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Obtaining Azosuperphosphate Fertilizer by Acid Processing of Low-grade Phosphorites of Central Kyzylkum

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ABSTRACT: This report sets out the results of the activation of low-grade phosphorite ore with concentrated solutions of ammonium sulfate. It was shown that treatment of the mineralized mass with a solution of ammonium sulfate leads to a sharp increase in the content of the assimilable form of P_2O_5 in phosphate raw materials. Granules of activated nitrogen-phosphorus fertilizers have sufficient strength, which meets the requirements of agriculture.

KEYWORDS: low-grade phosphorites , ammonization, highly carbonized, ammonium sulfate solution, decarbonization, decomposition.

I. INTRODUCTION

Introduction. In our republic, special attention is paid to the intensive development of agriculture. The “Strategy and further development of the Republic of Uzbekistan” outlines the most important tasks, especially modern agricultural technologies in the field of water and land resources [1].

During the implementation of these tasks, an important role is played by the production of mineral fertilizers. It is known that phosphoric fertilizers increase the yield of agricultural crops and improve their quality, increase the resistance of plants to lodging from dryness, accelerate their ripening. The modern industry of phosphorus-containing fertilizers in Uzbekistan is mainly based on low-grade phosphorites of Central Kyzyl-Kum. The development of intensive fast-moving technologies for the production of phosphorus-containing fertilizers with a reduced consumption of mineral acids and involving low-grade phosphorites in the processing with the existing shortage of mineral resources is an urgent and important issue today [2, p-182].

Finding effective ways to obtain assimilated phosphorus fertilizers from phosphorites that are difficult to enrich is of both scientific and practical importance. The process of converting phosphorites to a state that can be assimilated by the plant is by converting the phosphorus nutrient in it into a state that can be assimilated by the plant using various methods. In this regard, mechanical, chemical, mechano-chemical, thermal and microbiological methods of activation of phosphorite-containing P_2O_5 - phosphorus anhydride from non-vegetative form to plant assimilation have been developed in order to make efficient use of low-quality phosphorites and reagents[3, p-112].

II. LITERATURE SURVEY

In this paper we describe the processes of activation of low-grade phosphorites by mechanical and chemical methods and the production of superphosphate-class azosuperphosphates, which have a large number of acid-processed nutrient components. We have done some research on this in our previous work[4, 5].

The possibility of obtaining phosphorus fertilizers rich in micro and macronutrients using the method of mechanical activation has been studied worldwide. Lignosulfonate, zeolite and lignite were used as activating additives in phosphate raw materials, and when lignosulfonates were used as activating additives in this process, twice as much R_2O_5 was formed in plant absorption form as in mechanically activated phosphate ore with zeolite and lignite [6, 7, 8].

Despite the fact that the scientific and practical recommendations developed by scientists are sufficient, today the demand for phosphorus fertilizers in agriculture is not fully met. Another major reason for this is the declining supply of high quality phosphorites worldwide today. Based on the above problems, the creation of superphosphate fertilizers from low-grade phosphate raw materials, which are used today, is one of the main current problems.

III. METHODOLOGY AND EXPERIMENTAL RESULTS

Based on these problems, one of the main tasks in this article is to study the processes of mechanical and chemical decomposition of low-grade Central Kyzylkum phosphorites in Uzbekistan. To obtain azo superphosphate, we first investigated the chemical composition and properties of low-grade phosphorites of the Central Kyzyl- Kum. For laboratory experiments used low-grade phosphate raw materials called mineralized mass is a product of dry screening of phosphorite ore. The chemical composition of phosphorites is as follows: P_2O_{5gen} : 12,38-13,94%, P_2O_{5ass} : 1,95-1,99%; CaO 43,78-44,06%; MgO 1,96-2,11%; Fe_2O_3 1,78-1,80%; Al_2O_3 1,21-1,48%; CO_2 19,10-19,73%; SO_3 2,10-2,56%; F 0,42-1,45%; H_2O 1,17-1,78%.

