

Investigation of the Influence of Temperature of Washed Water on the Main Indicators of Fertilizer Precipitate

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Authors' contributions

This work was carried out in collaboration among all authors. Author ALG designed the study, performed the statistical analysis. Author BES wrote the protocol and wrote the first draft of the manuscript managed the analyses of the study. Author ShSN managed the literature searches. All authors read and approved the final manuscript.

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ABSTRACT

The influence of the temperature of washing water on the main indicators of fertilizer precipitates obtained on the basis of mineralized mass (MM) and phosphorite flour (PF) has been studied. For laboratory experiments, MM with the following composition (weight, %) were used: 14.60-P₂O₅, 43.99-CaO; 14.11-CO₂, 1.58-SO₃; CaO:P₂O₅=3.01 and PF, which has the following composition (weight,%): 17.76-P₂O₅, 47.51-CaO; 5.27-i.r.; CaO:P₂O₅=2,68. The optimal temperature of washing water can be considered 80-90°C of both phosphate raw materials (PRM). Under optimal parameters, the following formulations of fertilizer precipitates (weight.%) are obtained: P₂O_{5total}=21.60-22.65; P₂O_{5acceptable by citric acid}=11.42-12.19; P₂O_{5water solubility}=2.08-2.20; CaO_{total}=40.89-41.40; CaO_{acceptable by citric acid}=22.12-23.07; CaO_{water solubility}=4.03-4.26; N_{total}=1.69-1.79 based on MM and P₂O_{5total}=24.86-26.07; P₂O_{5acceptable by citric acid}=13.19-14.08; P₂O_{5water solubility}=2.40-2.57; CaO_{total}=45.19-45.41; CaO_{acceptable by citric acid}=24.54-25.37; CaO_{water solubility} =4.25-4.48;

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$N_{total}=1.78-1.90$ based on PF. The degree of washing of fertilizer precipitates from $Ca(NO_3)_2$ is 97.82-98.01 and 97.75-97.98%, respectively for MM and PF.

Keywords: Mineralized mass; phosphorite flour; nitric acid; acid concentration; temperature of washing water.

1. INTRODUCTION

Currently, the pressing tasks in the field of production of phosphorous fertilizers are: increase their production volume, expand the range, engage in processing of poorer raw materials in the content of P_2O_5 (PM) and phosphate waste (mineralized mass (MM), which is formed during thermal enrichment of the Central Kyzylkum (CK) phosphorites), and maximize the cost of production. To date, the phosphoric industry is experiencing a shortage of quality phosphate raw materials. In this regard, the search for methods of reduction of their production, involvement in the sphere of their production of non-conventional phosphorites, increasing the concentration and efficiency of phosphorus fertilizers is of great importance for the farming of the Republic. At the moment, scientific and research works on the acid processing phosphorites of the Central Kyzylkum for various types of fertilizers by scientists of Uzbekistan, including scientists of the Institute of General and Inorganic Chemistry of the Academy of Sciences of the Republic of Uzbekistan and the Tashkent Chemical and Technological Institute, are being successfully and actively conducted. Many works are devoted to hydrochloric acid processing with the production of single fertilizer - fertilizer precipitate [1-4]. The only drawback of these works is the problem of the disposal of calcium chloride solutions, which is formed during the processing of phosphorites with hydrochloric acid. In the work [5] the production of nitrogen-phosphoric fertilizers on the basis of the decomposition of poor phosphorites of Central Kyzylkum with a mixture of phosphoric and sulfuric acids is studied. The disadvantages of this work are the use of phosphoric and sulfuric acid, which are expensive in our country. The works [6-9] are devoted to the nitric acid processing of phosphorites of the Central Kyzylkum with the production of nitrocalcium phosphate fertilizers.

