$	T/K	x_1^{exptl}	$100\ (x_1^{exptl} - x_1^{calcd})/x_1^{exptl}$
	Benzene			Chloroform	
283.00	8.832	2.25	283.00	8.928	0.46
288.00	9.824	0.46	288.00	10.16	−0.01
293.00	10.91	1.53	293.00	11.44	−1.50
298.00	12.57	0.12	298.00	13.18	−0.45
303.20	14.32	0.10	303.10	15.26	0.94
308.00	15.84	−1.87	308.20	17.31	0.47
313.00	18.35	0.35	313.00	19.52	0.29
318.00	20.91	0.84	318.00	21.99	−0.34
323.00	23.46	−0.14	323.00	24.97	−0.09
	Acetone			1-Propanol	
283.00	4.448	4.16	283.00	0.817	−0.37
288.00	4.896	−1.63	288.00	0.903	−7.20
293.00	5.577	−3.94	293.00	1.154	1.13
298.00	6.872	1.94	298.40	1.339	−1.42
303.10	7.931	1.13	303.20	1.631	3.00
308.20	9.074	−0.32	308.00	1.882	2.28
313.00	10.38	−0.72	313.00	2.151	0.19
318.00	11.98	−0.68	318.00	2.483	−0.68
323.00	13.98	0.69	323.00	2.890	−0.55
	Isobutanol			Methylbenzene	
283.00	0.425	−11.7	283.00	6.295	−5.07
288.00	0.544	−10.1	288.00	8.088	−2.21
293.00	0.691	−8.97	293.00	9.674	−6.52
298.40	0.902	−6.43	298.00	12.39	−3.41
303.20	1.212	2.06	303.00	15.25	−4.18
308.00	1.563	6.27	308.00	19.52	−0.69
313.00	1.924	5.46	313.00	24.97	2.88
318.00	2.296	1.96	318.00	29.99	0.45
323.00	2.668	−4.12	323.00	36.96	0.80

experimental and calculated results is presented in Table 1. x_1^{calcd} represents the solubility calculated from eq 2, and x_1^{exptl} represents the experimental solubility values. The values of the

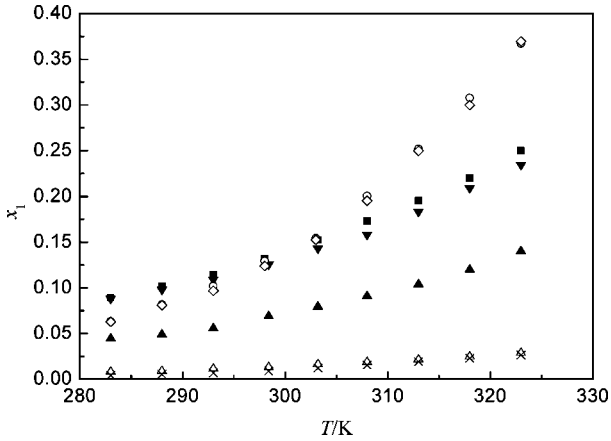

Figure 1. Mole fraction solubility of fluorene (x_1) in different solvents from (283.00 to 323.00) K. ▼, benzene; ■, chloroform; ▲, acetone; Δ, 1-propanol; ×, isobutanol; ◇, methylbenzene; ○, methylbenzene in the literature.[7]

Table 2. Curve-Fitting Parameters of Equation 2 for Fluorene in Benzene, Chloroform, Acetone, 1-Propanol, Isobutanol, and Methylbenzene from (283.00 to 323.00) K

solvent	A	B	C	100 rmsd
benzene	−97.752	2365.2	15.401	1.15
chloroform	−64.383	801.29	10.474	0.67
acetone	−64.406	487.16	10.545	2.16
1-propanol	−65.649	305.91	10.586	2.79
isobutanol	−96.548	710.90	15.709	7.12
methylbenzene	−101.009	1132.43	16.702	3.53

three parameters, A, B, and C, together with the root-mean-square relative deviations (rmsd), are listed in Table 2. The rmsd is defined as the following

$$\mathrm{rmsd} = \left\{ \frac{1}{N} \sum_{i=1}^{N} \left(\frac{x_i^{calcd} - x_i^{exptl}}{x_i^{exptl}} \right)^2 \right\}^{1/2} \quad (3)$$

where N is the number of experimental points.

Conclusions

From Tables 1 and 2 and Figure 1, we can draw the following conclusions: (1) The solubility of fluorene in benzene, chloroform, acetone, 1-propanol, isobutanol, and methylbenzene is a function of temperature, and it increases with an increase in temperature. (2) The best solubility of fluorene was shown in methylbenzene in this work. (3) The calculated solubility of fluorene sets a good coherence with the experimental values, and the experimental solubility and correlation equation in this work can be used for solvent selection and model research in the process of crystallization of fluorene.

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