

# STUDY OF THE DECOMPOSITION OF PHOSPHORITES WITH SULFURIC ACID

Abdurakhmanova Navbahor Kakhramanovna  
Candidate of Technical Sciences, Associate Professor  
Branch of the Russian State Gubkin University in Tashkent

Nazirova Rakhsamokhon Mukhtarovna  
Doctor of Technical Sciences (PhD), Associate Professor of the Department "Technology Storage and Primary Processing of Agricultural Products" of the Fergana Polytechnic Institute;

Mirsalimova Saodat Rakhmatjanovna  
Candidate of Chemical Sciences, Associate Professor, Head of the  
Department of Chemical Technology of the Fergana Polytechnic Institute.

## ANNOTATION:

**It is known that the phosphorites of the Central Kyzyl Kum are poor raw materials and have a high calcium modulus. The peculiar structural features characterizing the high reactivity of high-carbonate Kyzylkum phosphorites give reason to believe that the most optimal solution is to process them into effective fertilizers by chemical activation of phosphate raw materials.**

**Keywords: phosphorites, sulfuric acid, degree of decomposition, concentration, acid rate, acid activity, stoichiometry.**

## INTRODUCTION:

Phosphorus occupies a special place among the chemical elements. It is found in many minerals, primarily calcium phosphates. In living nature, it forms organophosphorus compounds, which serve as carriers of high-energy reactions that ensure the vital activity of living organisms. The role of phosphorus in wildlife is unique. Assessing it, Isaac Asimov wrote that you can find a replacement for coal, oil or iron, but there is no substitute for phosphorus. Plants take phosphorus from the soil and with each harvest the soil becomes impoverished. To preserve the fertility and

reproduction of agricultural products, it is necessary to replenish the stock of phosphorus in the soils, which is carried out by the introduction of mineral fertilizers. For these purposes, about 80 % of the extracted phosphates are used.

The revealed world reserves of phosphate ores are accounted for in more than 60 countries and are estimated at 63 billion tons of  $P_2O_5$ . Five countries (Morocco, USA, China, Russia and Kazakhstan) have 49.5 billion tons of  $P_2O_5$ , which is almost 80 % of the world's total reserves. The resources of phosphate raw materials in Uzbekistan amount to 190 million tons of  $P_2O_5$ . The  $P_2O_5$  content in phosphate raw materials produced in various countries of the world varies widely - from 21 % (Uzbekistan) to 38.2 % (Nauru).

The economic indicators of the production of phosphate fertilizers are directly related to the quality of the phosphate raw materials used. The best phosphate raw material in the world is apatite concentrate from the Khibiny deposit in Russia. It contains 39.4 %  $P_2O_5$ . Calcium module, i.e. the ratio of  $CaO: P_2O_5$ , it is minimal and equal to 1.32. There are no carbonates in it. Currently, only for him there is a technology for processing into any phosphorus-containing fertilizers with good technical and economic indicators. The

transition to the processing of poor phosphorites significantly worsens the technical and economic indicators of the production of phosphorus-containing fertilizers. For example, the production of 1 ton of  $P_2O_5$  in extraction phosphoric acid from Karatau phosphorites (24.5 %  $P_2O_5$ ,  $CaO: P_2O_5 = 1.56$ , 8.0%  $CO_2$ ) costs twice as much as from the Khibiny apatite concentrate. Phosphate concentrates containing at least 33%  $P_2O_5$  are marketed in the world market for the production of phosphorus fertilizers.

But only a few countries have rich raw materials. These are the USA, Russia, the countries of North-West Africa, the Middle East. Moreover, the deposits of such raw materials in these countries are already severely depleted. It is becoming more and more difficult and expensive to extract and process phosphate raw materials. Therefore, all over the world there is a search for ways to reduce the cost of the processes of obtaining phosphorus-containing fertilizers and to involve poor phosphate raw materials in the production.

Our Kyzylkum phosphorites are just such a poor raw material. The average sample of phosphorite from the Dzheroy-Sardarinsky deposit contains 16.2%  $P_2O_5$ , 17.7%  $CO_2$  and a calcium modulus ( $CaO: P_2O_5$ ) in it is 2.85. Such ore without concentration is not suitable for obtaining concentrated phosphorus fertilizers from it by acid extraction. The Kyzylkum phosphorite plant is now producing ordinary phosphorite flour with a content of 18%  $P_2O_5$  by dry dressing and a thermoconcentrate with a content of 28%  $P_2O_5$  by roasting. Ordinary phosphate rock is now sent to the Kokand superphosphate and Samarkand chemical plants for the production of simple ammoniated superphosphate. But the performance indicators of the Kokand plant at the same time fell sharply: the content of  $P_2O_5$  in the product dropped to 11%; there is a large

overconsumption of sulfuric acid; superphosphate is not granulated, the plant's capacity dropped from 47 thousand tons to 25.44 thousand 100%  $P_2O_5$ .