The first time in world practice Sh.Namazov and other scientists have developed a highly intensive and resource-saving technology for obtaining nitrocalcium phosphate fertilizer by decomposition of ordinary phosphate powder of

the Central Kyzylkum with an incomplete norm of nitric acid, which was applied at JSC "Samarkandkimyo". The advantages of nitric acid processing of phosphates are that it uses nitric acid bilaterally: as a source of active hydrogen ion and as a carrier of nitrogen - a useful compound of fertilizers. It does not impose high requirements on phosphate raw materials, as sulfuric acid processing. Its disadvantages: loss of nitrogen oxides into the gas phase; abundant foam formation when the carbonate phosphorites are decomposed; energy intensity of the process of releasing calcium nitrate; increase in the hygroscopic nature of the product with the presence of calcium nitrate in it. Despite the above mentioned disadvantages of nitric acid processing, this method is more promising and expedient. Therefore, in the works [10-12], the production of fertilizer precipitates was studied. As well as the influence of technological parameters on the qualitative indicators of the received fertilizers is investigated.

The influence of the amount of washing water and the concentration of nitric acid in the production of a chemical enriched phosphate concentrate-semiproduct for the production of concentrated single and complex fertilizers was studied in [13-14]. Based on these works, the study is important of the influence of the temperature of washing water and the concentration of nitric acid in the acid processing of the CK phosphorites. But in these work were not studied the influence of the temperature of washing water on the basic indices of fertilizer precipitates (P_2O_{5t} , CaO_t , $P_2O_{5w.s.}$, $CaO_{w.s.}$ and N_t) obtained from mineralized mass (MM) and phosphorite flour (PF).

It is known from the data [15] that the solubility of calcium nitrate in water strongly depends on temperature. For example, at 20°C solubility of nitrate of calcium is equal 128,8 g in 100 g water, at 30°C 149,4 g, at 40°C 189 g, at 60°C - 359 g, and at 100°C – 363 g.

It is known that calcium nitrate is a very hygroscopic substance and it impairs the product properties of the obtained fertilizers. Therefore, in order to maximally remove calcium nitrate from

the resulting fertilizer precipitates, we decided to study the effect of the temperature of the washing water.

This paper presents the results of studies to determine the effect of the temperature of washing water on the basic indices of fertilizer precipitates (P_2O_{5t} , CaO_t , $P_2O_{5w.s.}$, $CaO_{w.s.}$ and N_t) obtained from mineralized mass (MM) and phosphorite flour (PF).

2. METHODS AND MATERIALS

For laboratory experiments, MM with the following composition (weight, %) were used: 14.60 - P_2O_5 , 43.99 - CaO ; 14.11 - CO_2 , 1.58 - SO_3 ; 10.82 - i.r.; $CaO : P_2O_5 = 3.01$ and PF, having the following composition (weight,%): 17.76 - P_2O_5 , 47.51 - CaO ; 13.93 - CO_2 , 3.27 - SO_3 ; 5.27 - i.r.; $CaO : P_2O_5 = 2.68$. The concentration of nitric acid varied from 50 to 59%. The norm of nitric acid was taken 110% of stoichiometry for the formation of $CaHPO_4$. The decomposition of the phosphorus raw materials (PRM) was carried out on a laboratory installation consisting of a tubular glass reactor equipped with a screw mixer driven by an electric motor. The reactor without artificial heating, the temperature of the mixture developed to 35-40°C due to the heat of exothermic reactions. The duration of the decomposition process was 25-30 min. After decomposition, the required amount of water was added to the obtained nitrogen phosphate pulp to avoid negative influence during separation and deterioration of its filtration. After the addition of water, the nitrogen phosphate suspension was neutralized by the water solution $Ca(OH)_2$ to medium pH=4.5-5.0, after which the neutralized precipitate suspension was divided into solid and liquid phases on the Buchner funnel, with a discharge of 0.65 mm Hg. through two layers of filtering paper. The remaining water residue on the filter was washed once with different temperature of washing water 20, 30, 40, 50, 60, 70, 80, 90 and 100°C at the weight ratio PRM:H₂O = 1 : 2.0. The washed single fertilizers was dried together with the filter paper in the drying cabinet at a temperature of 90°C. Dried samples of single fertilizers were analyzed according to well-known methods [16].

