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OBTAINING NP-FERTILIZERS BASED ON THE THERMAL CONCENTRATE OF THE PRODUCT OF ACID DECOMPOSITION OF CHLORIDE AND AMMONIUM NITRATE

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Abstract. *The article presents the results of obtaining complex nitrogen-phosphorus (NP-) fertilizers with different ratios of nutrients based on the introduction of ammonium nitrate into a phosphorus concentrate obtained with a double-filtered chlorine-phosphorus porridge obtained by a thermal concentrate decomposed with chloride acid, studies at different costs of chloride acid.*

Introduction. It is necessary to provide the population with sufficient food in an environment where the world population is growing at a high rate, arable land resources and water supplies are declining. At the same time, it is necessary to effectively use chemical agents, including new types of mineral fertilizers and defoliants, which are one of the important factors in obtaining high and high-quality crop yields in a short time. In this regard, the efficient use of mineral fertilizers is of great importance. In this direction, it is important to increase production volumes and types of effective nitrogen, phosphorus and potash fertilizers, and develop a technology for their production.

Object and research methods. The preparation of complex nitrogen-phosphorus (NP-) fertilizers with different ratios of nutrients based on the addition of ammonium nitrate to the phosphorus concentrate obtained with double-filtered chlorine-phosphorus porridge, obtained by the thermoconcentrate decomposed with chloride acid, was studied in various ratios of chloride acid. Thermal concentrate, 31.4% hydrochloric acid and ammonium nitrate (or 89-92% ammonium nitrate solution from the neutralization recuperator) were used to study the process of obtaining complex NP fertilizers.

The interaction of the thermal concentrate with hydrochloric acid was carried out with intensive stirring in a glass reactor at a temperature of 20-30 °C for 15-20 minutes.Depending on the stoichiometric ratio of acid, perchloric acid was completely supplied in 4-7 minutes to decompose the thermal concentrate. However, the total duration of the process is 1-2 hours. Acidity was taken equal to 45-75% of stoichiometry. When calculating the acid standard, phosphate and calcite minerals (including free CaO) in the thermal concentrate were calculated for the formation of monocalcium phosphate and calcium chloride. The reaction of the washed phosphoconcentrate with acid proceeded without foaming. As a result of the decomposition of the thermal concentrate, the temperature rises to 65-85°C, depending on the acid level. To prevent the loss of phosphorus, which is considered a nutrient, in the processes of filtering, the porridge of chlorophosphoric acid formed during the decomposition of the thermal concentrate with hydrochloric acid was neutralized with gaseous ammonia to a pH value of 5-5.5. Using well-known methods [1-13], the processes of decomposition of the thermal concentrate in hydrochloric acid, the kinetics of decomposition, the determination of the content of nitrogen, phosphorus, potassium, calcium and moisture in the resulting porridge and the finished product are detailed.

Research results and discussion.A chemical analysis of the dependence of the chemical composition of the porridge of neutralized chlorophosphoric acid on the acid standard was carried out (Table 1).

Table 1

Dependence of the chemical composition of porridge processed from the thermal concentrate MH in perchloric acid on the acid level, %

Acidstandard	N	P_2O_5			CaO			H_2O	CI^-
		tot.	assim.	water sol.	tot.	assim.	water sol.		
Compositionofporridge									
45	0,28	14,33	6,67	-	31,04	16,96	11,42	35,23	14,48
55	0,40	13,01	7,50	-	28,17	18,72	12,67	38,07	16,07
65	0,55	11,94	8,08	1,07	25,79	19,82	14,17	41,2	17,43
75	0,62	10,75	8,26	1,29	23,29	20,99	15,31	43,83	18,77

Chemical analysis showed that the degree of thermal concentrate decomposition increases from 46.54 to 76.83% with an increase in acid content from 45 to 75%. At an acid level of 45%, the moisture content of the porridge is 35.23%, and the amount of chloride ions is 14.48%. With an increase in the acid level from 55 to 75%,

the moisture content increases from 1.08 to 1.25 times, and the content of chloride ions increases from 1.11 to 1.30 times. An increase in the level of acid has a positive effect on the fluidity of the resulting porridge.

The main composition of the products of the chloride-acid decomposition of the thermal concentrate (porridge) is (mono- and di-) calcium phosphates, calcium chloride and water. With an increase in the level of acid, the amount of calcium chloride and water in the porridge increases dramatically.

Chlorophosphorus porridge is the main intermediate for obtaining chlorate-containing defoliants, complex NP- and NPK-fertilizers. To obtain complex NP-fertilizers, phosphorus chlorine porridge was filtered 2 times in the ratio of water:porridge=1:1 under laboratory conditions. The filtrate (calcium chloride solution) formed during the 1st filtration was used as a raw material for obtaining a defoliant. The wet residue was reconstituted with water 1:1 and filtered a second time. The filtrate formed during the 2nd filtration process is used to filter the newly formed porridge of chlorophosphoric acid. The wet residue (phosphoconcentrate) formed in the 2nd filtration process was used to obtain complex NP- and NPK-fertilizers. Wet residues formed during the 1st and 2nd filtration of chlorophosphorus porridge, and intermediate products formed during their drying, were subjected to chemical analysis (Table 2).

