IJIAET International Journal of Innovative Analyses and Emerging Technology

| e-ISSN: 2792-4025 | http://openaccessjournals.eu | Volume: 1 Issue: 5

Production of Complex Fertilizers by the Method of Decomposition of Poor Kyzylkum Phosphorites with a Reduced Nitric Acid Rate

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ANNOTATION

It is known that nitric acid processing of phosphates is a more promising and economical way to obtain phosphorus-containing fertilizers. At the same time, nitric acid not only participates in the decomposition of phosphate raw materials, but also its anions remain in the final product in the form of a valuable nitrogen nutrient component. The process of obtaining phosphorus fertilizers consists in converting indigestible forms of phosphorus in phosphate raw materials into forms that are assimilable for plants. This is usually done by acidic methods, that is, the decomposition of raw materials with sulfuric, nitric, phosphoric or hydrochloric acids.

The nitric acid method of processing phosphate raw materials allows the complex use of raw materials components. The main advantage of nitric acid processing is that the chemical energy of nitric acid is not only used for the decomposition of phosphorites, but also NO_3- anions in the form of a nutrient component remain in the composition of complex fertilizers.

Key words: nitric acid, acid rate, decomposition, rheological properties, pulp, fertilizer, reactor.

The nitric acid method of processing phosphate raw materials allows the complex use of raw materials components. The main advantage of nitric acid processing is that not only the chemical energy of nitric acid is used to decompose phosphorites, but also NO_3- anions in the form of a nutrient remain in the complex fertilizer. The decomposition of phosphorite with a reduced norm of nitric acid consists in the fact that the water-insoluble neutral calcium salt of phosphoric acid - fluorapatite Ca_5 (PO_4) 3F is converted into soluble acid salts, mainly into dicalcium phosphate $CaHPO_4$.

$Ca_5 (PO_4) 3F + 6HNO_3 = Ca (H_2PO_4)_2 + CaHPO_4 + 3Ca (NO_3)_2 + HF + 357.85$ kJ / mol

Consequently, during the decomposition of unenriched phosphate rock with nitric acid, the reaction of dicalcium phosphate formation predominantly proceeds, and monocalcium phosphate is contained in the resulting products as an impurity. A significant part of the phosphate in the product is also presented as an incompletely decomposed raw material, the phosphate complex of which is activated and is soluble in soil solutions.

The activation of the phosphate complex of phosphate raw materials under the action of nitric acid is mainly explained by the leaching of isomorphically substituted CO_2 groups from the nodes of their crystal lattices, which makes them unstable due to the weakening of interatomic bonds in already deformed crystal lattices.

A model laboratory setup was created in the laboratory. The installation is shown in Fig. 1, which consists of a cylindrical reactor (3) with a capacity of 10 l (0.01 m^3), made of stainless steel grade X1810NT and a capacity (10 l) for acidic nitrocalcium phosphate pulp (6), a screw dispenser (2), pressure tank (4) for nitric acid. Nitric acid (55.6%) from the pressure tank (4) entered the reactor (3), where simultaneously from the bunker (1) through the screw feeder (2) the unenrichedKyzylkum phosphate rock was fed. The reactor was equipped with a stirrer, the rotation frequency of which was controlled using a LATR. The stirrer rotation speed was 250-300 rpm. The nitric acid was fed using a Mariotte flask (5). The feed rate of nitric acid and phosphate rock was 74.5 ml / min and 100 g / min, respectively. The decomposition process was carried out in a solid-phase mode. Therefore, in the experiments, abundant foaming was not observed. The resulting pulp was, depending on the norm of HNO_3, a medium-moving or easy-moving mass. Experiments on a model setup were carried out at HNO_3 rates of 37, 50, and 60% in terms of calcium oxide. For each

IJIAET International Journal of Innovative Analyses and Emerging Technology
| e-ISSN: 2792-4025 | http://openaccessjournals.eu | Volume: 1 Issue: 5

norm of HNO_3, acidic nitrocalcium phosphate pulps were obtained in sufficient quantities. Every four hours, an average sample was taken and analyzed for the content of the main components: P_2O_{5total}, P_2O_{5assim}, P_2O_{5water}, CaO_{total}, CaO_{ass}.,CaO_{aq}., N_{total}. according to standard techniques. The raw phosphate raw material used was the unfortified phosphate rock of the Central KyzylKum of the following composition, wt. %:

