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Research of the Process of Obtaining Organo-Mineral Fertilizer Based on Nitrogen Acid Decomposition of Non-Conditional Phosphorites of Central Kyzylkumes and Poultry Cultivation Waste

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Abstract: ***The article presents the results of decomposition of substandard phosphorites at a non-full rate of nitric acid (10-50% of stoichiometry on CaO), followed by its processing by bird droppings in various ratios. The optimal parameters for obtaining organic mineral fertilizers are determined, the technological scheme is given and the material balance is calculated, the possibility of obtaining nitrogen-phosphorus-humus fertilizers based on substandard phosphorites and bird droppings is shown, by composition (wt.%): P_2O_5 total - 6.53; P_2O_5 acceptable - 4.04; CaO total - 17.12; N - 4.21; organic matter - 28.38; humic acids - 9,12, fulvic acids - 5,25, water-soluble organic substances - 4,2; pH – 5,93.***

Keywords: chicken manure, sludge phosphorite, mineralized mass, phosphorus, calcium, humidity, humus substances, organic mineral fertilizer.

I. INTRODUCTION

Among the technology for the processing of phosphate mineral acids, nitric acid processing is a more economical method of producing fertilizers. When implementing this method, nitric acid not only participates in the decomposition of phosphate raw materials, but its anions remain in the final product in the form of a nutrient component - nitrogen. When phosphorites are decomposed by nitric acid, a solution is formed, the so-called nitric acid extract, consisting of calcium nitrate and free phosphoric acid, and, depending on the rate of nitric acid, underdeveloped phosphorite may be contained. The presence of excess calcium nitrate in the solution helps to neutralize the process of retrogradation, i.e. the transfer of digestible forms of phosphorus is indigestible and due to the presence of calcium nitrate in the final product, the latter has a high hygroscopicity.

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Most of the technologies developed by the nitric acid decomposition of phosphorites differ in the way they remove excess calcium ions. Depending on the method of removal of excess calcium nitrate, one-way nitrogen and phosphate fertilizers can be obtained, as well as complex, double or triple fertilizers with a wide range of nutrient ratios [1-5]. However, all the developed technological schemes associated with the removal of excess calcium nitrate in hardware and in operational terms are cumbersome and complex.

In agricultural practice and numerous agrochemical studies, the equivalence in efficiency of partially decomposed phosphorites and standard conventional fertilizers has been proved [6–8].

In [9], the technology of nitrocalcium phosphate fertilizer (NCPF) production is presented. The essence of the technological process consists in the decomposition of ordinary phosphorites with an incomplete rate of nitric acid, followed by the addition of water to the decomposition products and the neutralization of pulp with ammonia, drying, and granulation. The technology was introduced at JSC Samarqandkimyo, for the period 2006 - 2014, more than 700 thousand tons of NCPF were produced. Due to the high content of hygroscopic calcium nitrate, this technology was improved by partially removing calcium nitrate from ammoniated pulp and then obtaining solid activated NPCa and liquid fertilizers consisting of calcium nitrate solution [10-12].

In the works, a method [13,14] of the nitric-acid processing of carbonate phosphorites was proposed, which allows to obtain simultaneously enriched phosphate concentrate, precipitate, nitrogen-phosphorus and liquid nitrogen-calcium fertilizers and chemically precipitated calcium carbonate, however this technology has also not found practical application.

It is known that many organic acids are capable of forming hard soluble salts with calcium. In addition, organic additives to mineral fertilizers in the form of various high-molecular acids, such as humic, polycarboxylic, significantly reduce the process of retrogradation in the soil, which is very significant in the question of increasing the utilization rate of applied phosphorus fertilizers [15-18].

A source for the production of fertilizers with a certain degree of extraction of P_2O_5 at a reduced rate of nitric acid for the decomposition of phosphates can be bird droppings containing ammonium salts of organic acids

[19–21], which form poorly soluble compounds with calcium ion, are ameliorating soil.

At the same time, an organomineral fertilizer is obtained, which contributes to a better assimilation of the main nutrients by plants. Organic substances prevent the washing out of nitrate nitrogen from the soil and prevent the retrogradation of assimilable phosphates, create favorable conditions for the life of microorganisms, stimulate the growth and development of plants, which leads to an increase in crop yields [22-24].

