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Improvements of the Technology of Obtaining Complex Fertilizer From Low-Grade Phosphorites in the Central Kyzylkum

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ABSTRACT: The methods of nitric acid processing of phosphate raw materials are analyzed. A technology has been developed for nitric acid processing of phosphate raw materials for nitrogen-phosphorus fertilizer with the implementation of the process in a thickened pulp by adding sulfuric acid.

A technology for producing complex fertilizers with a wide range of N:P2O5:K2O ratios with improved physical and chemical parameters due to the introduction of various potassium salts into the process is proposed.

KEYWORDS: phosphorite, fertilizers, nitric acid, nitric acid processing, pulp, granule.

I. INTRODUCTION

Since 2005, JSC Samarqandkimyo has been using a pilot plant to develop the technology for producing nitrocalcium phosphate fertilizers (NKF) from unenriched Tashkur phosphate rock (16-18% P2O5) [1-7].

The process technology is based on the principle of decomposition of phosphate raw materials with non-concentrated nitric acid (57% HNO3) within the limits of 55-65% of the stoichiometry for complete binding of all CaO phosphate, evaporation of the resulting pulp without separating calcium nitrate from it, while simultaneously neutralizing the residual acidity with gaseous ammonia, granulation and drying of the resulting product.

The slurry evaporated in contact-type apparatuses has the following composition (mass %): P2O5-8-9; N-5-6, pulp density and temperature - 1.7-1.8 t/m3 and 75ºC, respectively.

The transition on the design flow diagrams of the granulation and drying unit for the production of ammophos using BGS devices to the NKFU 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 anhydrous salt forms. Calcium nitrate is thermally stable at temperatures below 420ºC. With an increase in temperature above 420ºC, 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:

$$Ca(NO_3)_2 \overset{>420^{\circ}C}{=} Ca(NO_2)_2 + O_2$$

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

$$Ca(NO_2)_2 \overset{>500^{\circ}C}{=} CaO + NO_2 + NO$$

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

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II. SIGNIFICANCE OF THE SYSTEM

The methods of nitric acid processing of phosphate raw materials are analyzed. The study of methodology is explained in section III, section IV covers the experimental results of the study, and section V discusses the future study and conclusion.

III. METHODOLOGY

The second feature of the mixture of NKFU salts during their drying is also due to the presence of calcium nitrate crystalline hydrates in it. the tetrahydrate form of calcium nitrate Ca(NO3)2 * 4H2O. As the temperature rises (>70 ºC), 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 fluid pulp [6-7].

Known methods of processing nitric acid pulp by removing excess calcium by introducing various components [3]. When sulfuric acid or sulfates are used to isolate excess calcium from nitric acid pulp, it is possible to obtain a product containing P2O5 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 part of the phosphorus remains in a water-soluble form.

IV. EXPERIMENTAL RESULTS

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

Usually used 47-50% nitric and 93% sulfuric acid. 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, 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 apparatuses similar to those described above for the production of nitrogen-phosphorus fertilizers using the nitrogen-sulphuric acid method. In this case, 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, it is possible to vary the proportion of water-soluble P2O5 in the fertilizer.

Although it is possible to obtain a fairly cheap product with a favorable ratio of nitrogen and phosphorus, containing a part of P2O5 in a water-soluble form, using the nitric-sulfuric acid method, nevertheless, the use of sulfuric acid in the process does not always seem rational. This is primarily due to the fact that sulfuric acid is used here mainly only to remove excess calcium from the nitric acid extract.

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

With the introduction of individual acids, simultaneously or as a mixture, natural phosphate decomposes in the course of the reaction by three acids (nitric, sulfuric and phosphoric). The chemical energy of sulfuric acid will decrease rapidly as calcium sulfate is formed, and phosphoric acid will be suppressed by increasing the concentration of its salts in solution. With the introduction of nitric acid first, and then sulfuric acid, the decomposition of part of the phosphate by nitric acid will actually end before the introduction of sulfuric acid.

By adding sulfuric acid to a pulp containing calcium nitrate and phosphate, as well as phosphoric acid, less favorable conditions for the release of nitrogen oxides can be created, because. in fact, an aggressive mixture of sulfuric and nitric acids is not formed in the reactor. In this case, the corrosive ability of the resulting pulp is reduced.

