

Solubility and density of *p*-dioxanone in organic solvent systems

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Abstract : The *p*-dioxanone can be used to make a variety of commercially useful products particularly where biodegradability is desired, such as fibers, sutures, molded articles, containers, medical devices and surgical clips, film and compostable trash bags. The mole fraction solubility of *p*-dioxanone in various solvents such as acetone, ethylacetate, tetrahydrofuran, methanol, ethanol, 1-propanol, 1-butanol and 1-pentanol were measured over the temperature range from 263.05 K to 288.45 K. The solubility of *p*-dioxanone increases with increasing polarity of the solvents.

Keywords : *p*-dioxanone, biodegradability, solubility, density

1. Introduction

The compound of *p*-dioxanone has utility, for example, as a monomer in the production of poly-*p*-dioxanone.

Various methods have been known for the manufacture of this *p*-dioxanone. One method has been developed by our group through the oxidative dehydrogenation of diethylene glycol over metallic catalyst on supported silica particles. Depending on the reaction condition, this crude *p*-dioxanone obtained included many minor impurities such as ethylene glycol, diethylene glycol, acetic acid, water and oligomers due to the high reaction temperature. Usually, exceptionally high purity over 99.9 % of *p*-dioxanone monomer

is required for polymerization to a high molecular weight polymer[1-3].

Solution crystallization[4] has been applied to the purification of this compound due to the property of thermally unstable ether group. For the effective removal of impurity and the kinetic of crystallization such as nucleation and crystal growth, selecting the proper solvent through the experiments of solubility and density is very important for the high purified *p*-dioxanone. But, there appear to be no previous measurements on the solubility, density for the systems including *p*-dioxanone.

In this study, the solubility and density of *p*-dioxanone in various solvents such as ethylacetate, tetrahydrofuran, acetone, methanol, ethanol, 1-propanol, 1-butanol and 1-pentanol were measured over the temperature range from 263.05 K to 298.55 K.

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2. Experiments

2.1. Materials

p-Dioxanone (PDX) solid below 301.7 K was prepared and purified in our pilot plant according to procedures in ref. 1 and 2. The purity was greater than 99.9 %. The solvents were from commercial sources (Aldrich, Merck), analytical reagent grade with a minimum purity of > 99 %. Before their use all compounds were stored over molecular sieves to avoid contamination with water. The materials were analyzed by flame-ionization detection gas chromatograph (FID, Hewlett Packard 5890 Series II) equipped with capillary columns (BP20 1.0 UM, SGE). The physical properties of the pure components applied are listed in Table 1. For *p*-dioxanone the enthalpy of fusion, $D_{fus}H_1$ was determined by differential scanning calorimetry (DSC; TA Instruments, USA) with an accuracy of $\pm 0.05 \text{ kJ}\cdot\text{mol}^{-1}$.

2.2. Measurement of solubility

The mixtures were prepared by mass using a Mettler AG 204 balance with an uncertainty of $\pm 0.0001 \text{ g}$. The estimated uncertainty in the mole fraction is less than $\pm 2 \times 10^{-4}$. The solubility of *p*-dioxanone in the various solvents (tetrahydrofuran, ethylacetate, acetone, methanol, ethanol,

1-propanol, 1-butanol, 1-pentanol) were measured using a polythermal method described in detail previously[6–8]. The experimental apparatus for solubility measurement of PDX was shown in figure 1. The equilibrium cell, a cylindrical glass vessel (50 mm inside diameter, 120 mm long), was placed, by ground-glass joint, in a triple-jacketed vessel. The outer jacket was evacuated, and either heated or cooled fluid from a thermostated bath could be circulated through the middle jacket. An ethylene glycol + water solution was thermostated and controlled by a thermoelectric Eurotherm 808 PID controller with $\pm 0.1 \text{ K}$ uncertainty. The cell had a perforated rubber stopper, through which a copper-constantan thermocouple was inserted. The contents were stirred with a magnetic spin bar. The cell was tightly sealed to protect the system from dust and moisture condensation. Mixtures of solute and solvent were cooled in the bath until an abundant amount of crystal was formed and then heated very slowly at less than $0.001 \text{ K}\cdot\text{min}^{-1}$ near the equilibrium temperature. The crystal disappearance temperature, detected visually, was measured with a calibrated thermocouple connected to a recorder (Yokogawa, 180 micro R). The thermocouples were calibrated with a calibrated thermometer from Fisher Scientific

