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Research Article

Solid-Liquid Phase Equilibria of the Ternary System (NaCl + CH_3OH + H_2O) at 298.15, 308.15, 318.15 K, and 0.1 MPa

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The phase equilibrium of the ternary system (NaCl + CH_3OH + H_2O) at 298.15, 308.15, 318.15 K, and 0.1 MPa has been investigated by the isothermal dissolution equilibrium method. Solubilities and physicochemical properties including refractive index (n_D) and density (ρ) in the ternary system were determined experimentally. According to the experimental data, the phase diagrams and diagrams of physicochemical properties versus sodium chloride concentration in the solvents at 298.15, 308.15, and 318.15 K were plotted, respectively. The experimental results showed that the system did not cause stratification and the equilibrium solid phase was anhydrous sodium chloride. Neither double salt nor solid solution was found at the three temperatures. The physicochemical properties of the ternary system change regularly with the increase of sodium chloride concentration in the solution, and the solvation effect of CH_3OH on NaCl was significant. Moreover, the calculated values of NaCl solubility data based on the CNIBS/R-K equations agreed well with the experimental results, and the thermodynamic functions in the dissolution process of NaCl in the CH_3OH-H_2O binary system were also calculated for further investigation of NaCl dissolution process.

1. Introduction

A large number of mixture containing sodium chloride (NaCl), lithium chloride (LiCl), and organic pollution are obtained in association with the production of special plastics. For example, during the production of polyphenylene sulfide (PPS), a large amount of by-product salt slurry containing NaCl, LiCl, and a small amount of oligomers were generated [1]. The green recycling of above high-value by-products is of great significance in the sustainable development of the plastics, salt chemical, and chloralkali industries [2]. For separating the inorganic salts, such as NaCl, from the by-product salt slurry, the phase equilibria of the related system are priorities to investigate.

Compared with vast data on the solubility and physicochemical properties of NaCl in aqueous electrolyte systems, the solubility and physicochemical properties data of NaCl in organic solvents are quite limited. Yang et al. [3] published the ternary system (NaCl + C_2H_5OH + H_2O) equilibrium at 293.15 K. They found the equilibrium solid phase was anhydrous sodium chloride, and no double salt and solid solution formed, especially, ethanol had been found to have strong salting-out effect on NaCl.

In this work, the solubilities of NaCl in the ternary parameters of (NaCl + CH_3OH + H_2O) were studied with the isothermal dissolution method at 298.15, 308.15, and 318.15 K for the first time. The related physicochemical properties including density, refractive index, and salting-out rate were measured. Moreover, the solubilities of NaCl, as well as the thermodynamic functions in the dissolution process of NaCl in the CH_3OH-H_2O binary system were also calculated for further investigation.

2. Experimental

2.1. Apparatus and Reagents. The apparatus used for the isothermal equilibrium in this work was designed in our laboratory and is shown in Figure 1. The experiments were

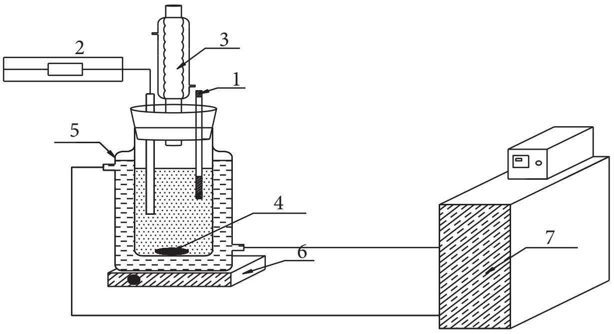

Figure 1: Apparatus used for the isothermal dissolution equilibrium: (1) sampler; (2) thermometer; (3) condenser; (4) magnetic stirring rod; (5) jacketed glass cell; (6) magnetic stirrer; and (7) temperature-controlled water bath.

carried out in a double-jacketed ground-glass cell with a volume of 100 cm^3. The temperatures of the cell were controlled by an external water bath circulator (K20-cc-NR, Huber, Germany) with an uncertainty of ±0.01 K [4]. To avoid the evaporation of methanol, a condensing unit was attached on the double-jacketed ground-glass cell.

The chemicals used in this study are shown in Table 1. It is worth mentioning that NaCl was recrystallized before used. Doubly deionized water (DDW) with pH 6.60 and conductivity less than 1×10^{-4} S·m^{-1} at room temperature (298.15 K) was used in this work.

