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ФАЗОВЫЕ РАВНОВЕСИЯ И КОНВЕРСИЯ СОЛЕЙ В СИСТЕМЕ Ba^{2+}, Na^{+} / NO_3^{-}, $HCOO^{-}$ – H_2O ПРИ 25 °С

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С целью установления оптимальных температурно-концентрационных параметров процесса конверсионного получения формиата бария в работе изучены фазовые равновесия в четырехкомпонентной взаимной системе Ba^{2+}, Na^{+} / NO_3^{-}, $HCOO^{-}$ – H_2O при 25 ºС. Исследования осуществляли изотермическим методом сечения, в качестве физического свойства жидкой фазы использовали показатель преломления. Выбор температуры исследования обусловлен тем, что в случае реализации процесса в промышленных условиях затраты энергии будут минимальными. Изучение фазовых равновесий в диагональных разрезах $NaHCOO$ – $Ba(NO_3)_2$ – H_2O и $NaNO_3$ – $Ba(HCOO)_2$ – H_2O показало, что в обеих системах присутствует область кристаллизации соли, которая не является исходным компонентом, что свидетельствует об отсутствии в исследуемой взаимной системе стабильной диагонали. В четырехкомпонентной системе все соли кристаллизуются в безводном состоянии. Большую часть диаграммы занимают области кристаллизации нитрата и формиата бария, области кристаллизации нитрата и формиата натрия незначительны вследствие высокой растворимости этих солей. Растворы, отвечающие составам эвтонических точек, являются конгруэнтно насыщенными. Анализ полученных данных показал, что нестабильной парой солей является формиат натрия и нитрат бария, так как их области кристаллизации не имеют общей границы. Пара солей формиат бария и нитрат натрия является стабильной, что подтверждает возможность протекания обменной реакции в сторону образования формиата бария. Полученные экспериментальные данные показали принципиальную возможность получения формиата бария из нитрата бария и формиата натрия, а также позволили обосновать параметры процесса конверсии.

Ключевые слова: формиаты и нитраты бария и натрия, взаимная водно-солевая система, фазовая диаграмма, конверсия солей, графические расчеты, температурно-концентрационные параметры

PHASE EQUILIBRIUM AND SALT CONVERSION IN Ba^{2+}, Na^{+} / NO_3^{-}, $HCOO^{-}$ – H_2O SYSTEM AT 25 °C

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Phase equilibria in the four-component reciprocal system Ba^{2+}, Na^{+} / NO_3^{-}, $HCOO^{-}$ – H_2O at 25 ºC were studied to establish the optimal temperature-concentration parameters of barium formate conversion production. The isothermal section method was used, the refractive index was used as physical property of liquid phase. The choice of temperature is due to minimum energy costs when implementing the process in industrial conditions. The study of phase equilibria in the $NaHCOO$ – $Ba(NO_3)_2$ – H_2O and $NaNO_3$ – $Ba(HCOO)_2$ – H_2O systems showed that in both systems there is field of salt crystallization, which is not initial component, which indicates absence stable diagonal in studied four-component system. In four-component system, all salts crystallize in an anhydrous state. Most of diagram is occupied by crystallization regions of barium nitrate and barium formate, crystallization regions of sodium nitrate and sodium formate are insignificant due to the high solubility of these salts. Solutions corresponding to the compositions of eutonic points are congruently saturated. Analysis of data obtained showed that sodium formate and barium nitrate are unstable pair of salts, since their crystallization regions do not have common boundary. A pair of barium formate and sodium nitrate is stable, which confirms the possibility of exchange reaction in direction of barium formate formation. The obtained experimental data showed fundamental possibility of barium formate obtaining from barium nitrate and sodium formate, and also made it possible to substantiate the parameters of the conversion process.

Key words: barium formate and nitrate, sodium formate and nitrate, reciprocal water-salt system, phase diagram, salt conversion, graphic calculations, temperature and concentration parameters

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INTRODUCTION

Using physicochemical analysis to build phase diagrams of polycomponent systems makes it easier to determine the temperature and concentration parameters of phase separation processes. Data on the mutual solubility of salts in a given temperature range allow us to theoretically support the sequence and modes of individual stages of technological processes such as lithium [1, 2] and boron [3, 4] compound extraction from natural brines, manganese extraction from industrial waste [5, 6], soda production waste disposal [7], extractive distillation processes [8] and synthesis of heat storage materials [9].

Conversion is the most environmentally safe and simple method to obtain water-soluble salts. Using two salts of the reciprocal system as starting materials, the exchange reaction produces two other salts as the final product. Salt production based on salt metathesis reactions includes stages of salt crystallisation and solution evaporation. The optimal temperature-concentration parameters for salt production are established based on solubility diagrams of multicomponent reciprocal water-salt systems.

