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Solid–liquid phase equilibria in the ternary systems $H_2O+ZnCl_2+NaCl$ at temperatures of 298, 313 and 333 K

SEVILAY DEMIRCI[1], VEDAT ADIGUZEL[1]* and OMER SAHIN[2]

[1]*Department of Chemical Engineering, Kafkas University, Kars 36100, Turkey and*
[2]*Department of Chemical Engineering, Siirt University, Siirt 56100, Turkey*

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Abstract: In this study, an economic separation method is suggested with the use of phase equilibria in order to ensure the recycling of $ZnCl_2$, the industrial waste amount of which is very high, and to prevent it from an environmental pollutant. Sodium chloride–zinc chloride–water systems were examined by the isothermal method at temperatures of 298, 313 and 333 K. The analyses of the liquid and solid phases were used to determine the composition of the solid phase using the Schreinemaker's graphic method. The solid–liquid phase equilibrium and viscosity data belonging to all the ternary systems were identified and the solubility and viscosity changes with temperature were compared. The viscosity values were inversely proportional to the temperature as the amount of $ZnCl_2$ in the solution increased. NaCl, 2NaCl $ZnCl_2$ $n$$H_2O$ (n: 2, 0), $ZnCl_2$ salts were observed at 298, 313 and 333 K in the solid phases that are in equilibrium with the liquid phase at the invariant point.

Keywords: crystallization region; invariant point; Schreinmakers method; solubility; viscosity.

INTRODUCTION

The solid-liquid phase equilibrium method is widely used, which includes increasing the yield of valuable chemicals and products in the salt industry. Moreover, this method makes the waste less harmful for the environment by increasing the rate of recycling and waste disposal. Thirdly, it enables a researcher to economically obtain valuable chemicals in fewer reaction steps in a lab setting.[1–3]

$ZnCl_2$ is widely applied in industry, such as for the production of paper, the metal industry, textiles, the synthesis of important chemicals, agriculture, water refinement, cosmetics and the drug industry.[4,5] However, industrial $ZnCl_2$ waste brings about economic and environmental problems especially in the steel ind-

* Corresponding author. E-mail: vedatnursen@gmail.com
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ustry. In this industry, one of the most effective and oldest methods is the hot-dip galvanizing method in order to increase the corrosion resistance of steel.[5] In the galvanizing process, the metal is cleaned from undesirable substances, such as rust and impurities in the acid bath. The amount of iron and zinc increases together in the acid reaction in the acid bath. The content of this impure solution is reported as 5–200 g L^{-1} zinc, 60–150 g L^{-1} iron and 10–80 g L^{-1} HCl.[6–9]

The issue of waste disposal is gaining significance in industry day by day together with the increase in environmental awareness and toughness of legislation. In this study, a separation process is suggested for $ZnCl_2$ with the change in solid–liquid phase equilibrium at different temperatures by benefiting from the ternary system H_2O+$ZnCl_2$+NaCl.

EXPERIMENTAL

Apparatus and reagents

NaCl (≥99.5 %) and $ZnCl_2$ (98 %) were purchased from Merck and used without further purification. The solution condition was provided for by pure water of pH 6.6 and conductivity <10^{-4} S m^{-1}.

Viscosity analyses were realized using a Brookfield DV2T viscometer (accuracy ±1.0 % of the full-scale range).

Titration measurements were conducted with Hirschmann Solarus automatic burette (accuracy ±0.2 %). A stabile experimental temperature was provided by a Polyscience branded cooler and mixer water bath (accuracy ±0.05 K).

Experimental methods

Phase equilibria were determined by the isothermal solubility saturation method.[10-13] Sealed insulated tubes containing saturated $ZnCl_2$ solutions disolved in 40mL pure water were placed on the disc.[13] NaCl salts were added to these tubes subsequently in increasing amounts until the invariant point was reached. The disc was mixed for one day in a heater and cooler circulator which is stable at the desired temperature (298, 313 and 333 K). The tubes were kept in the circulator until the phase separation was clearly seen. The samples were taken from the solid and liquid phases of all the tubes for ion analysis and viscosity determination. The solid phase compositions at all temperatures were detected according to the Schreinmaker's wet residue method.[10-14]

The same processes were repeated by adding $ZnCl_2$ salts to the saturated solutions of NaCl in increasing amounts until the invariant point was reached. All the tests were repeated three times for reliability of the test results. Results were expressed as ± standard deviation value.