Table 1. Physical properties of the chemicals applied[5]

compound	CAS number	T_{fus}/K	$r/(\text{g}\cdot\text{cm}^{-3})$	$D_{fus}H/(\text{kJ}\cdot\text{mol}^{-1})$	dielectric constants
<i>p</i> -dioxanone	[3041-16-5]	301.70	1.390	16.14	-
acetone	[67-64-1]	178.50	0.784	5.690	20.56
ethylacetate	[141-78-6]	10.48	0.895	10.48	6.05
tetrahydrofuran	[109-99-9]	164.76	0.882	8.540	7.58
methanol	[67-56-1]	175.47	0.786	3.215	32.66
ethanol	[64-17-5]	158.66	0.785	5.020	24.55
1-propanol	[72-23-8]	147.00	0.799	5.372	20.45
1-butanol	[71-36-3]	184.53	0.806	9.372	17.51
1-pentanol	[71-41-0]	195.00	0.810	10.50	13.9

(No. 15-078-7) with an uncertainty of ± 0.05 K. The uncertainty of the thermocouple measurements is believed to be ± 0.1 K. Some of the experiments were conducted in triplicate to check the reproducibility. The saturation temperature for a given mixture was uncertainty within ± 0.1 K.

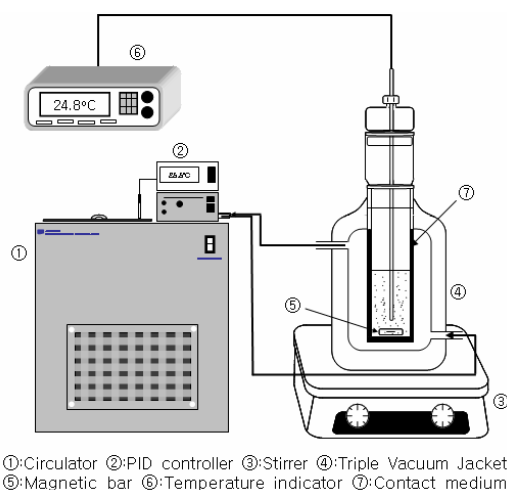


Figure 1. Experimental apparatus for the solubility measurement of PDX.

2.3. Measurement of density

The density of *p*-dioxanone in the various solvents (tetrahydrofuran, acetone, methanol, ethanol, 1-propanol, 1-butanol, 1-pentanol) was determined using a digital densimeter (Mettler Toledo, AG) with density measurement packages (No. 210260 and No. 238491), which uses the Archimedes' principle. The density of solution was determined using a sinker of known volume (10 mL). The sinker was weighed in air and then in the solution. The method was described in detail in the previous works[6]. Before measurement, the densimeter was calibrated with distilled water at the experimental temperatures. Some of the experiments were conducted to check the accuracy at the end of measurement. The densimeter in the above configuration appeared to have an uncertainty of ± 0.0002

$\text{g}\cdot\text{cm}^{-3}$. The bath temperature was measured with a calibrated thermocouple and maintained constant to ± 0.1 K at the desired temperature.

3. Results and discussions

3.1. Solubility

The mole fraction solubility of *p*-dioxanone in various solvents such as acetone, ethylacetate, tetrahydrofuran, methanol, ethanol, 1-propanol, 1-butanol and 1-pentanol were measured over the temperature range from 263.05 K to 288.45 K, and the values are listed in Table 2 and plotted in Figure 2. The solubilities of *p*-dioxanone increase with increase in the temperature and decrease with an increase in the number of carbon atoms in alcohols. This means that the solubility of *p*-dioxanone in alcohols increases with increasing polarity of the solvents. In different types of solvents, the temperature dependence of the solubility of *p*-dioxanone was in the order of acetone > ethylacetate \approx tetrahydrofuran > alcohols, which depended on the difference between interactions of solute and solvent.

It was found that exponential temperature dependence, as shown in Figure 2, permitted a linear interpolation. Thus, the mole fraction solubility x_1 of *p*-dioxanone was analyzed using the equation

$$x_1 = a \cdot e^{\beta(T/K)} \quad (1)$$

where T is the absolute temperature and a and β are parameters. The exponential expression describes satisfactorily the temperature dependence of the solubility, within the temperature range studied. The binary parameters for Eq. (1) with the relative standard deviation (RSD) and the absolute average deviation (AAD) are given in Table 3. For all studied systems the

obtained RSD and AAD are smaller than 0.065 and 0.010, respectively.

