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A.Zh. Allamuratova

A.U. Erkaev

A.M. Reimov

Saparbay Kazakhbaev

Dilshodbek Kurbiyazov

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IMPROVING TECHNOLOGY FOR PRODUCING COMPLEX FERTILIZER FROM LOW-GRADE PHOSPHORITES OF THE CENTRAL KYZYLKUM

A.Zh.Allamuratova¹, A.U.Erkaev², A.M.Reimov³, S.Kazakhbaev⁴, D.Kurbiyazov⁵

^{1,3,4,5}Karakalpak State University. Address: 230100, Street Abdirrov, 1, Nukus, Uzbekistan, Republic of Karakalpakstan.

²Tashkent Institute of Chemical Technology. Address: 100011, Navoi Street, 32, Tashkent, Uzbekistan.

Abstract: The methods of nitric acid processing of phosphate raw materials are analyzed. A technology has been developed for the nitric acid processing of phosphate raw materials into nitrogen-phosphorus fertilizer with the implementation of the process in a condensed pulp by adding sulfuric acid. A technology for producing complex fertilizers with a wide range of $N:P_2O_5:K_2O$ ratios with improved physico-chemical parameters due to the introduction of various potassium salts into the process is proposed.

Keywords: Complex fertilizers, phosphate raw materials, nitric acid, sulfuric acid, nitrogen-phosphorus fertilizer, calcium, nitrocalcium phosphate fertilizers, Central Kyzylkum.

Annotatsiya: Fosfat xom ashyosini nitrat kislota yordamida qayta ishlash usullari tahlil qilingan. Sulfat kislota qo'shilishi orqalin quyultirilgan pulpada jarayonni amalga oshirish bilan fosfat xom ashyosini azotli fosforli o'g'itga nitrat kislota yordamida qayta ishlash texnologiyasi ishlab chiqilgan. Jarayonga turli xil kaliy tuzlarini kiritish orqali yaxshilangan fizik-kimyoviy ko'rsatkichlar va $N:P_2O_5:K_2O$ nisbatlarining keng diapazoniga ega murakkab o'g'itlar ishlab chiqarishning texnologik sxemasi taklif etilgan.

Tayanch so'zlar: kompleks o'g'itlar, fosfat xom ashyosi, nitrat kislota, sulfat kislota, azotli fosforli o'g'itlar, kaltsiy, nitrokalsiy fosfatli o'g'itlar, Markaziy Qizilqum.

Аннотация: Проанализированы методы азотнокислотной переработки фосфатного сырья. Разработана технология азотнокислотной переработки фосфатного сырья на азотнофосфорное удобрение с осуществлением процесса в пульпе, сгущенной за счет добавления серной кислоты. Предложена технологическая схема производства сложных удобрений с улучшенными физико-химическими показателями и широким диапазоном соотношения $N:P_2O_5:K_2O$ за счет введения в процесс различных солей калия.

Ключевые слова: Комплексные удобрения, фосфатное сырье, азотная кислота, серная кислота, азотнофосфорное удобрение, кальций, нитрокальцийфосфатные удобрения, Центральные Кызылкумы.

Introduction

Our country has a powerful raw material base capable of practically meeting the needs of the national economy in mineral raw materials. One of the factors accelerating the agro-industrial complex is the effective use of mineral fertilizers used in agriculture. An important place in this is occupied by the expansion of the range of mineral fertilizers produced, the creation of cost-effective and environmentally efficient production technologies through the efficient use of raw materials reserves, as well as their introduction into the industry.

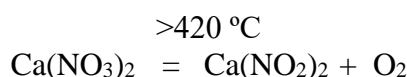
It is known that the current demand in our republic for phosphorus fertilizers is 25-30%. Because there are relatively few high-quality raw materials for obtaining the necessary phosphorus fertilizers. One of the most pressing issues is processing low-grade phosphorus-containing raw materials with the best possible technical and financial metrics in order to fulfill the expanding requirements of agriculture for phosphorus fertilizers. Since 2005, Samarqandkimyo OJSC has operated a pilot plant to test the technology for producing nitrocalcium phosphate fertilizers (NCPF) from unenriched phosphate rock Tashkura (16-18% P_2O_5) /1-7/.