Table 2

The chemical composition of products formed during the filtration of chlorine-phosphorus porridge, %

Acidstandard	N	P_2O_5			CaO			H_2O	CI^-
		tot.	assim.	water sol.	tot.	assim.	water sol.		
After 1st filtration									
45	0,45	23,10	10,76	-	34,11	11,41	2,49	27,26	3,15
55	0,73	24,01	13,68	-	31,06	13,63	2,46	27,17	3,12
65	1,18	25,62	17,34	2,31	28,47	15,66	3,53	27,08	3,33
75	1,56	27,07	20,80	3,25	24,11	18,35	4,05	27,00	3,51
After2ndfiltration									
45	0,47	24,30	11,31	-	33,39	9,51	0,12	27,01	0,15
55	0,78	25,26	14,56	-	30,22	11,88	0,12	26,86	0,16
65	1,25	27,10	18,35	2,41	27,53	13,97	1,15	26,46	0,24
75	1,66	28,80	22,13	3,45	22,97	16,83	1,61	25,99	0,32
Dry phosphoconcentrate									
45	0,65	33,07	15,40	-	45,43	12,94	0,16	0,68	0,20
55	1,04	34,31	19,78	-	41,04	16,13	0,16	0,67	0,21
65	1,67	36,36	24,61	3,27	36,93	18,75	1,54	1,34	0,32
75	2,2	38,14	29,30	4,57	30,43	22,29	2,13	1,98	0,42

After the 1st filtration of phosphorus porridge obtained at an acidity of 45%, the content of total phosphorus and calcium in it is 23.10 and 34.11%, respectively. Digestible phosphorus and calcium are 10.76 and 11.41%, respectively. In addition, the amount of calcium and chlorine ions in water-soluble form is 2.49% and 3.15%, respectively, compared to those in porridge with chlorophosphoric acid. With an increase in acidity from 55 to 75%, the amount of phosphorus and calcium in the assimilable form changes to 13.68-20.80 and 13.63-18.35%, respectively. The total amount of calcium decreases from 1.10 to 1.41 times. This shows that with an increase in the level of acid, the formation of calcium chloride in the thermal concentrate increases, and phosphorus is relatively rich in phosphorus concentrate obtained during the filtration process[8].

After the 2nd filtration, chlorine ions in the composition of the wet residue (phosphoconcentrate) are reduced by 10.97-21.00 times compared to the 1st filtration at the corresponding stoichiometric speeds. The amount of water is 25.99-27.01% depending on the acid level.

After the filtration processes, the phosphoconcentrate was dried. With an acidity of 75% in the dried phosphorus concentrate, the total amount of phosphorus is 38.14%, and 76.82% of it is in the absorbed form, total calcium is 30.43%, and its 73.25% is in the absorbed form. The amount of moisture in the dried phosphorus concentrate ranges from 0.68 to 1.98% in ascending order of acid number.

The resulting wet phosphorus concentrate is considered the main semi-finished product for the production of NP fertilizers. To obtain NP-fertilizers with nutrients in various ratios ($N:P_2O_5$=from 1:0.5 to 1:2), the phosphoconcentrate was treated with the required amount of ammonium nitrate solution (or ammonium nitrate). The obtained complex NP fertilizers were subjected to chemical analysis (Table 3).

Table 3
Chemical composition of complex NP-fertilizers based on phosphoconcentrate and ammonium nitrate, %

$N:P_2O_5$	N			P_2O_5			CaO			H_2O
	tot.	ammoniacal	nitrate	tot.	assim.	water sol.	tot.	assim.	water sol.	
at a stoichiometric ratio of acid 45%										
1:2	12,60	7,04	5,56	22,25	11,12	-	30,37	8,88	0,11	0,45
1:1	16,88	9,49	8,38	16,76	8,55	-	22,09	6,82	0,08	0,33
1:0,7	20,69	10,81	9,88	13,84	7,19	-	17,90	5,74	0,06	0,27
1:0,5	23,20	11,98	11,23	11,23	5,95	-	14,29	4,74	0,05	0,21
at a stoichiometric ratio of acid 55%										
1:2	11,64	6,17	5,46	23,28	13,97	-	27,84	11,12	0,11	0,45
1:1	17,34	8,93	8,4	17,34	10,57	-	20,74	8,43	0,08	0,34
1:0,7	20,33	10,38	9,94	14,23	8,82	-	17,02	7,03	0,07	0,28
1:0,5	22,97	11,66	11,31	11,48	7,23	-	13,74	5,76	0,05	0,22
at a stoichiometric ratio of acid 65%										
1:2	12,24	6,681	5,55	24,48	17,13	2,32	24,86	14,60	1,09	0,9
1:1	18	9,412	8,58	18	12,78	1,71	18,28	10,88	0,80	0,66
1:0,7	20,96	10,82	10,14	14,67	10,56	1,39	14,9	7,88	0,65	0,54
1:0,5	23,54	12,04	11,5	11,77	8,59	1,12	11,95	6,41	0,52	0,43
at a stoichiometric ratio of acid 75%										
1:2	12,75	7,11	5,63	25,49	20,39	3,18	20,34	17,56	1,47	1,32
1:1	18,54	9,8	8,73	18,54	15,02	2,32	14,79	12,91	1,07	0,96
1:0,7	21,47	11,17	10,3	15,03	12,32	1,88	11,99	9,11	0,87	0,78
1:0,5	24,00	12,34	11,65	12,01	9,96	1,50	9,573	7,36	0,69	0,62

