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NamMTI ILMIY-TEXNIKA JURNALI TAHRIR HAY'ATI A'ZOLARI

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OPTIMAL AMOUNTS AND CONCENTRATIONS OF CALCIUM NITRATE SOLUTION IN THE PRODUCTION OF PHOSPHATE FERTILIZERS

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Abstract: The effect of the quantity and concentration of calcium nitrate solutions on the qualitative parameters of fertilizer precipitates obtained on the basis of mineralized mass (MM) was studied. For laboratory experiments, we used MM with the following composition (wt.%): 14.60 - P₂O₅, 43.99 - CaO; 14.11 - CO₂, 1.58 - SO₃; 10.82 - n.d. ; CaO: P₂O₅ = 3.01. The optimal ratios and concentrations of calcium nitrate were determined: the ratio of MM : solution Ca(NO₃)₂ = 1 : 2.0 and the concentration of calcium nitrate solution - 10 and 15%. With optimal parameters, a fertilizer precipitate of the following composition (wt.%) is obtained: P₂O₅total = 23.60-23.68; P₂O₅ac.c.a. by citric acid = 12.67-12.72; P₂O₅water solubility = 2.10-2.11; CaOtotal = 43.36-43.39; CaOac.c.a. by citric acid = 24.19-24.24; CaOwater solubility = 4.24-4.35; N = 1.77-1.82. The degree of removal of Ca(NO₃)₂ from fertilizer precipitates is 97.07-97.42%. This produces the main filtrate containing 20.70-22.52% Ca(NO₃)₂.

Keywords: mineralized mass, nitric acid, acid concentration, amount and concentration of calcium nitrate solution.

Introduction. It is known that at present the most pressing problems in the field of phosphate fertilizer production are the following: increasing production volumes, expanding the product range, involving poorer raw materials with low P₂O₅ content in processing, and minimizing production costs. Currently, the phosphate industry of Uzbekistan is experiencing a shortage of high-quality phosphate raw materials. In this regard, the search for methods to reduce production costs, the involvement of substandard phosphorites in processing, and increasing the concentration and efficiency of phosphate fertilizers are of great importance for the agriculture of the republic.

In recent years, Uzbek scientists have been purposefully conducting research on the acid processing of Central Kyzylkum (CK) phosphorites to obtain various types of fertilizers.

The works of Shamuratova M.R., Namazov Sh.S., and others are devoted to obtaining a single phosphate fertilizer—fertilizer precipitate—by hydrochloric acid processing [1–5]. The main drawback of these studies is the problem of utilizing calcium chloride solutions formed during the processing of phosphorites with hydrochloric acid. In studies [6, 7], the production of concentrated phosphate fertilizers—double

superphosphate—by a cyclic method was investigated. The disadvantages of these works include the use of phosphoric acid, which is expensive under local conditions, as well as the use of a large volume of circulating solution.

Studies [8–11] are devoted to the nitric acid processing of CK phosphorites to obtain nitro-calcium phosphate fertilizers, and this technology has been implemented at JSC “Samarkandkimyo.” The main advantage of nitric acid processing of phosphates is that nitric acid is used in a dual role: as a source of active hydrogen ions and as a carrier of nitrogen, a beneficial component of fertilizers. Another advantage of nitric acid is that it does not impose strict requirements on phosphate raw materials, unlike sulfuric acid processing. Its disadvantages include losses of nitrogen oxides into the gas phase, intense foaming during the decomposition of carbonate phosphorites, high energy consumption in the separation of calcium nitrate, and increased hygroscopicity of the product when calcium nitrate is present. Despite the above-mentioned drawbacks, nitric acid processing is considered the most promising and expedient method.

Therefore, studies [12, 13] investigated the production of fertilizer precipitates based on CK phosphorites and nitric acid. These works examined the influence of the main technological parameters on the quality indicators of the obtained single fertilizers.