P_2O_5 total CaO CO_2 F

Unfortified phosphate rock 17.65 47.48 15.2 1.81

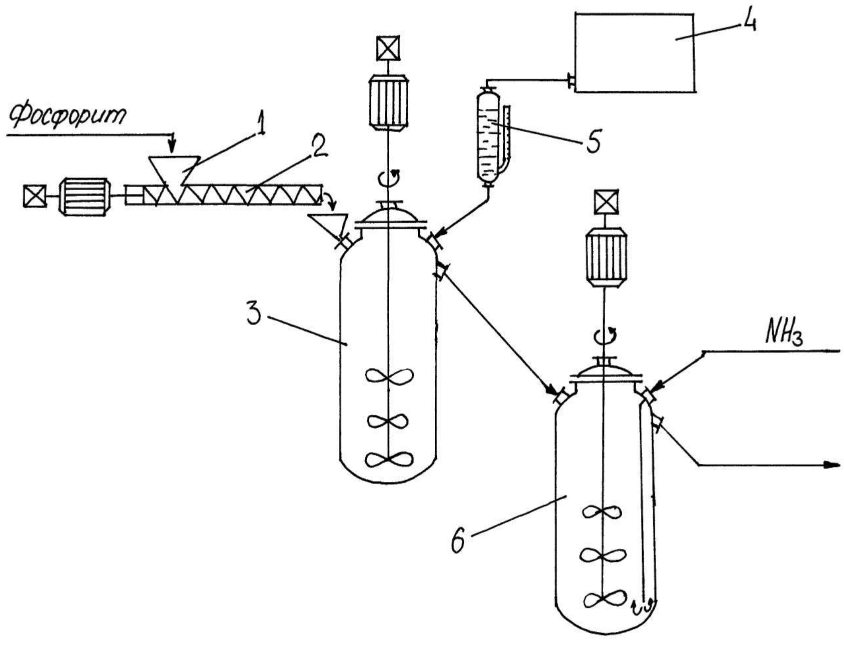
As can be seen from the data in Table 1, the composition of nitrocalcium phosphate pulps to a certain extent depends on the norm of nitric acid.

Table 1. Chemical composition of acidic nitrocalcium phosphatefat

Pulp

Samplerate	NormHNO3, %	P_2O_5, %		$\frac{P_2O_{5\,усв.}}{P_2O_{5\,общ.}} \cdot 100$ %	CaO, %			N, %
		gen.	assim.		gen.	assim.	water.	
When using unfortified phosphate rock								
	37	9,01	4,12	45,72	24,21	18,86	8,6	4,48
1	50	8,21	4,52	55,05	22,08	17,84	8,26	5,52
	60	7,65	4,83	63,14	20,58	17,13	8,13	6,17
	37	9,27	4,09	44,12	24,28	18,92	8,87	4,46
2	50	8,25	4,59	55,63	22,10	17,85	8,25	5,50
	60	7,60	4,79	63,03	20,60	17,16	8,14	6,20
	37	9,04	4,14	45,80	24,19	18,84	8,59	4,50
3	50	8,19	4,56	55,68	22,17	17,92	8,30	5,57
	60	7,70	4,86	63,11	20,56	17,12	8,13	6,15

With an increase in the HNO_3 norm, the relative content of the assimilable form of P_2O_5 increases, which indicates an increase in the amount of decomposed phosphates in the pulp composition. An increase in the relative content of the aqueous form of calcium with an increase in the norm of nitric acid is observed in all experiments, regardless of the type of phosphate raw material.



Pic. 1. Scheme of a continuously operating model plant for the production of nitrocalcium phosphate fertilizer

IJIAET International Journal of Innovative Analyses and Emerging Technology

| e-ISSN: 2792-4025 | http://openaccessjournals.eu | Volume: 1 Issue: 5

The rheological properties (density, viscosity) of nitrocalcium phosphate slurries (NCPP) play an important role in their further processing for fertilizers.