The results of the experiment showed that at the level of acidity of 45% and the ratio of nitrogen and phosphorus $N:P_2O_5$=1:0.5, the composition of the complex NP-fertilizer formed $N_{tot.}$ – 23.20%, $P_2O_{5tot.}$ – 11.23%, $P_2O_{5assim.}$ – 5.95%, $CaO_{tot.}$ - 14.29% and $CaO_{assim.}$ - 4.74%. When changing the ratio of nitrogen and phosphorus in the fertilizer from $N:P_2O_5$=1:0.7 to 1:2 $P_2O_{5tot.}$ – from 1.23 to 1.98 times, $CaO_{tot.}$ increases - from 1.25 to 2.12 times. Also, an increase in ammonium nitrate in the fertilizer (from 1:2 to 1:0.5) increases the amount of nitrogen in the form of nitrate in its content up to 2 times.

These laws are repeated for other stoichiometric ratios of perchloric acid. As the level of acidity increases, the amount of nutrients in their content increases in the same proportions as nitrogen and phosphorus. For example, at an acidity level of 45% and a ratio of nitrogen and phosphorus $N:P_2O_5$=1:1, the sum of nutrients ($\sum N+P_2O_5+CaO_{assim.}$) is 42.46%. When the acid level is 75%, the amount of nutrients is 50.59%

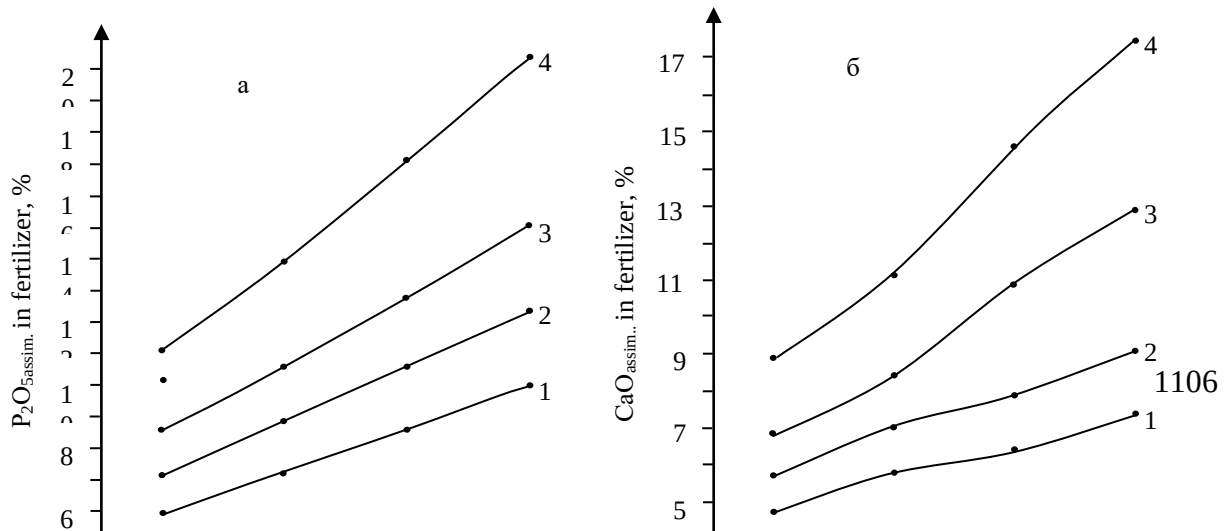

Figure 1. Dependence of the change in the amount of P_2O_{5assim}. (a) and CaOassim. (b) in the composition of the fertilizer from the acidity standard. The ratio of nitrogen and phosphorus in the fertilizer N:P 1–1:0.5; 2–1:0.7; at 3–1:1 and 4–1:2.

The dependence of the forms of phosphorus and calcium absorbed by plants on changes in the rate of perchloric acid and the $N:P_2O_5$ ratio is shown in Figure 1.

Conclusion. It can be seen that with an increase in the rate of perchloric acid, the form of phosphorus and calcium absorbed by the plant increases. It can be observed that the ratio of nitrogen and phosphorus in fertilizers obtained at the same $N:P_2O_5$ rates varies from 1:2 to 1:0.5, that is, the degree of decomposition of the phosphorus concentrate increases with an increase in the content of ammonium nitrate in the fertilizer. This can be explained by an increase in the form of phosphorus absorbed by the plant in relation to its total content in fertilizers.

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