2.2. Experimental Method. The phase equilibrium of the ternary system was studied with the isothermal dissolution method [5]. At the beginning, series of H_2O and methanol were exactly preweighted. The weight of H_2O was m_W, and the weight of methanol was m_M. Then, H_2O and methanol with known composition were added to the 100 mL jacketed glass cell and capped tightly. The jacketed glass cell was placed in the external water bath, whose temperature was set at a certain value (298.15 ± 0.01, 308.15 ± 0.01, and 318.15 ± 0.01 K). After stirring 1 h, a certain amount of NaCl was added into the jacketed glass cell and stirred for 36 h. A 0.5 cm^3 sample of the clarified supernatant was taken from the liquid phase of the jacketed glass cell with a pipette at regular intervals for chemical analysis. It was worthy saying that the magnetic stirrer was allowed to rest for 1 h in order to ensure the separation of the solid and liquid phase before sampling. If the compositions of the liquid phase in the bottle became constant, it indicated that the equilibrium was achieved.

2.3. Analytical Method. Briefly, the solubilities of sodium chloride in the binary solvent mixtures were measured by the gravimetric method using high precision balance (OHAUS, USA, precision of 0.1 mg) with standard uncertainty of 0.2 mg at 0.681 level of confidence [6]. The weight of the empty bottle was m_0, and the weight of the bottle with solution (supernatant that has achieved equilibrium) was m_1. The bottle with liquid samples was first dried in an oven at 388.15 K. After a fine grind of this NaCl solid, again the fine NaCl was dried at 388.15 K until the mass did not change. In addition, the dried NaCl solid was tested by infrared spectroscopy, and the result (Figure S1) showed the characteristic peaks of both of H_2O and CH_3OH were not observed. The result above indicated that the solid NaCl obtained did not trap or embed some of water and/or methanol molecules. At this time, the weight of the bottle was obtained and kept constant as m_2. All results were the average of 3 repeated tests. The content of each component in the equilibrium supernatant system is calculated by the following equations:

$$
\begin{aligned}
w_{NaCl} &= \frac{m_2 - m_0}{m_1 - m_0},\\
w_{H_2O} &= \frac{m_W (m_1 - m_2)}{(m_W + m_M)(m_1 - m_0)},\\
w_{CH_3OH} &= \frac{m_M (m_1 - m_2)}{(m_W + m_M)(m_1 - m_0)},
\end{aligned}
\tag{1}
$$

where w_{H_2O} and w_{CH_3OH} are the mass fractions of water and methanol in the equilibrium supernatant system, respectively. All experimental data containing m_0, m_1, m_2, m_w, and m_M are given in Table S1 in the supporting information.

The densities (ρ) were measured by the automatic oscillating U-tube densimeter (DMA 4500, Anton Paar, Austria, precision of 1.0×10^{-5} g·cm^{-3}) with standard uncertainty of 0.5 mg·cm^{-3} at 0.681 level of confidence [7]. The refractive indices (n_D) were measured by an Abbe refractometer (model WZS-1, Shanghai, precision ± 0.0001) with standard uncertainty of 0.001 at 0.681 level of confidence [8]. The solid phase minerals were identified by an X-ray diffractometer (MSAL XD-3, Beijing) and BX51 digital polarizing microscope (Olympus, Japan). All measurements of the above physicochemical properties were maintained in a supper thermostatic water bath that controlled at the desired temperature (298.15 ± 0.01, 308.15 ± 0.01, and 318.15 ± 0.01 K).

3. Results and Discussion

3.1. For the Ternary System NaCl + CH_3OH + H_2O. In order to verify the reliability of our experimental method, we compared the equilibrium solubility, refractive index, and density data with the reported binary system (NaCl + H_2O) at different temperatures, and the results are shown in Table 2. The confidence interval of NaCl solubility (mass fraction) in water was 26.43 ± 0.15, 26.67 ± 0.09, and 26.80 ± 0.11 at 298.15, 308.15, and 318.15 K, respectively. The results above proved our experimental method was reliable.

The solubility and physicochemical properties of the ternary system (NaCl + CH_3OH + H_2O) at 298.15, 308.15, and 318.15 K are shown in Table 3. On the basis of the experimental solubility data in Table 3, the equilibrium phase diagrams of the ternary system (NaCl + CH_3OH +

Table 1: Chemicals used in this study.