The tables and graphics were formed after conducting the mathematical calculations necessary for interperation all the data and results.

Analytical methods

Cl^- and Zn^{2+} concentrations were determined respectively with Mohr and EDTA according to complexometric titration methods.[15] Na^+ amounts were calculated according to the total ion balance.

RESULTS AND DISCUSSION

Solubility data of H_2O–$ZnCl_2$–NaCl system at 298 K

The solubility and viscosity values of NaCl–H_2O and $ZnCl_2$–H_2O binary systems were detected respectively as 26.42 % NaCl and 80.13 % $ZnCl_2$, 1.55 mPa s and 156.84 mPa s at 298 K. The solid phases belonging to these compositions were found as NaCl and $ZnCl_2$.

NaCl, $ZnCl_2$ and H_2O compositions and viscosity in the invariant points of H_2O+$ZnCl_2$+NaCl system at 298 K were, respectively, F: 17.99, 39.70 and 42.31 % and 5.78 mPa s; E_1: 4.68, 67.68 and 27.64 % and 31.92 mPa s. The solid phase of invariant point consisted of NaCl, $ZnCl_2$ and 2NaCl $ZnCl_2$ $2H_2O$ salts.

The solubility and viscosity data belonging to this ternary system are given in Table I and Figs. 1 and 2.

TABLE I. SLE data for the ternary H_2O–$ZnCl_2$–NaCl system at 298 K and 0.102 MPa; standard uncertainties u are $u(\rho)$ = 0.001 g cm^{-3}, $u(T)$ = 0.05 K, $u_r(P)$ = 5 % and $u(w)$ = 0.01; NC: NaCl, DS: 2NaCl $ZnCl_2$ $2H_2O$, ZC: $ZnCl_2$

No.	Content, mass. %				Content, mol.%		η / mPa s	Equilibrium salt
	Liquid phase		Solid phase					
	NaCl	$ZnCl_2$	NaCl	$ZnCl_2$	NaCl	$ZnCl_2$		
1	26.42	0.00	95.08	0.00	100	0.00	1.55	NC
2	24.50	14.81	89.04	2.60	79.50	20.50	1.79	NC
3	22.61	25.70	95.04	1.28	67.24	32.76	3.07	NC
4	20.36	33.95	93.05	3.45	58.29	41.71	4.18	NC
5	19.20	36.94	82.37	8.04	54.75	45.25	4.85	NC
6F	17.99	39.70	34.05	44.50	51.34	48.66	5.78	NC+DS
7	14.15	47.03	34.00	47.30	41.22	58.78	8.12	DS
8	10.77	53.40	31.44	48.37	32.00	68.00	11.53	DS
9	8.04	59.30	30.08	50.70	23.95	76.05	17.43	DS
10	6.75	62.20	29.46	52.35	20.17	79.83	21.10	DS
11E_1	4.68	67.68	17.48	66.36	13.88	86.12	31.92	DS+ZC
12	3.46	70.71	2.87	76.07	10.24	89.76	45.18	ZC
13	2.56	73.22	2.17	76.97	7.57	92.43	78.55	ZC
14	0.00	80.13	0.00	86.18	0.00	100	156.84	ZC

There are three crystallization areas in Fig. 1: 1) CAF corresponding to the crystallization area of NaCl; 2) FPE_1 corresponding to the crystallization area of 2NaCl $ZnCl_2$ $2H_2O$; 3) E_1BD corresponding to crystallization area of $ZnCl_2$. Point A and B are the invariant points of the binary systems NaCl−H_2O and $ZnCl_2$−H_2O. Point E_1 and F represent an invariant of the ternary systems. The curves AF, FE_1 and E_1B represent the saturation curves. X and Y points correspond to the percent amount of $ZnCl_2$ and NaCl in the double salt, respectively.

