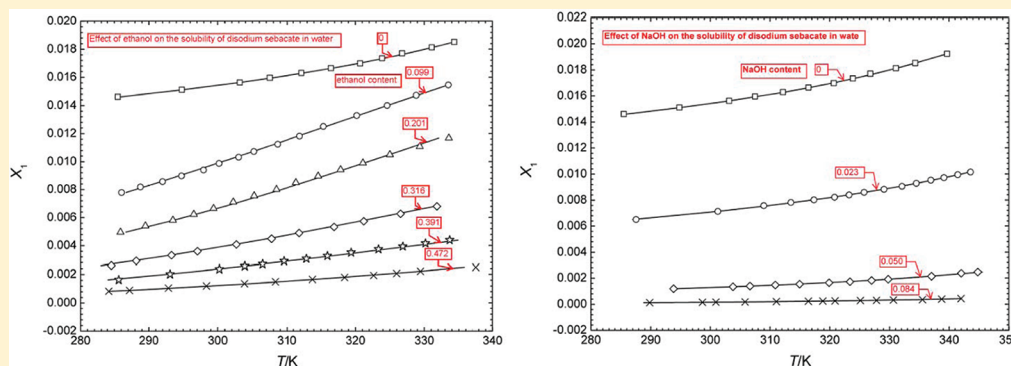# Effects of NaOH and Ethanol on the Solubility of Disodium Decanedioate in Water

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ABSTRACT: To investigate the effects of NaOH and ethanol on the solubility of disodium decanedioate in water, the solubilities of disodium decanedioate in two aqueous solutions (aqueous NaOH solutions and aqueous ethanol solutions) were determined in a temperature range of (284 to 345) K. The solute-free mole fraction of NaOH and ethanol in the solvent mixtures extended from 0 to 0.084 and 0 to 0.472, respectively. With the increase of the mole fractions of ethanol (or NaOH) in solvent mixtures, the solubilities decreased obviously. Alcohol-out and alkali-out phenomena could be evidently observed, between which the alkali-out effect was more prominent. The determined data were correlated with the new electrolyte nonrandom two-liquid (E-NRTL) model, and the root-mean-square deviations of solubility temperature varied from (0.26 to 0.91) K.

INTRODUCTION

Decanedioic acid is extremely valuable for the production of nylon, alkyd resins, plasticizers, cosmetics, and biological agents, and so forth.[1−4] It is mainly prepared from castor oil by caustic fusion method in China, which involves saponification, cracking, neutralization, and acidification processes.[5,6] Some research has been conducted to improve the traditional process.[7,8] Disodium decanedioate (NaOOC-(CH$_2$)$_8$COONa, CAS No. 17265-14-4), which is a white powdered crystal, is generated in cracking process and then acidified to sebacic acid. In the current industrial process, excess NaOH is used in the cracking reaction to keep the high reaction yield. Therefore the cracking residue contains about 0.5 mass fraction disodium decanedioate, 0.2 mass fraction sodium aliphatate, near 0.2 mass fraction free NaOH, and trace sodium phenate (phenol is used as a diluent in the cracking reaction). The free NaOH needs to consume extra strong acid H$_2$SO$_4$ in the following neutralization process and forms sodium sulfate which will be discharged with wastewater and pollute the environment. Some methods have been developed to treat the sebacic acid wastewater.[9−11] In fact, if we can separate disodium decanedioate out of the cracking residue and recycle free NaOH to the cracking reaction, not only the NaOH consumption can be reduced, but also the H$_2$SO$_4$ consumption and the sodium sulfate production can be reduced. Finally, the effluent wastewater and production cost

of sebacic acid will be decreased greatly. Solubility, as an important thermodynamic property, is always treated as essential information in a separation process. We have been focusing on the improvement of the production process of sebacic acid and have systematically determined the solubility data of sebacic acid in several pure solvents and binary solvent mixtures.[12,13] To design the recovering and recycling free NaOH process, solubility data of disodium decanedioate are needed. However, no relative solubility data can be found in popular reference books and databases. Therefore, the solubilities of disodium decanedioate in aqueous ethanol and aqueous NaOH solutions at different temperatures were measured by a dynamic method in a temperature range from (284 to 345) K. The experimental data were correlated with the new electrolyte nonrandom two-liquid (E-NRTL) model[14] which could provide reliable results for mixed solvent electrolyte systems.[14−16]