The RSD is defined by

$$RSD = \left[\frac{1}{N} \sum_{i=1}^N \left(\frac{X_{1,i}(cal) - X_{1,i}}{X_{1,i}} \right)^2 \right]^{(1/2)} \quad (2)$$

and the AAD is defined by

$$AAD = \frac{1}{N} \sum_{i=1}^N |X_{1,i}(cal) - X_{1,i}| \quad (3)$$

where N is the number of data points.

Solubility data can be used to estimate the activity coefficient through the equilibrium relationship. If no solid–solid transition occurs in the temperature range, the following equation can be used to calculate the

solid–liquid–phase equilibria [9–10].

$$\ln x_1^L \gamma_1^L = - \frac{\Delta_{fus} H_1}{R} \left(\frac{1}{T} - \frac{1}{T_{fus,1}} \right) \quad (4)$$

where x_1^L is the mole fraction of component 1 (*p*-dioxanone) in the liquid phase, γ_1^L is the activity coefficient of component 1 in the liquid phase, $\Delta_{fus} H_1$ is the molar enthalpy of fusion of component 1, $T_{fus,1}$ is the melting temperature of component 1, T is the absolute temperature of the mixture, and R is the universal gas constant. The activity coefficients γ_1^L can be calculated using Eq. (4) from experimentally determined solution composition x_1^L and temperature T .

Table 2. Experimental solubilities of *p*-dioxanone (x_1) in various solvents

acetone		ethylactate		tetrahydrofuran		methanol	
x_1	T/K	x_1	T/K	x_1	T/K	x_1	T/K
0.2766	263.05	0.1701	263.55	0.1531	263.25	0.0557	263.15
0.3114	265.55	0.2049	268.25	0.1752	265.55	0.0693	265.75
0.3326	268.15	0.2311	270.85	0.2000	268.15	0.0864	268.05
0.3608	270.55	0.2713	273.05	0.2311	271.05	0.1084	270.95
0.4019	273.15	0.3020	276.05	0.2683	273.45	0.1400	273.45
0.4395	275.65	0.3469	278.15	0.2958	275.55	0.1876	275.75
0.4859	278.45	0.3859	280.95	0.3437	278.15	0.2394	278.05
0.5325	280.75	0.4471	283.45	0.3905	281.05	0.3166	280.85
0.5778	283.25	0.5090	286.05	0.4561	283.15	0.4022	283.15
0.6371	285.55	0.5637	288.15	0.5264	286.05	0.5067	285.65
0.6813	288.45	0.6425	290.85	0.6017	288.35	0.6212	288.45
ethanol		1-propanol		1-butanol		1-pentanol	
x_1	T/K	x_1	T/K	x_1	T/K	x_1	T/K
0.0145	263.45	0.0055	263.15	0.0029	263.15	0.0012	263.15
0.0215	265.65	0.0086	265.55	0.0043	265.75	0.0017	265.75
0.0272	268.15	0.0120	268.45	0.0058	268.05	0.0029	268.25
0.0390	270.95	0.0173	270.65	0.0091	270.55	0.0040	270.85
0.0492	273.15	0.0233	273.15	0.0134	273.45	0.0052	273.15
0.0660	275.75	0.0345	275.65	0.0227	275.95	0.0091	275.95
0.1016	278.25	0.0468	278.45	0.0287	278.15	0.0142	278.25
0.1501	280.75	0.0761	280.65	0.0536	280.55	0.0219	280.75
0.2101	283.15	0.1131	283.15	0.0681	283.25	0.0291	283.05
0.3119	285.75	0.1757	285.95	0.1137	285.95	0.0466	285.65
0.4094	288.35	0.2376	288.05	0.1529	288.15	0.0631	288.15

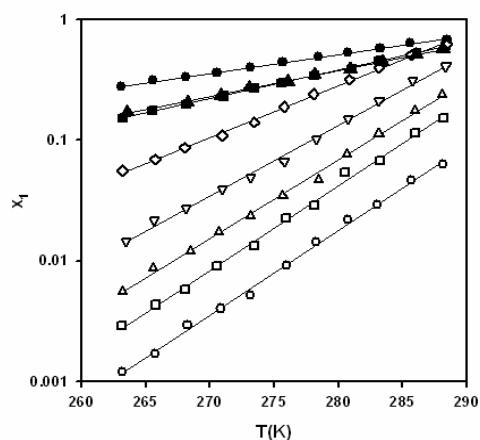


Figure 2. Mole fraction solubility of *p*-dioxanone(1) with various solvents : acetone (●), ethylacetate(▲), tetrahydrofuran (■), methanol (◇), ethanol (▽), 1-propanol (△), 1-butanol (□), 1-pentanol (○).