Chemical name	Source	Initial mass fraction purity	Purification method	Final mass fraction purity	Analysis method
NaCl	[a]A.R.	0.995	Recrystallization	0.998	Chemical analysis and ICP-OES[c]
CH_3OH	[b]A.R.	0.998	None	—	—

[a] A.R.: the Group of States Chemical Reagent Co., Ltd.; [b] A.R.: the Tianjin Fuyu Fine Chemical Co. Ltd.; [c] ICP-OES: inductively coupled plasma optical emission spectrometer.

Table 2: Solubilities, refractive index, and density of the binary system (NaCl + H_2O) at 298.15, 308.15, and 318.15 K and $p = 0.1$ MPa.[a]

T/K	Solubility, 100 w[b]	Refractive index, n_D	Density, ρ ($g\cdot cm^{-3}$)	Reference
298.15	26.30	1.3796	1.1978	[9]
	26.37	1.3810	1.1983	[10]
	26.45	—[c]	—	[11]
	26.47	1.3801	1.19755	This work
308.15	26.59			[11]
	26.66	1.3766	1.1936	[9]
	26.62	—	1.19350	[12]
	26.63	1.3789	1.19362	This work
318.15	26.69	1.3766	1.1889	[9]
	26.80	1.3776	1.18959	This work

[a] Standard uncertainties u are $u(T) = 0.1$ K and $u(p) = 0.005$ MPa [13]; [b] w, mass fraction, respectively. [c] Not detected.

H_2O) at 298.15, 308.15, and 318.15 K are shown in Figures 2–4.

Points A_1 in Figure 2, A_2 in Figure 3, and A_3 in Figure 4 represented the solubilities of NaCl in pure water in mass fraction (100 w) with 26.47, 26.63, and 26.80 at 298.15, 308.15, and 318.15 K, respectively. Similarly, Points B_1 in Figure 2, B_2 in Figure 3, and B_3 in Figure 4 represents the solubilities of NaCl in pure methanol in mass fraction (100w) with 1.36, 1.37, and 1.38 at 298.15, 308.15, and 318.15 K, respectively. There was no turning point and no double salt and solid solution in this system at different temperatures. The equilibrium solid phase was anhydrous sodium chloride. It can be seen from Figure 5 that the solubilities of NaCl increased with the increasing of the percentage of H_2O in the mixed solvent at three temperatures, i.e., the solubilities of NaCl decreased with the increase of the percentage of CH_3OH.

On the basis of the physicochemical property data (densities and refractive indices) in Table 3, the diagrams of physicochemical properties versus sodium chloride content in the ternary system at 298.15, 308.15, and 318.15 K were plotted in Figures 6(a) and (b). It was found that densities and refractive indices in this ternary system at three temperatures change regularly with the increasing of sodium chloride content. Generally, the densities and the refractive indices have the same varying trend. Both of them increased with the increasing of sodium chloride content at each temperature.

3.2. Salting-Out Rate.

According to the results above, the solubilities of NaCl decreased with the increase of the percentage of CH_3OH. It suggested that methanol had strong salting-out effect on NaCl. The salting-out effects of methanol on NaCl can be expressed by salting-out rate (SOR). SOR [14] is identified in the following equation:

$$\mathrm{SOR}\,(\%) = \frac{w_{\mathrm{NaCl-W}} - w_{\mathrm{NaCl-Mix}}}{w_{\mathrm{NaCl-W}}} \times 100\%, \tag{2}$$

where $w_{\mathrm{NaCl\text{-}W}}$ is the mass fraction of NaCl in saturated sodium chloride pure water solution and $w_{\mathrm{NaCl\text{-}Mix}}$ expresses the mass fraction of NaCl in the equilibrium supernatant system.

According to the data in Table 3, the values of SOR were calculated by the above equation in accordance with the solubilities of NaCl, and the curve of SOR versus the mass fraction of methanol in mixture solvent at 298.15, 308.15, and 318.15 K is shown in Figure 7. The SOR was increased gradually with the increasing of methanol content in mass fraction.

