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Research Article

Phase Equilibria and Phase Diagrams for the Aqueous Ternary System Containing Sodium, Chloride, and Metaborate Ions at 288.15 and 308.15 K and 0.1 MPa

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Salt-water phase diagram of the ternary system ($NaCl + NaBO_2 + H_2O$) is significant in separate boron resources from brine. Solubilities and physicochemical properties bifurcated to density and refractive index for the ternary system at 288.15 and 308.15 K and 0.1 MPa were investigated experimentally by means of isothermal dissolution. According to the experimental data, the phase diagrams for this system at two temperatures were constructed and their physicochemical properties were drawn. There both contain two invariant points, three univariant isotherm dissolution curves, and three crystallization regions corresponding to sodium metaborate tetrahydrate ($NaBO_2{\cdot}4H_2O$, Smt), double salt teepleite ($NaCl{\cdot}NaBO_2{\cdot}2H_2O$, Te), and halite (NaCl, Ha) for ($NaCl + NaBO_2 + H_2O$). At the invariant points of this ternary system, the coexisting solid phases are both for (Smt + Te) and (Te + Ha), respectively. It was found that the double salt of teepleite was incongruent at 288.15 K and then transformed into congruent at 308.15 K. Comparing the phase diagrams at 288.15 and 308.15 K, it reveals that the area of the crystallization region of $NaBO_2{\cdot}4H_2O$ decreased with the increase of temperature obviously, while the area of $NaCl{\cdot}NaBO_2{\cdot}2H_2O$ increased sharply and that of NaCl increased lightly. The refractive indices and densities of the system at two temperatures are similarly increased firstly and then decreased with the increase of NaCl concentration in the solution and attain extreme points at both invariant points. Besides, the calculated values of densities and refractive indices utilizing the empirical equations are in accordance with the experimental data at 288.15 and 308.15 K.

1. Introduction

Boron and its compounds can be used as inorganic functional materials in many high-tech fields because of their special properties such as light weight, flame retardant, high hardness, high strength, heat resistance, and abrasion resistance [1, 2]. China is rich in boron resources; the total reserves ranked the fourth in the world. With the depletion of solid mineral resources, salt lake mineral resources become a strategic resource of our country. In addition, China is famous for its abundant mineral salt resources, and there are more than 1,000 salt lakes distributed in the west of China. Among them, salt lakes on the Qinghai-Tibet Plateau are rich in high concentrations of lithium and boron ions [3]. The boron resource will be further enriched in the brine after evaporating and extracting sodium and potassium salts in brines. A study on the phase equilibrium of the boron-containing salt-water system at different temperatures provides a theoretical basis for the separation and recovery of boron resources.

Studies on the phase equilibrium of salt-water systems have made undeniable contributions to effectively exploit salt lake resources. Therefore, more and more scholars were focused on phase equilibria and phase diagrams of those systems of salt lake brines and underground brines in recent years. Most of the salt lakes in the Qaidam Basin in Qinghai belong to the complex salt-water system of Li^+, Na^+, K^+, Mg^{2+}//Cl^-, SO_4^{2-}, and borate–H_2O [4], such as the ternary system ($LiCl + LiBO_2 + H_2O$) and ($Li_2SO_4 + LiBO_2 + H_2O$) at 323.15 K [5], ($Li_2SO_4 + K_2SO_4 + H_2O$) at 288.15 K [6], the quaternary system (Li^+, Mg^{2+}//Cl^-, SO_4^{2-} −H_2O) [7], and (Na^+, K^+, Li^+//$B_4O_7^{2-}$ − H_2O) at 273.15 K [8], as well as the quinary system (Li^+, Na^+, K^+, Mg^{2+}/SO_4^{2-} −H_2O) at 298.15 K [9] and (Na^+, K^+, Li^+//SO_4^{2-}, $B_4O_7^{2-}$ − H_2O) at 273.15 K [10]. Although the ternary system ($NaCl + NaBO_2 + H_2O$) at 308.15 K has been reported in 1929 [11], the solubility data are uncompleted except for the two boundary and the invariant point datum in the literature. In this work, the isothermal solubilities and the corresponding physicochemical properties of the aqueous solution containing densities and refractive indices for the ternary system ($NaCl + NaBO_2 + H_2O$) at 288.15 and 308.15 K were demonstrated for the first time.