The degree of washing of precipitate from $Ca(NO_3)_2$ was calculated by the formula,

$$\varphi = \left(1 - \frac{m_{precip} \cdot \omega_{Ca(NO_3)_2}}{m_{PRM} \cdot 2,9286 \cdot \omega_{CaOtotal}} \right) \cdot 100\% \quad \text{where}$$

m_{PRM} - the mass of the initial phosphorite raw material, $\omega_{CaOtotal}$ - the mass of the CaO in the initial PRM, m_{precip} - the mass of the obtained fertilizer precipitate, $\omega_{Ca(NO_3)_2}$ - the mass fraction of $Ca(NO_3)_2$ in the fertilizer precipitate. 2.9286 – ratio of molar masses $Ca(NO_3)_2$ and CaO accordingly to reaction of $CaO + 2HNO_3 = Ca(NO_3)_2 + H_2O$.

3. RESULTS AND DISCUSSION

The results of obtained data on reception of fertilizer precipitates on the basis of MM are given in tables 1,2. The tables results shows that at the concentration of HNO_3 50% with a change of temperature of washing water from 20°C to 100°C, i.e., with an increase the temperature of washing water the content of P_2O_{5t} in samples of fertilizer precipitates increases from 21.14 to 21.66%, the content of CaO_t decreases from 41.37 to 40.87%, and the number of $P_2O_{5w.s.}$, $CaO_{w.s.}$ and N_t decreases from 2.35 to 2.07, from 4.27 to 4.01% and from 1.90 to 1.68% respectively. The degree of washing of fertilizer precipitate from $Ca(NO_3)_2$ increases from 97.22 to 98.05%. This indicates that due to the removal of calcium nitrate from a wet product, the quality of fertilizer precipitate is improved. When the temperature of washing water 20°C with increases concentrations of nitric acid from 50 to 59%, the nitrogen content in the precipitates increases from 1.90 to 1.99% and the degree of washing of precipitate from $Ca(NO_3)_2$ decreases from 97.22 to 96.59%.

There is a sufficient amount of $Ca(NO_3)_2$ remains in the composition of the precipitates, which is a hygroscopic substance. When a high temperature of water is used (for example, at temperatures – 80 and 90°C), the $Ca(NO_3)_2$ content in the precipitation is reduced to a minimum. So, the optimal temperatures of washing water could be considered 80 and 90°C. A similar pattern is observed in the case of high concentrations of HNO_3 55 and 59%. However, the quality of fertilizer precipitates is reduced accordingly due to incomplete washing $Ca(NO_3)_2$. Based on these data, it can be assumed that all concentrations of nitric acid are optimal. The use of higher concentrations of HNO_3 is due to the fact at lower acid concentrations formed a huge amount of foam, which reduces the productivity of the reactors.

Table 1. The main composition of fertilizer precipitates

Temperature of washing water	20			30			40			50		
Concentration of nitric acid	50	55	59	50	55	59	50	55	59	50	55	59
P ₂ O _{5t} .	21.14	21.68	22.08	21.18	21.73	22.12	21.27	21.82	22.23	21.36	21.91	22.32
P ₂ O _{5ac.c.a.} by 2 %-citric acid	11.66	11.97	12.41	11.64	11.94	12.38	11.60	11.1	12.34	11.56	11.87	12.29
P ₂ O _{5 w.s.}	2.35	2.39	2.42	2.32	2.36	2.39	2.28	2.32	2.36	2.23	2.27	2.32
CaO _t .	41.37	41.58	41.84	41.32	41.53	41.80	41.27	41.49	41.75	41.18	41.40	41.66
CaO _{ac.c.a.} by 2%-citric acid	22.39	22.72	23.34	22.35	22.69	23.31	22.32	22.66	2.28	22.28	22.62	23.24
CaO _{w.s.}	4.27	4.38	4.53	4.24	4.34	4.49	4.21	4.30	4.45	4.18	4.27	4.41
N _t .	1.90	1.94	1.99	1.87	1.91	1.97	1.84	1.88	1.94	1.81	1.85	1.91
Degree of washing of precipitate from Ca(NO ₃) ₂ , %	97.22	97.05	96.59	97.40	97.32	97.23	97.58	97.53	97.42	97.69	97.62	97.51