NKFU from Tashkur phosphorites has very poor physical properties: it is highly hygroscopic, smears, poorly disperses and is not suitable for mechanized application to the soil and fertilizer mixing. To improve the physicochemical properties of NKFU, it is necessary to improve the technological process of obtaining and explore various options for its conditioning. The developed technology of nitric acid processing of carbonized phosphorites with the addition of retur or water can significantly improve the physicochemical properties of NKFU, but this is an insufficient measure /1/. Therefore, it is additionally necessary to coat its granules with effective and accessible inorganic salts. Inorganic salts

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and their mixtures with organic substances can be used as modifying additives. They inhibit crystallization or dissolution during storage, change its hygroscopicity or hinder polymorphic transformations. We have chosen the following ways to improve the physicochemical properties of NKFU: deep drying and rapid cooling; processing of NKFU with inert additives; coating of NKFU 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: moisture sorption 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, particle sizes and flowability. To improve the physical and chemical properties of products, as surface modifying or conditioning additives that reduce caking of crystalline or granular mineral fertilizers, water-insoluble inert hydrophilic mineral powders, organic hydrophobic and polymeric substances, and surfactants are used.

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

The chemistry of the process of obtaining nitrocalcium phosphate fertilizer (NKFU) and physicochemical studies in this area show that NKFU is a very complex system consisting of a solid and liquid phase, in which a number of changes occur: decomposition, maturation, retrogradation, evaporation, moisture absorption, salt crystallization, etc.

In view of the foregoing, 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 norm 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 fed into the obtained pure nitric acid pulp, due to which the additional decomposition of the phosphate raw material and its drying takes place. In the resulting reaction pulp, the content of the liquid phase is 2-4 times less compared to the known technology, which contributes to 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 at the same time retur is supplied in the amount necessary to maintain moisture in the mass of 5-25%, in order to granulate it in a plate-shaped (or screw) granulator or 5-30% of water is supplied from the weight of the entire mixture of pulp 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 physico-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, and at a moisture content of 23.0%, a creamy consistency is formed, which makes it difficult to further process the pulp. It is more expedient to carry out granulation in a screw or disc granulator in the presence of a retur.

Experimental data showed that with a recycle ratio 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 effect 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
Chemical composition of feedstock

Name components	Content of components, mass. %								
	P_2O_5	CaO	Al_2O_3	Fe_2O_3	MgO	F	CO_2	K_2O	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	-	-
pulverized fraction	18,55	45,01	0,96	0,78	0,90	2,2	15,0	-	-
Nitrogen acid	-	-	-	-	-	-	-	-	56,0
chloride Potassium	-	-	-	-	-	-	-	60,0	-

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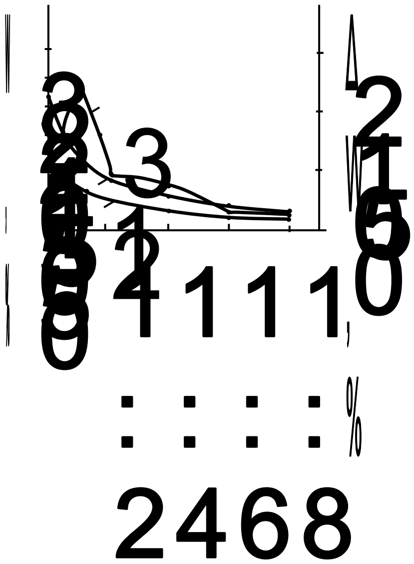
At the same time, the additives of potassium salts, especially sulfate salts, turned out to be the best. It has been established that at a consumption of 3-12 g of H2SO4 per 100 g of the product, the hygroscopicity of the product crystals changes from highly hygroscopic to hygroscopic. In the experiments, various types of phosphate raw materials from the Central KyzylKum, sulfuric and nitric acids, potassium chloride and ammonia were used, the composition of which is given in Table 1. The experiments were carried out on a laboratory setup consisting of a reactor with stirrers placed in a thermostat. The norms of nitric and sulfuric acid, as well as their total norms, varied within 5-40, 7-92 and 22-123%, respectively; 3). Within the range of acid norms, the following indicators were obtained: $P2O5_{total}$ - 8.40-13.5%; $P2O5_{ass}P2O5_{total}$.=0.30:0.95%; N_{total} - 0.90-7.17%; the amount of nutrients - 12.85-16.8%; moisture content of the product without drying stage - 0.1-12.9%; Products. obtained with a total rate of acidic reagents of not more than 47%, are activated nitrogen-phosphorus fertilizers, since the ratio of $P2O5_{ass}/P2O5_{total}$ does not exceed 50%, despite the fact that the sum of nutrient components reaches up to 16.33% at a content of up to 4.43% N_{total}. With an increase in the norm of sulfuric acid from 23 to 93% at the same norms of nitric acid $P2O5_{ass}$/ $P2O5_{total}$. increases from 40, 50.60 to 91.92.95%, product moisture decreases from 3; 3.6; 12.9 to 0.1; 0.3; 7.98, respectively, for 10, 20, 30% norms of nitric acid. Increasing the norm of nitric acid more than 30% and the norm of sulfuric acid more than 70% is undesirable, because the moisture content of the product exceeds 6.03% and it becomes strongly acidic, which requires deep ammonization, and the content of nutrients decreases. Sieve analysis of the obtained product shows that the granulometric composition strongly depends on the moisture content of the product and the norms of sulfuric acid (Table 3). With an increase in the moisture content of the product, the proportion of the 2-3 mm fraction reaches 56; 60, 58% at sulfuric acid rates of 96, 74, 60.5%, respectively.