3.3. Correlation of the Solubility of NaCl and the Composition of Mixed Solvents.

Acree [15] proposed a CNIBS/R-K equation, which was shown in the following equation (3). This equation could study the correlation of the solubility of a solute and the composition of the mixed solvent at isothermal temperature:

$$\ln x_A = x_B^0 \ln\left(x_A\right)_B + x_C^0 \ln\left(x_A\right)_C + x_B^0 x_C^0 \sum_{i=0}^{N} S_i\left(x_B^0 - x_C^0\right)^i, \tag{3}$$

where x_{A} is the molar solubility of the solute in the mixed solvent; x_{B}^0 and x_{C}^0, respectively, represent the molar ratio of the solvents B and C in the mixed solvent in the absence of solute; $(x_{\mathrm{A}})_{\mathrm{B}}$ and $(x_{\mathrm{A}})_{\mathrm{C}}$ represent the saturated molar solubility of solute in the pure solvent B and C, respectively; N represents the number of the component; S_{i} is the parameter of the model.

Our system was the two-component mixed solvent system, and then N equals 2 and x_{C}^0 can be replaced by

Table 3: The experimental solubilities and the physicochemical properties in the ternary system ($NaCl + CH_3OH + H_2O$) at 298.15, 308.15, and 318.15 K, and $p = 0.1$ MPa.[a]

No.	Composition of liquid phase, $100\,w$[b]			Physicochemical properties		Equilibrium solid phase
	CH_3OH	H_2O	NaCl	Refractive index, n_D	Density, ρ (g/cm^3)	
				298.15 K		
1, A_1	0.00	73.53	26.47	1.3801	1.19755	NaCl
2	4.74	70.98	24.28	1.3773	1.16931	NaCl
3	8.00	69.13	22.87	1.3755	1.15040	NaCl
4	16.13	64.34	19.53	1.3717	1.10901	NaCl
5	24.84	58.85	16.31	1.3678	1.06641	NaCl
6	35.03	52.09	12.88	1.3623	1.02088	NaCl
7	44.53	45.35	10.12	1.3579	0.98190	NaCl
8	54.38	38.05	7.57	1.3531	0.94401	NaCl
9	65.97	28.98	5.05	1.3475	0.90274	NaCl
10	77.05	19.70	3.25	1.3426	0.86571	NaCl
11	87.86	10.07	2.07	1.3374	0.83531	NaCl
12, B_1	98.64	0.00	1.36	1.3301	0.79767	NaCl
				308.15 K		
1, A_2	0.00	73.37	26.63	1.3789	1.19362	NaCl
2	4.04	71.17	24.79	1.3762	1.16933	NaCl
3	7.80	68.99	23.21	1.3748	1.14854	NaCl
4	15.87	64.23	19.90	1.3701	1.10590	NaCl
5	25.19	58.31	16.50	1.3658	1.06084	NaCl
6	34.53	52.06	13.41	1.3608	1.01920	NaCl
7	44.73	44.89	10.37	1.3560	0.97650	NaCl
8	55.97	36.55	7.48	1.3500	0.93320	NaCl
9	66.13	28.60	5.27	1.3451	0.89573	NaCl
10	76.97	19.62	3.41	1.3399	0.85862	NaCl
11	87.66	10.19	2.15	1.3338	0.82366	NaCl
12, B_2	98.63	0.00	1.37	1.3270	0.78797	NaCl
				318.15 K		
1, A_3	0.00	73.20	26.80	1.3776	1.18959	NaCl
2	3.90	71.04	25.06	1.3752	1.16633	NaCl
3	7.69	68.79	23.51	1.3732	1.14571	NaCl
4	15.97	63.84	20.19	1.3689	1.10219	NaCl
5	24.84	58.19	16.97	1.3649	1.05944	NaCl
6	34.55	51.75	13.70	1.3594	1.01414	NaCl
7	44.55	44.71	10.74	1.3547	0.97288	NaCl
8	55.09	36.98	7.93	1.3481	0.92982	NaCl
9	65.92	28.59	5.49	1.3430	0.88964	NaCl
10	76.18	20.18	3.64	1.3373	0.85332	NaCl
11	87.64	10.18	2.18	1.3310	0.81509	NaCl
12, B_3	98.62	0.00	1.38	1.3244	0.77871	NaCl

[a] Standard uncertainties u are $u(T) = 0.1$ K and $u(p) = 0.005$ MPa; $u(w)$ for NaCl, CH_3OH, and H_2O are 0.0049, 0.0005, and 0.0005 in mass fraction, respectively; $u(x)$ for n_D and ρ are 0.001 and 0.5 mg·cm^{-3}, respectively; [b] w, mass fraction.