Experimental

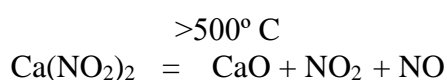
The process technology is based on the principle of decomposition of phosphate raw materials with non-concentrated nitric acid (57% HNO_3) within the limits of 55-65% of stoichiometry for complete binding of all CaO phosphate, evaporation of the resulting pulp without separating calcium nitrate from it while simultaneously neutralizing residual acidity in gaseous ammonia, granulation and drying of the resulting product.

The pulp evaporated in contact-type devices has the following composition (wt.%): P_2O_5 -8-9; N-5-6, pulp density and temperature - 1.7-1.8 t/m³ and 75 °C, respectively.

The transition on the design technological schemes of the granulation and drying unit for the production of ammophos using BGS devices to the NCPF mode should be carried out based on the special properties of nitrophosphates. The solid phase of the product mainly consists of dicalcium phosphate (20-25%), monocalcium phosphate (1-5%), calcium nitrate (50-55%) based on the anhydrous forms of salts. Calcium nitrate is thermally stable at temperatures below 420°C. With an increase in temperature above 420°C, even with short contacts of small particles with flue gases, due to their thermal stability, they undergo thermal decomposition with the formation of calcium nitrite and the release of free oxygen:



Further heating of the mass leads to the decomposition of nitrite with the formation of nitrogen oxides:



The occurrence of these reactions is involved in the beginning of the process of so-called “cigar-shaped combustion”. The entire mass inside the apparatus begins to burn without a flame, leaving behind a white powder (CaO), which leads to loss of product. In addition, the gas phase becomes very aggressive towards the materials of the BGS, gas paths and fans and intense corrosion begins.

The second feature of the mixture of NCPF salts during drying is also due to the presence of calcium nitrate crystal hydrates in it. In cases of spraying NCPF pulp into a drum, when the water content reaches more than 25% and the temperature is below 70°C, during the evaporation of water from the adhering layer of pulp on the surfaces of the rotor particles the tetrahydrate form of calcium nitrate $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ crystallizes. As the temperature rises (>70°C), the tetrahydrate, releasing one molecule of water, begins to melt in it (incongruent melting) and over time the entire mass inside the apparatus turns into a flowing pulp [6-7].

There are known methods for processing nitric acid pulp by removing excess calcium by introducing various components [3]. When using sulfuric acid or sulfates to separate excess calcium from nitric acid pulp, a product can be obtained containing P_2O_5 partially or completely in a water-soluble form [8]. This is achieved by binding the required amount of calcium in the form of sulfate, released into the solid phase from the solution, and the remaining calcium is not enough to form completely dicalcium phosphate and therefore all or parts of the phosphorus remain in water-soluble form.

According to the nitric-sulfuric acid method, phosphate decomposition is carried out with a mixture of nitric and sulfuric acids in a molar ratio.

Usually, 47-50% nitric and 93% sulfuric acid are used. The total rate of acids (in equivalents) is 130-160% of the stoichiometric amount. To reduce the release of oxides and reduce corrosion of equipment, the phosphate is first decomposed with nitric acid, and then sulfuric acid is added and then the resulting pulp is neutralized with ammonia.

The process is most often carried out in devices similar to those described above to produce nitrogen-phosphorus fertilizers using the nitric-sulfuric acid method. In this case, the phosphate is decomposed in the first two reactors with nitric acid, sulfuric acid is added to the third and fourth reactors, and in subsequent reactors the pulp is neutralized with ammonia, mixed with ammonia or lime

and dried in a drum dryer. By changing the amount of sulfuric acid introduced into the system, you can vary the proportion of water-soluble P_2O_5 in the fertilizer.

Although in terms of nitrogen – with the sulfuric acid method and it is possible to obtain a fairly cheap product with a favorable ratio of nitrogen and phosphorus, containing part of P_2O_5 in water-soluble form, however, the use of sulfuric acid in the process does not always seem rational. This is explained primarily by the fact that sulfuric acid is used here mainly only to remove excess calcium from the nitric acid extract.

Methodology for introducing sulfuric acid into the process in various schemes. The decomposition of phosphates with a mixture of nitric and sulfuric acids varies: either sulfuric acid is introduced mixed with nitric acid (in different proportions), or first sulfuric acid, then nitric, or sulfuric acid together with ammonia after partial decomposition of the phosphate with nitric acid, sometimes even after partial neutralization with ammonia.