EXPERIMENTAL SECTION

Materials. All chemicals used were ACS grade or better with assays of 0.99 mass purity. Sebacic acid (0.995 mass purity plus) and NaOH (ACS grade, 0.97 mass purity) were purchased

from Aladdin-Reagent, Shanghai Crystal Pure Industrial Co., Ltd., Shanghai, China, while ethanol (0.996 mass purity plus) was purchased from Guangfu Chemical Reagents Co., Tianjin, China. The reagents above were used without further purification.

Disodium decanedioate was prepared by reacting sebacic acid with NaOH in aqueous solution. Sebacic acid was mixed in deionized water and heated up to 363 K; then newly prepared 20 mol·kg^{-1} NaOH solution was added slowly with continuously stirring by a mechanical stirrer. The most suitable feed molar ratio (sebacic acid/NaOH), reaction time, and operating temperature were 1:2.1, 2 h, and 368 K, respectively. After isothermal reaction under operating conditions, the solution was vaporized, cooled to room temperature, filtered, and dried to constant weight for 3 h. The obtained disodium decanedioate was then recrystallized in water (Microprocessor pH meter was used to confirm no remaining NaOH). The disodium decanedioate used in the experiment has a purity over 0.99 mass fraction, which was confirmed by high-performance liquid chromatography (HPLC) combined with infrared (IR) spectroscopy.

The concentrations of aqueous NaOH solutions were analyzed by titration using newly prepared aqueous potassium hydrogen phthalate solution, and the determination was repeated five times. In the present work, deionized water was generated from a HETHC laboratory water purification system with a resistivity less than 18.2 MΩ·cm.

Apparatus and Procedure. All measurements were made on freshly prepared solutions, which were prepared gravimetrically using an AND model GR-120 analytical balance. The accuracy of the given composition was ± 0.0001 g. The solubility determination was made by the dynamic method[17] combined with a laser technique. Predetermined solute and solvent were mixed and heated slowly with a continuously stirring inside a jacketed glass vessel. The temperature of the system, which was detected by a platinum resistance thermometer Pt-100 (calibrated with an accuracy of ± 0.01 K), was controlled by a refrigerated/heating circulator (Julabo FP45-HE, Germany, temperature stability ± 0.01 K). The heating rate would be less than 0.1 K·h^{-1} near the solid−liquid equilibrium (SLE) temperature. A laser monitoring system (manufactured by Department of Physics, Peking University) was run to determine the SLE temperature, at which the solid just disappeared and the intensity of the laser beam reached a maximum. The uncertainty of temperature measurement was ± 0.3 K, while the uncertainty of mole fraction solubility was ± 2·10^{-6}.

RESULTS AND DISCUSSION

Experimental solubilities of disodium decanedioate in aqueous ethanol solutions and aqueous NaOH solutions are given in Tables 1 and 2, respectively, where T^{exp} is the experimentally measured temperature; x_1 is the mole fraction solubility; x_3^0 is the solute-free mole fraction of ethanol in solvent mixtures; and x_4^0 is the solute-free mole fraction of NaOH in solvent mixtures.