Metal formates are used in a variety of industries due to their unique properties. Because saturated solutions of potassium and sodium formates have negative crystallisation temperatures, these salts are part of antifreeze additives and coolants [10]; calcium formate is used as a cement modifier [11], chlorine-free fertiliser, E238 preservative in the food industry, in veterinary medicine, etc. Formates of transition and rare earth metals are used as starting materials to produce high-temperature superconductors, catalysts, and nanosized metal powders and their oxides [12, 13].

The problem of using and processing limited-demand production waste and by-products is currently relevant. For example, sodium formate (crystalline or as a saturated solution), a by-product of pentaerythritol and chloroform production [14], can be used as a starting component in the production of metal formates.

It has been proven that potassium formate can be obtained through salt metathesis reactions:

$$KAn + NaHCOO \leftrightarrows KHCOO + NaAn,$$

where An – SO_4^{2-}, CO_3^{2-}, HCO_3^- [15, 16]. The conversion process at optimal temperature and concentration parameters revealed that potassium formate, due to its high solubility, remains in the solution. The sodium salt settles out, and after separation, a saturated solution of potassium formate with a low impurity content is obtained.

The following reaction allows for the production of alkaline earth metal formates by conversion:

$$MAn_2 + 2NaHCOO \leftrightarrows M(HCOO)_2 + 2NaAn,$$

where M – Mg, Ca, Sr, Ba, An – Cl^- or NO_3^-. The study of phase equilibria in diagonal sections of the quaternary systems MAn_2 – NaHCOO – H_2O at 25 °C revealed that, in addition to the initial salt crystallisation fields, there is a crystallisation field of an alkaline earth metal formate. To date, the quaternary reciprocal systems Ca^{2+}, Na^+//Cl^- (NO_3^-), $HCOO^-$ – H_2O have been studied, and a conversion method for obtaining calcium formate has been developed [17-19].

Quaternary reciprocal systems that include formates of other alkaline earth metals and sodium should be examined in order to determine the optimal temperature-concentration parameters of salt conversion. For example, highly pure barium formate can be used to manufacture capacitor materials and high-temperature superconductors. Ceramics and other high-tech industries can also benefit from it.

There is a well-known method for producing barium formate by reacting crystalline barium carbonate with formic acid, which includes evaporation of the reaction mass, separation of the end product, its purification, and drying. A number of patents describe conversion methods for producing barium formate from sodium formate and salts or barium hydroxide in an aqueous solution using the reaction:

$$BaAn_2 + 2NaHCOO \leftrightarrows Ba(HCOO)_2 + 2NaAn,$$

where An – Cl^-, PO_4^{3-}, S^{2-}, SO_4^{2-}, OH^-. Thus, crystalline barium formate and sodium salt are obtained by using various barium salts.

This paper presents the findings of a phase equilibria study in the quaternary reciprocal system Ba^{2+}, Na^+/NO_3^-, $HCOO^-$ – H_2O at 25 °C and confirms the possibility of producing barium formate from sodium formate and barium nitrate by conversion.

EXPERIMENTAL PART

We used the following salts in the study: sodium formate (technical grade, Metafrax Chemicals, Russia) purified by double recrystallisation followed by drying at 105 °C to constant weight; sodium and barium nitrates and barium formate (analytical grade, LLC "REAHIM JSC", Russia).

We used the isothermal section method [20] to study the solubility in water-salt systems in order to obtain comparable experimental data. The method is based on the determination of any easily measurable physical property of the liquid phase of weighed mixtures prepared from the system's components at a given temperature. These mixtures must fall somewhere along the composition triangle. Following the measurements, a functional dependence is graphed, which connects the physical property and the content of one of the system components. On the graph, each type of equilibrium corresponds to a different functional line. Points of line intersection indicate the composition that corresponds to the boundary of different phase fields. We determined the salient points using graphical interpolation based on a linear approximation of individual sections of the obtained dependences. The described method determines solubility with an accuracy of 0.5 wt.%. We used the refractive index as a physical property of the liquid phase because it is easily measured and yields accurate results.

We conducted the experiment as follows. Tightly sealed test tubes containing weighed mixtures (5.0000 g each with an analytical balance weighing accuracy of ±0.0002 g) were thermostated at 25 °C ± 0.2 °C until equilibrium was established. The equilibrium was indicated by the consistency of the liquid phase's refractive index over time. For two or three samples of one solution, the refractive index was measured on ИРФ-454Б (JSC "Kazan Optical and Mechanical Plant", Russia) refractometer with an accuracy of ±0.0005. The accepted value was the arithmetic mean of all measurements.