2. Experimental

2.1. Reagents. The reagents containing auxiliary reagents used through this experiment were all of analytical grade provided by Sinopharm and are listed in Table 1. In this work, the fresh CO_2-free double deionized water with the conductivity for about 8×10^{-5} S·m^{-1} as well as pH 6.60 at 298.15 K was used to artificially synthesize the complexes and analysis chemical.

2.2. Apparatus. An ultrapure system (AWL-0502-U, Aquapro International Company LLC) was applied to provide the double deionized water. A high precision electronic analytical balance (AR224CN, OHAUS Corporation) of 220 g capacity was available for sampling with a precision of ±0.02 mg. A magnetic stirring thermostat (XHC-500-8A, Beijing Fortunejoy Science Technology Co., Ltd.) for temperature control was employed with a precision of 0.01 K. For determining the densities (ρ), an Anton Paar digital vibrating-tube densimeter (DMA 4500, Anton Paar Co., Ltd.) that can automatically control the sample cell temperature within 0.01 K was applied with a precision of ±0.01 mg·cm^{-3}. In order to measure the refractive index (n_D), a high precision refractometer (Abbemat-550, Anton Paar Co., Ltd.) with a precision of ±0.000002 was used. An X-ray powder diffractometer (XRD, MSAL XD-3, Beijing Purk Instrument Co., Ltd., China) was employed to identify the solid phases using the LTK-450 accessory that makes the transition temperature ranging from 80 K to 723 K available.

2.3. Experimental Method. The experimental phase equilibrium was investigated by isothermal dissolution. In brief, a sequence of complexes according to the binary system invariant points were added into the 300 cm^3 glass bottles, weighed at once, and sealed tightly; after that, the bottles were placed at (288.15 ± 0.01 or 308.15 ± 0.01) K in the thermostatic water bath with stirring at 120 rpm to speed up the equilibria of those compounds. About 5.0 cm^3 clear supernatant was taken out by a special sampler from the liquid part of each bottle for refractive index determination. It is worth mentioning that the stirrer was demanded to temporarily close for 2 h before sampling for the purpose of ensuring the solid-liquid separation. It manifested that the equilibrium was achieved once the composition of the liquid phase was constant. The enough upper clear liquids were removed and preserved in order to analyze chemical composition and measure the physicochemical properties including densities and refractive indices. The equilibrium solid phase was identified by XRD.

Table 1: Chemicals used in this study.

Chemicals	Purity in mass fraction	Source	Analysis method
NaCl	0.995	Sinopharm Chemical Reagent Co., Ltd	Volumetric method for Cl^-
$NaBO_2$	0.990	Sinopharm Chemical Reagent Co., Ltd	Gravimetric method for BO_2^-

2.4. Analytical Method. In the presence of a mixed indicator of phenylazoformic acid, 2-phenylhydrazide, and bromophenol blue, the concentration of Cl^- was determined by titration with a standard solution of mercury nitrate and the uncertainty of the method was ±0.003 in mass fraction [12]. And, the concentration of the BO_2^- was determined by means of gravimetric analysis of the sodium hydroxide standard solution in the existence conditions of mixture indicators of methyl red plus phenolphthalein and the superabundant mannitol with the uncertainty of ±0.0005 in mass fraction [12]. The Na^+ concentration was calculated using the anion-cation balance and evaluated by an inductively coupled plasma optical emission spectrometer (ICP-OES, Prodigy, Leman Co., USA) with a standard uncertainty of 0.005 (0.68 level of confidence) in mass fraction.

Moreover, the densities (ρ) were measured after checking with the air and fresh CO_2-free double deionized water by a DMA 4500 densimeter with an uncertainty of ±0.15 mg·cm^{-3} and the refractive indices (n_D) were measured by a Abbemat-550 refractometer with an uncertainty of ±0.00003. And, the thermostat (K20-cc-NR, Huber, Germany) was employed to control all the measurements at the expected temperature with ±0.01 K.