This paper presents the results of laboratory studies aimed at determining the effect of the amount and concentration of calcium nitrate solutions on the quality indicators of fertilizer precipitates (total P_2O_5 , available P_2O_5 , available CaO, water-soluble CaO, and N).

Objects and Methods of Research. For laboratory experiments, MM was used with the following composition (wt.%): 14.60 P_2O_5 , 43.99 CaO, 14.11 CO_2 , 1.58 SO_3 , 10.82 insoluble residue; CaO : P_2O_5 = 3.01. For the decomposition of phosphorites, 58.78% nitric acid was used. The nitric acid dosage was taken as 110% of the stoichiometric amount required for the formation of $CaHPO_4$. For washing the precipitate pulp, calcium nitrate solutions of various concentrations—5, 10, 15, 20, and 25%—were used.

The decomposition of MM was carried out in a laboratory setup consisting of a tubular glass reactor equipped with a screw stirrer driven by an electric motor. The reactor was operated without external heating; the temperature of the mixture rose to 35–40 °C due to the heat of exothermic reactions. The duration of the decomposition process was 25–30 minutes. After decomposition, the required amount of calcium nitrate solution was added to the resulting nitric-phosphate pulp in order to avoid adverse effects during phase separation and filtration, as well as to obtain a more concentrated $Ca(NO_3)_2$ solution. After the addition of the $Ca(NO_3)_2$ solution, the acidic nitric-phosphate suspension was neutralized with an aqueous $Ca(OH)_2$ solution to a pH of 4.5–5.0. The neutralized precipitate suspension was then separated into solid and liquid phases using a Büchner funnel under a vacuum of 0.65 mm Hg through two layers of filter paper.

The moist precipitate remaining on the filter was washed once with hot water (80–90 °C) at a weight ratio of MM : H_2O = 1.0 : 2.0. The washed precipitate was dried together with the filter paper in a drying oven at 90 °C. The dried samples of single fertilizers were analyzed using standard methods [14].

The degree of washing of the precipitate from $\text{Ca}(\text{NO}_3)_2$ was calculated using the formula: $\varphi = \left(1 - \frac{m_{\text{precip}} \cdot \omega_{\text{Ca}(\text{NO}_3)_2}}{m_{\text{ini.phos.}} \cdot 2,9286 \cdot \omega_{\text{CaOtot.}}}\right) \cdot 100\%$

$m_{\text{fos.}}$ is the mass of the initial phosphorite;

$\omega_{\text{CaOtotol.}}$ is the mass fraction of CaO in the initial phosphorite;

$m_{\text{precip.}}$ is the mass of the obtained fertilizer precipitate;

$\omega_{\text{Ca}(\text{NO}_3)_2}$ is the mass fraction of $\text{Ca}(\text{NO}_3)_2$ in the fertilizer precipitate.

The factor 2.9286 represents the ratio of the molar masses of $\text{Ca}(\text{NO}_3)_2$ and CaO according to the reaction: $\text{CaO} + 2\text{HNO}_3 = \text{Ca}(\text{NO}_3)_2 + \text{H}_2\text{O}$.

Discussion of Results. The results of laboratory experiments on obtaining fertilizer precipitates based on MM are summarized in Table 1. The data show that as the weight ratio MM : $\text{Ca}(\text{NO}_3)_2$ changes from 1.0 : 1.5 to 1.0 : 3.0, i.e., with an increase in the amount of washing liquid relative to phosphorite, the total P_2O_5 content in fertilizer precipitate samples increases from 23.69 to 24.35%, while the water-soluble P_2O_5 content decreases from 2.21 to 2.06%. At the same time, the contents of water-soluble CaO and nitrogen decrease from 4.20 to 3.92% and from 1.75 to 1.63%, respectively. The degree of washing of the fertilizer precipitate from $\text{Ca}(\text{NO}_3)_2$ increases from 97.02 to 98.78%. This indicates that the removal of calcium nitrate from the moist product improves the quality of the fertilizer precipitate.