It is necessary to note the present time in many countries of the world there is a shortage of high-quality phosphate raw materials and phosphate fertilizers. In 2018, enterprises of Uzkimyosanoat JSC of the Republic of Uzbekistan produced 153.8 thousand tons of phosphate fertilizers (in terms of 100% P_2O_5). And the need for agriculture is 691.7 thousand tons of P_2O_5. These figures indicate that the availability of phosphate fertilizers is insufficient. Currently, Kyzylkum phosphorite Combine enriches highly carbonated phosphorites of Central Kyzylkum and produces waste in the form of off-balance ore with a content of 13-15% P_2O_5 and sludge phosphate with a content of 10-12% P_2O_5. The total volume of accumulated waste phosphorites already reaches 15 million tons.

In the interaction of phosphate raw materials with an incomplete norm of nitric acid, calcium carbonates decompose in the first place:

$$CaCO_3 + 2HNO_3 \rightarrow Ca(NO_3)_2 \cdot H_2O + CO_2$$

At the same time, but to a lesser extent, the phosphate mineral decomposes:

$$Ca_5F(PO_4)_3 + 2HNO_3 + 5H_2O \rightarrow Ca(NO_3)_2 + 7H_3PO_4 + HF$$

As a result of the hydrolysis of monocalcium phosphate, dicalcium phosphate is also formed:

$$Ca(H_2PO_4)_2 \cdot H_2O + H_2O \rightarrow CaHPO_4 \cdot 2H_2O + H_3PO_4$$

In poultry organic acids, the most reactive are carboxyl groups. Therefore, when litter is added to the product of nitric acid decomposition of phosphates, their interaction with nitrate and other calcium salts can be expressed by the following equations:

$$2R\,COOH + Ca(NO_3)_2 \longrightarrow (R\,COO)_2\,Ca + 2HNO_3$$
$$2R\,COOH + Ca(H_2PO_4)_2 \longrightarrow (R\,COO)_2\,Ca + 2H_3PO_4$$
$$2R\,COOH + 2CaHPO_4 \longrightarrow (R\,COO)_2\,Ca + Ca(H_2PO_4)_2$$
$$2R\,COOH + Ca_3(PO_4)_2 \longrightarrow (R\,COO)_2\,Ca + 2CaHPO_4$$

II. MATERIALS AND METHODS

To study the processes of obtaining organic fertilizers, poultry droppings having the composition (wt.%) Were used as organic raw materials: moisture – 64,78; ash – 11,29; organic substances – 23,93; humic acids – 1,04; fulvic acids – 7,27; water soluble organic matter -1,28; P_2O_5 – 1,25; N – 0,95; K_2O – 0,74; CaO – 1,55. And as the phosphate raw material used mineralized mass and sludge phosphorite. Before use, they were ground to a particle size of 0,25 mm. The composition of substandard phosphorites is shown in table 1.

Table I. The chemical composition of substandard phosphorites

Types of phosphate raw materials	The content of components, weight. %									$P_2O_{5accep.}$ / $P_2O_{5total.}$ 100 %
	P_2O_5	CaO	Al_2O_3	Fe_2O_3	MgO	F	CO_2	SO_3	H.O.	
Mineralized mass	14,33	43,02	1,18	1,38	1,19	1,85	14,70	2,22	13,23	9,01
Sludge phosphorite	11,57	41,08	1,84	1,42	0,61	1,52	20,91	0,46	14,9	11,50

From the table. 1 it can be seen that substandard phosphorites of the Central Kyzyl Kum are characterized by a low phosphorus content (11.5-14.33% P_2O_5), a high carbonate content (14.70-20.91% CO_2) and a high calcium modulus (CaO: P_2O_5 = 3, 0-3.55).

Chemical analysis of bird droppings, mineralized mass, sludge phosphorite and products of their processing was carried out by the following methods.

Humidity was determined according to GOST 26712-85, ash content according to GOST 26714-85 and organic matter according to GOST 27980-80. The amount of the water-soluble fraction of organic substances extracted from the products with water was determined by filtration and evaporation in a water bath, drying the solid residue to a constant weight, and then burning it to determine the ash content and subtract it. Humic acids were isolated by treating the products with 0.1 N alkali solution followed by acidification of the solution with mineral acid [26]. The solid phase after separation of alkali-soluble organic substances from it contains residual organic matter. It was thoroughly washed with disco, dried to constant weight and the content of organic substances was determined. The difference between the amounts of alkali-soluble organic substances and humic acids gives us the content of fulvic acids.

All P_2O_5 forms were determined by the gravimetric method by precipitation of the phosphate ion with a magnesia mixture in the form of magnesium ammonium phosphate, followed by calcination of the precipitate at 1000-1050 ° C according to GOST 20851.2-75. Digestible P2O5 forms were determined by solubility in both 2% citric acid and 0.2 M Trilon B.