$(1 - x_B^0)$. x_A is the molar solubility of the NaCl in the mixed methanol + water system. x_B^0 and x_C^0 are referred to the molar fraction of methanol and water in the binary methanol + water system recalculated considering a NaCl-free supernatant, respectively. $(x_A)_B$ and $(x_A)_C$ represent the saturated molar solubility of NaCl in the pure methanol and water, respectively. In this way, Equation (3) can be changed to Equation (4):

$$\begin{aligned}\ln x_A - x_B^0 \ln (x_A)_B - \left(1 - x_B^0\right) \ln (x_A)_C \\ = x_B^0\left(1 - x_B^0\right)\left[S_0 + S_1\left(2x_B^0 - 1\right) + S_2\left(2x_B^0 - 1\right)^2\right].\end{aligned} \tag{4}$$

According to Equation (4), the calculated parameters and multiple correlation coefficient R^2 at different temperatures are presented in Table 4. All the R^2 values are distributed in the range of 0.9983–0.9986. The results inferred the calculated values agreed well with the experimental data, and this agreement showed that the parameter S_i obtained in this work were reliable and can be used to calculate any solubilities of NaCl in the mixed solvent ($CH_3OH + H_2O$) in the corresponding temperatures.

3.4. Calculated Thermodynamic Functions.

To exploit the valuable NaCl from organic solvents, the thermodynamic functions for the dissolution process of NaCl in the mixed solvent ($CH_3OH + H_2O$) are also essential. Hence, we calculated the relevant thermodynamic functions [16]

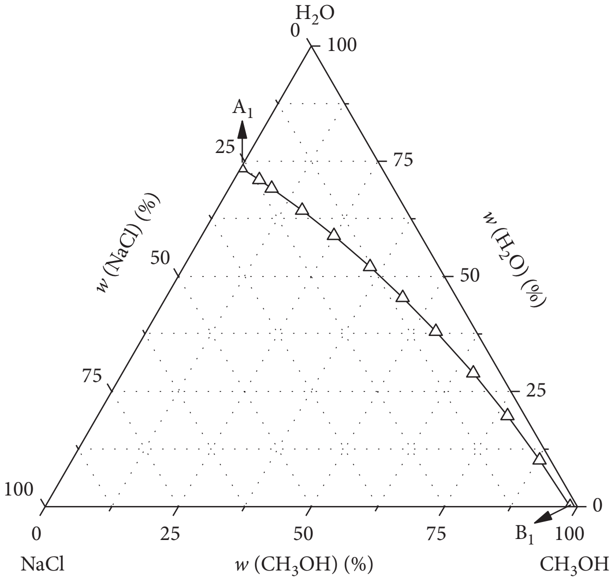

Figure 2: Phase diagram of the ternary system (NaCl + CH$_3$OH + H$_2$O) at 298.15 K.

during the dissolution process of NaCl in the mixed solvent, such as Gibbs free energy ($\Delta_{\text{sol}}G$), entropy ($\Delta_{\text{sol}}S$), and enthalpy ($\Delta_{\text{sol}}H$), and the following correlation equations were used [17]:

$$T_{\text{mean}} = \frac{n}{\sum_{i=1}^{n}(1/T)},$$

$$x_A = \frac{(m_A/M_A)}{(m_A/M_A + m_W/M_W + m_M/M_M)},$$

$$\Delta_{\text{sol}}H = -R\left(\frac{\partial \ln x_A}{\partial(1/T - 1/T_{\text{mean}})}\right),$$

$$\Delta_{\text{sol}}G = -RT_{\text{mean}} \times \text{intercept}, \tag{5}$$

$$\Delta_{\text{sol}}S = \frac{\Delta_{\text{sol}}H - \Delta_{\text{sol}}G}{T_{\text{mean}}},$$

$$\zeta_H = \frac{|\Delta_{\text{sol}}H|}{|\Delta_{\text{sol}}H| + |T_{\text{mean}} \times \Delta_{\text{sol}}S|},$$

$$\zeta_{\text{TS}} = \frac{|T_{\text{mean}} \times \Delta_{\text{sol}}H|}{|\Delta_{\text{sol}}H| + |T_{\text{mean}} \times \Delta_{\text{sol}}S|},$$