3. Results and Discussion

A comparison on the solubilities for the binary subsystems ($NaCl + H_2O$) and ($NaBO_2 + H_2O$) at 288.15 and 308.15 K in the experiments and literatures is shown in Table 2. These results indicate that the experimental process and analytical results are authentic and dependable.

3.1. Phase Diagrams and Physicochemical Properties of the Ternary System ($NaCl + NaBO_2 + H_2O$). The solubilities and physicochemical properties of the aqueous solution containing density and refractive index of the ternary system ($NaCl + NaBO_2 + H_2O$) at $T = 288.15$ and 308.15 K are presented in Table 3. In addition, the phase diagrams at 288.15 K with the solid line and 308.15 K with the dash line based on the above data are plotted in Figure 1. The composition of the solubilities of NaCl and $NaBO_2$ is expressed in mass fraction.

Table 2: Solubilities of the binary subsystems ($NaCl + H_2O$) and ($NaClO_3 + H_2O$) at $T = 288.15$ and 308.15 K and $p = 0.1$ MPa.[a]

System	$T = 288.15$ K		$T = 308.15$ K	
	100 w^b	Reference	100 w^b	Reference
$NaCl + H_2O$	26.35	[13]	26.66	[14]
	26.35	[15]	26.63	[16]
	26.37	This work	26.69	This work
$NaBO_2 + H_2O$	18.38	[17]	26.30	[18]
	18.75	This work	26.25	This work

[a]Standard uncertainties u are $u(T) = 0.01$ K, $u(p) = 0.005$ MPa. $u(x)$ for NaCl and $NaBO_2$ is 0.0037 and 0.00028 in mass fraction, respectively. w^b = mass fraction.

Table 3: Solubilities, densities, and refractive indices of the ternary system ($NaCl + NaBO_2 + H_2O$) at $T = 288.15$ and 308.15 K and 0.1 MPa.[a]