Figures 1 and 2 represent plots of x_1 versus temperature at different x_3^0 or x_4^0 values. In Figure 1, the experimental solubilities increase with temperature in all concentration ranges. The addition of ethanol, from 0 to 0.472 solute-free mole fractions, results in a prominent decrease in solubility, which shows the ethanol-out effect. As shown in Figure 2, all of the experimental determined solubility data increase with temperature and diminish with NaOH content (alkali-out

Table 1. Experimental Solubility x_1 at Temperature T^{exp} and Solute-Free Mole Fraction of Ethanol x_3^0 for the System of Disodium Decanedioate (1) + Water (2) + Ethanol (3)^a

$x_1 \cdot 10^3$	T^{exp}/K	$x_1 \cdot 10^3$	T^{exp}/K	$x_1 \cdot 10^3$	T^{exp}/K
		$x_3^0 = 0.099$			
7.7780	286.05	9.8525	300.21	12.498	315.37
8.1754	288.79	10.299	302.99	13.279	320.20
8.5669	292.00	10.711	305.20	13.991	324.61
8.9640	294.75	11.229	308.63	14.712	328.83
9.3870	297.93	11.812	311.90	15.437	333.50
		$x_3^0 = 0.201$			
4.9595	285.90	7.1045	302.20	9.4143	317.87
5.3984	289.50	7.5473	305.25	9.8937	321.10
5.8129	293.51	8.0050	308.45	10.492	325.03
6.2269	296.53	8.4904	311.36	11.072	329.35
6.6527	299.39	8.9537	314.60	11.673	333.57
		$x_3^0 = 0.316$			
2.5893	284.57	4.0728	302.65	5.7295	321.23
2.9132	288.19	4.4891	307.75	6.2616	326.58
3.3171	293.29	4.8983	311.85	6.7891	331.85
3.5952	297.07	5.3247	316.91		
		$x_3^0 = 0.391$			
1.6231	285.65	2.8987	309.55	3.7296	323.36
2.0015	293.10	3.0689	312.91	3.9285	326.90
2.3426	300.25	3.2670	315.95	4.1565	330.20
2.5522	303.90	3.5002	319.39	4.3912	333.71
2.6967	306.47				
		$x_3^0 = 0.472$			
0.8273	284.20	1.3582	303.83	1.9260	322.65
0.8962	287.21	1.4958	308.30	2.0701	326.01
1.0485	292.86	1.6571	313.78	2.2136	329.47
1.1954	298.37	1.7965	318.45	2.4850	337.60

^{a}Standard uncertainties u are $u(T) = 0.3$ K, $u(x) = 2 \cdot 10^{-6}$.

Table 2. Experimental Solubility x_1 at Temperature T^{exp} and Solute-Free Mole Fraction of NaOH x_4^0 for the System of Disodium Decanedioate (1) + Water (2) + NaOH (4)^a

$x_1 \cdot 10^3$	T^{exp}/K	$x_1 \cdot 10^3$	T^{exp}/K	$x_1 \cdot 10^3$	T^{exp}/K
		$x_4^0 = 0.000$			
14.607	285.50	16.289	312.18	17.700	326.80
15.100	294.81	16.639	316.45	18.120	331.10
15.614	303.16	16.978	320.70	18.503	334.35
15.943	307.50	17.341	323.90	19.222	340.67
		$x_4^0 = 0.023$			
6.4976	287.55	8.2202	320.80	9.2821	334.50
7.1237	301.25	8.3909	323.25	9.5256	336.95
7.5643	308.95	8.5900	325.75	9.7086	339.15
7.8076	313.49	8.8210	329.10	9.9416	341.45
8.0094	317.35	9.0523	332.10	10.138	343.60
		$x_4^0 = 0.050$			
1.1816	293.85	1.5327	314.95	1.9089	329.81
1.3298	303.80	1.6350	319.90	2.1307	337.07
1.3840	306.65	1.7125	323.40	2.3611	342.05
1.4653	310.89	1.8069	327.03	2.4669	344.81
		$x_4^0 = 0.084$			
0.1113	289.85	0.2207	316.42	0.3136	330.67
0.1429	298.69	0.2377	318.85	0.3515	335.55
0.1522	300.93	0.2455	320.90	0.3848	338.77
0.1722	305.83	0.2798	325.15	0.4213	342.01
0.1966	311.05	0.2924	327.83		