where m_A, m_W, and m_M are the mass (g) of sodium chloride, water, and methanol from Table 3, respectively; M_A, M_W, and M_M are the relative molar mass of sodium chloride, water, and methanol, respectively; ζ_H and ζ_{TS} present the enthalpy compensation and entropy compensation of NaCl dissolved in the CH$_3$OH-H$_2$O binary solvents, respectively; n is the number of studied temperature points. Intercept is obtained from the linear fit between $\ln x_A$ and $(1/T - 1/T_{\text{mean}})$. The temperature was selected in the range of 298.15–318.15 K; as a result, the value of T_{mean} was 307.93 K.

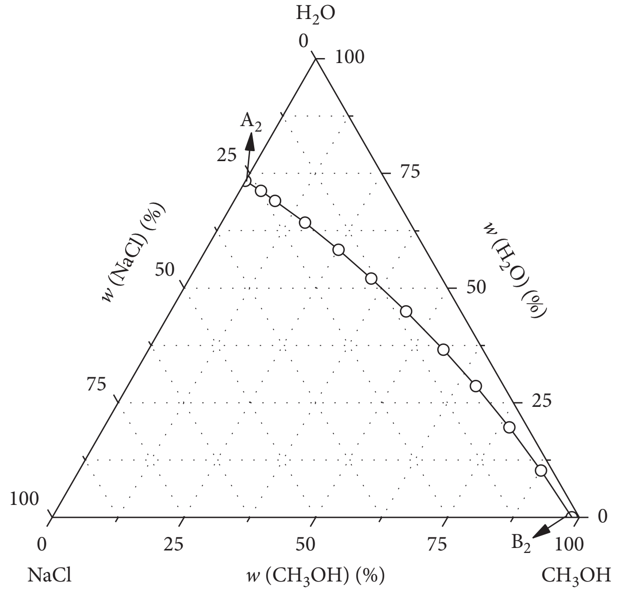

Figure 3: Phase diagram of the ternary system (NaCl + CH$_3$OH + H$_2$O) at 308.15 K.

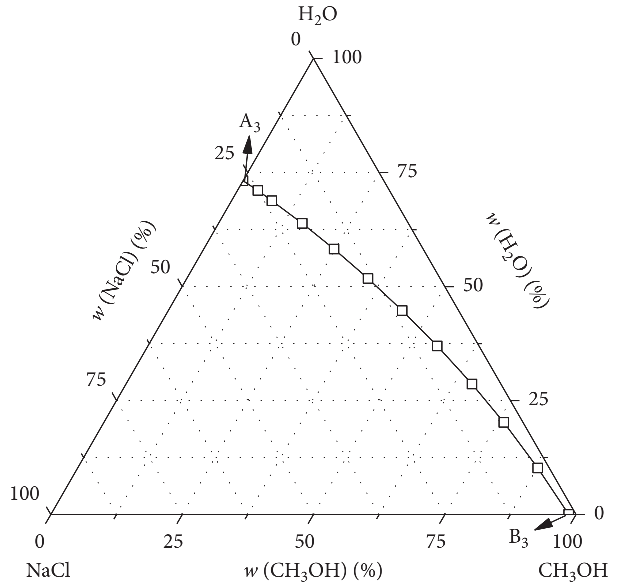

Figure 4: Phase diagram of the ternary system (NaCl + CH$_3$OH + H$_2$O) at 318.15 K.

The calculated thermodynamic functions of NaCl dissolved in the CH$_3$OH-H$_2$O binary system are presented in Table 5. No matter how the mixture ratios of CH$_3$OH-H$_2$O binary system changed, the values of $\Delta_{\text{sol}}H$ were all greater than zero, suggesting that the dissolution of NaCl in CH$_3$OH-H$_2$O binary system was an endothermal process. In addition, the maximum ζ_H was 0.400, which was much lower than the minimum ζ_{TS} (0.600). This result inferred that the contribution of ζ_{TS} to $\Delta_{\text{sol}}G$ is more significant than that of ζ_H, and enthalpy has limited effect on $\Delta_{\text{sol}}G$ while entropy is the major contributor.