The determination of CaO content was carried out complexometrically: by titration with a 0.05 N solution of Trilon B in the presence of a fluorexon indicator. Nitric acid with a concentration of 59% was used to decompose substandard phosphorites. Norms of nitric acid varied in the range of 10-50% of stoichiometry for the decomposition of CaO phosphate raw materials. The experiments were carried out as follows, nitric acid was slowly poured into a glass reactor, in which a sample of fossy was located. The contact duration of the components was 40 minutes, after which poultry manure was added to the pulp, and stirring was continued for 60 minutes.

Drying was carried out at 80 °S until the moisture content in the finished product was 10-13%. Processing of products of nitric acid decomposition by bird droppings was carried out in the range of weight ratios of bird droppings to mineralized mass and slurry phosphorite from 100: 10 to 100: 40. The results are shown in tables 2 and 3.

III. DISCUSSION OF RESEARCH RESULTS

The tables show that the higher the norm of nitric acid and the more bird droppings taken, the less P_2O_5 total in the product, but the higher the relative content of the assimilable form of P_2O_5, nitrogen, organic substances and humic acids. As can be seen from the table. 2 when the ratio of bird droppings: mineralized mass = 100: 10 and the norm of nitric acid 10% of stoichiometry is obtained organomineral fertilizer containing P_2O_5 total – 5,05%; P_2O_5acceptable– 1,54%, N – 2,27 %; organic matter – 45,58%; humic acids – 2,51 %, fulvic acids –9,83 %, water-soluble organic substances –2,3 %. At the same ratio of droppings to mineralized mass, but at a rate of acid of 50%, a fertilizer containing P_2O_5 total – 4,74%; P_2O_5acceptable – 3,61%; N – 3,67%; organic matter – 42,82%; humic acids – 2,36 %; fulvic acids –9,24 %; water soluble organic matter –2,16 %. Fertilizers of similar composition are obtained during the processing of sludge phosphorite (Table 3). The tables show that the more acid and bird droppings are taken, the more complete the decomposition of phosphate raw materials. So, for a sludge phosphorite with an acid rate of 10% of stoichiometry and a weight ratio of litter to sludge phosphoriteof 100: 30, the product has (wt.%): P_2O_5 total – 6,11 %; P_2O_5acceptable – 2,24%, CaO total. – 18,07 %, N – 2,25 %, organic matter – 31,62 %, humic acids –1,81%, fulvic acids – 6,87 %, water-soluble organic substances 1,67, and with an acid rate of 50% and a weight ratio of litter to sludge phosphorite100: 30 the product has (wt.%): P_2O_5 total – 6,35 %; P_2O_5acceptable – 4,61 %, CaO total. – 16,63 %, N –4,84 %, organic substances – 16,63 %, humic acids – 1,52 %, fulvic acids 5,95 %, water-soluble organic substances 1,39 %.

Table II. The composition of the organic fertilizer obtained activation of mineralized mass with nitric acid and bird droppings

Norm HNO_3,%	$P_2O_{5total.}$ %	$\frac{P_2O_{5accep.}}{P_2O_{5total.}}$ 100 %	CaO_{total}, %	Organic matter,%	Fulvic acids, %	Humic acids, %	Water-soluble organic substances,%	N,%	pH
The mass ratio of bird droppings and mineralized mass = 100 : 10									
10	5,05	30,53	11,13	45,58	9,83	2,51	2,30	2,27	7,10
20	4,96	45,22	10,95	44,85	9,67	2,47	2,26	2,64	6,61
30	4,88	55,02	10,77	44,13	9,52	2,43	2,23	2,99	6,02
40	4,81	67,33	10,61	43,46	9,37	2,39	2,19	3,34	5,50
50	4,74	76,14	10,45	42,82	9,24	2,36	2,16	3,67	4,47
The mass ratio of bird droppings and mineralized mass = 100 : 20									
10	6,32	29,81	15,70	37,06	7,99	2,04	1,87	2,18	7,22
20	6,15	43,68	15,30	36,11	7,79	1,99	1,82	2,77	6,70
30	6,00	52,81	14,90	35,18	7,59	1,94	1,78	3,33	6,14
40	5,85	65,42	14,55	34,34	7,41	1,89	1,73	3,87	5,74
50	5,72	74,73	14,21	33,54	7,24	1,85	1,69	4,38	4,65
The mass ratio of bird droppings and mineralized mass = 100 : 30									
10	7,19	28,44	18,83	31,23	6,74	1,72	1,58	2,12	7,31
20	6,96	42,05	18,22	30,22	6,52	1,66	1,53	2,86	6,78
30	6,73	50,98	17,64	29,25	6,31	1,61	1,48	3,56	6,23
40	6,53	61,89	17,12	28,38	6,12	1,56	1,43	4,21	5,96
50	6,35	72,65	16,63	27,57	5,95	1,52	1,39	4,84	4,82
The mass ratio of bird droppings and mineralized mass = 100 : 40									
10	7,87	27,02	21,23	27,13	5,85	1,49	1,37	2,08	7,39
20	7,58	40,98	20,44	26,12	5,63	1,44	1,32	2,94	6,93
30	7,30	49,25	19,69	25,16	5,43	1,39	1,27	3,74	6,35
40	7,05	58,81	19,02	24,31	5,24	1,34	1,23	4,48	5,77
50	6,82	69,02	18,41	23,52	5,07	1,30	1,19	5,18	5,10