Table 4 gives mole fractions, equilibrium temperature and activity coefficients. Table 4 shows that in nearly all the cases, positive deviation from ideal solution ($\gamma_1 > 1$) are encountered in the systems showed. Such deviation is attributed to the interaction of a chemical nature between the *p*-dioxanone and solvent.

3.2. Density of *p*-dioxanone + solvents solution

The experimental densities for the *p*-dioxanone(1) + tetrahydrofuran, + ethylacetate, + acetone, + methanol, + ethanol, + 1-propanol, + 1-butanol and + 1-pentanol were measured in the range of from 263.05 K to 298.55 K and $x_1 = 0.0291$ to 0.6801 and are listed in Table 5. As a typical example, densities of *p*-dioxanone + acetone solution as functions of mole fraction and temperature were shown in Figure 3. Densities of this solution decreases linearly with increasing temperature. The densities for *p*-dioxanone + solvent systems were correlated by Eq. (5) as a function of absolute temperature, T , and *p*-dioxanone mole fraction, x_1 as below:

$$\rho / (Kg \cdot m^{-3}) = K_1 + K_2 x_1 + K_3 (T/K) + K_4 x_1 (T/K) \quad (5)$$

The values of coefficients k_1 , k_2 , k_3 , and k_4 are listed in Table 6 with the RSD and AAD between the measured value and the calculated value, which is defined in Eq. (2) and (3).

Table 3. Values of parameters for equation (1) in various solvents

system	a	b / K^{-1}	RSD	AAD
acetone	2.184010^{-5}	0.0359	0.017	0.004
tetrahydrofuran	9.875710^{-8}	0.0541	0.017	0.005
methanol	2.621110^{-13}	0.0989	0.040	0.010
ethanol	5.765210^{-18}	0.1346	0.065	0.005
1-propanol	4.165810^{-20}	0.1498	0.062	0.003
1-butanol	9.413810^{-22}	0.1616	0.065	0.002
1-pentanol	3.540210^{-22}	0.1620	0.062	0.001

Table 4. Activity coefficient of *p*-dioxanone in various solvents

acetone			ethylacetate			tetrahydrofuran		
<i>T</i> /K	x_1^L	γ_1^L	<i>T</i> /K	x_1^L	γ_1^L	<i>T</i> /K	x_1^L	γ_1^L
263.05	0.2758	1.41	263.55	0.1662	2.371	263.25	0.1513	2.58
265.55	0.3016	1.38	268.25	0.2094	2.140	265.55	0.1713	2.43
268.15	0.3312	1.35	270.85	0.2381	2.019	268.15	0.1972	2.27
270.55	0.3610	1.32	273.05	0.2653	2.919	271.05	0.2307	2.09
273.15	0.3963	1.29	276.05	0.3075	1.788	273.45	0.2626	1.96
275.65	0.4335	1.26	278.15	0.3010	1.701	275.55	0.2942	1.85
278.45	0.4793	1.22	280.95	0.3915	1.588	278.15	0.3387	1.71
280.75	0.5206	1.19	283.45	0.4428	1.493	281.05	0.3962	1.57
283.25	0.5695	1.15	286.05	0.5032	1.398	283.15	0.4439	1.48
285.55	0.6185	1.12	288.15	0.5581	1.324	286.05	0.5193	1.35
288.45	0.6863	1.08	290.85	0.6374	1.234	288.35	0.5881	1.26
methanol			ethanol			1-propanol		
<i>T</i> /K	x_1^L	γ_1^L	<i>T</i> /K	x_1^L	γ_1^L	<i>T</i> /K	x_1^L	γ_1^L
263.15	0.0526	7.41	263.45	0.0145	27.10	263.15	0.0055	70.84
265.75	0.0681	6.15	265.65	0.0195	21.42	265.55	0.0079	52.72
268.05	0.0855	5.21	268.15	0.0273	16.38	268.45	0.0121	37.25
270.95	0.1138	4.23	270.95	0.0398	12.11	270.65	0.0169	28.28
273.45	0.1458	3.53	273.15	0.0535	9.54	273.15	0.0246	20.75
275.75	0.1830	2.98	275.75	0.0759	7.19	275.65	0.0357	15.25
278.05	0.2297	2.52	278.25	0.1062	5.47	278.45	0.0543	10.76
280.85	0.3030	2.05	280.75	0.1487	4.16	280.65	0.0755	8.17
283.15	0.3804	1.72	283.15	0.2054	3.19	283.15	0.1098	5.97
285.65	0.4871	1.43	285.75	0.2915	2.39	285.95	0.1671	4.20
288.45	0.6426	1.16	288.35	0.4136	1.79	288.05	0.2288	3.22
1-butanol			1-pentanol					
<i>T</i> /K	x_1^L	γ_1^L	<i>T</i> /K	x_1^L	γ_1^L			
263.15	0.0028	139.15	263.15	0.0012	324.68			
265.75	0.0042	99.72	265.75	0.0018	232.65			
268.05	0.0061	73.09	268.25	0.0026	172.42			
270.55	0.0092	51.82	270.85	0.0040	120.13			
273.45	0.0146	35.23	273.15	0.0058	88.01			
275.95	0.0219	25.05	275.95	0.0092	59.63			
278.15	0.0313	18.53	278.25	0.0134	43.39			
280.55	0.0461	13.35	280.75	0.0200	30.94			
283.25	0.0713	9.22						
285.95	0.1102	6.36						
288.15	0.1573	4.70						