A similar trend is observed when using calcium nitrate solutions of other concentrations. For example, when using a 5% calcium nitrate solution, as the weight ratio MM : $\text{Ca}(\text{NO}_3)_2$ solution changes from 1.0 : 1.5 to 1.0 : 3.0, i.e., with an increase in the amount of calcium nitrate solution relative to phosphorite, the P_2O_5 content in fertilizer precipitate samples increases from 23.60 to 24.08%, while the water-soluble P_2O_5 content decreases from 2.19 to 2.04%. The contents of water-soluble CaO and nitrogen decrease from 4.25 to 3.97% and from 1.77 to 1.66%, respectively. The degree of washing of the fertilizer precipitate from $\text{Ca}(\text{NO}_3)_2$ increases from 96.84 to 98.56%. However, in this case, the quality of the fertilizer precipitates slightly deteriorates due to incomplete leaching by $\text{Ca}(\text{NO}_3)_2$ solutions compared with washing using pure water.

Table 1. Main composition of fertilizer precipitates

Main chemical composition of fertilizers, %								
Concentration $\text{Ca}(\text{NO}_3)_2$, %	$\text{P}_2\text{O}_5^{\text{total}}$	Available			Available		N	Degree of washing of precipitates from $\text{Ca}(\text{NO}_3)_2$, %
		P_2O_5 by 2% citric acid	$\text{P}_2\text{O}_5^{\text{water}}$	$\text{CaO}^{\text{total}}$	CaO by 2% citric acid	$\text{CaO}^{\text{water}}$		
Ratio MM : H₂O = 1 : 1,5								
	23,69	12,72	2,21	43,16	24,01	4,20	1,75	97,02
Ratio MM : p-p $\text{Ca}(\text{NO}_3)_2 = 1 : 1,5$								
5	23,60	12,68	2,19	43,17	24,06	4,25	1,77	96,84
10	23,52	12,67	2,18	43,19	24,14	4,34	1,81	96,59

15	23,44	12,63	2,16	43,21	24,19	4,46	1,86	96,25
20	23,32	12,58	2,14	43,23	24,24	4,92	2,05	95,12
25	23,20	12,55	2,11	43,26	24,31	5,29	2,20	94,54
Ratio MM : H₂O = 1 : 2,0								
	23,85	12,78	2,15	43,41	24,11	4,10	1,71	97,85
Ratio MM : p-p Ca(NO₃)₂ = 1 : 2,0								
5	23,76	12,74	2,13	43,33	24,12	4,15	1,74	97,66
10	23,68	12,72	2,11	43,36	24,19	4,24	1,77	97,42
15	23,60	12,67	2,10	43,39	24,24	4,35	1,82	97,07
20	23,48	12,63	2,08	43,41	24,29	4,80	2,01	95,93
25	23,36	12,59	2,06	43,45	24,34	5,16	2,16	95,34
Ratio MM : H₂O = 1 : 2,5								
	24,02	12,85	2,11	43,59	24,16	4,01	1,67	98,68
Ratio MM : p-p Ca(NO₃)₂ = 1 : 2,5								
5	23,92	12,80	2,09	43,53	24,17	4,06	1,70	98,47
10	23,84	12,78	2,08	43,66	24,25	4,15	1,74	98,25
15	23,78	12,74	2,07	43,68	24,30	4,25	1,78	97,90
20	23,64	12,69	2,05	43,71	24,35	4,69	1,96	96,75
25	23,52	12,65	2,02	43,79	24,39	5,04	2,11	96,15
Ratio MM:H₂O=1:3,0								
	24,35	13,01	2,06	44,07	24,38	3,92	1,63	98,78
Ratio MM: p-p Ca(NO₃)₂=1:3,0								
5	24,08	12,86	2,04	43,72	24,40	3,97	1,66	98,56
10	24,01	12,84	2,01	43,81	24,47	4,07	1,70	98,35
15	23,94	12,80	1,99	43,83	24,52	4,16	1,74	98,01
20	23,80	12,78	1,97	43,84	24,57	4,58	1,91	97,02
25	23,68	12,71	1,93	43,86	24,61	4,93	2,06	96,46