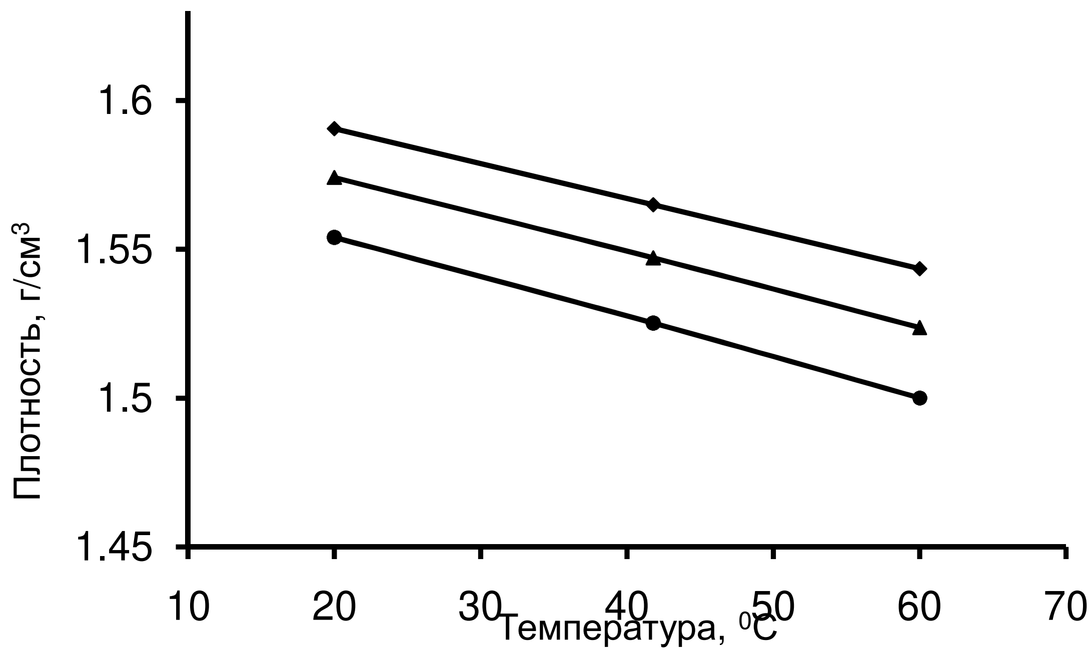
It should be noted that part of the absorption liquid formed during the production of phosphorus-containing fertilizers is returned to the reactor, where the stage of decomposition of the phosphate raw material with an acid reagent is carried out. At the same time, the moisture content in NKFP, depending on the HNO_3 norm, ranges from 35-38%. And the moisture content in the NKFP samples obtained on the model setup averages 26-28%. In this regard, before determining the rheological properties of NKFP, water was added to them based on the calculation that the moisture content in the pulp was at the level of 35%. The density of the NCPP was measured by the pycnometric method at temperatures of 20, 40, and 60 ° C.

The NKFP viscosity was determined using a VPZh viscometer with a diameter of 3.16 mm. The values of the density and viscosity of the LCPP are shown in Figures 2. From the data in Figure 2 it can be seen that the values of the LCPP density with an increase in the HNO_3 norm at the same temperature decrease. An increase in temperature from 20 to 60^0C at all norms of HNO_3 contributes to a decrease in density by an average of 1.09 - 1.03 times. With an increase in the HNO_3 norm, the NCPP viscosity (Fig. 3) noticeably increases. An increase in temperature from 20 to 6^0C, the value of viscosity decreases on average 2.8 - 3.4 times.

Thus, based on the results obtained, it can be assumed that acidic NCPP obtained at rates of HNO_3 in the range of 37-60% have sufficient fluidity, which makes it possible to carry out both the stage of decomposition of phosphorites with nitric acid and their further processing into solid granular fertilizers without special technological difficulties. The results of experiments on the decomposition of phosphorites with an incomplete norm of HNO_3 on a model unit under continuous conditions showed practically very close values to the results obtained under laboratory conditions and on a model laboratory unit of periodic action.

Further, acidic NCPP was subjected to ammonization with ammonia to pH 3.8 and 4.0. The resulting ammoniated mass was dried in a drying oven at 110–102^0C to a residual moisture of 1–1.5%. Fertilizer samples (NKFU) were analyzed for the content of the main components, the results of which are shown in Table 2.

The data in Table 2 show that the composition of fertilizers obtained under laboratory conditions, on a model plant of periodic action and on a model plant in discontinuous conditions, are very close to each other. Among the obtained NKFU samples from the point of view of agrochemistry and economy of initial raw materials, it seems to us that the best are fertilizers obtained at a rate of HNO_3 of 50 and 60%.



Pic. 2. Dependence of the density of nitrocalcium phosphate slurries at different temperatures. HNO_3 rate,%: 1- 37, 2-50 and 3-60. Feedstock: unfortified phosphate rock.