Number	Composition of liquid phase (100 w^b)			Physicochemical properties						Equilibrium solid phase[c]
				n_D			ρ (g·cm^{-3})			
	NaCl	$NaBO_2$	H_2O	Exp.	Cal.	Error (%)	Exp.	Cal.	Error (%)	
$T = 288.15$ K										
1, A	0.00	18.75	81.25	1.381165	1.380946	0.02	1.23257	1.233083	−0.04	Smt
2	2.19	16.84	80.97	1.381333	1.379982	0.10	1.23445	1.225448	0.73	Smt
3	4.48	16.31	79.21	1.382766	1.382761	0.00	1.23691	1.237727	−0.07	Smt
4	6.23	15.29	78.48	1.383543	1.383297	0.02	1.23841	1.238616	−0.02	Smt
5	8.37	14.96	76.67	1.386051	1.386332	−0.02	1.25052	1.252547	−0.16	Smt
6	9.80	14.31	75.89	1.386602	1.387243	−0.05	1.25164	1.255849	−0.34	Smt
7	11.73	13.51	74.76	1.388240	1.388682	−0.03	1.25933	1.261443	−0.17	Smt
8, E_1	15.78	12.81	71.41	1.393743	1.394239	−0.04	1.28321	1.287257	−0.32	Smt + Te
9	16.45	11.91	71.64	1.392724	1.393132	−0.03	1.28220	1.280336	0.15	Te
10	19.06	9.30	71.64	1.390829	1.391105	−0.02	1.26806	1.266143	0.15	Te
11, E_2	23.69	5.29	71.02	1.389759	1.389125	0.05	1.25242	1.250018	0.19	Te + Ha
12	24.67	4.15	71.18	1.387503	1.387945	−0.03	1.23924	1.242539	−0.27	Ha
13	24.90	3.07	72.03	1.385798	1.385571	0.02	1.23060	1.229566	0.08	Ha
14, B	26.37	0	73.63	1.381019	1.380294	0.05	1.20238	1.200096	0.19	Ha
$T = 308.15$ K										
1, A′	0	26.25	73.75	1.393555	1.392905	0.05	1.30969	1.309373	0.02	Smt
Ref.[d]	0	26.30	73.7	—[e]	—	—	—	—	—	Smt
2	3.05	24.73	72.22	1.394734	1.394722	0.00	1.31580	1.315954	−0.01	Smt
3	4.48	24.61	70.91	1.395337	1.397000	−0.12	1.32017	1.327283	−0.54	Smt
4	5.26	23.59	71.15	1.396353	1.395947	0.03	1.32141	1.320209	0.09	Smt
5	6.24	23.53	70.23	1.397057	1.397563	−0.04	1.32325	1.328297	−0.38	Smt
6	8.20	21.92	69.88	1.398424	1.397209	0.09	1.32899	1.323756	0.39	Smt
7, E_1'	9.71	21.90	68.39	1.400152	1.399867	0.02	1.34505	1.337236	0.58	Smt + Te
Ref.[d]	9.40	22.40	68.20	—	—	—	—	—	—	Smt + Te
8	15.11	15.03	69.86	1.392665	1.393046	−0.03	1.29003	1.291311	−0.10	Te
9	19.52	9.94	70.53	1.388626	1.388750	−0.01	1.26231	1.261882	0.03	Te
10, E_2'	23.92	5.86	70.22	1.386720	1.386832	−0.01	1.24574	1.246036	−0.02	Te + Ha
Ref.[d]	23.80	5.90	70.30	—	—	—	—	—	—	Te + Ha
11	24.47	4.62	70.91	1.384967	1.384853	0.01	1.23394	1.234590	−0.05	Ha
12	25.21	3.16	71.63	1.382760	1.382689	0.01	1.22125	1.222015	−0.06	Ha
13	26.07	1.48	72.45	1.380175	1.380219	0.00	1.20791	1.207774	0.01	Ha
14, B′	26.69	0.00	73.31	1.377920	1.377802	0.01	1.19398	1.194236	−0.02	Ha
Ref.[d]	26.60	0	73.40	—	—	—	—	—	—	Ha

[a]The standard uncertainties u are $u(T) = 0.01$ K, $u(p) = 0.005$ MPa, $u(\rho) = 0.15$ mg·cm^{-3}, and u (n_D) = 0.00003; $u(x)$ for NaCl and $NaBO_2$ is 0.0037 and 0.00028 in mass fraction, respectively. w^b, in mass fraction. [c]Smt, $NaBO_2·4H_2O$; Te, $NaCl·NaBO_2·2H_2O$; Ha, NaCl. [d]For reference [11]. [e]Without data in Reference [11].

In Figure 1, for the ternary system ($NaCl + NaBO_2 + H_2O$) at 288.15 and 308.15 K, (a) there are three crystallization regions corresponding to sodium metaborate tetrahydrate ($NaBO_2·4H_2O$, Smt), double salt teepleite ($NaCl·NaBO_2·2H_2O$, Te), and halite (NaCl, Ha), $NaCl·NaBO_2·2H_2O$ (Te) and NaCl (Ha) for ($NaCl + NaBO_2 + H_2O$) system at 288.15 and 308.15 K. Points A and A′ are the solubilities of the single salt of $NaBO_2$ in mass fraction (100 w) of 18.75 at 288.15 K and 26.25 at 308.15 K. Similarly, points B and B′ are the solubilities of the single salt of NaCl in mass fraction (100 w) corresponding to 26.37 at 288.15 K and 26.69 at 308.15 K. According to the phase diagrams of this ternary system at 288.15 and 308.15 K, the system