The weight ratio **MM : Ca(NO₃)₂ solution = 1.0 : 2.0** can be considered optimal, since at this ratio all quality indicators of the fertilizer precipitate samples remain satisfactory. Below this ratio, the quality of the fertilizers deteriorates, and at higher ratios, more diluted calcium nitrate solutions are obtained. With an increase in the concentration of the calcium nitrate washing solution, all quality parameters of the fertilizer precipitates worsen.

For example, at a ratio of **MM : Ca(NO₃)₂ solution = 1 : 1.5**, increasing the concentration of calcium nitrate solution from 5% to 25% causes the total P₂O₅ content in the fertilizer precipitate samples to decrease from 23.60% to 23.20%, the water-soluble P₂O₅ content decreases from 2.19% to 2.11%, while the contents of water-soluble CaO and N increase from 4.25% to 5.29% and from 1.77% to 2.20%, respectively. The degree of washing of the fertilizer precipitate from Ca(NO₃)₂ decreases from 96.84% to 94.54%. This indicates that using highly concentrated calcium nitrate solutions for leaching from fertilizer precipitate suspensions is not advisable.

The most suitable concentration of the calcium nitrate solution is **10–15%**, as this produces more concentrated Ca(NO₃)₂ solutions that can be easily processed into solid fertilizers. When using water or a 5% Ca(NO₃)₂ solution, the quality indicators of the fertilizer precipitates are much better than when using 10–15% Ca(NO₃)₂ solutions.

However, this leads to the formation of more diluted $\text{Ca}(\text{NO}_3)_2$ solutions, which require significant capital investment for processing into target products. Therefore, the optimal concentration of the $\text{Ca}(\text{NO}_3)_2$ solution can be considered 10–15%.

In further studies, we examined the chemical composition of the liquid phases formed during leaching of calcium nitrate from the precipitate suspensions (main filtrate) and during washing of wet precipitates with water (Table 2). The obtained data are consistent with the results presented in Table 1.

Table 2. Main composition of liquid phases

Concentration $\text{Ca}(\text{NO}_3)_2$, %	Main chemical composition of fertilizers, %					
	Main filtrate			Filtrate after washing with water		
	$\text{P}_2\text{O}_{5\text{water}}$	$\text{CaO}_{\text{water}}$	N	$\text{P}_2\text{O}_{5\text{water}}$	$\text{CaO}_{\text{water}}$	N
Ratio MM:H₂O=1:1,5						
	0,78	3,41	1,55	0,35	1,02	0,44
Ratio MM: s-n $\text{Ca}(\text{NO}_3)_2=1:1,5$						
5	0,76	5,12	2,41	0,34	1,39	0,62
10	0,73	7,85	3,78	0,33	1,52	0,69
15	0,70	8,54	4,13	0,31	1,84	0,86
20	0,68	10,92	5,32	0,30	1,97	0,92
25	0,65	12,29	6,02	0,29	2,15	1,03
Ratio MM:H₂O=1:2,0						
	0,72	2,96	1,34	0,32	0,89	0,38
Ratio MM: s-n $\text{Ca}(\text{NO}_3)_2=1:2,0$						
5	0,69	4,62	2,17	0,31	1,27	0,57
10	0,66	7,07	3,40	0,30	1,39	0,64
15	0,64	7,69	3,72	0,29	1,68	0,78
20	0,62	9,82	4,78	0,28	1,80	0,84
25	0,59	11,06	5,41	0,26	1,96	0,93
Ratio MM:H₂O=1:2,5						
	0,65	2,37	1,06	0,24	0,71	0,30
Ratio MM: s-n $\text{Ca}(\text{NO}_3)_2=1:2,5$						
5	0,63	4,16	1,95	0,23	1,15	0,53
10	0,61	6,34	3,05	0,22	1,26	0,59
15	0,58	6,92	3,34	0,21	1,53	0,72
20	0,57	8,81	4,29	0,21	1,64	0,78
25	0,54	9,92	4,85	0,20	1,79	0,86
Ratio MM:H₂O=1:3,0						
	0,59	1,98	0,87	0,21	0,60	0,26
Ratio MM: s-n $\text{Ca}(\text{NO}_3)_2=1:3,0$						
5	0,57	3,74	1,76	0,20	1,06	0,49
10	0,55	5,65	2,72	0,19	1,17	0,55
15	0,53	6,21	3,01	0,18	1,40	0,66
20	0,52	7,91	3,85	0,18	1,50	0,71
25	0,49	9,07	4,34	0,17	1,65	0,79