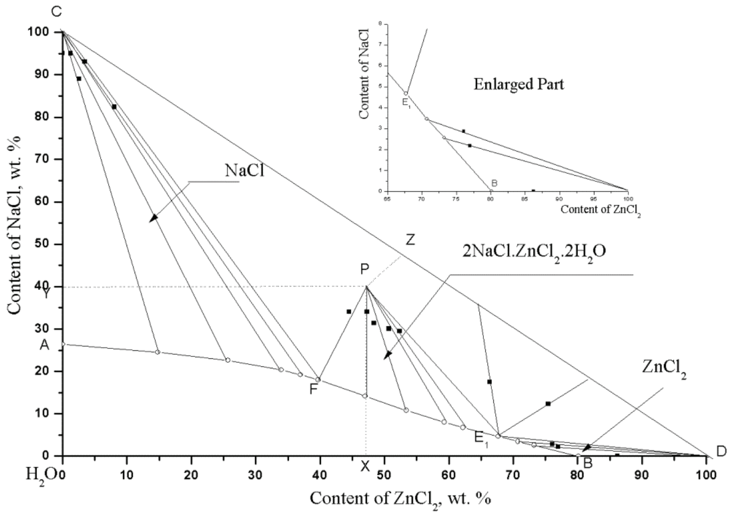

Fig. 1. SLE diagram for the ternary H_2O–$ZnCl_2$–NaCl system at 298 K and 0.102 MPa.

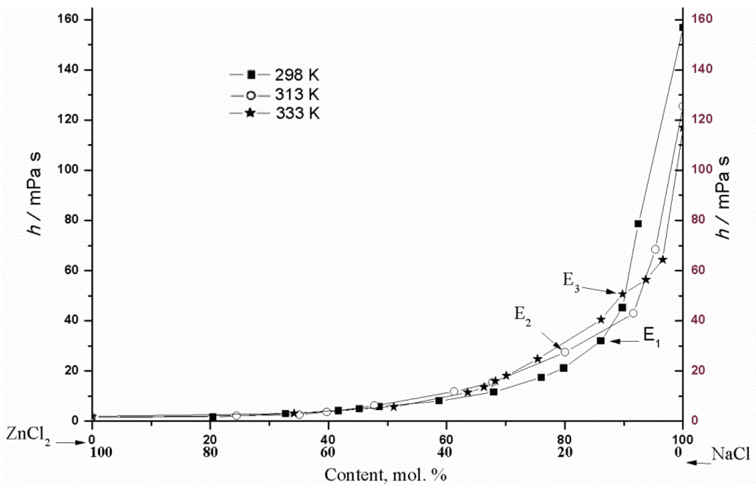

Fig. 2. Viscosity diagram for the ternary H_2O–$ZnCl_2$–NaCl system at 298–333 K and 0.102 MPa.

Solubility data of H_2O–$ZnCl_2$–NaCl system at 313 K

The solubility and viscosity values of NaCl–H_2O and $ZnCl_2$–H_2O binary systems were determined respectively as 27.25 % NaCl and 81.10 % $ZnCl_2$, 1.62 and 117 mPa s at 313 K. The solid phases belonging to these compositions were found as NaCl and $ZnCl_2$.

NaCl, $ZnCl_2$ and H_2O compositions and viscosity in the invariant point of H_2O+$ZnCl_2$+NaCl system at 313 K were, respectively, F: 19.16, 40.89 and 39.95

% and 6.20 mPa s; E_2: 6.86, 64.15 and 28.99 % and 27.42 mPa s. The solid phase of invariant point consists of NaCl, $ZnCl_2$ and 2NaCl $ZnCl_2$ salts.

The solubility and viscosity data belonging to this ternary system are given in Table II and Figs. 2 and 3.

