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Study of the Process of Obtaining Microelement-containing Chlorine-Free Liquid Nk-Fertilizers

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ABSTRACT: In order to obtain liquid fertilizers with trace elements, the solubility of the components in the K^+, NH^{4+}// NO^{3-} – H_2O system at 25ºC was studied. The compositions were selected and the rheological properties of the solutions were studied from the solubility diagram of the system. As a result of the research, stable transparent liquid fertilizers are obtained with a crystallization temperature of less than 10ºC, the content of the sum of nutrients (N + K)% from 7,10% (N-3,46%, K-3,64%) to 12,51% (N-8,63%, K-3,88%).

KEYWORDS: System, component, solution, crystallization, density, viscosity, potassium nitrate, ammonium nitrate, liquid, fertilizer, trace element.

I. INTRODUCTION

Liquid complex fertilizers are a necessary product in the innovative technology of intensive farming. The program for the development of agriculture, water saving and the development of drip irrigation provides for the development and involvement in agricultural turnover of lands that have lost their physical properties due to non-core or intensive exploitation. To help in solving this issue, liquid complex fertilizers adapted to the tasks set will help [1].

Liquid complex fertilizers (LCF) are solutions that contain nutrients. In addition to them, pesticides and growth stimulants can be added to the mixture [2-4]. The advantage of liquid complex fertilizers over solid ones lies in the ease of production and lower costs in terms of capital investment, storage and transportation. The proportion of nutrients that make up the FCS can be adjusted depending on the properties of the soil.

In this regard, for the physicochemical substantiation of the process of obtaining chlorine-free complex nitrogen-potassium fertilizers, we studied and theoretically analyzed the three-component system K^+, NH^{4+}// NO_3^- – H_2O by the isothermal method at 25ºC.

II. METHODS AND MATERIALS.

Fertilizer samples were prepared by dissolving the solid components in a thermostated glass reactor at 20-30°C. First, the calculated amount of water, potassium nitrate, and ammonium nitrate was added; after ammonization, a microelement (ME) was added to a predetermined value. The resulting FCF was analyzed for nitrogen content and potassium oxide by well-known methods [5–6]. The specific mass of samples of the studied compounds and solutions was determined by the pycnometric method [7] using a capillary pycnometer with a volume of 10 cm^3. To determine the volume, the pycnometer was filled with bidistilled water, thermostated at 25ºC, and weighed. Knowing the weight of the dry pycnometer, the density of water at 25ºC, and the weight of the pycnometer filled with water, its volume was

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calculated. Weighing was carried out with an accuracy of ±0.00005 mg. The results are presented with an accuracy of ±0,1kg/m^3.

The pH of the solution medium was measured according to the procedure [8] on an EV-74 universal ion meter.

The kinematic viscosity of the solutions was determined on a VPG capillary viscometer [9] with a capillary diameter of 1,16–2,75 mm. Results accuracy±0.0001•10^{-1} mm^2/s.

III. RESULTS AND DISCUSSION.

The equilibrium of the phases in the system was established with continuous stirring and temperature control after 6-8 hours. Based on the chemical analysis of liquid and solid phases and interpolation of literature data [10-11], an isothermal diagram of the solubility of the system under study at 25°C was constructed (Figure 1).

The liquidus curve of the solubility diagram splits into three branches, corresponding to the crystallization of two initial components - KNO_3, NH_4NO_3 and a compound of composition $3KNO_3$•NH_4NO_3.

The rectilinear rays emanating from the liquidus line of this compound intersect at a point located on the anhydrous side of the concentration triangle. This indicates that this compound does not contain water of crystallization.

The rays of the $3KNO_3$•NH_4NO_3 compound, connecting the pole of the complex with the origin, cross the branch of its crystallization. This indicates the congruent solubility of the $3KNO_3$•NH_4NO_3 compound in water without decomposition. Therefore, it can be recrystallized from aqueous solutions.