Figure 1 shows a 3D graph illustrating the effect of the calcium nitrate solution concentration and the ratio MM : $\text{Ca}(\text{NO}_3)_2$ solution on the total P_2O_5 and water-soluble CaO contents in the fertilizer precipitate samples. From these data, it is evident that increasing the concentration of the calcium nitrate solution worsens the quality of the fertilizer precipitate samples, while increasing the MM : $\text{Ca}(\text{NO}_3)_2$ solution ratio improves it.

Figure 2 (a, b) shows the effect of the calcium nitrate solution concentration and the ratio MM : $\text{Ca}(\text{NO}_3)_2$ solution on the $\text{Ca}(\text{NO}_3)_2$ content in the main filtrates and filtrates after washing with water. These data indicate that increasing the concentration of the calcium nitrate solution raises the $\text{Ca}(\text{NO}_3)_2$ concentration in both filtrates, whereas increasing the MM : $\text{Ca}(\text{NO}_3)_2$ solution ratio decreases the calcium nitrate concentration.

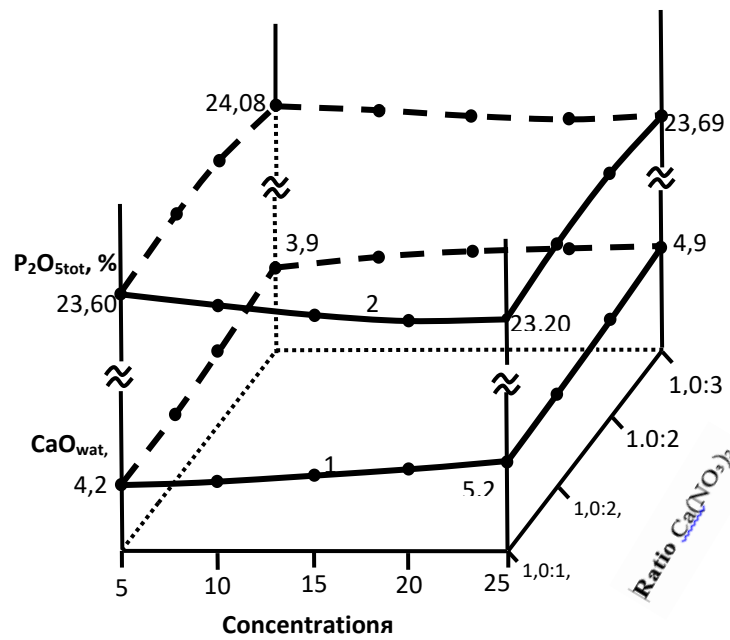
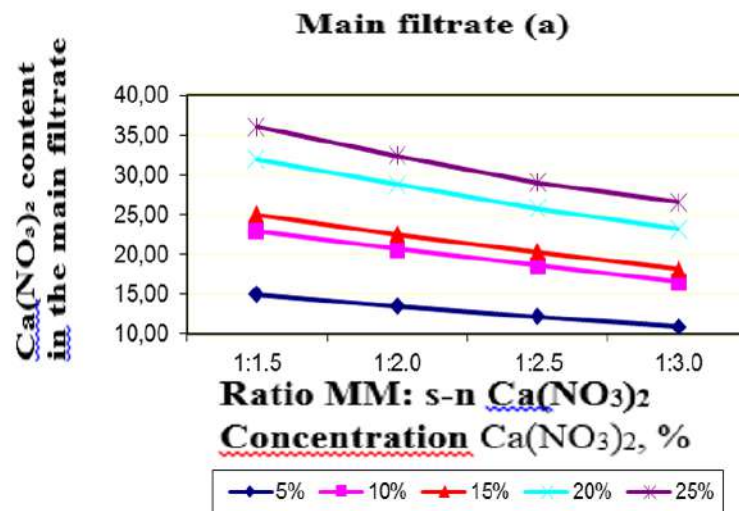