TABLE II. SLE data for the ternary H_2O–$ZnCl_2$–NaCl system at 313 K and 0.102 MPa; standard uncertainties u are $u(\rho)$ = 0.001 g/cm^3, $u(T)$ = 0.05K, $u_r(P)$ = 5% and $u(w)$ = 0.01w NC: NaCl, DS2: 2NaCl.$ZnCl_2$, ZC: $ZnCl_2$

No.	Content, mass. %				Content, mol. %		η / mPa s	Equilibrium salt
	Liquid phase		Solid phase					
	NaCl	$ZnCl_2$	NaCl	$ZnCl_2$	NaCl	$ZnCl_2$		
1	27.25	0.00	96.08	0.00	100	0.00	1.62	NC
2	22.92	17.33	87.01	2.80	75.52	24.48	2.14	NC
3	21.78	27.46	80.74	6.80	64.91	35.09	2.60	NC
4	20.97	32.27	91.32	3.60	60.24	39.76	3.64	NC
5F	19.16	40.89	38.90	50.15	52.21	47.79	6.20	NC+DS2
6	13.5	49.88	35.62	52.51	38.69	61.31	11.81	DS2
7	11.26	55.29	32.00	52.72	32.20	67.80	15.50	DS2
$8E_2$	6.86	64.15	29.57	60.00	19.96	80.04	27.42	DS2+ZC
9	2.78	70.05	2.90	76.60	8.38	91.62	43.00	ZC
10	1.52	72.7	2.04	77.00	4.65	95.35	68.40	ZC
11	0.00	81.1	0.00	83.12	0.00	100	125.5	ZC

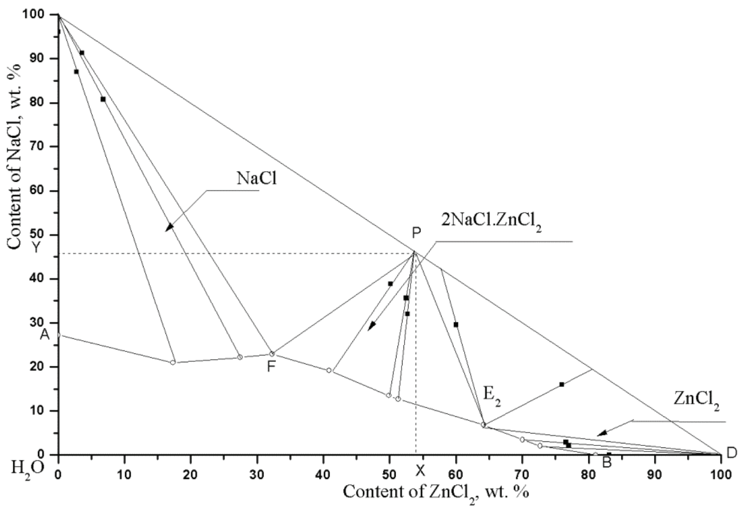

Fig. 3. SLE diagram for the ternary H_2O–$ZnCl_2$–NaCl system at 313 K and 0.102 MPa.

There are three crystallization areas in Fig. 3: 1) CAF corresponding to the crystallization area of NaCl; 2) FPE_2 corresponding to the crystallization area of 2NaCl·$ZnCl_2$; 3) E_2BD corresponding to crystallization area of $ZnCl_2$. Point A and B are the invariant points of the binary systems NaCl−H_2O and $ZnCl_2$−H_2O.

The point E_2 and F represent an invariant of the ternary systems. The curves AF, FE_2 and E_2B represent the saturation curves.

X and *Y* points correspond to the percent amount of $ZnCl_2$ and NaCl in thr double salt, respectively.

Solubility data of the H_2O–$ZnCl_2$–NaCl system at 333 K

When the temperature was brought to 333 K, it was observed that NaCl whose solubility value is 27.24 % in the NaCl–H_2O system decreases to 18.99 (F), 3.57 % (E_3) and the solubility of $ZnCl_2$ whose solubility is 83.2 % in $ZnCl_2$––H_2O system decreases to 46.22 (F), 73.03 % (E_3). It is also determined that the viscosity values change respectively from 1.92 mPa s to 5.76 (F), 50.67 mPa s (E_3) and from 125.5 mPa.s to 5.76 (F), 50.67 mPa s (E_3) when the invariant point is reached. The solid phase of the invariant point consists of NaCl, $ZnCl_2$ and $2NaCl·ZnCl_2$ salts.

The solubility and viscosity data belonging to this ternary system are given in Table III and Figs. 2 and 4.