When individual acids are introduced, simultaneously or as a mixture, natural phosphate is decomposed during the reaction by three acids (nitric, sulfuric and the resulting phosphoric). The chemical energy of sulfuric acid will decrease rapidly as calcium sulfate is formed, and phosphoric acid will be suppressed as the concentration of its salts in solution increases. When introducing first nitric acid and then sulfuric acid, the process of decomposition of a part of the phosphate with nitric acid will actually end before introduction of sulfuric acid.

By adding sulfuric acid to a pulp containing calcium nitrate and phosphate, as well as phosphoric acid, it is possible to create less favorable conditions for the release of nitrogen oxides, because an aggressive mixture of sulfuric and nitric acids does not form in the reactor. In this case, the corrosive ability of the resulting pulp decreases.

Tashkur phosphorites has very poor physical properties: it is highly hygroscopic, smears, dissipates poorly and are not suitable for mechanized application to the soil and fertilizer mixing. To improve the physicochemical properties of NCPF, it is necessary to improve the technological process of production and explore various options for its conditioning. The developed technology for nitric acid processing of carbonized phosphorites with the addition of retour or water can significantly improve the physicochemical properties of NCPF, but this is an insufficient measure/1/. Therefore, it is additionally necessary to coat its granules with effective and accessible inorganic salts. Inorganic salts and their mixtures with organic substances can be used as modifying additives. They inhibit crystallization or dissolution during storage, change its hygroscopicity or impede polymorphic transformations.

We have chosen methods for improving the physicochemical properties NCPF: deep drying and rapid cooling; treatment of NCPF with inert additives; coating of NCPF granules with sulfate, phosphate and carbonate salts in the presence and absence of organic substances. After the completion of the main production processes for obtaining crystalline products during their storage in warehouses and transportation, various secondary physical and chemical processes occur: sorption of moisture from the air or drying, recrystallization due to hydration, dehydration or polymorphic transformations, etc. This can lead to a significant change in the consumer qualities of crystalline products. Products, particle sizes and flowability. To improve the physicochemical properties of products, water-insoluble inert hydrophilic mineral powders, organic hydrophobic and polymeric substances, and surfactants are used as surface modifying or conditioning additives that reduce caking of crystalline or granular mineral fertilizers.

These substances absorb moisture located on the surface of the grains and, thereby, prevent the occurrence of phase contacts. Therefore, these additives must be hygroscopic and have a large moisture capacity.

The chemistry of the process for producing nitrocalcium phosphate fertilizer (NCPF) and physical and chemical research in this area show that NCPF is a very complex system consisting of a solid and liquid phase, in which a number of changes occur: decomposition, ripening, retrogradation, evaporation, moisture absorption, crystallization of salts, etc.

Taking into account the above, we have developed a method in which decarbonization and decomposition of phosphate raw materials with nitric acid with a concentration of at least 45% and a rate of 25-100% are carried out in one apparatus, and the time of its supply to the reaction zone is carried out in equal portions for 2-4 minutes at active mixing.

Then sulfuric acid is supplied to the resulting pure nitric acid pulp, due to which the phosphate raw material is further decomposed and dried. In the resulting reaction pulp, the content of the liquid phase is 2-4 times less compared to the known technology, which facilitates the process in a thick pulp /4/. Therefore, during the decomposition of carbonate-containing phosphate raw materials, abundant foaming and the release of nitrogen oxides into the gas phase are not observed. The resulting thick mass or its mixture with neutralizing additives is fed into the mixer, where retur is simultaneously supplied in the amount necessary to maintain moisture in the mass of 5-25%, for the purpose of granulating it in a plate (or screw) granulator or 5-30% of water from weight of the entire pulp mixture in order to carry out the process of granulation and drying on the BGS.

Practice shows that the process [1-3] granulation in BGS is technologically difficult to implement, because the resulting product exhibits poor physical and chemical properties.

As shown earlier / 2/ the content of total and free water in the decomposition products depends on the rate and concentration of nitric acid. Moreover, at a humidity of 23.0%, a creamy consistency is formed, which complicates further processing of the pulp. It is better to carry out granulation in a screw or disk granulator in the presence of a retour.

Experimental data have shown that with a retur multiplicity and a moisture content of 23% in the initial product, the moisture content of the resulting mixture approaches 1.5-2.2%, i.e. The granulation process can be carried out on a plate or screw granulator.

The work [9-14] shows the influence of inorganic salts on the physicochemical properties of nitrogen-phosphorus fertilizers obtained on the basis of nitric acid decomposition of phosphorites of the Central Kyzylkum.