Rice . Changes in the moisture content of the product in depends on the multiplicity of reture

The moisture content of the product depends on the recycle ratio (Table 3). With a reture ratio of 1:6 to 1:8, the moisture content of the product is minimal and is less than 1.5%.

A technology has been developed that allows varying the composition of complex fertilizers with improved physical and chemical properties over a wide range of the ratio of nutrient 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, pulp evaporation to a density of at least 1750 kg/m3, addition of 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 retur and cooling.

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Table 2

The composition of the products of nitric-sulfuric acid processing of ordinary phosphate rock of the Central Kyzylkum

№	Nitric acid		Sulphuric acid		total norm acids,%	Content P_2O_5 wt,%		P_2O_5 total./. P_2O_5 Ass.	Content nitrogen, wt.%			Degree decarbonization %	Sum. pitatelny component.%
	Concen tration %	Norm, %	Concen tration, %	Norm, %		total.	Ass.		Nit.	Amm onia.	Total.		
1.	34	11	92	23	33	13,50	5,4	40,1	1,97	-	1,97	75	15,47
2.	54	11	92	70	80	10,54	8,22	78,2	15,7	1,50	3,04	95	13,58
3.	54	11	92	93	103	9,36	8,55	91,3	1,37	2,90	4,27	97,5	13,63
4.	54	21	92	23	45	12,80	6,40	50,4	3,75	1,05	4,80	86	17,6
5.	54	21	92	70	91	9,34	7,66	82,3	2,73	1,81	4,54	96	13,88
6.	54	21	92	93	114	8,43	7,76	92,1	2,46	3,01	5,47	98	13,9
7.	54	31	92	23	53	11,54	6,92	60,1	5,05	1,21	6,26	90	17,8
8.	54	31	92	70	100	8,89	8,0	90,1	3,89	2,02	5,9	97	14,79
9.	54	31	92	93	123	8,14	7,73	95,1	3,56	3,51	7,07	98	15,21
10	54	15	92	58,5	73,5	10,61	7,75	73,4	2,17	2,03	4,20	89	14,81
11	54	15	92	11,7	20,7	14,82	5,04	34,1	3,03	-	3,03	68	17,85
12	54	15	65	32,7	47,7	13,31	6,79	51,1	2,73	1,15	3,88	86,5	17,19
13	54	16	65	7,6	23,7	14,68	4,55	31,1	3,24	-	3,24	65,0	17,92
14	54	16	65	15,2	31,2	13,82	5,25	38,2	3,03	-	3,03	71,0	16,85
15	50	16	65	22,8	38,8	12,94	5,69	44,3	2,83	1,03	3,86	77,0	16,80
16	50	15	65	7,6	22,6	13,70	4,11	30,4	2,78	-	2,78	60,0	16,48
17	50	10	65	15,2	25,2	13,93	4,60	33,4	1,88	-	1,88	67,1	15,81
18	50	5	65	22,8	27,8	13,93	4,88	35,2	0,94	-	0,94	70,02	14,87

The amount of nitric acid and potassium salts per 100 kg of phosphorite at a given mass ratio N: P2O5: K2O is calculated by the formula:

A_{HNO3} \u003d (C_{P2O5} * 63/14) * (100 / C_{HNO3}) * (P_N / P_{P2O5});