Table 5. Experimental densities of *p*-dioxanone (x_1) in various solvents

acetone			tetrahydrofuran			methanol		
T/K	x_1	$\rho/\text{Kg}\cdot\text{m}^{-3}$	T/K	x_1	$\rho/\text{Kg}\cdot\text{m}^{-3}$	T/K	x_1	$\rho/\text{Kg}\cdot\text{m}^{-3}$
298.05	0.6801	1134	298.05	0.6027	1105	298.05	0.6010	1164
293.05	0.6801	1140	293.05	0.6027	1109	293.05	0.6010	1171
288.05	0.6801	1147	288.05	0.6027	1114	288.05	0.6010	1177
283.05	0.6801	1153	283.05	0.6027	1119	283.05	0.6010	1183
298.05	0.4985	1052	298.05	0.5088	1068	298.05	0.4010	1070
293.05	0.4985	1058	293.05	0.5088	1073	293.05	0.4010	1077
288.05	0.4985	1064	288.05	0.5088	1078	288.05	0.4010	1083
283.05	0.4985	1070	283.05	0.5088	1082	283.05	0.4010	1090
278.05	0.4985	1077	278.05	0.5088	1087	278.05	0.4010	1096
298.05	0.3021	963	298.05	0.2977	988	298.05	0.2508	1001
293.05	0.3021	969	293.05	0.2977	992	293.05	0.2508	1007
288.05	0.3021	975	288.05	0.2977	996	288.05	0.2508	1013
283.05	0.3021	981	283.05	0.2977	1000	283.05	0.2508	1019
278.05	0.3021	988	278.05	0.2977	1004	278.05	0.2508	1026
273.05	0.3021	994	273.05	0.2977	1008	273.05	0.2508	1032
298.05	0.0989	871	298.05	0.1238	921	298.05	0.0947	928
293.05	0.0989	877	293.05	0.1238	925	293.05	0.0947	934
288.05	0.0989	884	288.05	0.1238	928	288.05	0.0947	941
283.05	0.0989	890	283.05	0.1238	932	283.05	0.0947	946
278.05	0.0989	896	278.05	0.1238	935	278.05	0.0947	953
273.05	0.0989	902	273.05	0.1238	939	273.05	0.0947	959
268.05	0.0989	908	268.05	0.1238	942	268.05	0.0947	965
298.05	0.0501	849	298.05	0.0501	892	298.05	0.0501	907
293.05	0.0501	855	293.05	0.0501	896	293.05	0.0501	913
288.05	0.0501	861	288.05	0.0501	899	288.05	0.0501	919
283.05	0.0501	867	283.05	0.0501	903	283.05	0.0501	925
278.05	0.0501	874	278.05	0.0501	906	278.05	0.0501	932
273.05	0.0501	880	273.05	0.0501	910	273.05	0.0501	938
268.05	0.0501	886	268.05	0.0501	913	268.05	0.0501	944
263.05	0.0501	892	263.05	0.0501	916	263.05	0.0501	950
ethylacetate			ethanol			1-propanol		
T/K	x_1	$\rho/\text{Kg}\cdot\text{m}^{-3}$	T/K	x_1	$\rho/\text{Kg}\cdot\text{m}^{-3}$	T/K	x_1	$\rho/\text{Kg}\cdot\text{m}^{-3}$
298.55	0.5638	1098	298.05	0.4080	1029	298.05	0.2389	918
293.05	0.5638	1104	293.05	0.4080	1035	293.05	0.2389	922
288.05	0.5638	1110	288.05	0.4080	1040	288.05	0.2389	927
283.05	0.5638	1115	283.05	0.4080	1046	283.05	0.2389	932
298.55	0.4471	1065	298.05	0.3109	975	298.05	0.1357	873
293.05	0.4471	1072	293.05	0.3109	980	293.05	0.1357	876
288.05	0.4471	1078	288.05	0.3109	986	288.05	0.1357	882
283.05	0.4471	1083	283.05	0.3109	992	283.05	0.1357	887
278.05	0.4471	1090	278.05	0.3109	998	278.05	0.1357	892
298.05	0.3013	1001	298.05	0.2131	921	298.05	0.1053	860
293.05	0.3013	1007	293.05	0.2131	926	293.05	0.1053	864
288.05	0.3013	1012	288.05	0.2131	932	288.05	0.1053	869
283.05	0.3013	1018	283.05	0.2131	938	283.05	0.1053	874