Table 1

Name components	Chemical composition of feedstock								
	Content of components, mass. %								
	P ₂ O ₅	CaO	Al ₂ O ₃ _ _ _	Fe ₂ O ₃ _ _ _	Mg O	F	CO ₂ _	K ₂ O	HNO ₃ _ _
Phosphorite flour	17.20	46.30	1.23	1.04	1.70	1.98	15.5	-	-
Washed Concentrate	24.10	46.40	0.34	0.47	0.93	2.44	9.5	-	-
Dust-like Fraction	18.55	45.01	0.96	0.78	0.90	2.2	15.0	-	-
Nitrogen acid	-	-	-	-	-	-	-	-	56.0
Chloride Potassium	-	-	-	-	-	-	-	60.0	-

In this case, the best additives turned out to be potassium salts, especially sulfate ones.

It has been established that with the consumption of 3- 12 r H₂SO₄ per 100 g of product, the hygroscopicity of the product crystals goes from highly hygroscopic to hygroscopic.

Results and discussion

In the experiments carried out, various types of phosphate raw materials of the Central Kyzylkum, sulfuric and nitric acid, potassium chloride and ammonia, the composition of which is given in Table 1.

The experiments were carried out in a laboratory setup consisting of a reactor placed in a thermostat with stirrers.

The rates of nitric and sulfuric acid, as well as their total rates, varied within 5-40, 7-92 and 22-123%, respectively; the influence of rates on the degree of decomposition of phosphate raw materials, the content of nutrients and the moisture content of the resulting fertilizer was studied (Table 2, 3).

Within the varying norms of acids, the following indicators were obtained: P_2O_5 total - 8.40-13.5%; $P_2O_{5ass} / P_2O_{5total} = 0.30$: 0.95%; N_{total} - 0.90-7.17 %; sum of nutritional components - 12.85-16.8%; product humidity without the drying stage is 0.1-12.9%;

Products, obtained with a total rate of acid reagents of no more than 47%, are activated nitrogen-phosphorus fertilizers, since the ratio of P_2O_5 dry / P_2O_5 total does not exceed 50%, despite the fact that the sum of nutritional components reaches up to 16.33% with a content of up to 4.43% N_{tot} .

With an increase in the rate of sulfuric acid from 23 to 93 % at the same rates of nitric acid P_2O_{5ass} / P_2O_5 general increases from 40, 50, 60 to 91, 92, 95%, product moisture content decreases from 3; 3.6; 12.9 to 0.1; 0.3; 7.98, respectively, for 10, 20, 30% nitric acid.

Increasing the nitric acid rate by more than 30% and the sulfuric acid rate by more than 70% is undesirable, because the moisture content of the product exceeds 6.03% and it becomes strongly acidic, which requires deep ammoniation, and the content of nutritional components decreases.

Sieve analysis of the resulting product shows that the particle size distribution strongly depends on the moisture content of the product and the norms of sulfuric acid (Table 3).

With increasing product humidity, the proportion of the 2-3 mm fraction reaches 56; 60, and 58% at sulfuric acid rates of 96, 74, 60.5%, respectively.

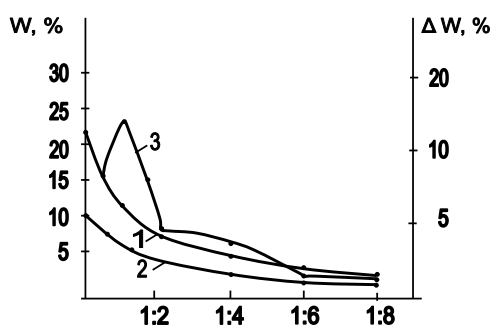


Fig. 1. Changes in the moisture content of the product depend on the return rate.

The moisture content of the product depends on the return rate (Table 3). When the retur ratio is from 1:6 to 1:8, the moisture content of the product is minimal and is less than 1.5%.

Technology has been developed that allows variation in the composition of complex compounds fertilizers with improved physicochemical properties within a wide range of ratios of nutritional components (Table 4).