RESULTS AND DISCUSSION

The following reaction can be used to obtain barium formate by conversion:

$$Ba(NO_3)_2 + 2NaHCOO \leftrightarrows Ba(HCOO)_2 + 2NaNO_3.$$

The value of the solubility product of reciprocal salt pairs can be used to estimate the reaction direction [21]. The equality of the solubility products of the salts of each of the reversible pairs is a necessary condition for the solution saturated simultaneously with four salts, according to the van 't Hoff rule. If the solubility product of one of the salt pairs is greater than the other at a given temperature, then these salt pairs are in unstable equilibrium with one another. One salt pair shifts from sediment to solution, forming a second salt pair. Reference data in salt–water binary systems were used to calculate the solubility products of salts in the temperature range of 0-75 °C [22, 23].

According to Table 1, the solubility product of a salt pair with barium formate is greater than that of a salt pair with barium nitrate. Thus, the production of barium formate by conversion is impossible because the equilibrium in the salt metathesis reaction will be shifted towards the formation of sodium formate and barium nitrate. However, such a theoretical calculation does not always allow making a final decision on the feasibility of the conversion process.

Table 1

Solubility products of reciprocal salt pairs (mol/1000 g of water)

Таблица 1. Произведения растворимости взаимных пар солей (моль/1000 г воды)

t, °C	$[Ba(NO_3)_2]^3 \cdot [NaHCOO]^4$	$[Ba(HCOO)_2]^3 \cdot [NaNO_3]^4$
0	$1.18 \cdot 10^1$	$8.57 \cdot 10^3$
25	$2.81 \cdot 10^3$	$3.41 \cdot 10^4$
50	$2.27 \cdot 10^4$	$1.30 \cdot 10^5$
75	$1.31 \cdot 10^5$	$5.14 \cdot 10^5$

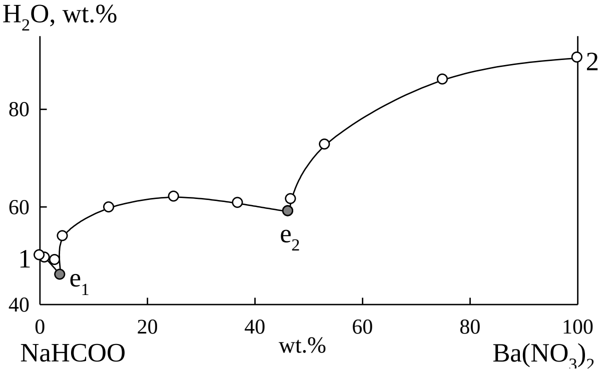

Fig. 1. Isotherm of solubility of the $Ba(NO_3)_2 – NaHCOO – H_2O$ system at 25 °C Crystallisation lines: 1–E1 – NaHCOO, E1–E2 – $Ba(HCOO)_2$, E2–2 – $Ba(NO_3)_2$

Рис. 1. Изотерма растворимости системы $Ba(NO_3)_2 – NaHCOO – H_2O$ при 25 °C Линии кристаллизации: 1-e1 – NaHCOO, e1-e2 – $Ba(HCOO)_2$, e2-2 – $Ba(NO_3)_2$

Thus, we examined the solubility in the ternary systems $Ba(NO_3)_2 – NaHCOO – H_2O$ and $Ba(HCOO)_2 – NaNO_3 – H_2O$, which are diagonal sections of the quaternary reciprocal system $Ba^{2+}, Na^+//HCOO^-, NO_3^- – H_2O$. Further research will be carried out at 25 °C because, in this case, the process's implementation in an industrial environment will require minimal energy consumption.

According to studies, in both diagonal sections, there is a crystallisation field of a salt that is not the initial component of the system (Fig. 1, 2). Thus, at 25 °C, there is no stable diagonal line in the $Ba^{2+}, Na^+//HCOO^-, NO_3^- – H_2O$ system. The phase diagram of the quaternary system can be used to reach a final conclusion about the possibility of obtaining barium formate and determine the concentration parameters of the process.