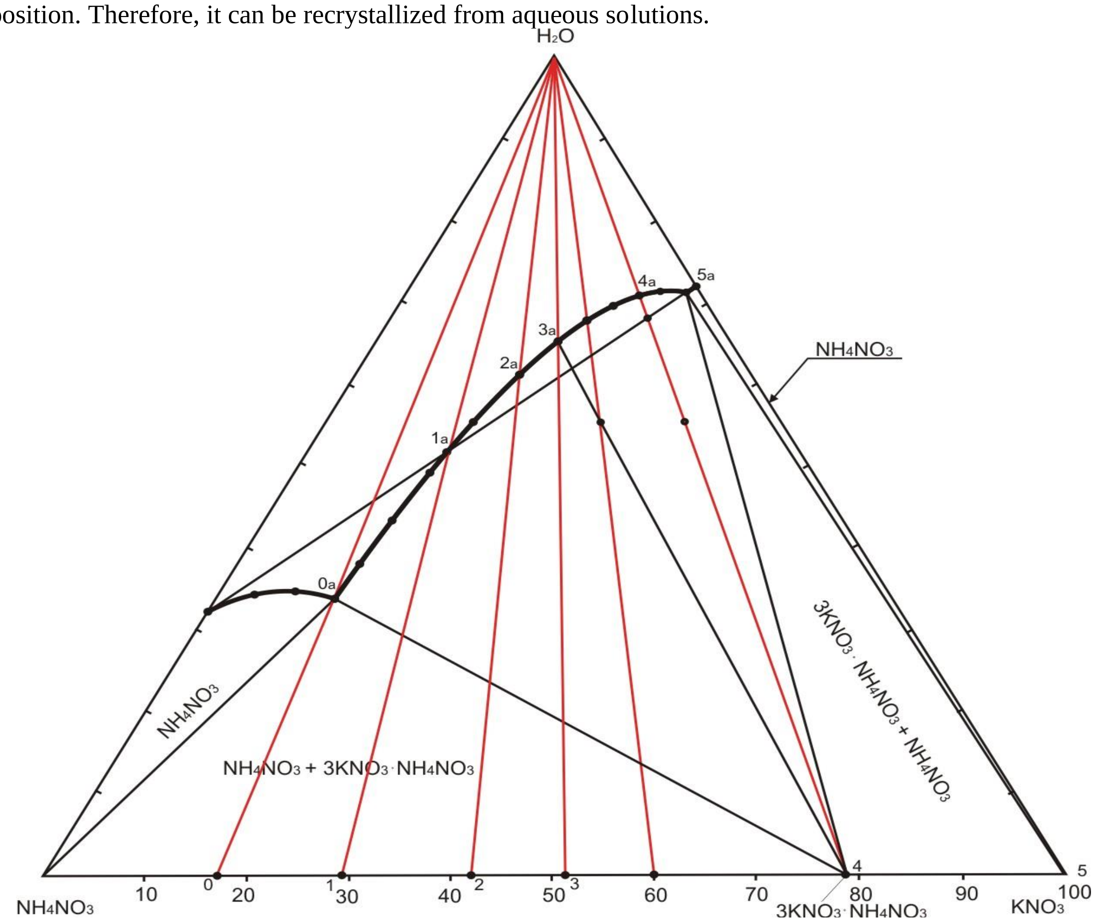

Figure 1. Isothermal diagram of the solubility of the ammonium nitrate-potassium nitrate-water system at 25ºC

The chemical analysis of the solid phase isolated from the supposed region of $3KNO_3$•NH_4NO_3 crystallization confirms its formation in the ammonium nitrate–potassium nitrate–water system.

Chemical analysis of the resulting compound gave the following results:

Found, in %: K -30,48; N-19,39; NO_3-14,45.

For $3KNO_3$•NH_4NO_3 calculated, in %: K -30,54; N-20,88; NO_3-15,66.

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Further, in order to obtain liquid fertilizers containing microelements in their composition, we selected and studied the rheological properties (Table 1) of solutions from the solubility diagram of the ammonium nitrate - potassium nitrate - water system at 25°C, which are marked on the diagram with conditional numbers 0a-4a.

Table 1
Rheological properties of the resulting solutions based on the solubility diagram of the ammonium nitrate-potassium nitrate-water system

№ p/p	Solution composition, %			Temp. crist,°C	Viscosity, mm^2/s	pH	Refractive index	Density g/cm^3
	NH_4NO_3	KNO_3	H_2O					
1[a]	31,63	15,37	53,00	12,3	1,2717	5,41	1,3906	1,2550
2[a]	22,29	16,21	61,5	13,8	1,2237	5,26	1,3778	1,2110
3[a]	15,93	17,07	67,00	12,5	1,1436	5,39	1,3696	1,1914
4[a]	13,50	14,50	72,00	10,6	1,1902	5,53	1,3634	1,1590
0[a]	-	27,50	82,50	25,5	1,1650	6,95	1,3665	1,2579

It follows from the data in Table 1 that with a decrease in the concentration of NH_4NO_3 from 31,63 to 13,50 the crystallization temperature, viscosity, refractive index, and density decrease from 12,3 to 10,6; from 1,2717 to 1,902; from 1,3906 to 1,3634; from 1,2550 to 1,590, respectively. A pure 27,5% solution has a higher crystallization temperature of -25,5°C and a density of -1,2579 g/cm^3. The study of the rheological properties of solutions with and without the addition of trace elements showed that they can be transported by existing pumping devices without any difficulty.

When obtaining microelement-containing liquid fertilizers, solutions obtained by nitric acid decomposition of spent catalysts in the production of nitric acid in the chemical industry were used. The decomposition of spent catalysts was carried out in a laboratory setup consisting of a tubular glass reactor equipped with a paddle stirrer driven by a motor. The required amount of nitric acid was placed in the reactor, and the calculated amount of spent catalysts was added with vigorous stirring. The temperature of the reaction mass was maintained at 45-50°C using a contact thermometer for 30 minutes.

Table 2 below shows the elemental composition of the spent catalyst waste from the production of nitric acid grade AMS.