The process includes decarbonization and acid decomposition of phosphate raw materials simultaneously (in one apparatus) at a rate of 25-100% of stoichiometry in terms of CaO, neutralization to a pH of at least 3.5, evaporation of the pulp to a density of at least 1750 kg/ m³, adding potassium salts at a mass ratio of N:P₂O₅:K₂O equal to (0.5-1.5):1.0:(0.5-1.5), granulation in the presence of retour and cooling. The amount of nitric acid and potassium salts on 100 kg phosphorite at a given mass ratio N:P₂O₅:K₂O is calculated by the formula:

$$A_{HNO_3} = (C_{P_2O_5} * 63/14) * (100 / C_{HNO_3}) * (P_N / P_{P_2O_5});$$

$$A_{f/s} = 100 * P_{P_2O_5};$$

$$A_{k.sol} = 0.5 (C_{P_2O_5} * M_{k.salt} / C_{K_2O}) * (P_{K_2O} / P_{P_2O_5});$$

where; A_{HNO_3} , $A_{f/s}$, $A_{potassium\ salt}$ - the amount of nitric acid, phosphate raw materials and potassium salts; $C_{P_2O_5}$, C_{K_2O} - content of P₂O₅ and K₂O in phosphate raw materials and potassium salt, respectively; C_{HNO_3} - concentration of nitric acid; M_{salt} - m alecular mass of potassium salts; P_N , $P_{P_2O_5}$, P_{K_2O} - numerical values given mass ratio of N, P₂O₅, K₂O, respectively.

Table 2

Composition of products of nitrogen and sulfuric acid processing of rows of phosphate rock in the Central Kyzylkum region

No.	Nitric acid		Sulfuric acid		Amount norm to islot, %	Content P ₂ O ₅ wt. %, %		Content nitrogen, wt. %, %			Degree decarbon - ization %	Sum . p ita- body comp. %
	Con- cent- walkie- talkie %	Norma %	Con- cent- walkie- talkie, %	Norma %		Genera l _	Ass. %	P ₂ O ₅ General _ / . P ₂ O ₅ Usv .	Nit.	And ammon iacal.	General	
1.	34	11	92	23	33	13.50	5.4	40.1	1.97	-	1.97	15.47
2.	54	11	92	70	80	10.54	8.22	78.2	15.7	1.50	3.04	13.58
3.	54	11	92	93	103	9.36	8.55	91.3	1.37	2.90	4.27	13.63
4.	54	21	92	23	45	12.80	6.40	50.4	3.75	1.05	4.80	17.6
5.	54	21	92	70	91	9.34	7.66	82.3	2.73	1.81	4.54	13.88
6.	54	21	92	93	114	8.43	7.76	92.1	2.46	3.01	5.47	13.9
7.	54	31	92	23	53	11.54	6.92	60.1	5.05	1.21	6.26	17.8
8.	54	31	92	70	100	8.89	8.0	90.1	3.89	2.02	5.9	14.79
9.	54	31	92	93	123	8.14	7.73	95.1	3.56	3.51	7.07	15.21
10	54	15	92	58.5	73.5	10.61	7.75	73.4	2.17	2.03	4.20	14.81
11	54	15	92	11.7	20.7	14.82	5.04	34.1	3.03	-	3.03	17.85
12	54	15	65	32.7	47.7	13.31	6.79	51.1	2.73	1.15	3.88	17.19
13	54	16	65	7.6	23.7	14.68	4.55	31.1	3.24	-	3.24	17.92
14	54	16	65	15.2	31.2	13.82	5.25	38.2	3.03	-	3.03	16.85
15	50	16	65	22.8	38.8	12.94	5.69	44.3	2.83	1.03	3.86	16.80
16	50	15	65	7.6	22.6	13.70	4.11	30.4	2.78	-	2.78	16.48
17	50	10	65	15.2	25.2	13.93	4.60	33.4	1.88	-	1.88	15.81
18	50	5	65	22.8	27.8	13.93	4.88	35.2	0.94	-	0.94	14.87

Table-3

The composition of the products of nitric acid processing of rows of phosphate rock in the Central Kyzylkum region

Sample numbers correspond intentions tab 1	pH	Moistness, %	Product composition % _			
			3 mm _	2 mm _	0.5 mm _	< 0.5 mm
1.	3.6	3.1	26	30	34	20.5
2.	0.2	0.5	24	36	25	10
3.	-	0.2	20	38	26	15
4.	1.0	3.6	33	44	15	16
5.	-	1.6	36	47	4.7	7.4
6.	-	0.3	30	34.4	21.6	12.2
7.	0.8	12.9	41.0	45.7	6.8	14.0
8.	-	0.1	46.0	28.1	14.4	6.5
9.	-	8.0	36	24.5	27.0	11.5
10	0.3	0.2	46.0	27.3	26.8	12.5
11	4.1	13.8	33.5	59.1	6.7	5.8
12	0.9	3.8	20.9	62.6	10.1	0.7
13	4.4	10.8	28.5	57.9	2.8	3.3
14	3.3	11.0	33	42.9	24.3	11.41
15	2.0	11.2	37	40.6	14.9	01
16	4.2	11.7	-	-	-	7.5
17	4.5	8.40	-	-	-	-
18	4.0	6.9	-	-	-	-