Table III. The composition of the organic fertilizer obtained activation of sludge phosphorite with nitric acid and bird droppings

Norm HNO_3, %	$P_2O_{5total.}$ %	$P_2O_{5accep.}$ / $P_2O_{5total.}$ 100 %	CaO_{total}, %	Organic matter,%	Fulvic acids, %	Humic acids, %	Water-soluble organic substances,%	N, %	pH
The mass ratio of bird droppings and sludge phosphorite = 100 : 10									
10	4,52	40,12	10,76	46,15	10,02	2,64	2,43	2,46	7,65
20	4,45	54,68	10,59	45,41	9,86	2,60	2,40	2,83	6,92
30	4,38	63,81	10,42	44,68	9,70	2,56	2,36	3,18	6,53
40	4,31	74,38	10,26	44,01	9,56	2,52	2,32	3,52	5,88
50	4,25	83,46	10,11	43,36	9,41	2,48	2,29	3,85	4,93
The mass ratio of bird droppings and sludge phosphorite = 100 : 20									
10	5,46	38,54	15,10	37,53	8,15	2,15	1,98	2,33	7,76
20	5,32	51,78	14,71	36,56	7,94	2,09	1,93	2,92	7,04
30	5,19	60,84	14,33	35,62	7,73	2,04	1,88	3,48	6,61
40	5,06	72,38	13,99	34,77	7,55	1,99	1,83	4,01	6,01
50	4,95	80,95	13,67	33,96	7,38	1,95	1,79	4,52	5,06
The mass ratio of bird droppings and sludge phosphorite = 100 : 30									
10	6,11	36,71	18,07	31,62	6,87	1,81	1,67	2,25	7,87
20	5,91	49,39	17,49	30,59	6,64	1,75	1,61	2,99	7,13
30	5,72	56,77	16,93	29,62	6,43	1,70	1,56	3,68	6,72
40	5,55	68,32	16,43	28,74	6,24	1,65	1,52	4,33	6,14
50	5,40	78,41	15,96	27,92	6,06	1,60	1,47	4,95	5,18
The mass ratio of bird droppings and sludge phosphorite = 100 : 40									
10	6,62	35,62	20,35	27,47	5,97	1,57	1,45	2,20	7,99
20	6,37	47,09	19,59	26,45	5,74	1,51	1,39	3,05	7,25
30	6,14	54,32	18,88	25,48	5,53	1,46	1,34	3,84	6,83
40	5,93	65,82	18,24	24,61	5,34	1,41	1,30	4,58	6,26
50	5,74	75,98	17,64	23,81	5,17	1,36	1,26	5,28	5,29