The content of all forms of phosphorus (total, assimilable, and water-soluble) in phosphate raw materials and finished products was determined according to the standard method. The determination of sulfates in phosphorite was carried out by the weight method, deposition in the form of barium sulfate. Carbon dioxide was determined by treating a sample of the sample with hydrochloric acid, followed by absorption of CO_2 with an alkali solution. Moisture was determined by drying to constant weight at a temperature of 80-105°C [9; 10; 11; 12].

In the experimental processes, ammonium sulfate was first added with concentrated sulfuric acid and then to the decomposition process using various methods. The effects of ammonium sulfate on decomposition processes were studied.

Depending on the concentration and norm of sulfuric acid, the calculated amount of phosphorite was treated with acid by mixing thoroughly for 20 minutes to determine the degree of decomposition of the phosphate raw material. After cooling, the obtained superphosphates were subjected to chemical analysis in the prescribed manner. The experimental results are shown in Tables 1 and 2.

Obtaining fertilizers by decomposing low-grade phosphorites at an incomplete rate of concentrated sulfuric acid differs from the classical methods in that upon the completion of the interaction of the components, the resulting mass is constantly fragmented into small aggregated particles, which, in turn, are continuously enveloped with excess phosphate feedstock.

Table 1

Degree of decomposition of unenriched phosphate rock depending on the concentration and norm of sulfuric acid

Norm H_2SO_4 , %	Content P_2O_5 , % in ready product			CO_2 , %	H_2O , %	Coefficient decomposition, %
	gen	ass	water			
Phosphorite decomposition with 70% sulfuric acid						
20	16,62	3,59	0,99	12,80	1,94	21,60
50	14,38	7,56	2,62	8,90	3,10	52,57
70	13,10	9,58	3,96	7,20	5,48	73,12
80	12,58	10,30	4,58	4,30	7,36	81,87
Phosphorite decomposition with 80% sulfuric acid						
20	17,14	3,81	1,21	11,80	0,36	22,22
50	14,56	8,02	2,88	8,10	2,32	55,08
70	13,42	10,04	4,17	6,10	3,57	74,81
80	12,95	10,70	5,14	3,90	4,27	82,62
Phosphorite decomposition with 93 % sulfuric acid						
20	17,10	3,98	1,53	11,30	0,83	23,27
50	14,80	8,32	3,10	7,80	1,35	56,21
70	13,60	10,71	4,39	5,70	2,45	78,75
80	13,17	11,14	5,43	3,43	2,68	84,58

Table2

The degree of decomposition of poor phosphorites depending on the concentration and norm of sulfuric acid

Norm H ₂ SO ₄ , %	Content P ₂ O ₅ , % in ready product			CO ₂ ,%	H ₂ O, %	Coefficient decomposition, %
	gen	ass	water			
Phosphorite decomposition with 70% sulfuric acid						
20	10,97	1,63	0,47	16,75	2,00	14,85
50	9,45	4,60	1,23	12,61	3,81	48,67
70	8,75	6,05	2,15	9,28	4,79	69,14
80	8,56	6,30	2,74	6,35	5,45	73,59
Phosphorite decomposition with 80% sulfuric acid						
20	10,99	1,85	0,57	16,22	0,58	16,83
50	9,80	4,92	1,48	10,51	1,93	50,20
70	8,92	6,32	2,54	7,04	2,10	70,85
80	8,18	6,42	2,84	4,88	2,31	78,48
Phosphorite decomposition with 93 % sulfuric acid						
20	11,30	2,10	0,75	15,63	0,04	18,58
50	10,15	5,38	1,94	9,75	1,65	53,00
70	9,49	6,94	2,98	5,91	1,83	73,12
80	9,14	7,36	3,36	4,18	2,05	80,52

The interaction of unenriched phosphates with sulfuric acid proceeds very easily and practically ends in 5-7 minutes. The process is exothermic, the temperature, depending on the norm of sulfuric acid, rises to 35-80 ° C.