Table 2. The main composition of fertilizer precipitates

Temperature of washing water	60			70			80			90			100		
Concentration of nitric acid	50	55	59	50	55	59	50	55	59	50	55	59	50	55	59
P ₂ O _{5t} .	21.46	22.00	22.42	21.56	22.09	22.51	21.60	22.07	22.61	21.64	22.10	22.65	21.66	22.12	22.67
P ₂ O _{5ac.c.a.} by 2 %-citric acid	11.50	11.82	12.24	11.45	11.79	12.21	11.43	11.77	12.19	11.42	11.75	12.17	11.43	11.76	12.18
P ₂ O _{5 w.s.}	2.19	2.22	2.28	2.15	2.19	2.25	2.10	2.15	2.22	2.08	2.13	2.20	2.07	2.12	2.19
CaO _t .	41.08	41.30	41.56	40.99	41.25	41.48	40.91	41.16	41.40	40.89	41.14	41.38	40.87	41.12	41.36
CaO _{ac.c.a.} by 2%-citric acid	22.21	22.56	23.19	22.18	22.51	23.15	22.13	22.47	23.07	22.12	22.46	23.05	22.11	22.45	23.04
CaO _{w.s.}	4.13	4.22	4.36	4.09	4.18	4.32	4.04	4.13	4.26	4.03	4.12	4.25	4.01	4.10	4.23
N _t .	1.77	1.81	1.86	1.74	1.78	1.83	1.70	1.74	1.79	1.69	1.73	1.78	1.68	1.72	1.77
Degree of washing of precipitate from Ca(NO ₃) ₂ , %	97.78	97.71	97.60	97.88	97.81	97.70	97.98	97.91	97.80	98.01	97.93	97.82	98.05	97.96	97.85

In further studies, PF was used with the above composition. The methods of laboratory experiments were identical as in the case of MM. The obtained data are summarized in tables 3,4. The tables results shows that at the concentration of HNO_3 50% with an increase the temperature of washing water from 20°C to 100°C the content of P_2O_5 t. in samples of fertilizer precipitates increases from 24.30 to 24.92%, the content of CaO_t decreases from 45.56 to 45.17%, and the number of $\text{P}_2\text{O}_{5w.s.}$, $\text{CaO}_{w.s.}$ and N_t decreases from 2.63 to 2.38, from 4.48 to 4.23% and from 1.97 to 1.77% respectively. The degree of washing of fertilizer precipitate from $\text{Ca}(\text{NO}_3)_2$ increases from 97.45 to 98.00%. This indicates that due to the removal of calcium nitrate from a wet product, the quality of fertilizer precipitate is improved. When the temperature of washing water 20°C with increases concentrations of nitric acid from 50 to 59%, the nitrogen content in the precipitates increases from 1.97 to 2.09% and the degree of washing of precipitate from $\text{Ca}(\text{NO}_3)_2$ decreases from 97.45 to 97.20%.

In this case, CaO_t and $\text{CaO}_{ac.c.a.}$ are decreased proportionally by 2% citric acid from 45.56 to

45.17% and from 24.73 to 25.52%, respectively. The content of $\text{CaO}_{w.s.}$ in the received fertilizer samples are decreased from 4.48 to 4.23%, i.e. decreases by 1.06 times. And the nitrogen content decreases from 1.97 to 1.77%, i.e. it is decreased by 1.11 times. The degree of washing of fertilizer precipitate from $\text{Ca}(\text{NO}_3)_2$ increases from 97.45 to 98.00%. A similar pattern is observed at other acid concentrations. All acid concentrations are optimal. So, the optimal temperatures of washing water could be considered 80 and 90°C, as in the case of MM.