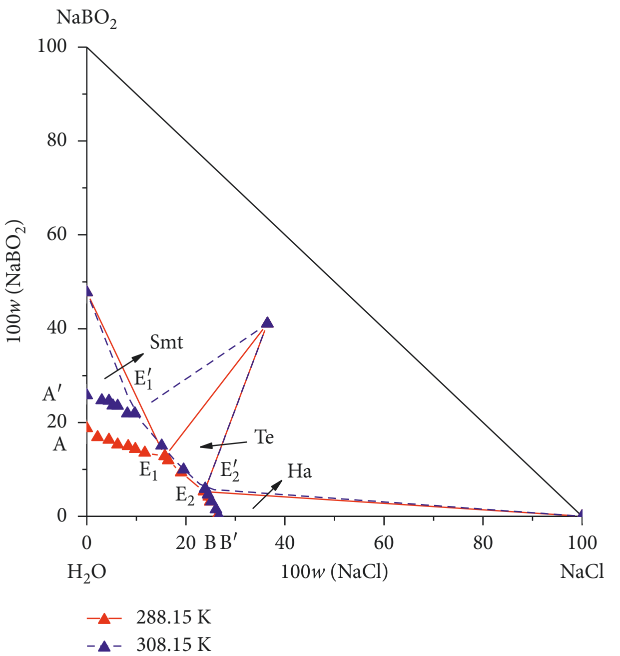

Figure 1: Comparison of the phase diagram of the ternary system $(NaCl + NaBO_2 + H_2O)$ between 288.15 and 308.15 K at $p = 0.1$ MPa. (▲) Experimental point; (—) solubility curve at 288.15 K; (- -) solubility curve at 308.15 K; Smt, $NaBO_2{\cdot}4H_2O$; Te, $NaCl{\cdot}NaBO_2{\cdot}2H_2O$; Ha, NaCl.

at two temperatures pertains to hydrate type I. The area of crystallization region of $NaBO_2{\cdot}4H_2O$ decreased obviously with the temperature increase and that of $NaCl{\cdot}NaBO_2{\cdot}2H_2O$ changed oppositely, while the area of crystallization region of NaCl increased lightly. The solubility of these three compounds is positively correlated with temperature shown in Figure 1. It is worth noting that the compositions of two invariant points (Smt + Te) and (Te + Ha) as well as two edge points at 308.15 K from literature [11] have been listed in Table 3 compared with the data obtained in this work. (b) There are two invariant coexisted solid phase points E_1 (288.15 K) and E_1' (308.15 K) with $(NaBO_2{\cdot}4H_2O + NaCl{\cdot}NaBO_2{\cdot}2H_2O$, i.e., Smt + Te) as well as E_2 (288.15 K) and E_2' (308.15 K) with $(NaCl + NaCl{\cdot}NaBO_2{\cdot}2H_2O$, i.e., Ha + Te). The X-ray diffraction patterns of the invariant points E_1/E_1' and E_2/E_2' are shown in Figure 2. (c) Both of these phase diagrams include three isothermal curves AE_1, E_1E_2, and BE_2 at 288.15 K and AE_1', $E_1'E_2'$ and BE_2' at 308.15 K corresponding to minerals of $NaBO_2{\cdot}4H_2O$, $NaCl{\cdot}NaBO_2{\cdot}2H_2O$ and NaCl, respectively. (d) Because there is no intersection with $E_1'E_2'$ when connecting the composition point of pure water and the location point of double salt teepleite (Te) at 288.15 K, double salt teepleite at 288.15 K is an incongruent double salt. However, this double salt is transformed into a congruent double salt at 308.15 K.

On the basis of the densities and refractive indices in Table 3, diagram of the physicochemical properties with sodium chloride composition in the solution as the abscissa at 288.15 and 308.15 K is constructed in Figure 3. As can be seen from the figure, the densities and refractive indices in