It was found that with an increase in the norm and concentration of sulfuric acid, the degree of decomposition of phosphorite increases. With a change in the concentration of sulfuric acid from 70 to 93%, the decomposition coefficient increases by only 1.02-1.07 times, and when its norm changes from 20 to 80%, K_p increases on average by 3.65 times. The decomposition of phosphates below 70% sulfuric acid leads to the formation of a product with a low decomposition coefficient, and with an increase in its norm above 40% from stoichiometry, a smearing mass is formed. Based on the results of the experiment, it was found that in the studied range of sulfuric acid concentrations up to its 50% norm, a powdery product is obtained containing 14-17% of total P₂O₅, of which 20-50% are in the form assimilated by plants. With an increase in the norm of sulfuric acid, the granulometric composition of simple superphosphate improves. So, for example, at a 70% norm of sulfuric acid, the content of the 1-4 mm fraction is 50-60%, and at 80% it is 70-75%. The samples lack particles larger than 10 mm. The decomposition of poor phosphorites by concentrated sulfuric acid practically does not differ from that of unenriched phosphum. The decomposition coefficient of poor phosphorites is 5-10% lower compared to phosphates. This is due to the presence of large fractions of phosphorite and a high content of carbonate minerals.

In this regard, to study the possibilities of obtaining a complex fertilizer, azo superphosphate from low-grade phosphate central Kyzyl-Kum, when they are decomposed with a lower norm acid in the presence of ammonium sulfate, the solubility of the latter in solutions of different concentrations of sulfuric acid depending on temperature was determined. During the experiment, ammonium sulfate and its solutions with a concentration of 5 to 20% and 100 g of phosphorite samples were used to determine the decomposition efficiency of phosphorites using mineral salts. The acidic saline solution was prepared by dissolving ammonium sulfate in 93% concentrated sulfuric acid. In Table 3, a solution of ammonium sulfate in sulfuric acid was prepared under normal room conditions.

Table 3

Solubility of ammonium sulfate in sulfuric acid, at room temperature

№	content	Ammonium Sulfate Contents 100 g H ₂ SO ₄ , g	The composition of the solution, wt. %		
			H ₂ SO ₄	(NH ₄) ₂ SO ₄	H ₂ O
1	1- content	5	88,57	4,76	6,67
2	2- content	10	84,56	9,09	6,35
3	3- content	15	80,87	13,04	6,09
4	4- content	20	77,50	16,67	5,83

Acid decomposition of the samples was carried out under laboratory conditions at room temperature, with constant stirring of the phosphorite samples and the acidic saline solution for 5–20 min. The main nutrients in the complex phosphorus fertilizer obtained as a result of the experiment - various forms of phosphorus anhydride (simple, plant-absorbing part), nitrogen were chemically analyzed. Studies show that as the concentration of ammonium sulfate in solution increases, so does the rate of decomposition of phosphorites. The results of the analysis are presented in Table 4 below.

Table 4
Analysis of samples of low-grade phosphorite on the basis of different percentage solutions of ammonium sulfate-sulfuric acid

№	Norm H ₂ SO ₄	pH (based on 10% suspension)		P ₂ O ₅ , %		N, %	$\frac{P_2O_5(ass)}{P_2O_5(gen)}$
		pH _{beg}	pH _{end}	P ₂ O _{5gen}	P ₂ O _{5ass}		
1- content							
1	100	4,1	6,9	10,74	8,56	0,66	79,10
2	80	4,2	7,0	11,46	8,03	0,53	70,10
3	60	4,3	7,2	12,30	5,65	0,39	45,90
2- content							
1	100	4,2	6,8	10,31	8,38	1,25	81,30
2	80	4,3	6,9	11,00	7,78	0,99	70,71
3	60	4,4	7,2	11,74	5,49	0,75	46,80
3- content							
1	100	3,8	6,7	9,96	8,31	1,81	83,52
2	80	3,9	6,8	10,60	7,65	1,43	72,17
3	60	4,0	7,1	11,50	5,58	1,07	48,52
4- content							
1	100	3,2	6,8	8,62	7,45	2,30	86,13
2	80	3,4	6,9	10,01	7,43	1,83	74,20
3	60	3,6	7,2	10,64	5,33	1,37	50,12

As can be seen from the table above, an increase in the rate of solution used in the process leads to an increase in the rate of decomposition of the raw material. When the rate of 1-component solution was increased on a certain interval from 60 to 100, the decomposition rate was observed to increase from 45.90% to 79.10%, and when the 2-component solution was used, the decomposition rate was increased from 46.80% to 81.30%. When the norms of solutions 3 and 4 were increased from 60 to 100%, they increased by 1.72 times, respectively, to 83.52 and 86.13%. The initial absorbable form of P₂O₅ in the raw material was found to increase 2.73 and 4.39 times, respectively, in the stoichiometric amount of solution during decomposition.