TABLE III. SLE data for the ternary H_2O–$ZnCl_2$–NaCl system at 333 K and 0.102 MPa; standard uncertainties *u* are $u(\rho)$ = 0.001 g/cm^3, $u(T)$ = 0.05K, $u_r(P)$ = 5% and $u(w)$ = 0.01; NC: NaCl, DS2: $2NaCl.ZnCl_2$, ZC: $ZnCl_2$

No.	Content, mass. %				Content, mol. %		η / mPa s	Equilibrium salt
	Liquid phase		Solid phase					
	NaCl	$ZnCl_2$	NaCl	$ZnCl_2$	NaCl	$ZnCl_2$		
1	27.24	0.00	89.22	0.00	100	0.00	1.92	NC
2	23.00	27.83	88.63	3.78	65.84	34.16	3.11	NC
3F	18.99	46.22	31.66	49.38	48.93	51.07	5.76	NC+DS2
4	14.16	57.61	29.66	55.49	36.43	63.57	11.42	DS2
5	13.01	59.80	32.84	56.03	33.66	66.33	13.60	DS2
6	12.33	61.80	29.19	57.27	31.75	68.25	16.08	DS2
7	11.54	63.03	28.29	58.23	29.92	70.08	18.12	DS2
8	9.42	67.38	30.06	59.98	24.58	75.42	24.78	DS2
9	4.99	72.24	28.75	61.47	13.86	86.14	40.50	DS2
10E_3	3.57	73.03	19.28	70.59	10.22	89.78	50.67	DS2+ZC
11	2.17	75.50	1.87	82.12	6.28	93.72	56.40	ZC
12	1.19	78.30	0.82	81.11	3.41	96.59	64.33	ZC
13	0.00	83.20	0.00	88.89	0.00	100	117	ZC

There are three crystallization areas in Fig. 4: 1) CAF corresponding to the crystallization area of NaCl, 2) FPE_3 corresponding to the crystallization area of $2NaCl.ZnCl_2$, 3) E_3BD corresponding to the crystallization area of $ZnCl_2$. Point A and B are the invariant points of the binary systems NaCl−H_2O and $ZnCl_2$−H_2O. Point E_3 and F represent invariants of the ternary systems. The curves AF, FE_3 and E_3B represent the saturation curves.

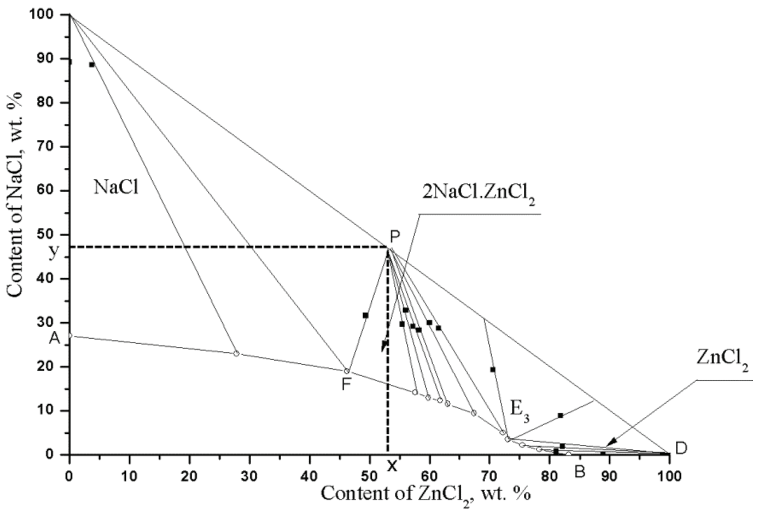

Fig. 4. SLE diagram for the ternary H_2O–$ZnCl_2$–NaCl system at 333 K and 0.102 MPa.

The points *X* and *Y* correspond to the percent amount of $ZnCl_2$ and NaCl in double salt, respectively.

Comparison of H_2O–$ZnCl_2$–NaCl systems between 273 and 373 K

The composition change belonging to six temperatures in total were compared also by using the data between the temperatures 273 and 373 K, belonging to the ternary system NaCl–$ZnCl_2$–H_2O from the literature.[12,16–18]

When the binary systems of NaCl–H_2O were examined between 273 and 373 K, the mass percentage composition change of the solubility of NaCl shows a low value of 2 %. However, it could be seen that mass percentage composition change of temperature and solubility was a noteworthy value of 19.32 % in the binary system of $ZnCl_2$.