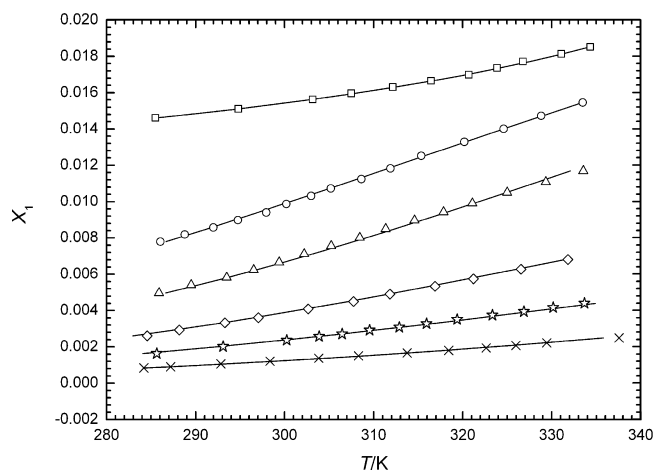^{a}Standard uncertainties u are $u(T^{exp}) = 0.3$ K, $u(x) = 2 \cdot 10^{-6}$.

Figure 1. Mole fraction solubilities of disodium decanedioate in binary ethanol + water solvent mixtures. The solute-free mole fraction of ethanol x_3^0 is: □, 0 (pure water); ○, 0.099; △, 0.201; ◇, 0.316; ☆, 0.391; ×, 0.472; —, solubility curve calculated from the new E-NRTL model.
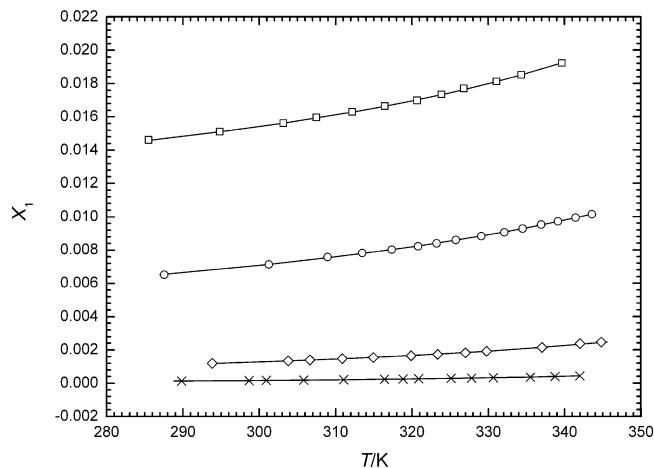

Figure 2. Mole fraction solubilities of disodium decanedioate in aqueous NaOH solutions. The solute-free mole fraction of NaOH x_4^0 is: □, 0 (pure water); ○, 0.023; ◇, 0.050; ×, 0.084; —, solubility curve calculated from the new E-NRTL model.

effect). Comparing with Figure 1, the decrease made by the NaOH addition is much more obvious. It demonstrates that, in the solvent mixtures of this paper, the alkali-out effect is more prominent than the ethanol-out effect.

The SLE is described as follows, in which solubility is defined by the solubility product of disodium decanedioate in the aqueous solution K_s:

$$K_s = a_+^{\nu+} a_-^{\nu-} = (\gamma_+^* x_+)^{\nu+} (\gamma_-^* x_-)^{\nu-} \tag{1}$$

where $\nu+$ and $\nu-$ indicate the electrolyte stoichiometric number of cations and anions, respectively; a, γ^*, and x are the activity, the unsymmetric activity coefficient (the superscript * denotes

an unsymmetric reference), and the mole fraction of the different species, respectively. Equation 1 is fit for aqueous, organic, or mixed solvent electrolyte solutions.[18]

In the SLE of disodium decanedioate + NaOH + water, the temperature dependence of the solubility product is assumed to be represented by the van't Hoff type equation[19] as follows:

$$\ln K_s = A + B/T \tag{2}$$

where T is the absolute temperature in Kelvin; A and B are constants and denote the thermal parameters of the pure salts.