And also from table data (Tables 1, 2 and 3,4) it is visible, that similar regularity is observed both at use of PF, but at the same time the content of P_2O_{5t} in received samples of single fertilizers more, than in case of single fertilizers received from MM. The fertilizer obtained on the basis of PF in all parameters has advantages over fertilizer on the basis of MM. For example, the content of P_2O_{5t} in the temperature of washing water 80°C (the concentration of acid 50%) is 24.86% against 21.60% in fertilizer obtained on the basis of MM, and at the temperature of washing water 90°C in the same concentration of acid it is 24.90% against 21.64%.

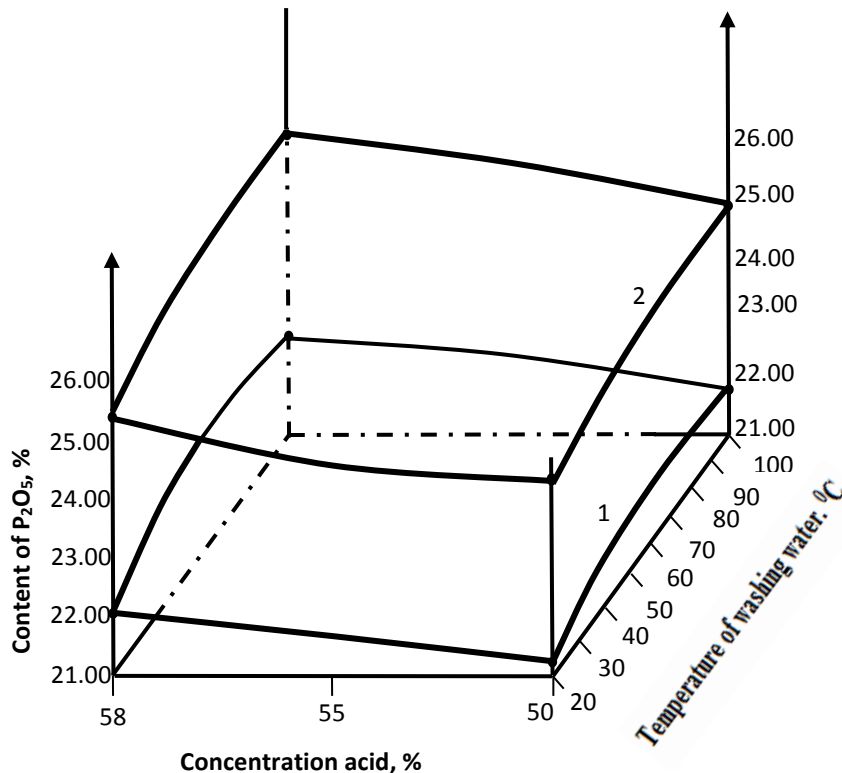


Fig. 1. Influence of nitric acid concentration and temperature of washing water on content of P_2O_{5t} in samples of fertilizer precipitates: 1-based MM; 2- based on PF

Table 3. The main composition of fertilizer precipitates

Temperature of washing water	20			30			40			50		
Concentration of nitric acid	50	55	59	50	55	59	50	55	59	50	55	59
P ₂ O _{5t} .	24.30	25.06	25.67	24.45	25.10	25.72	24.61	25.15	25.79	24.67	25.20	25.85
P ₂ O _{5ac.c.a.} by 2 %-citric acid	13.31	13.68	14.18	13.30	13.67	14.17	13.29	13.66	14.17	13.28	13.65	14.16
P ₂ O _{5 w.s.}	2.63	2.69	2.77	2.60	2.66	2.74	2.58	2.63	2.71	2.55	2.60	2.68
CaO _t .	45.56	45.66	45.79	45.51	45.61	45.74	45.46	45.56	45.69	45.42	45.52	45.64
CaO _{ac.c.a.} by 2%-citric acid	24.73	25.03	25.59	24.71	25.01	25.57	24.69	24.98	25.54	24.67	24.95	25.51
CaO _{w.s.}	4.48	4.57	4.69	4.45	4.54	4.67	4.42	4.51	4.64	4.39	4.48	4.61
N _t .	1.97	2.01	2.09	1.95	1.99	2.07	1.93	1.97	2.04	1.91	1.95	2.01
Degree of washing of precipitate from Ca(NO ₃) ₂ , %	97.45	97.37	97.20	97.49	97.42	97.25	97.57	97.50	97.36	97.61	97.55	97.41