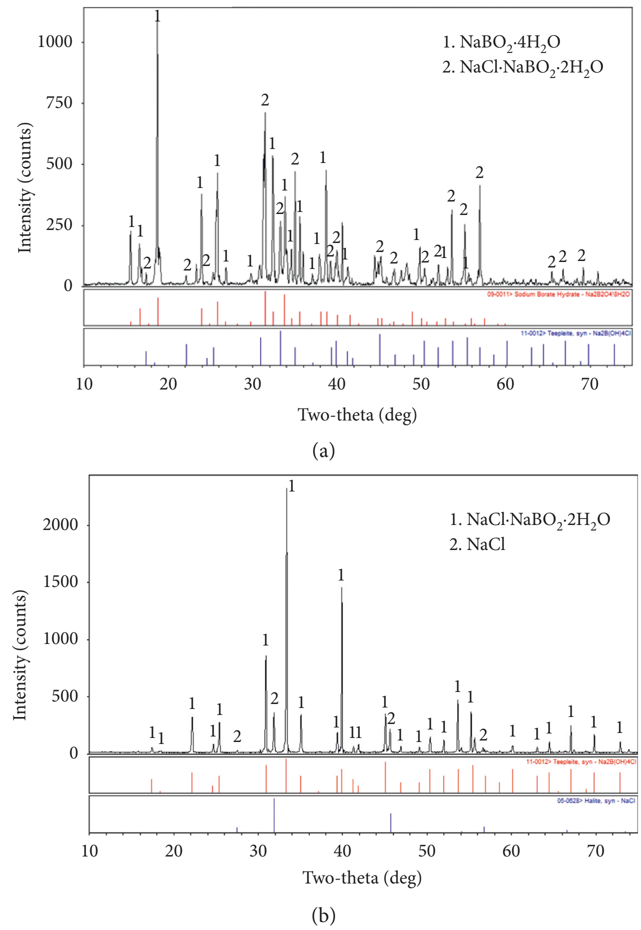

Figure 2: X-ray diffraction photograph of the invariant points for the ternary system $(NaCl + NaBO_2 + H_2O)$. (a) Invariant points E_1 and E_1' with $(NaBO_2{\cdot}4H_2O + NaCl{\cdot}NaBO_2{\cdot}2H_2O)$. (b) Invariant points E_2 and E_2' with $(NaCl + NaCl{\cdot}NaBO_2{\cdot}2H_2O)$.

the ternary system at two temperatures uniformly increased primarily from single salt points A or A′ of $NaBO_2$ to reach the first cosaturated invariant points E_1 or E_1' of (Smt + Te) and then dropped significantly with the increase of sodium chloride concentration to reach another cosaturated invariant points E_2 or E_2' of (Te + Ha), and further sharply decreased to another single salt points B or B′ of NaCl. These physicochemical properties reached extreme values at two-salt cosaturated invariant points.

4. Empirical Equations for Density and Refractive Index

Empirical equations for predicting density and refractive index of the salt-water system were applied to have correlation with the experimental results [18]:

$$\ln\left(\frac{d}{d_0}\right) = \sum A_i \times w_i, \qquad \ln\left(\frac{D}{D_0}\right) = \sum B_i \times w_i, \tag{1}$$