ethylacetate			ethanol			1-propanol		
T/K	x_1	$r/\text{Kg}\cdot\text{m}^{-3}$	T/K	x_1	$r/\text{Kg}\cdot\text{m}^{-3}$	T/K	x_1	$r/\text{Kg}\cdot\text{m}^{-3}$
278.05	0.3013	1024	278.05	0.2131	943	278.05	0.1053	878
273.05	0.3013	1029	273.05	0.2131	948	273.05	0.1053	883
298.05	0.2010	964	298.05	0.0501	830	298.05	0.0501	836
293.05	0.2010	969	293.05	0.0501	836	293.05	0.0501	840
288.05	0.2010	976	288.05	0.0501	841	288.05	0.0501	845
283.05	0.2010	981	283.05	0.0501	847	283.05	0.0501	849
278.05	0.2010	986	278.05	0.0501	852	278.05	0.0501	854
273.05	0.2010	992	273.05	0.0501	858	273.05	0.0501	859
268.05	0.2010	998	268.05	0.0501	863	268.05	0.0501	864
1-butanol			1-pentanol					
T/K	x_1	$r/\text{Kg}\cdot\text{m}^{-3}$	T/K	x_1	$r/\text{Kg}\cdot\text{m}^{-3}$			
298.05	0.1529	877	298.05	0.0631	847			
293.05	0.1529	881	293.05	0.0631	851			
288.05	0.1529	885	288.05	0.0631	855			
283.05	0.1529	890	283.05	0.0631	858			
298.05	0.1137	862	298.05	0.0501	841			
293.05	0.1137	866	293.05	0.0501	845			
288.05	0.1137	870	288.05	0.0501	849			
283.05	0.1137	874	283.05	0.0501	854			
278.05	0.1137	878	278.05	0.0501	858			
298.05	0.0501	836	295.55	0.0291	835			
293.05	0.0501	840	290.55	0.0291	839			
288.05	0.0501	845	285.55	0.0291	843			
283.05	0.0501	849	280.55	0.0291	847			
278.05	0.0501	853	275.55	0.0291	851			
273.05	0.0501	858	270.55	0.0291	855			