Fig. 1. The effect of calcium nitrate solution concentration and the MM : $\text{Ca}(\text{NO}_3)_2$ solution ratio on the total P_2O_5 and water-soluble CaO contents in fertilizer precipitate samples



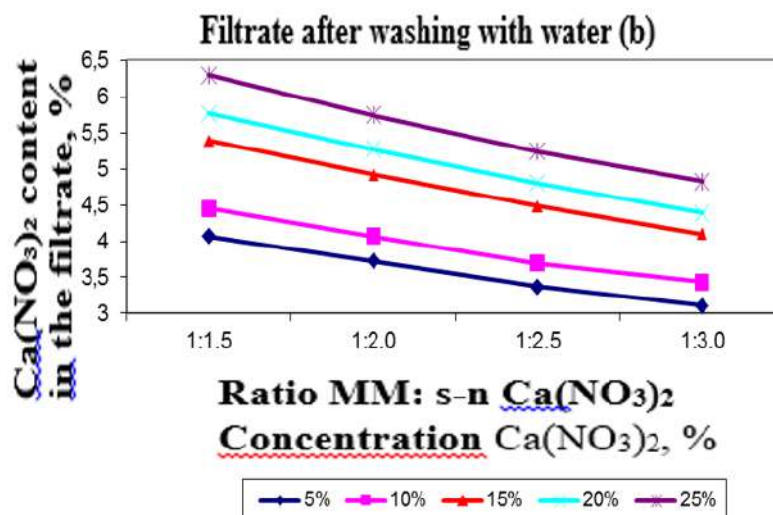


Figure 2 (a, b) shows the effect of the calcium nitrate solution concentration and the ratio **MM : Ca(NO₃)₂ solution** on the Ca(NO₃)₂ content in the main filtrates and filtrates after washing with water. These data indicate that increasing the concentration of the calcium nitrate solution raises the Ca(NO₃)₂ concentration in both filtrates, whereas increasing the **MM : Ca(NO₃)₂ solution** ratio decreases the calcium nitrate concentration

Conclusion: The influence of the amount and concentration of calcium nitrate solutions on the quality indicators of single fertilizers—fertilizer precipitates obtained from mineralized mass (MM)—was studied. Based on laboratory experiments, the optimal ratio and concentrations of calcium nitrate were determined, where the ratio **MM : Ca(NO₃)₂ solution = 1 : 2.0** and the calcium nitrate solution concentration of **10 and 15%** are considered optimal.

Under these optimal parameters, the compositions of fertilizer precipitates (wt.%) are as follows:

- Total P₂O₅ = 23.60–23.68
- Available P₂O₅ (by citric acid) = 12.67–12.72
- Water-soluble P₂O₅ = 2.10–2.11
- Total CaO = 43.36–43.39
- Available CaO (by citric acid) = 24.19–24.24
- Water-soluble CaO = 4.24–4.35
- Nitrogen (N) = 1.77–1.82

The degree of washing of fertilizer precipitates from Ca(NO₃)₂ is 97.07–97.42%. At the same time, the main filtrate contains 20.70–22.52% Ca(NO₃)₂.

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