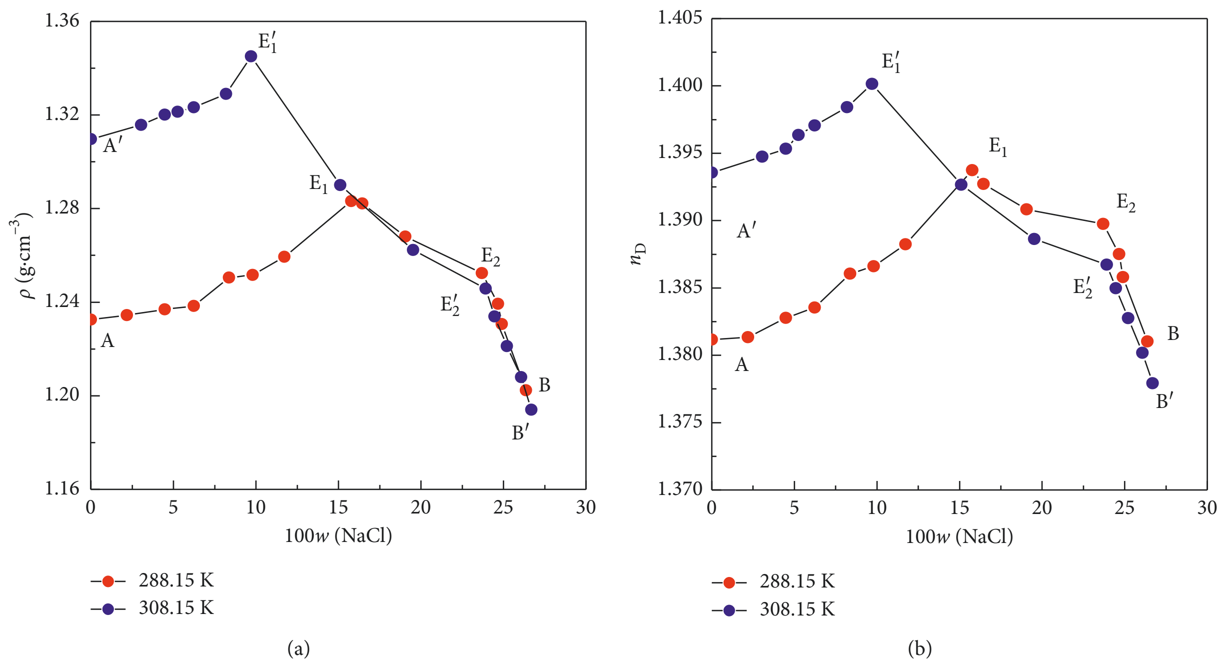

Figure 3: Diagram of physicochemical property versus sodium chloride content for the ternary system ($NaCl + NaBO_2 + H_2O$) at 288.15 and 308.15 K. (•) Experimental points at 288.15 and 308.15 K; (a) density versus NaCl composition; (b) refractive index versus NaCl composition.

where d_0 and D_0 refer to the density values and refractive indices of the pure water at the same temperature, respectively. The d_0 values are 0.999099 $g \cdot cm^{-3}$ at 288.15 K and 0.994028 $g \cdot cm^{-3}$ at 308.15 K, respectively [19]. The D_0 values are 1.33339 and 1.33131 at 288.15 and 308.15 K, respectively [19]. In these equations, d and D stand for the densities and refractive indices of the aqueous solution at the same given temperature. w_i is the mass fraction of component i in the equilibrium aqueous solutions of the system.

For density calculation, the calculated empirical constants A_i of NaCl and $NaBO_2$ are 0.00695118 and 0.0112224 at 288.15 K and 0.00687510 and 0.0104967 at 308.15 K, respectively. And, for refractive index calculation, constant B_i of NaCl and $NaBO_2$ are 0.00131103 and 0.00186903 at 288.15 K and 0.00128610 and 0.00172297 at 308.15 K, respectively. The experimental values and calculated results are all shown in Table 3 for comparison, and the relative deviations between the experimental and calculated values are within 0.73%.

5. Conclusions

The solubilities and physicochemical properties of aqueous solution including density and refractive index of the ternary system ($NaCl + NaBO_2 + H_2O$) at 288.15 and 308.15 K were measured by means of the isothermal dissolution equilibrium method. According to the obtained experimental data, the phase diagrams and the diagram of the physicochemical properties with the sodium chloride concentration as the abscissa at 288.15 and 308.15 K were constructed. It can be seen that the area of the crystallization regions of $NaBO_2$ decreased obviously, while the area of $NaCl \cdot NaBO_2 \cdot 2H_2O$ increased sharply and that of NaCl increased lightly with temperature increase via comparing the phase diagrams of this ternary system at the two temperatures. The densities and refractive indices of the ternary system ($NaCl + NaBO_2 + H_2O$) at 288.15 and 308.15 K similarly increased firstly and then decreased with the increase of NaCl concentration in the solution and reached the extreme values at the invariant points. The calculated values of densities and refractive indices using the empirical equations agree well with the experimental results at both temperatures.

Data Availability

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

Additional Points

Solubilities of the system ($NaCl + NaBO_2 + H_2O$) were determined for the first time. Congruent and incongruent double salts of teepleite are found in this system. Densities and refractive indices of the ternary system were revealed. Calculated density and refractive index fit well with experimental data.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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