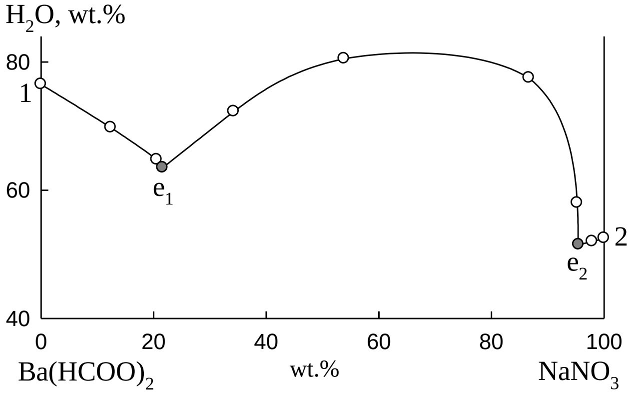

Fig. 2. Isotherm of solubility of the $Ba(HCOO)_2 – NaNO_3 – H_2O$ system at 25 °C Crystallisation lines: 1–E1 – $Ba(HCOO)_2$; E1 –E2 – $Ba(NO_3)_2$; 2–E2 – $NaNO_3$

Рис. 2. Изотерма растворимости (системы $Ba(HCOO)_2 – NaNO_3 – H_2O$ при 25 °C Линии кристаллизации: 1-e1 – $Ba(HCOO)_2$; e1-e2 – $Ba(NO_3)_2$; 2-e2 – $NaNO_3$

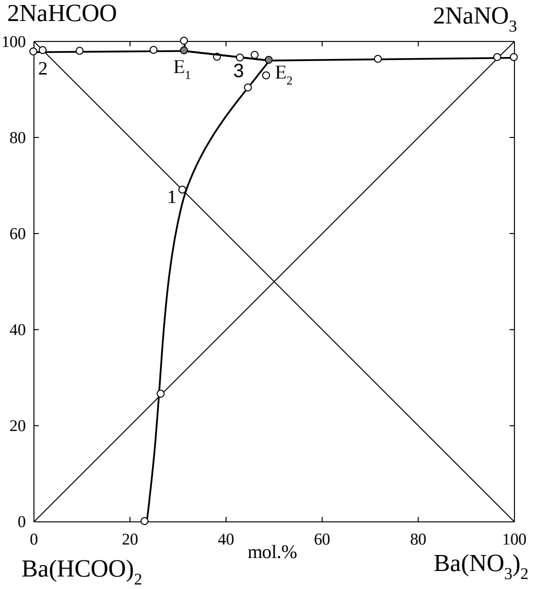

Fig. 3. Perspective projection of the phase diagram of the quaternary reciprocal system $Ba^{2+}, Na^+ // HCOO^-, NO_3^- – H_2O$ at 25 °C

Рис. 3. Перспективная проекция фазовой диаграммы четырехкомпонентной взаимной системы $Ba^{2+}, Na^+ // HCOO^-, NO_3^- – H_2O$ при 25 °C

Experiment data on solubility in contour systems and sections allowed us to make a phase diagram of the $Ba^{2+}, Na^+//HCOO^-, NO_3^- – H_2O$ system (Fig. 3, Table 2). It has been established that all salts crystallise in anhydrous form at 25 °C. The crystallisation fields of $Ba(NO_3)_2$ and $Ba(HCOO)_2$ take up the majority of the diagram. The crystallisation fields of NaHCOO and $NaNO_3$ are insignificant due to high solubility of these salts.

The lines of doubly saturated solutions cross at invariant equilibrium points E_1-E_2. Congruently saturated solutions correspond to eutonic compositions. During isothermal evaporation of salt solutions, solid phase crystallisation will end at point E_2, where the sediment will contain a mixture of $NaNO_3$, $Ba(HCOO)_2$, and $Ba(NO_3)_2$.

The phase diagram analysis revealed that the unstable pair at 25 °C is NaHCOO–$Ba(NO_3)_2$, because the crystallisation fields of these salts do not share a common boundary. The stability of the $Ba(HCOO)_2$ – $NaNO_3$ salt pair confirms the possibility of the exchange reaction progressing to the formation of $Ba(HCOO)_2$.

Table 2

Composition of the saturated solutions of the Ba^{2+}, Na^+ // $HCOO^-$, NO_3^- – H_2O system at 25 °C

Талица 2. Состав насыщенных растворов системы Ba^{2+}, Na^+ // $HCOO^-$, NO_3^- – H_2O при 25 °C