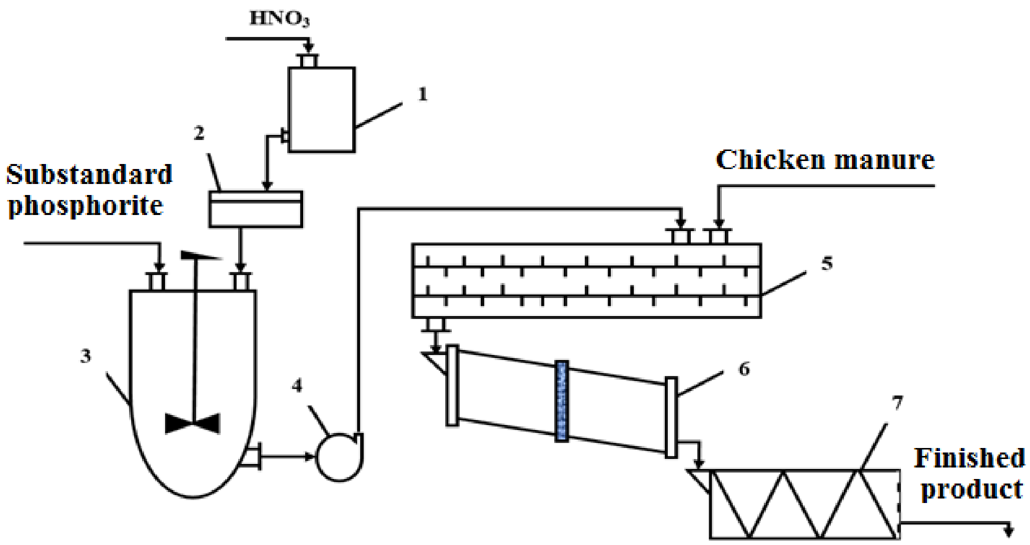

Fig. 1 Schematic diagram of the process of obtaining organic fertilizers:
1 - HNO_3 capacity; 2 - automatic hub; 3 - reactor; 4 - pump; 5 - auger mixer; 6 - drum dryer; 7 - press granulator.

At the request of agriculture, phosphorus fertilizer must have a high content of total and assimilable forms of P_2O_5, and the relative content of the water-soluble form of P_2O_5 should be at least 50%. Based on this requirement, the optimal norm of nitric acid for the decomposition of non-codicphosphorites is 40% of stoichiometry, and the optimum ratio of bird droppings: substandard phosphorite can be considered 100 : 30, at which the relative content of P_2O_5acceptable–61,89 % (table. 2). At the same time, organomineral fertilizers using a mineralized mass have the composition (wt.%): P_2O_5 total – 6,53; P_2O_5acceptable - 4,04; CaO total – 17,12; N – 4,21; organic matter -28,38; humic acids –1,56, fulvic acids 6,12, water-soluble organic substances 1,42, and using sludge phosphorite(wt.%): P_2O_5 total – 5,55; P_2O_5 acceptable – 3,79; CaO total – 16,43; N – 4,33; organic matter – 28,74; humic acids –1,65, fulvic acids –6,24, water-soluble organic substances –1,52.

Based on the experiments, a technological scheme was developed (Fig. 1) and the material balance of production of one ton of organic mineral fertilizers was calculated (Fig. 2). Nitric acid with a concentration of 59% from the storage enters the pressure tank 1, then to the automatic concentrator 2, where it is diluted with water. From the concentrator, the acid is sent to reactor 3, where fossil feed is fed. After the fossyre is decomposed with nitric acid at its incomplete rate, the resulting mass is fed to the mixer screw 4, where bird droppings are simultaneously fed. After thorough mixing, the resulting mass is sent to the drum dryer 5, then to the press granulator 6.

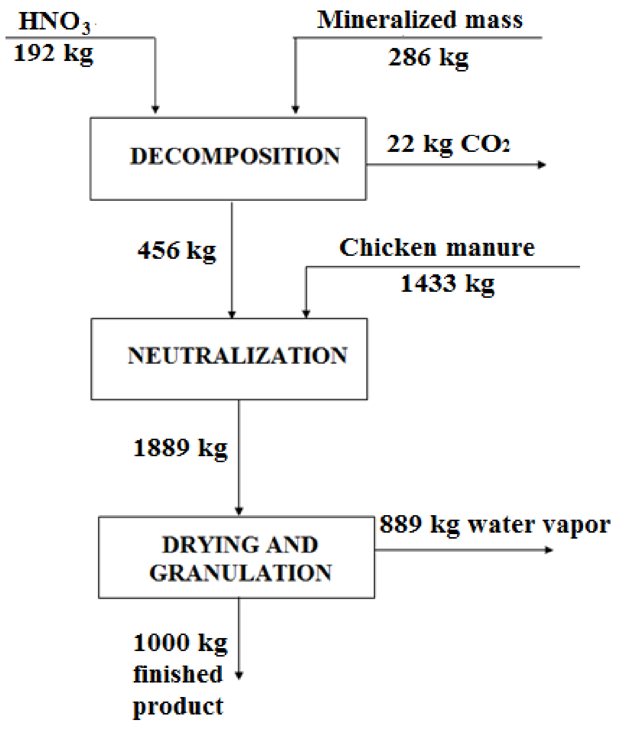

Fig. 2 Material balance of the production of organic fertilizers.

The optimal technological mode of the process of obtaining organic fertilizer is as follows:

- concentration of nitric acid,% ……………… .. 58;
- HNO_3 norm from stoichiometry,% .. …….................. 40;
- the duration of the decomposition process, min 40;
- weight ratio of fresh bird droppings to fossyry. 100 : 30;
- the duration of the processing of decomposition products fossyry nitric acid, min ………………… 60;
- the drying temperature of the product, °C .... 80.

IV. CONCLUSION

Thus, the study convincingly shows that poultry farms using a small amount of nitric acid can intensively process poultry manure and mineralized mass into organic fertilizers.

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