Table 4. The main composition of fertilizer precipitates

Temperature of washing water	60			70			80			90			100		
Concentration of nitric acid	50	55	59	50	55	59	50	55	59	50	55	59	50	55	59
P ₂ O _{5t} .	24.71	25.25	25.90	24.76	25.30	25.94	24.86	25.40	26.04	24.90	25.43	26.07	24.92	25.46	26.09
P ₂ O _{5ac.c.a.} by 2 %-citric acid	13.25	13.63	14.14	13.23	13.61	14.11	13.20	13.59	14.08	13.19	13.57	14.06	13.18	13.56	14.05
P ₂ O _{5 w.s.}	2.52	2.58	2.66	2.48	2.54	2.62	2.42	2.48	2.57	2.40	2.46	2.55	2.38	2.44	2.53
CaO _t .	45.38	45.47	45.59	45.31	45.40	45.51	45.22	45.31	45.41	45.19	45.28	45.38	45.17	45.26	45.35
CaO _{ac.c.a.} by 2%-citric acid	24.65	24.93	25.48	24.61	24.88	25.44	24.56	24.83	25.39	24.54	24.81	25.37	24.52	24.79	25.35
CaO _{w.s.}	4.36	4.45	4.58	4.32	4.41	4.54	4.27	4.36	4.48	4.25	4.34	4.46	4.23	4.31	4.42
N _t .	1.88	1.92	1.98	1.85	1.89	1.95	1.80	1.84	1.90	1.78	1.82	1.88	1.77	1.80	1.86
Degree of washing of precipitate from Ca(NO ₃) ₂ , %	97.68	97.63	97.48	97.75	97.68	97.55	97.95	97.88	97.75	97.98	97.91	97.79	98.00	97.93	97.81

A similar pattern is observed at other acid concentrations. Fig. 1 shows a large plot of the effect of acid concentration and the temperature of washing water on the content of P_2O_{5t} in samples of fertilizer precipitates obtained on the basis of MM (1) and PF (2). These charts clearly show the effect of nitric acid concentration and the increase in the temperature of washing water on the content of P_2O_{5t} in samples of fertilizer precipitates. The graph shows that the temperature of washing water has a more effective effect on the contents of P_2O_{5t} in fertilizer precipitates than the concentration of nitric acid. Because, the solubility of calcium nitrate in water strongly depends on temperature.

For example, with an increase in the temperature of washing water from 20 to 100°C at a concentration of nitric acid 50% of the content of P_2O_{5t} in samples of fertilizer precipitates is increased by 0.52% in the case of MM. With an increase in the acid concentration from 50 to 59% at the temperature of washing water of the content of P_2O_5 in samples of fertilizer precipitates increases by 0.94%. It is known that

phosphorous-containing fertilizer used in agriculture in its composition should contain at least 50% of the acceptable forms of P_2O_5 from total P_2O_5 . All samples of received fertilizers based on MM and PF meet this requirement.

It is known that the amount of $CaO_{w.s}$ in the obtained precipitates plays an important role, since it determines the hygroscopic nature of the received fertilizers. Therefore, the content of the fertilizer obtained should be minimal. Fig. 2 shows the effect of the temperature of washing water and the concentration of nitric acid on $CaO_{w.s}$ content in samples of fertilizer precipitates obtained on the basis of MM and PF.

Content $CaO_{w.s}$ in the obtaining fertilizer precipitates should be no more than 4.50%. This data shows that $CaO_{w.s}$ content in all acid concentrations and temperature of washing water is from 4.01 to 4.53% and from 4.23 to 4.69% for precipitates is appropriate on the basis of MM and PF. At the optimal temperature of washing water, it is from 4.03 to 4.26% for MM and from 4.25 to 4.48% for PF.