In mixed solvent system disodium decanedioate + ethanol + water, eq 2 needs to be modified because the solubility product is not only the function of salt (solute). The solubility product is a function of temperature, salt (solute), and solvent. Considering the effect of solvent, the solubility product of disodium decanedioate in water is different from the solubility product of disodium decanedioate in ethanol. A simple linear function is used to describe the effect of solvent composition. Therefore, for a mixed solvent system, eq 2 can be modified as follows:

$$\ln K_s' = Cx_3^0 + D(1 - x_3^0) + [Ex_3^0 + F(1 - x_3^0)]/T \tag{3}$$

where C, D, E, and F are constants derived from the thermal parameters and gas constant. The parameters A to F are determined by correlating the experimental data and given in Table 3.

The experimentally determined data are regressed by the new E-NRTL model[14] which yields simple activity coefficient expressions for both molecular and ionic species. The unsymmetric activity coefficients of ion i is represented by the combination of the Pitzer–Debye–Hückel (PDH) equation and the NRTL equation as follows:

$$\ln \gamma_i^* = \ln \gamma_i^{*PDH} + \ln \gamma_i^{*lc} \qquad i = m, c, a \tag{4}$$

where m, c, and a refer to molecular components, cationic species, and anionic species, respectively. The PDH equation for long-range interaction contributions in the electrolyte system, γ_i^{*PDH}, is given according to the paper of Chen et al.[20] published in 1982, while the NRTL equations for short-range interaction contribution of cationic species (c) γ_c^{lc} and anionic species (a) γ_a^{lc} are written according to Chen and Song's expression in 2009.[14]

In this work, the NRTL nonrandomness factor parameter α is fixed as a constant 0.2. The binary interaction energy parameter τ_{ij} is expressed to be a function of temperature with two model parameters a_{ij} and b_{ij} as follows:

$$\tau_{ij} = a_{ij} + b_{ij}/T \tag{5}$$

both i and j are used for all of the species. Comparing with the expression of τ_{ij} with a single parameter function, this improvement makes the average root-mean-square deviation σ diminished from (1.9 to 0.65) K for disodium decanedioate + ethanol + water systems and from (2.18 to 0.45) K for

Table 3. Parameters of K_s Equations for Disodium Decanedioate in Different NaOH + Water and Ethanol + Water Solvent Mixtures (Defined by eqs 2 and 3)

system	A	B/K	C	D	E/K	F/K
NaOH + water	−9.9049	−292.21				
ethanol + water			32.313	−9.9049	−22192	−292.21

disodium decanedioate + NaOH + water systems. The model parameters a_{ij} and b_{ij} are determined by correlating the experimental data and listed in Table 4.

Table 4. New E-NRTL Model Parameters a_{ij} and b_{ij} for Disodium Decanedioate + Ethanol + Water and Disodium Decanedioate + NaOH + Water Systems (Defined by eq 5)

i	j	a_{ij}	b_{ij}/K	a_{ji}	b_{ji}/K
disodium decanedioate	water	0.0000	−200.28	18.847	−14946
disodium decanedioate	ethanol	22.767	−7283.6	−45.363	−2154.6
disodium decanedioate	NaOH	4.0908	−1248.2	1058.9	8760.1
NaOH	water	−1.0513	−190.22	63.566	135.42
ethanol	water	−45.942	−1894.2	24.852	−4980.3

The activity coefficients for the short-range interaction γ_c^{lc} and γ_a^{lc} need to be normalized to the unsymmetric activity coefficient γ_c^{*lc} and γ_a^{*lc} by the activity coefficients at infinite dilution aqueous solutions $\gamma_c^{lc,\infty}$ and $\gamma_a^{lc,\infty}$ as follows:

$$\ln \gamma_l^{*lc} = \ln \gamma_i^{lc} - \ln \gamma_i^{lc,\infty} \qquad i = c, a \qquad (6)$$

In the disodium decanedioate + NaOH + water systems, the infinite dilution aqueous solution reference state is expressed as:

$$\ln \gamma_i^{lc,\infty} = \ln \gamma_i^{lc}(x_m \to 1) = z_i(G_{im}\tau_{im} + \tau_{mi}) \qquad (7)$$

where i refers to a or c, and m is water.