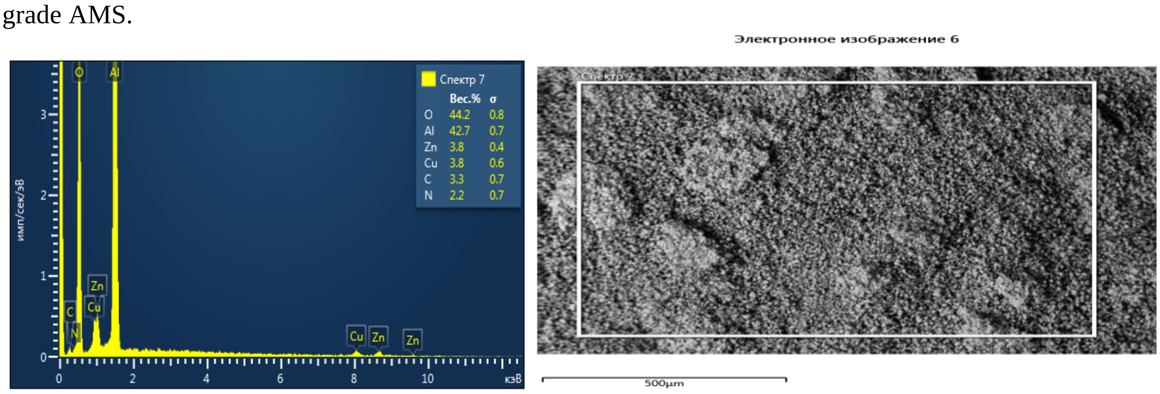

Figure 2. Energy dispersive spectrum of spent catalyst

Table 2
Elemental analysis of spent catalyst

Element	weight .%	Sigma weight .%	Standard name
C	3,33	0,72	C Vit
N	2,22	0,68	BN
O	44,17	0,75	SiO_2
Al	42,70	0,70	Al_2O_3
Cu	3,77	0,55	Cu
Zn	3,80	0,44	Zn
total:	100,00		

As can be seen from Table 2, the spent catalyst has the following chemical composition (wt %): O, 44,17; A1 – 42,70;Zn-3,80;Cu-3,77;C–3,33;N– 2,22; (table 2). Before use, it was ground to a particle size of less than 0.20 mm.

After decomposition with nitric acid, the liquid phase was analyzed for elemental composition, which is given in Table 3. As follows from Table 3, the liquid phase has and contains: P-0,0014; K-0,2900; Ca-0,0010; Mg-0,0010 g/l macroelements and B-0,00032; Cu-120,0; Zn-0,3600; Co-0,0700; Mn-0,0150 and Fe-0,0340 g/l of microelements, respectively.

Table 3
Elemental composition of the liquid phase after the decomposition of spent catalysts

Catalyst grade	Macronutrients, g/l				Trace elements, g/l					
	Mg	**P**	**K**	**Ca**	**Cu**	**Zn**	**B**	**Mn**	**Fe**	**Co**
AMS	0,0010	0,0014	0,2900	0,0010	120,0	0,360000	0,00032	0,0150	0,0340	0,0700

Table 4
Rheological properties of the resulting solutions based on the solubility diagram of the ammonium nitrate-potassium nitrate-water system with the addition of trace elements

Later, the rheological properties of solutions with the addition of trace elements were also studied (table 4)

№ p/p	Solution composition , %				Temp. crist,℃	Viscosity , mm^2/c	pH	Refractive index	Density g/cm^3
	NH_4NO_3	KNO_3	H_2O	ME					
1	15,82	7,69	26,50	26,50	+4,0	1,709	5,130	1,394	1,200
2	11,15	8,10	30,75	30,75	-1,0	1,773	5,170	1,386	1,234
3	7,97	8,53	33,50	33,50	+1,0	1,927	5,210	1,380	1,214
4	6,75	7,25	36,00	36,00	-2,0	1,751	5,230	1,379	1,203
5	-	27,50	41,25	41,25	+20,0	1,276	5,280	1,380	1,227

The data obtained show that, in contrast to solutions with additions of trace elements, with a decrease in the concentration of NH_4NO_3 from 15,82 to 6,75%, with an increase in the content of trace elements from 26,50 to 36,00%, the crystallization temperature sharply decreases from +4,0 to –(-2,0), and the density decreases to a lesser extent. , within 1,200-1,234 g/cm^3, and the viscosity increases within 1,709-1,927 mm^2/s. The crystallization temperature of a solution containing 27,50% KNO_3 and 41,25% trace elements drops sharply to 20ºC.

IV. CONCLUSION

Thus, as a result of the research, stable transparent liquid fertilizers are obtained with a crystallization temperature of less than 10ºC, the content of the sum of nutrients (N + K)% from 7,10% (N-3,46%, K-3,64%) to 12,51% (N-8,63%,K-3,88%) and trace elements,g/l(Cu-60,0;Zn-0,18; B-0,00016; Mn-0,0075; Fe-0,017; Co-0,035).

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