In addition, it was observed that the solubility of NaCl was between 3.57––10.21 % and the solubility of $ZnCl_2$ was between 60.37–77.25 % when the invariant point data of the ternary systems were examined (Table IV, Fig. 5).

The solubility of NaCl and $ZnCl_2$ increased with increasing temperature. All the ternary systems contained the double salt. The structure of the double salt was of the form $2NaCl\cdot ZnCl_2\cdot 2H_2O$ at low temperatures (273 and 298 K). Nevertheless, it turned into a double salt $2NaCl\cdot ZnCl_2$ above 313 K (313–373 K) as the temperature increased. Obviously, the hydrated double salt turned into an anhydrous double salt with increasing temperature.

Comparing the present study with data from Zhang *et al.*[18] at 298 K, it was observed that the solubility of the solid phase compositions and double salts were compatible with each other.

TABLE IV. SLE data for the ternary H_2O–$ZnCl_2$–NaCl systems at 273–333 K

T / K	Invariant point, mass %		Solid phase	Reference
	NaCl	$ZnCl_2$		
273	26.25	0.00	NaCl	Adiguzel *et al.*[12]
	19.83	32.27	NaCl+2NaCl $ZnCl_2$ $2H_2O$	
	5.24	60.37	$ZnCl_2$·$2H_2O$+2NaCl.$ZnCl_2$·$2H_2O$	
	0.00	66.60	$ZnCl_2$·$2H_2O$	
298	26.71	0.00	NaCl	Zhang *et al.*[18]
	25.22	36.23	NaCl+2NaCl·$ZnCl_2$·$2H_2O$	
	6.24	77.25	$ZnCl_2$+2NaCl·$ZnCl_2$·$2H_2O$	
	0.00	80.77	$ZnCl_2$	
298	26.42	0.00	NaCl	In this study
	17.99	39.70	NaCl+2NaCl·$ZnCl_2$·$2H_2O$	
	4.68	67.68	$ZnCl_2$+2NaCl·$ZnCl_2$·$2H_2O$	
	0.00	80.13	$ZnCl_2$	
313	27.25	0.00	NaCl	In this study
	19.16	40.89	NaCl+2NaCl·$ZnCl_2$	
	6.86	64.15	$ZnCl_2$+2NaCl·$ZnCl_2$	
	0.00	81.10	$ZnCl_2$	
323	27.22	0.00	NaCl	Zhang *et al.*[17]
	25.08	40.17	NaCl+2NaCl·$ZnCl_2$	
	8.55	76.09	$ZnCl_2$+2NaCl·$ZnCl_2$	
	0.00	82.25	$ZnCl_2$	
333	27.24	0.00	NaCl	In this study
	18.99	46.22	NaCl+2NaCl·$ZnCl_2$	
	3.57	73.03	$ZnCl_2$+2NaCl·$ZnCl_2$	
	0.00	83.20	$ZnCl_2$	
373	28.25	0.00	NaCl	Zhang *et al.*[16]
	24.90	46.07	NaCl+2NaCl·$ZnCl_2$	
	10.21	72.97	$ZnCl_2$+2NaCl·$ZnCl_2$	
	0.00	85.92	$ZnCl_2$	

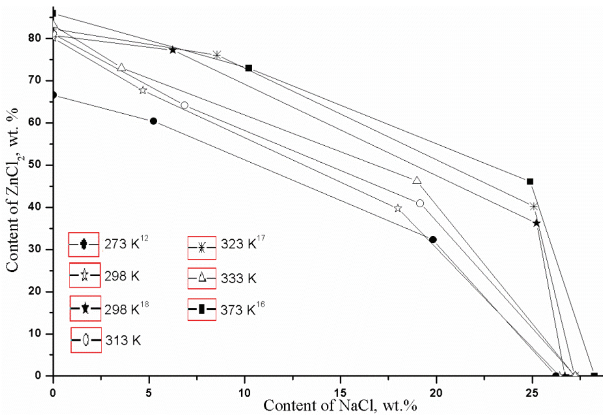

Fig. 5. Comparison between the SLE diagrams for the ternary H_2O–$ZnCl_2$–NaCl system at 273–373 K.