$A_{f/s}$ = 100 * P P2O5;

$A_{k.salt}$ \u003d 0.5 (C_{P2O5} * $M_{c.salt}$/C_{K2O}) * (P_{K2O}/P_{P2O5});

where; A_{HNO3}, A f/s, Ak.salt - the amount of nitric acid, phosphate raw materials and potassium salts;

C_{P2O5}, C_{K2O}-contents of P2O5 and K2O in phosphate raw materials and potassium salt, respectively;

C_{HNO3} - concentration of nitric acid;

$M_{total.salt}$ - molecular weight of potassium salts

P_N, P_{P2O5}, P_{K2O} - numerical values of the specified mass ratio of N, P2O5, K2O, respectively.

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Table 3
The composition of the products of nitric acid processing of ordinary phosphorite flour of the Central Kyzylkum

Sample numbers correspond to the names of the tab. 1	pH	Humidity, %	Network composition of products %			
			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	-	-	-	-

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 turned out to be the following:

Components Points (α) Qualitative assessment of hygroscopicity

K_2SO_4 >1 Practically non-hygroscopic

K_2HPO_4. >1 Virtually non-hygroscopic

K_2CO_3 >1 Practically non-hygroscopic

KNO_3 (3.23) 3-5 Hygroscopic

KCl (3.62) 3-5 Hygroscopic

Potassium salts are added to the mixer, where at the same time stripped off melt enters 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).

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Table-4
The composition of complex fertilizers obtained from phosphorites of the Central Kyzylkum

Mass. ratio $N:P_2O_5:K_2O$	Type of phosphorites	View potash salts	Norm, HNO_3, %	Mass ratio original components A_{HNO3}: $A_{f/s}$: $A_{K.salt}$	Component content%			Nutrient amount components, %
					N	P_2O_5	K_2O	
1:1:1 1,5:1:1 1:1,5:1	Washed dried. concentrate	K_2SO_4	103,5 160,0 103,5	192,9:100:77,59 298,3:100:77,59 192,9:150:77,59	8,84 10,89 7,2	8,83 7,26 10,76	8,84 7,3 7,2	26,5 25,5 25,5
1:1:1 1,5:1:1 1:1,5:1	-	KCl	103,5 160,0 103,5	192,9:100:56,86 298,3:100:56,86 192,9:150:56,86	9,53 11,6 7,7	9,53 7,72 11,43	9,53 7,7 7,7	28,5 27,0 27,0
1:1:1 1,5:1:1 1:1,5:1	-	K_2CO_3	103 160 103	192,9:100:48,86 298,3:100:48,86 192,9:150:48,86	9,84 11,9 7,9	9,84 7,92 11,9	9,84 7,92 7,9	29,5 27,7 27,7
1:1:1 1,5:1:1 1:1,5:1	pulverized fraction	K_2SO_4	79 119 79	148,8:100:59,74 223,47:100:59,74 1489,48:150:59,74	7,62 9,76 6,33	7,62 6,51 9,49	7,62 6,51 6,33	22,86 22,78 22,78
1:1:1 1,5:1:1 1:1,5:1		KCl	79 119 79	148,8:100:43,78 223,47:100: 43,78 1489,48:150:43,78	8,2 10,3 6,9	8,16 6,9 10,3	8,2 6,89 6,9	24,6 24,1 24,1

The process of granulation in the presence of reture must be carried out to maintain the moisture content in the mass is not more than 1-8%.

Reducing the density of the melt to less than 1750 kg/m3 leads to a complication of the granulation process and a deterioration in the properties of the obtained fertilizers due to an increase in their moisture content.

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

With an increase in the mass ratio above 1.5 : 1.0 : 1.5, the degree of decomposition of phosphorite and the amount of P2O5usv decrease. as part of a complex fertilizer, the consumption of nitric acid and, accordingly, ammonia for acid neutralization increases.

V. CONCLUSION AND FUTURE WORK

Thus, as a result of the implementation of the proposed option, while maintaining all the advantages of the existing /1-3/ methods, the drying stage is reduced, the technology allows you to control the composition of complex fertilizers in a wide range of nutrient components ratio and its physico-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. Savings are achieved by improving the quality of the product and the use of cheap highly carbonized phosphorites (unenriched phosphate ore, mineralized mass, washed dried concentrate and pulverized fraction).

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