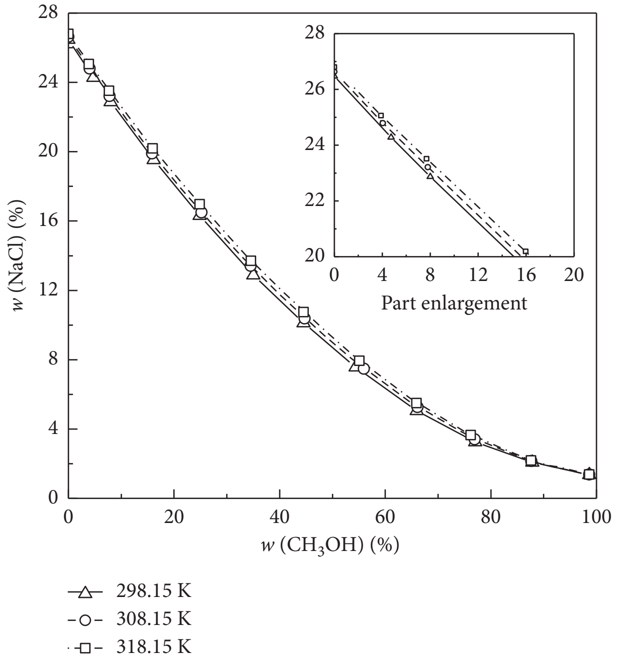

Figure 5: Comparison of the sodium chloride content of the ternary system(NaCl + CH_3OH + H_2O) at 298.15, 308.15, and 318.15 K; (△), experimental point at 298.15 K; (—), solubility curve at 298.15 K; (○), experimental point at 308.15 K; (- -), solubility curve at 308.15 K; (□), experimental point at 318.15 K; (-··-), solubility curve at 318.15 K.

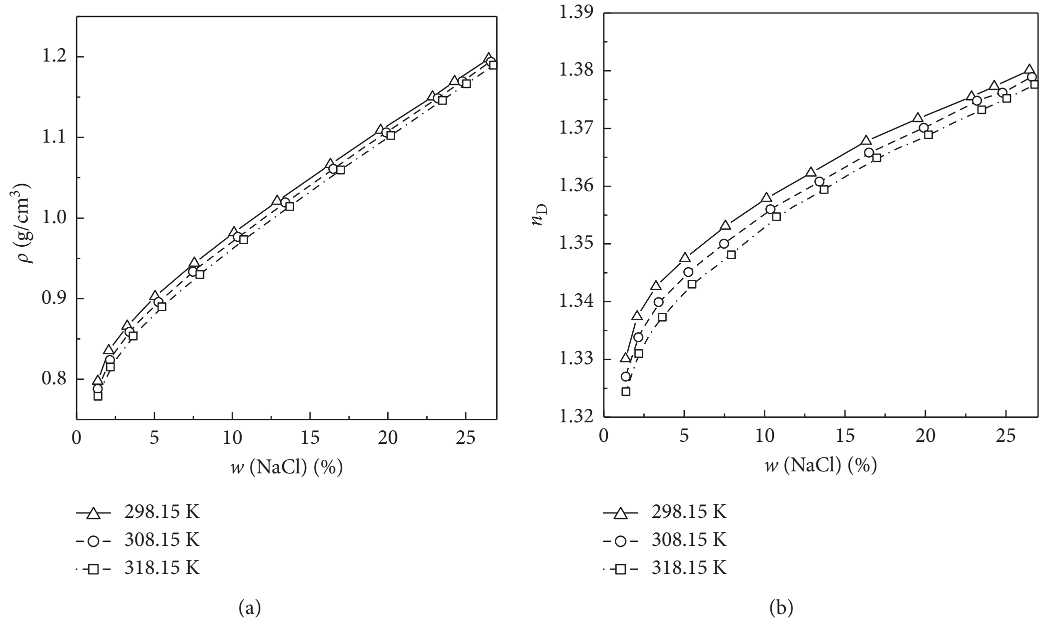

Figure 6: Comparison of the physicochemical properties versus sodium chloride content for the ternary system (NaCl + CH_3OH + H_2O) at 298.15, 308.15, and 318.15 K; (△), experimental point at 298.15 K; (○), experimental point at 308.15 K; (□), experimental point at 318.15 K: (a) density versus NaCl composition; (b) refractive index versus NaCl composition.