In subsequent experimental processes, low-grade phosphorite was decomposed at different rates of sulfuric acid and introduced into the process by spraying with an aqueous solution of 20% ammonium sulfate (5%, 10%, 15%, 20%) relative to the amount of mass formed. The results were chemically analyzed (Table 5).

Table 5
The composition of the samples granulated with an aqueous solution of ammonium sulfate

Number content	P ₂ O ₅ , %			$\frac{P_2O_5(ass)}{P_2O_5(gen)}$	CO ₂ , %	N, %
	P ₂ O _{5gen}	P ₂ O _{5ass}	P ₂ O _{5water}			
H ₂ SO ₄ norm 100						
1-content	10,82	9,13	1,65	84,38	6,21	0,26
2- content	10,34	8,97	2,12	86,72	5,91	0,51
3- content	9,86	8,65	3,01	87,68	4,15	0,74
4- content	8,42	7,46	3,45	88,60	3,82	1,00
H ₂ SO ₄ - norm 80						
1- content	11,52	7,57	0,96	65,71	8,12	0,38
2- content	11,09	7,38	1,32	66,54	7,01	0,67
3- content	10,78	7,31	2,45	67,81	6,64	0,76

4- content	10,19	6,99	3,08	68,59	4,88	1,03
H ₂ SO ₄ - norm 60						
1- content	12,35	5,29	0,91	42,80	12,21	0,45
2- content	11,84	5,17	1,29	43,66	12,00	0,74
3- content	11,45	5,22	2,15	45,58	10,01	1,01
4- content	10,34	4,97	2,66	48,06	9,00	1,12

The results of the analysis show that after 100% decomposition of sulfuric acid, the composition of fertilizers obtained by granulation using a 5-20% aqueous solution of (NH₄)₂SO₄ -ammonium sulfate, the total amount of P₂O₅ in the fertilizer increased by 10.82 with increasing concentration of (NH₄)₂SO₄. - decreased to 8.42%, and the decomposition rate increased from 84.38% to 88.60%, respectively. When stichiometric acid standards of 60-80 are used in the decomposition process, the total amount of P₂O₅ in the solution (NH₄)₂SO₄ decreases with increasing concentration of to 12.35-10.34% and 11.52-10.19%, respectively, and vice versa. levels increased to 42.80-48.06% and 65.71 to 68.59%, respectively. Also, the plant assimilation form of P₂O₅ in the primary raw material was 1.95%, after the decomposition process, this figure increased by 2.54 and 4.68 times, respectively, to 4.97 and 9.13%, respectively, with an increase in the amount of stichiometric acid.

In subsequent studies, low-grade phosphorites were mechano-chemically activated for 30 min with seemingly crushed ammonium sulfate, and then the products were obtained by decomposition of them at different rates of acid-stoichiometric quantities and chemical analysis (Table 6).Compounds were prepared for this process in ascending order of (NH₄)₂SO₄; Ammonium sulphate was activated by adding 5% of phosphorite content in composition 1, 10% in content 2, 15% in content 3 and 20% in content 4.

Table 6
Results of decomposition of mechanically activated samples with sulfuric acid

Number content	P ₂ O ₅ , %			$\frac{P_2O_5(ass)}{P_2O_5(gen)}$	CO ₂ , %	N, %
	P ₂ O _{5gen}	P ₂ O _{5ass}	P ₂ O _{5water}			
H ₂ SO ₄ norm 100						
1-content	10,54	9,61	1,72	91,14	6,01	0,70
2-content	10,28	9,39	2,20	91,34	5,61	1,36
3-content	9,84	9,18	3,05	93,29	4,05	1,98
4-content	8,54	8,04	3,49	94,15	3,02	2,56
H ₂ SO ₄ - norm 80						
1-content	11,46	7,93	0,98	69,20	8,02	0,76
2-content	11,06	7,73	1,34	69,89	6,85	2,24
3-content	10,51	7,58	2,47	72,12	6,54	3,35
4-content	10,11	7,36	3,13	72,79	4,08	4,46
H ₂ SO ₄ - norm 60						
1-content	12,28	5,57	0,94	45,35	10,01	0,84
2-content	11,48	5,40	1,29	47,03	9,00	2,45
3-content	11,24	5,47	2,17	48,66	8,01	3,66
4-content	10,07	5,09	2,68	50,54	7,71	4,85