In saturated solution, mol.%					$n_D^{25°}$	Salt composition of the solutions, mol.%				Equilibrium solid phase
$NaNO_3$	NaHCOO	$Ba(NO_3)_2$	$Ba(HCOO)_2$	H_2O		$NaNO_3$	NaHCOO	$Ba(NO_3)_2$	$Ba(HCOO)_2$	
0.0	22.9	0.0	0.5	76.5	1.3975	0.0	97.8	0.0	2.2	NaHCOO + $Ba(HCOO)_2$
0.5	22.5	0.0	0.5	76.6	1.3945	2.0	96.1	0.0	2.0	NaHCOO + $Ba(HCOO)_2$
1.7	20.8	0.5	0.0	76.9	1.4010	7.5	90.4	2.1	0.0	NaHCOO + $Ba(HCOO)_2$
6.8	19.7	0.0	0.5	73.0	1.4050	25.0	73.0	0.0	1.9	NaHCOO + $Ba(HCOO)_2$
0.0	0.0	1.7	5.6	92.7	1.3850	0.0	0.0	23.2	76.8	$Ba(NO_3)_2$ + $Ba(HCOO)_2$
0.0	2.4	2.4	4.2	91.1	1.3810	0.0	26.5	26.5	47.0	$Ba(NO_3)_2$ + $Ba(HCOO)_2$
3.9	4.8	0.0	3.9	87.5	1.3915	31.0	38.0	0.0	31.0	$Ba(NO_3)_2$ + $Ba(HCOO)_2$
6.4	10.2	1.8	0.0	81.6	1.3880	34.9	55.3	9.7	0.0	$Ba(NO_3)_2$ + $Ba(HCOO)_2$
10.1	12.7	1.8	0.0	75.4	1.4020	41.2	51.5	7.2	0.0	$Ba(NO_3)_2$ + $Ba(HCOO)_2$
15.8	0.0	0.6	0.0	83.6	1.4150	96.6	0.0	3.4	0.0	$NaNO_3$ + $Ba(NO_3)_2$
15.4	0.6	0.6	0.0	83.5	1.3910	93.1	3.4	3.4	0.0	$NaNO_3$ + $Ba(NO_3)_2$
15.7	6.5	0.9	0.0	76.9	1.4070	67.9	28.3	3.8	0.0	$NaNO_3$ + $Ba(NO_3)_2$
8.6	18.8	0.0	0.0	72.7	1.4070	31.4	68.6	0.0	0.0	$NaNO_3$ + NaHCOO
11.2	14.0	0.8	0.0	74.1	1.4080	43.1	53.9	3.0	0.0	$NaNO_3$ + $Ba(HCOO)_2$
10.7	16.3	0.0	0.9	72.1	1.4130	38.2	58.4	0.0	3.3	$NaNO_3$ + $Ba(HCOO)_2$
19.31	40.959	0.0	1.2	38.5	1.4080	31.4	66.6	0.0	2.0	NaHCOO + $Ba(HCOO)_2$ + $NaNO_3$
33.32	31.96	0.0	2.7	32.0	1.4020	49.0	47.0	0.0	4.0	$Ba(NO_3)_2$ + $Ba(HCOO)_2$ + $NaNO_3$

We determined the concentration parameters of the $Ba(HCOO)_2$ production using the phase diagram of the quaternary system Ba^{2+}, Na^+//$HCOO^-$, NO_3^- – H_2O (Fig. 3). To crystallize $Ba(HCOO)_2$ from the initial reaction mixture, its composition must fall within the crystallization field of this salt and be located on an unstable diagonal line between points 1 and 2. In this case, as a result of the exchange reaction, $Ba(HCOO)_2$ will be released into the solid phase, and the mother liquor's composition will lie on the line of doubly saturated solutions relative to $Ba(HCOO)_2$ and NaHCOO (E_1–point 2) or relative to $Ba(HCOO)_2$ and $NaNO_3$ (E_1–point 3). Graphical calculations showed that if the initial mixture composition is close to point 1, and the resulting mother liquor composition is close to point 3, the maximum yield of $Ba(HCOO)_2$ is possible. The $NaHCOO:Ba(NO_3)_2$ ratio in point 1 is 2.2:1, which corresponds to the stoichiometric coefficients in the previously mentioned salt metathesis reaction. The method's disadvantage is a large amount of forming mother liquor, which is nearly twice as heavy as the $Ba(HCOO)_2$ sediment.

CONCLUSIONS

The mutual solubility of salts in the quaternary reciprocal system Ba^{2+}, Na^+/NO_3^-, $HCOO^-$ – H_2O at 25 °C was studied for the first time. The concentration boundaries of salt crystallization fields were determined, as were the coordinates of triple-saturated eutonic points. The system does not form crystalline hydrates, double salts, or solid solutions at the temperature used in this study. The $Ba(HCOO)_2$ – $NaNO_3$ – H_2O system is the stable diagonal.

The fundamental possibility of obtaining $Ba(HCOO)_2$ from NaHCOO and $Ba(NO_3)_2$ by the conversion method is demonstrated using experimental data on the mutual solubility of salts and graphical calculations on the phase diagram.

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