In the disodium decanedioate + ethanol + water mixed-solvent systems, the infinite dilution mixed-solvent solution reference state is used. In this case, the $\ln \gamma_i^{lc,\infty}$ is shown as:

$$\ln \gamma_i^{lc,\infty} = \ln \gamma_i^{lc}(x_a \to 0 \text{ and } x_c \to 0) \qquad (8)$$

as a result

$$\frac{1}{Z_i}\ln \gamma_i^{lc,\infty} = \frac{X_m G_{im}}{X_m + X_{m'}G_{m'm}}$$
$$\left(\tau_{im} - \frac{X_{m'}G_{m'm}\tau_{m'm}}{X_m + X_{m'}G_{m'm}} \right)$$
$$+ \frac{X_{m'}G_{im'}}{X_{m'} + X_m G_{mm'}}\left(\tau_{im'} \right.$$
$$\left. - \frac{X_m G_{mm'}\tau_{mm'}}{X_{m'} + X_m G_{mm'}} \right)$$
$$+ \frac{X_m G_{mi}\tau_{mi} + X_{m'}G_{m'i}\tau_{m'i}}{X_m G_{mi} + X_{m'}G_{m'i}} \qquad (9)$$

where i refers to a or c, and m and m' are the molecular components water and ethanol.

The model parameters are estimated using the Nelder−Mead simplex method, in which the root-mean-square deviation σ between the experimental equilibrium temperature and the calculated equilibrium temperature is taken as the object function.[21]

The calculation results are expressed in Figures 1 and 2 with solid lines. The root-mean-square deviations for each solvent system are listed in Table 5, which range from (0.26 to 0.91) K, and the average root-mean-square deviations for disodium decanedioate + ethanol + water systems and disodium

Table 5. Root-Mean-Square Deviations σ from the Description by the New E-NRTL Model

solvent system	σ/K	solvent system	σ/K
water + 0.099 mole fraction ethanol	0.26	pure water	0.27
water + 0.201 mole fraction ethanol	0.67	water + 0.023 mole fraction NaOH	0.33
water + 0.316 mole fraction ethanol	0.56	water + 0.050 mole fraction NaOH	0.62
water + 0.391 mole fraction ethanol	0.66	water + 0.084 mole fraction NaOH	0.57
water + 0.472 mole fraction ethanol	0.91		

decanedioate + NaOH + water systems are 0.65 and 0.45 K, respectively. Relative differences of the experimental temperature T^{exp} from those calculated with the new E-NRTL model T^{cal} at different operating temperatures are illustrated in Figure 3, which shows acceptable results for both the mixed solvent
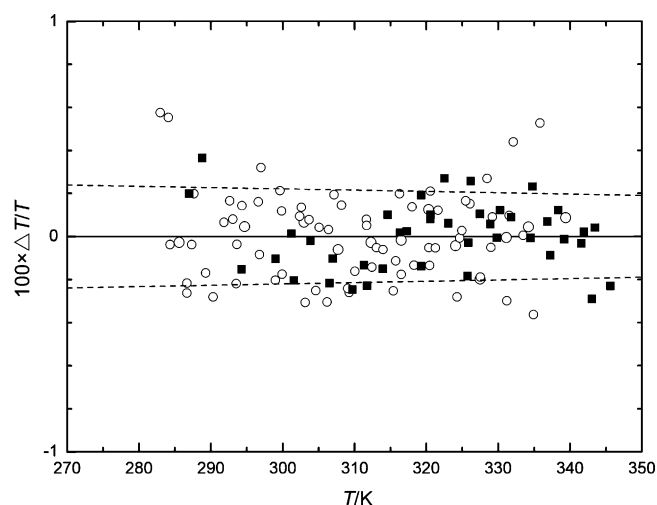

Figure 3. Relative differences $\Delta T/T = (T^{exp} - T^{cal})/T^{cal}$ of the experimental determined temperature T^{exp} from calculated temperature T^{cal} with the new E-NRTL model. ■, the results from NaOH + water systems; ○, the results from ethanol + water systems. The dashed lines imply the expanded uncertainties of our measurements, which is estimated to be 0.6 K.

systems and the multicomponent electrolyte systems studied in this paper.