When studying the composition of samples decomposed at 100% sulfuric acid, it was observed that the total amount of R₂O₅ in it decreased by 10.54-8.54%, and the decomposition coefficient increased from 91.14% to 94.15%. In the decomposition of phosphorite, the total amount of P₂O₅ in the content decreases from 12.28 to 10.07% and from 11.46 to 10.11%, while the rate of decomposition is 45.35-50.54%, respectively, when the rate of sulfuric acid increases by 60-80%. and 69.20-72.79%. As a result of these studies, the amount of 1.95% absorbent P₂O₅ in the raw material increased 2.61-4.95 times after decomposition, from 5.09 to 9.61%.

The effectiveness of ammonium sulfate in the decomposition rate of phosphorites has been scientifically substantiated as a result of the study. The inclusion of ammonium sulfate leads to an increase in the absorbable form of P₂O₅ in the raw material from 1.56 to 4.95 times. However, we know that ammonium sulfate reacts with the monocalcium phosphate formed in the composition to form NH₄H₂PO₄, resulting in the formation of large amounts of calcium sulfate in the samples. With this in mind, in the later stages of the study, the processes of melting ammonium sulfate salt at

normal (room) conditions at the highest value in sulfuric acid of different concentrations were studied. As the concentration of sulfuric acid in the process increased, a decrease in the solubility of ammonium sulfate was observed (Table 7).

Table 7
Solubility of ammonium sulfate in sulfuric acid, at room temperature

№	Conc. H ₂ SO ₄ , %	Ammonium Sulfate Content в 100 g H ₂ SO ₄ , g	The composition of the solution, wt. %		
			H ₂ SO ₄	(NH ₄) ₂ SO ₄	H ₂ O
1	65	43.0	45,77	30,28	23,95
2	75	40.0	53,57	28,57	17,86
3	85	35.0	62,96	25,92	11,12
4	93	30.0	71,30	23,12	5,58

Taking into account the high degree of decomposition in concentrated sulfuric acid during the processing of phosphorites and the increase in water-soluble forms of P₂O₅ in the finished product, as well as sulfuric acid concentrations in a solution of ammonium sulfate in sulfuric acid were 53.57-71.30%. The concentrations of ammonium sulfate in the selected solutions are 28.57%, 25.92% and 23.12%, respectively. Experiments were carried out on the basis of different (15-60) norms of stichiometric quantities of solutions.

Table 8.

Decomposition of unenriched fosphates with an incomplete norm of sulfuric acid in the presence of ammonium sulfate

Norm H ₂ SO ₄ , %	Content P ₂ O ₅ , % In ready product			CO ₂ , %	N, %	H ₂ O, %	Coefficient, decomposition, %
	general.	ass	water.				
Decomposition with a solution of the content 53,57% H ₂ SO ₄ -28,57% (NH ₄) ₂ SO ₄ -17,86% H ₂ O							
15	16,70	3,69	1,05	12,10	0,96	0,16	22,09
40	14,02	7,50	2,67	8,40	2,01	2,61	53,49
50	12,62	9,33	3,97	6,90	2,54	3,36	73,93
60	12,16	10,07	5,69	4,10	2,79	3,94	82,81
Decomposition with a solution of the content 62,96% H ₂ SO ₄ -25,92% (NH ₄) ₂ SO ₄ -11,12% H ₂ O							
15	16,97	3,87	1,31	11,1	0,77	0,56	22,80
40	14,50	8,15	2,95	7,80	1,66	0,84	56,20
50	13,18	9,92	4,22	5,70	2,27	1,49	75,26
60	12,69	10,54	5,11	3,20	2,33	2,02	83,05
Decomposition with a solution of the content 71,30% H ₂ SO ₄ -23,12% (NH ₄) ₂ SO ₄ -5,58% H ₂ O							
15	17,10	4,10	1,59	10,9	0,59	0,41	23,97
40	15,06	8,59	3,29	7,30	1,31	0,49	57,03
50	13,88	10,98	5,91	5,10	1,69	0,63	79,10
60	13,48	11,68	5,90	3,01	1,88	0,79	86,64

Table 9.