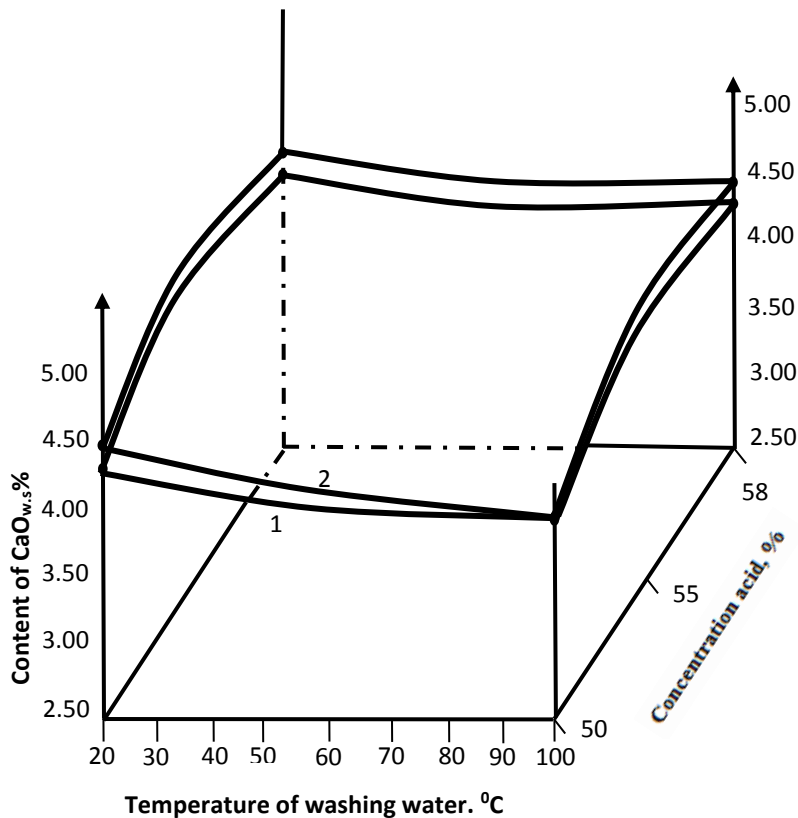


Fig. 2. Influence of the temperature of washing water and the concentration of nitric acid on the content of $CaO_{w.s}$. In samples of fertilizer precipitates: 1-based on MM; 2- based on PF.

4. CONCLUSION

The possibility of involving poorer raw materials with P_2O_5 (PM) and phosphate waste (MM) in nitric acid processing has been shown. The influence of the temperature of washing water and the concentration of nitric acid on the qualitative parameters of single fertilizers - fertilizer precipitate, obtained on the basis of mineralized mass (MM) and phosphorite flour (PF) has been studied. The optimal temperature of washing water was determined, which leads an improvement in the quality of the fertilizer precipitate. The optimal temperature of washing water can be considered 80-90°C of both phosphate raw materials (PRM). Under optimal parameters, the following formulations of fertilizer precipitates (weight.%) are obtained: P_2O_5 total.=21.60-22.65; P_2O_5 acceptable by citric acid=11.42-12.19; P_2O_5 water solubility=2.08-2.20; CaO total.=40.89-41.40; CaO acceptable by citric acid.=22.12-23.07; CaO water solubility=4.03-4.26; N total.=1.69-1.79 based on MM and P_2O_5 total.=24.86-26.07; P_2O_5 acceptable by citric acid=13.19-14.08; P_2O_5 water solubility=2.40-2.57; CaO total=45.19-45.41; CaO acceptable by citric acid=24.54-25.37; CaO water solubility =4.25-4.48; N total.=1.78-1.90 based on PF. The degree of washing of fertilizer precipitates from $Ca(NO_3)_2$ is 97.82-98.01 and 97.75-97.98%, respectively for MM and PF.

Based on laboratory experiments, the possibility of processing MM and PF for fertilizer precipitates that meet all the requirements of agriculture is shown.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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