According to the calculated results, when 0.084 solute-free mole fraction NaOH present in aqueous solution, the solubility is diminished from 0.0144 (in pure water) to 0.000086 at 280 K. However, 0.472 solute-free mole fraction ethanol is needed to give rise to the solubility decrease to 0.000738 at the same temperature. A conclusion can be drawn that a small amount of NaOH added into the solvent can decrease the solubility dramatically, while more ethanol is needed to reach the same result.

It can be seen from Figure 1 that the temperature dependence for ethanol mole fractions of 0.099 and 0.201 show anomalous trends compared with other systems. To study this behavior quantitatively, the relationship between temperature dependence of solubility and mole fraction x_3^0 or x_4^0 is expressed in Figure 4, in which κ^* is used to represent the temperature dependence of solubility. κ^* is the average slope of each curve in Figures 1 and 2. Because the relations between
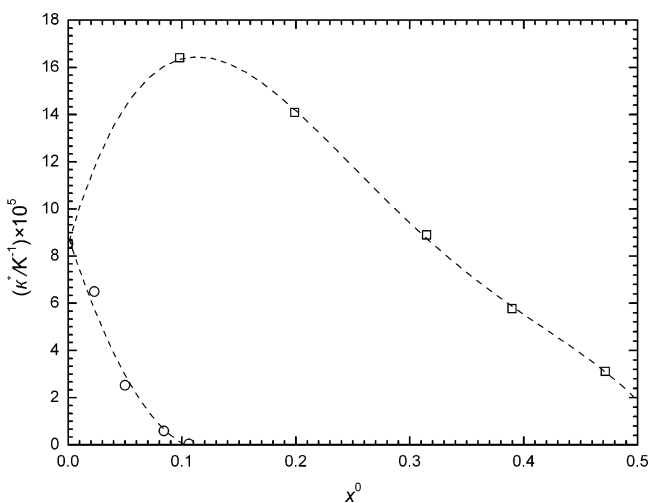
Figure 4. Plot of κ^* versus the solute-free mole fraction x^0. x^0 is: $\square$, x_3^0; $\bigcirc$, x_4^0. Dotted lines represent the trends of the points.

solubility and temperature in Figures 1 and 2 are slight curves, κ^* is calculated by solubility difference dividing temperature change in the present measurement range. As shown in Figure 4, κ^* is dramatically decreased with the increase of x_4^0. However, the increase of x_3^0 makes the value of κ^* first climb up and then decline. The maximum value of κ^* occurs at the point that x_3^0 is about 0.1. This behavior for ethanol gives reason to the anomaly in Figure 1 in which the average slopes for the curves of 0.099 and 0.201 are larger than that of other curves. In the systems containing ethanol, a high value of κ^* means more effective crystallization, by which more products can be obtained during a separation process in the industrial application.

CONCLUSIONS

Solubility data for two ternary aqueous systems disodium decanedioate + ethanol + water and disodium decanedioate + NaOH + water have been determined in a temperature range of (284 to 345) K. In all solvent systems, solubilities increased with temperature and evidently diminished with addition of either ethanol or NaOH, between which the effect of NaOH was more prominent. The temperature dependence of solubility kept decreasing while the content of NaOH increased. However, with the addition of ethanol, the influence of the temperature on solubility increased first and then decreased, and the maximum value occurred when the ethanol solute-free mole fraction was about 0.1. In that case, the crystal separation process could be more efficient.

Calculated solubility data of disodium decanedioate were generated using the new E-NRTL model with the model parameters gained from regression. The average root-mean-square deviation was 0.65 K for disodium decanedioate + ethanol + water and 0.45 K for disodium decanedioate + NaOH + water systems, while the values for each system ranged from (0.26 to 0.91) K.

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