4. Conclusion

The solubilities and physicochemical properties including refractive index and density of the ternary mixture solvent system (NaCl + CH_3OH + H_2O) at 298.15, 308.15, and 318.15 K were investigated using the isothermal dissolution equilibrium method. Based on the experimental data, the equilibrium phase diagrams and diagrams of physicochemical properties versus composition were plotted. It was found that there was no turning point and no double salt and solid solution, and the equilibrium solid phase is anhydrous sodium chloride. The physicochemical properties of the ternary system at three temperatures show regular change with the increase of sodium chloride concentration in

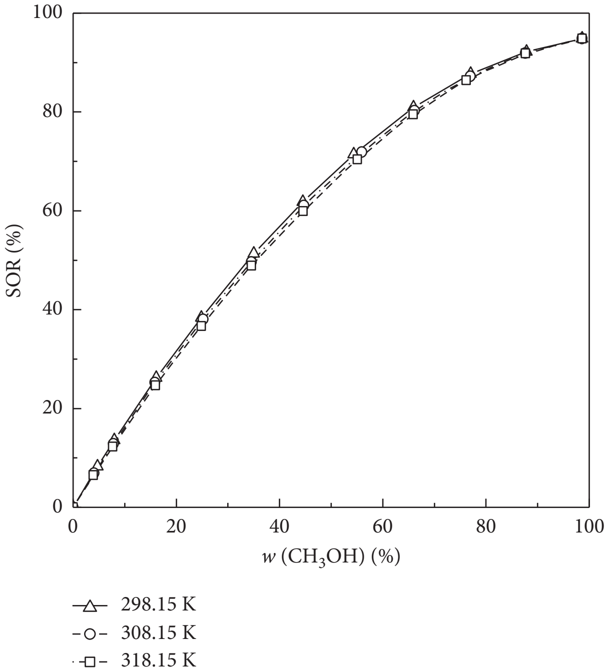

Figure 7: Salting-out rate (SOR) versus methanol content for the ternary system (NaCl + CH_3OH + H_2O) at 298.15, 308.15, and 318.15 K; (△), experimental point at 298.15 K; (○), experimental point at 308.15 K; (□), experimental point at 318.15 K.

Table 4: Parameters of CNIBS/R-K model for solubility in solvent mixtures.

T/K	S_0	S_1	S_2	R^2
298.15	0.065	−0.810	−0.464	0.9984
308.15	0.236	−0.751	−0.505	0.9986
318.15	0.363	−0.785	−0.585	0.9983

Table 5: Calculated various thermodynamic functions of NaCl dissolved in different mixture ratios of CH_3OH-H_2O binary system.

$x^0_{CH_3OH}$	$\Delta_{sol}H$/(kJ·mol^{-1})	$\Delta_{sol}G$/(kJ·mol^{-1})	$\Delta_{sol}S$/(J·mol^{-1}·K^{-1})	ζ_H	ζ_{TS}
0.00	0.599	5.879	−17.148	0.102	0.898
0.10	1.310	6.423	−16.605	0.204	0.796
0.20	2.107	6.998	−15.885	0.301	0.699
0.30	2.819	7.625	−15.607	0.370	0.630
0.40	3.320	8.311	−16.207	0.400	0.600
0.50	3.531	9.052	−17.928	0.390	0.610
0.60	3.416	9.829	−20.827	0.348	0.653
0.70	2.984	10.613	−24.773	0.281	0.719
0.80	2.293	11.360	−29.444	0.202	0.798
0.90	1.443	12.014	−34.331	0.120	0.880
1.00	0.579	12.508	−38.737	0.046	0.954

solution. Methanol had strong salting-out effect on NaCl, and the SOR was increased gradually with the increasing of methanol content in the mixture solvent ternary system. The calculated values of NaCl solubility data based on the CNIBS/R-K equations agreed well with the experimental results. In addition, the thermodynamic functions in the dissolution process of NaCl in the CH_3OH-H_2O binary system were also calculated for further investigation of NaCl dissolution process. Generally, studies on phase equilibria and phase diagrams of the ternary systems (NaCl + CH_3OH + H_2O) at 298.15, 308.15, 318.15 K, and 0.1 MPa could provide the based thermodynamic data to exploit the valuable inorganic salts from organic solvents.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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Supplementary Materials

The experimental data including the FT-IR spectra of NaCl are presented in Figure S1, and the weight data containing m_0, m_1, m_2, m_W, and m_M are presented in Table S1. (*Supplementary Materials*)

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