Table 6. Values of parameters for equation (5) in various solvents

solvent	k_1	k_2	k_3	k_4	RSD	AAD
tetrahydrofuran	1067.2	564.7	-0.6498	-0.537	3.6×10^{-4}	0.30
acetone	1197.8	442.6	-1.2472	0.036	3.3×10^{-4}	0.29
ethylacetate	1218.5	399.4	-1.1017	-0.103	4.0×10^{-4}	0.39
methanol	1253.2	493.9	-1.2410	-0.089	3.7×10^{-4}	0.29
ethanol	1133.9	557.2	-1.1131	-0.002	4.2×10^{-4}	0.29
1-propanol	1086.1	474.7	-0.9131	-0.135	5.2×10^{-4}	0.31
1-butanol	1073.5	352.1	-0.8635	0.159	4.0×10^{-4}	0.34
1-pentanol	1067.2	179.3	-0.8283	0.804	5.7×10^{-4}	0.30

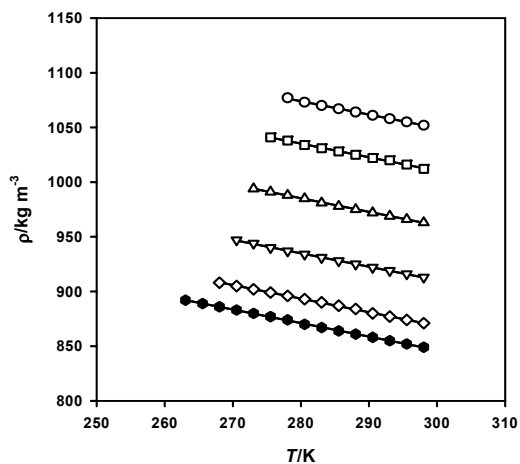


Figure 3. Density of *p*-dioxanone(1)-acetone solution as a function of mole fraction x_1 : (○)=0.4985, (□)=0.4123, (△)=0.3021, (▽)=0.1911, (◇)=0.0989, (●)=0.0501.

4. Conclusions

The solubilities of *p*-dioxanone were increased with increase in the temperature and decreased with an increase in the number of carbon atoms in alcohols. The temperature dependence of the solubility of *p*-dioxanone was in the order of acetone > ethylacetate ≈ tetrahydrofuran > alcohols, which depended on the difference between interactions of solute and solvent.

The experimental densities for the *p*-dioxanone + tetrahydrofuran, + ethylacetate + acetone, + methanol, + ethanol, + 1-propanol, + 1-butanol and + 1-pentanol were decreased linearly with increasing temperature in the range of from 263.05 K to 298.55 K and $x_1 = 0.0291$ to 0.6801.

Reference

1. J. H. Lee, H. R. Kim, Y. H. Han, S. J. Jeong, Y. J. Seo and B. R. Choi, Korean

- Patent KR 2003-0050471, (2003).
2. C. U. Kim, K. J. Kim and Y. H. Cheon, Korean Patent KR 2004-0033122, (2004).
3. C. G. Moyers and M. P. Farr, US Patent Number 5,675,022, (1997).
4. A. Mersmann, "Crystallization Technology Handbook", Marcel Dekker, Inc., New York, (1994).
5. J. A. Riddick, W. B. Bunger and T. K. Sakano, "Techniques of Chemistry, Organic Solvents", 4th ed.; John Wiley and Sons: New York, 1, 190 (1986).
6. K. J. Kim, M. J. Kim, J. M. Lee, H. S. Kim and B. S. Park, Experimental Solubility and Density for 3-Nitro-1,2,4-Triazol-5-One + C1 to C7 1-Alkanols, *Fluid Phase Equilib.*, **146**, 261 (1998).
7. K. J. Kim, M. J. Kim, J. M. Lee, S. H. Kim, H. S. Kim and B. S. Park, Solubility, Density and Metastable Zone Width of the 3-Nitro-1,2,4-triazol-5-one +Water System, *J. Chem. Eng. Data*, **43**, 65 (1998)
8. K. J. Kim, C. H. Lee and S. K. Ryu, "Solubility of Thiourea in C₁-C₆ 1-Alcohols", *J. Chem. Eng. Data*, **39**(2), 228 (1994).
9. J. Gmehling and B. Kolbe, "Thermodynamics", VCH: Weinheim, (1992).
10. A. Jakob, R. John, C. Rose and J. Gmehling, Solid-Liquid Equilibria in Binary Mixtures of Organic Compounds, *Fluid Phase Equilib.*, **113**, 117 (1995).
11. J. M. Prausnitz, R. N. Lichtenthaler and E. G. Azevedo, "Molecular Thermodynamics of Fluid-Phase Equilibria", Prentice-Hall, Inc.: Englewood Cliffs, NJ, Chapter 9 (1986).