Decomposition of poor phosphorites with an incomplete norm of sulfuric acid in the presence of ammonium sulfate

Norm H ₂ SO ₄ , %	Content P ₂ O ₅ , % In ready product			CO ₂ , %	N, %	H ₂ O, %	Coefficient, decomposition, %
	general.	ass.	water.				
Decomposition with a solution of the content 53,57% H ₂ SO ₄ -28,57% (NH ₄) ₂ SO ₄ -17,86% H ₂ O							
15	11,07	1,67	0,63	16,5	0,98	1,01	15,08
40	9,25	4,63	1,41	12,0	2,07	2,98	50,05
50	8,37	5,93	2,15	8,9	2,61	4,10	70,84
60	8,06	6,03	2,66	5,91	2,88	4,55	74,44
Decomposition with a solution of the content 62,96% H ₂ SO ₄ -25,92% (NH ₄) ₂ SO ₄ -11,12% H ₂ O							
15	11,16	1,95	0,66	15,9	0,79	0,98	17,47
40	9,69	4,99	1,64	10,1	1,74	1,17	51,49
50	8,87	6,41	2,67	6,64	2,22	1,63	72,26
60	8,53	6,81	3,08	4,15	2,45	2,19	79,83



Decomposition with a solution of the content 71,30% H ₂ SO ₄ -23,12% (NH ₄) ₂ SO ₄ -5,58% H ₂ O							
15	11,43	2,26	0,96	15,1	0,61	0,65	19,77
40	10,17	5,62	2,12	9,00	1,38	0,78	55,26
50	9,48	7,15	3,11	4,88	1,80	0,99	75,42
60	9,10	7,49	3,45	3,82	1,97	1,26	82,30

The results of the study show that when enriched phosphorite is decomposed in an acid concentration of 75% in a solution of 28.57% of ammonium sulfate in sulfuric acid, from 22.09 to 82.81%, ie 3.75 times, ammonium sulfate in 85% concentration of acid in accordance with the 15-60 standard. at a suitable rate of 25.92% solution from 22.80 to 83.05%, ie 3.64 times, and when decomposed using a solution containing 23.12% ammonium sulfate in 93% concentrated acid, from 23.97 to 86, It was found to increase by 64%, or 3.61 times. The decomposition process of low-grade phosphorites is almost indistinguishable from the decomposition process of unenriched phosphorites. However, studies based on the norms of solutions of the above composition have shown that the decomposition rate of low-grade phosphorites increases in proportion to the acid concentration; A 28.57% solution of 75% H₂SO₄ with a change of 15-60% to 4.93 times, a 25.92% solution of 85% H₂SO₄ with a change of 15-60% to 4.57 times and 93% A 23.12% solution of H₂SO₄ has been shown to increase 4.16 times with a 15-60% change in the norm.

IV. CONCLUSION

It has been established that in all the studied norms for the decomposition of phosphorite by sulfuric acid, a non-caking friable product is obtained and has good marketable properties that satisfy the requirements of agriculture. When poor phosphorites are decomposed at a 60% norm of sulfuric acid in the presence of ammonium sulfate, a complex fertilizer is obtained containing 1.5-2.5% nitrogen and 8-9% total phosphorus. Studies have established that a product from poor phosphorites with their moisture content up to 5% has good properties, i.e. 100% friability remains. To further improve the properties and increase the nitrogen nutrient content in the synthesized product - azosuperphosphate, it is recommended to use the compositions obtained in table 5 obtained at higher temperatures. Based on the studies, the optimal technological parameters of the main stage are established - the process of decomposition of low-grade phosphorites: norm of sulfuric acid-60-80% of stoichiometry; Content for decomposition: 71,30% H₂SO₄-23,12% (NH₄)₂SO₄-5,58% H₂O; interaction time-5-10 min; product granulation time-10-20 min.

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