CONCLUSIONS

It was stated that the solubility of $ZnCl_2$ decreases from 66.6 to 60.37 % and the solubility of NaCl decreases sharply from 26.25 to 5.24 % to the invariant point as shown in the ternary system of NaCl–$ZnCl_2$–H_2O at 273 K, as conducted by Adiguzel *et al.*[12]

Zhang *et al.* implied that the solubility of $ZnCl_2$ decreases from 80.77 % to 77.25 % to the invariant point and the solubility of NaCl decreases from 26.71 to 6.24 %. These results show a sharper drop compared to that of $ZnCl_2$ in the ternary system at 298 K.[18]

It was observed in the ternary system at 298 K that the solubility of $ZnCl_2$ decreases from 80.13 to 67.68 % to the invariant point. On the other hand, the solubility of NaCl decreases significantly from 26.42 to 4.68 % compared to that of $ZnCl_2$.

It was concluded in the ternary system at 313 K that the solubility of $ZnCl_2$ decreases from 81.1 to 64.15 % to the invariant point and the solubility of NaCl decreases from 27.25 to 6.86 % and show a similar change with $ZnCl_2$.

The solubility of $ZnCl_2$ at 333 K changed from 83.2 to 73.03 % to the invariant point. The solubility of NaCl decreased 27.04 to 3.57 %. The solubility change of NaCl is greater at this temperature.

It was reported that the solubility of $ZnCl_2$ decreases from 82.25 to 76.09 % and the solubility of NaCl decreases from 27.22 to 8.55 % according to the ternary system of NaCl–$ZnCl_2$–H_2O at 323 K as studied by Zhang *et al.*[17]

It was found that the solubility of $ZnCl_2$ decreases from 85.92 to 72.97 % and the solubility of NaCl decreases from 28.25 to 10.21 % in the study regarding the ternary system of NaCl–$ZnCl_2$–H_2O at 373 K, which was conducted by Zhang *et al.*[16]

When the solubility change data of the ternary system of H_2O+$ZnCl_2$+NaCl between 273–373 K temperature was compared, it could be clearly seen that $ZnCl_2$ had a salting-out effect on NaCl. In addition, it was seen that the viscosity values are inversely proportional to the temperature as the amount of $ZnCl_2$ in the solution increased when the viscosity values of the ternary systems are compared in this study.

In conclusion, this study and literature data suggest that the process for the separation of $ZnCl_2$ by adding NaCl in the calculated amount to the solution and/or by decreasing the temperature of the solution of NaCl and $ZnCl_2$ occur at temperatures between 273 and 373 K. Therefore, an economic separation method could be developed by the method of phase equilibrium using NaCl, which is cheap and easily accessible, in order to provide or the recycling of $ZnCl_2$, the industrial waste amount of which is very high and to prevent it from posing a threat for the environment.

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И З В О Д

РАВНОТЕЖА ФАЗА ЧВРСТО–ТЕЧНО ТЕРНЕРНОГ СИСТЕМА $H_2O+ZnCl_2+NaCl$ НА ТЕМПЕРАТУРАМА ОД 298, 313 И 333 К

SEVILAY DEMIRCI[1], VEDAT ADIGUZEL[1] и OMER SAHIN[2]

[1]*Department of Chemical Engineering, Kafkas University, Kars 36100, Turkey* и [2]*Department of Chemical Engineering, Siirt University, Siirt 56100, Turkey*

У овом раду је предложена сепарациона метода заснована на фазној равнотежи, којом се постиже економски оправдана рециклажа $ZnCl_2$, присутног у значајним количинама у индустријском отпаду, а у циљу спречавања загађења животне средине. Растворљивост систему натријум-хлорид + цинк-хлорид + вода је испитивана у изотермским условима на температурама 298, 313 и 333 K. Schreinemakers графичка метода је коришћена за анализу течне и чврсте фазе и за одређивање састава испитиваног система. У раду су дати подаци за равнотежу чврсто–течно, као и подаци за вискозност и растворљивост тернарног система. Такође испитиван је утицај температуре на вискозност и растворљивост. Вредности вискозности су биле обрнуто пропорционалне температури, како се количина $ZnCl_2$ у раствору повећавала.

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