Table-4

Composition of complex fertilizers obtained from phosphorites of the Central Kyzylkum

Mass. with relation. N :P ₂ O ₅ :K ₂ O	Type of phosphorites	View potassium salts	Norm, HNO ₃ , %	Mass ratio original components A _{NNO3} : A _{f/s} : A _{k.s ol}	Contents of components %			Amount of nutrients components, %
					N	P ₂ O ₅	K ₂ O	
1:1:1	Washed dried. Concentrate	K ₂ SO ₄ _	103.5	192.9:100:77.59	8.84	8.83	8.84	26.5
1.5:1:1			160.0	298.3:100:77.59	10.89	7.26	7.3	25.5
1:1.5:1			103.5	192.9:150:77.59	7.2	10.76	7.2	25.5
1:1:1	-	KCl	103.5	192.9:100:56.86	9.53	9.53	9.53	28.5
1.5:1:1			160.0	298.3:100:56.86	11.6	7.72	7.7	27.0
1:1.5:1			103.5	192.9:150:56.86	7.7	11.43	7.7	27.0
1:1:1	-	K ₂ CO ₃	103	192.9:100:48.86	9.84	9.84	9.84	29.5
1.5:1:1			160	298.3:100:48.86	11.9	7.92	7.92	27.7
1:1.5:1			103	192.9:150:48.86	7.9	11.9	7.9	27.7
1:1:1	Dust fraction	K ₂ SO ₄	79	148.8:100:59.74	7.62	7.62	7.62	22.86
1.5:1:1			119	223.47:100:59.74	9.76	6.51	6.51	22.78
1:1.5:1			79	1489.48:150:59.74	6.33	9.49	6.33	22.78
1:1:1		KCl	79	148.8:100:43.78	8.2	8.16	8.2	24.6
1.5:1:1			119	223.47:100: 43.78	10.3	6.9	6.89	24.1
1:1.5:1			79	1489.48:150:43.78	6.9	10.3	6.9	24.1

As a potassium additive, its sulfate, nitrate, phosphate, carbonate, chloride salts and their mixtures are used, the hygroscopic efficiency of which on a 10-point scale was as follows:

Components	Points (α)	Qualitative assessment of hygroscopicity
K ₂ SO ₄	>1	Almost non-hygroscopic
K ₂ HPO ₄	>1	Almost non-hygroscopic
K ₂ CO ₃	>1	Almost non-hygroscopic
KNO ₃	(3.23) 3-5	Hygroscopic
KCl	(3.62) 3-5	Hygroscopic

Potassium salts are added to the mixer, where the evaporated melt is simultaneously supplied in the amount necessary to maintain the mass ratio $N:P_2O_5:K_2O$ equal to $(0.5 - 1.5) : 1.0 : (0.5 - 1.5)$.

Process granulation in the presence of retur must be carried out to maintain the moisture content in the mass of no more than 1-8%.

Decrease in melt density – to less than 1750 kg/m^3 leads to a complication of the granulation process and a deterioration in the properties of the resulting fertilizers due to an increase in their moisture content.

A decrease in the mass ratio $0.5:1.0:0.5$ leads to a complication of the decomposition process and a decrease in the decomposition coefficient of phosphorite .

With increasing mass ratio above $1.5:1.0:1.5$ the degree of decomposition of phosphorite and the amount of $P_2O_{5\text{ass}}$ decrease. as part of a complex fertilizer, the consumption of nitric acid and, accordingly, ammonia to neutralize the acid increases.

Conclusion

Thus, as a result of the implementation of the proposed option, while maintaining all the advantages of existing /1-3/ methods, the drying stage is reduced, the technology allows regulate the compositions of complex fertilizers within a wide range of the ratio of nutritional components and its physical and chemical properties. The resulting complex fertilizer has good agrochemical properties for all types of crops.

Technical and economic calculations indicate the profitability of the proposed method . Economies are achieved by improving the quality of the product and the use of cheap highly carbonized phosphorites (raw phosphate ore, mineralized mass, washed dried concentrate and dust fraction).

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