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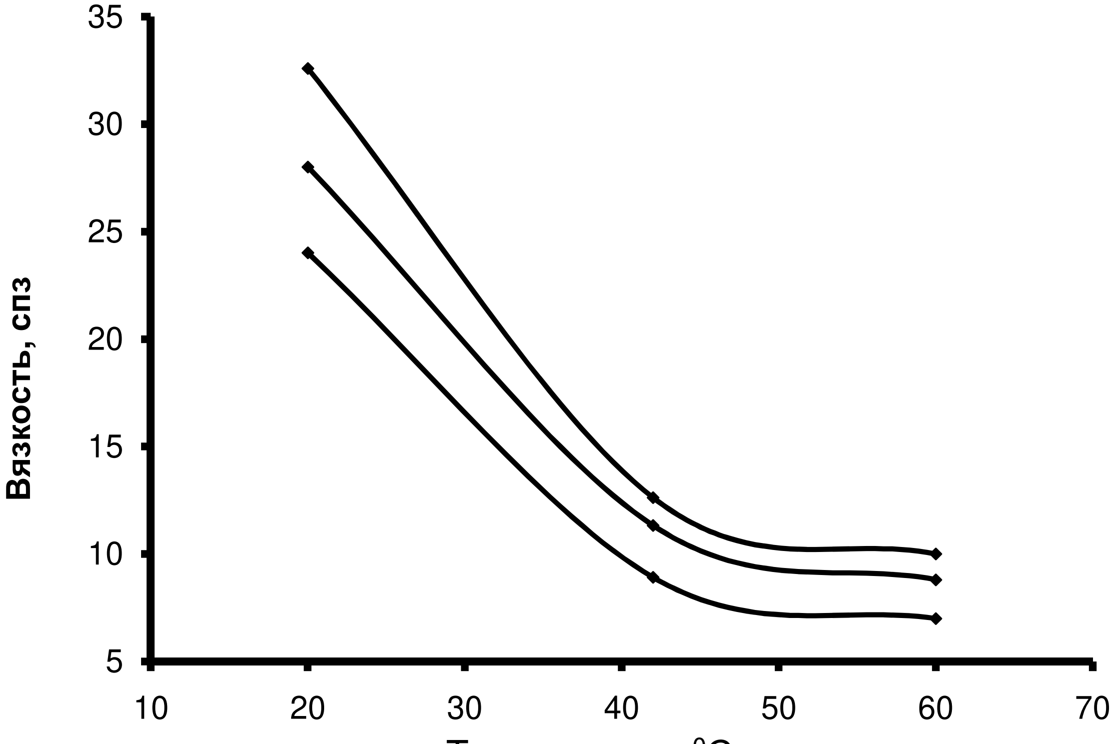

Fig. 3.Dependence of the viscosity of nitrocalcium phosphate slurries at different temperatures. HNO_3 rate,%: 1- 37, 2-50 and 3-60. Feedstock: unfortified phosphate rock.

Table 2 Chemical composition of nitrocalcium phosphate fertilizers

Samplerate	Norm HNO_3, %	P_2O_5, %			$\frac{P_2O_{5\,усв.}}{P_2O_{5\,общ.}} \cdot 100$ %	CaO, %			N, %
		gen.	assim.	water		gen.	assim.	water	
NKFU based on ordinary phosphate rock									
1	40	15,11	6,9	0,5	45,66	40,64	31,61	14,55	7,74
	50	14,01	7,68	0,75	54,82	37,68	29,77	13,60	9,87
	60	13,05	8,10	0,70	62,07	35,1	29,06	13,55	11,57
2	40	15,19	6,87	0,49	45,23	40,86	31,8	14,64	7,70
	50	13,97	7,66	0,71	54,83	37,57	29,72	13,79	9,9
	60	13,10	8,16	0,79	62,30	35,23	29,19	13,52	11,55
3	40	15,07	6,86	0,51	45,52	40,53	31,49	14,47	7,80
	50	14,1	7,79	0,77	55,25	37,92	30,02	13,95	9,86
	60	13,11	8,14	0,79	62,10	35,26	29,27	13,72	11,60

Based on the results of a series of experiments carried out on a larger continuous model plant, the following main technological parameters of the process of obtaining nitrocal-phosphate fertilizer are recommended:

Temperature of HNO_3 entering the reactor, ^{o}C 20-35

The norm of nitric acid from stoichiometry,% 50-60

HNO_3 concentration,% 47-58

Decomposition temperature, ^{o}C 40-50

Duration of the process, min 30-45

Mass ratio phosphorite: absorption liquid 1: 0.3 - 0.4

Temperature of the ammonization process NKFP, ^{o}C 50-60

ISSN 2792-4025 (online), Published under Volume: 1 Issue: 5 in October-2021

Duration of ammonization, min 3-4

Drying and granulation temperature, ^{0}C 100-110

Composition of nitrocalcium phosphate fertilizers:

Ntotal,% 9.86 - 12.90

P_2O_5total,% 13.05 - 17.54

P_2O_5usv,% (according to Trilon B) 7.62 - 9.12

P_2O_5св,% (by 2% - solution of citric acid) 10.43 - 11.07

CaO total,% 31.41 - 40.86

CaO ass% (by 2% citric acid solution) 20.98 - 30.02

CaOaq,% 10.3 - 13.95

CaO assimilated / CaO total,% (by 2% citric acid solution) 61.2 - 83.01

CaO water / CaO total,% 30.1 - 41.01

Nutrient ratio, N: P_2O_5 1: 1.1 ÷ 2.1

The amount of nutrients,%. 23.0 ÷ 29.0

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ISSN 2792-4025 (online), Published under Volume: 1 Issue: 5 in October-2021