In the production of ordinary phosphorite flour by the dry dressing method, large-tonnage production wastes are generated - mineralized mass (12-14%  $P_2O_5$ ) and dusty fraction (18%  $P_2O_5$ ), which are still stored until future times.

There is no more important task for scientists and production workers now how to find economical ways processing of ordinary phosphorite flour, mineralized mass and dust-like fraction of phosphorites of the Central Kyzyl Kum into effective single phosphorus and complex fertilizers. Analysis of the published literature has shown that this can be done using the so-called non-traditional methods of processing phosphate raw materials, namely, methods of chemical, mechanochemical and microbiological activation of phosphate raw materials.

Chemical activation implies the decomposition of raw materials with an incomplete norm of an acid reagent (sulfuric and nitric acids) to obtain undecomposed phosphates, but containing a high percentage of assimilable  $P_2O_5$ . This also includes the task of intensifying the production of simple superphosphate, as well as the use of phosphoric acid decomposition to obtain such a promising complex fertilizer as ammophosphate.

To study the process of obtaining simple superphosphate by the chamber method, sulfuric acid decomposition of phosphate raw materials from Central Kyzylkum and maturation of acidic superphosphate were carried out as follows. A predetermined amount of acid of a certain concentration was poured into a porcelain glass equipped with a paddle stirrer. The acid was preheated to a selected temperature in the range from 65 to

75 °C, depending on the concentration of H<sub>2</sub>SO<sub>4</sub>. Phosphorite was dosed into the heated acid for one minute. Large (up to 10-12 mm) and fine cellular (3-4 mm) foam is formed in the reactor, which disintegrates within 1-3 minutes. With the addition of sulfuric acid, the foam height increased, reaching 2/3 of the slurry layer height. The interface between the pulp and foam is clear. After that, the pulp in a glass was stirred for another 5-7 minutes, then the resulting sour mass was kept in an oven for 1.5-2 hours at 110-120 °C (to approach the temperature regime of maturation in the current production of simple superphosphate by the chamber method).

To establish the optimal technological parameters of the process of opening phosphate raw materials with the maximum decomposition coefficient, the experiments were carried out in the range of sulfuric acid concentration of 56-64 % and the norm of 80-100 % of stoichiometry. The stoichiometric rate of sulfuric acid for the decomposition of phosphate raw materials to dihydrogen phosphate-calcium was calculated taking into account the content of P<sub>2</sub>O<sub>5</sub>, CaO, and CO<sub>2</sub> in it. for the experiments, phosphorites with a particle size of 0.2-0.4 mm were used. The obtained samples of acidic superphosphates were subjected to chemical analysis for the content of various forms of P<sub>2</sub>O<sub>5</sub> and moisture according to generally known methods.

The rate of decomposition of phosphorites by sulfuric acid depends on many factors, in particular, on the concentration and rate of acid, the ratio of the solid and liquid phases, the time of mixing and ripening, etc. Sulfuric acid decomposition of phosphorite is a heterogeneous process that occurs at the interface between the solid and liquid phases. The process covers the following stages:

- A) diffusion of sulfuric acid to the surface of phosphorite;
- B) chemical interactions of reagents;

C) diffusion of the resulting reaction products in the system.

The concentration of sulfuric acid directly affects the reaction rate, the amount of the liquid phase and its initial composition. Therefore, it is of great importance in the decomposition of phosphorites. The more concentrated the acid, the greater the heat of reaction and the more pronounced the water. If a highly diluted sulfuric acid is used, the temperature rise will be negligible and the reaction rate will be reduced as a result. In addition, the superphosphate must be dried to remove excess moisture. When determining the optimal acid concentration, one should take into account not only the reaction rate, but also the physical properties of the product.

Table 1 shows the chemical and analytical data on the composition of fresh acidic superphosphates based on unrefined ore, washed and thermoconcentrates and the results of calculating the decomposition coefficient of phosphate raw materials depending on the concentration and rate of sulfuric acid. The data obtained show that with an increase in the concentration of sulfuric acid from 56 to 60 %, the degree of decomposition of the washed concentrate increases at a 100 % rate from 88.6 to 90.6 % (Fig. 1). this is due to an increase in the activity of acid hydrogen ions in the system. A further increase in the concentration of sulfuric acid leads to an increase in the concentration of the formed calcium sulfate in the liquid phase, thereby reducing the reaction rate and, accordingly, the degree of decomposition of the washed concentrate. The degree of P<sub>2</sub>O<sub>5</sub> conversion is 86.5 % when a concentration of 64.5 % is used for the decomposition of sulfuric acid.

The same picture is observed during the decomposition of the original phosphorite ore and thermoconcentrate. Moreover, the interaction of ore with sulfuric acid proceeds faster compared to washed concentrate. The

highest value of the degree of ore decomposition of 93.7 % is achieved with a sulfuric acid concentration of 58%. A significant decrease in the decomposition rate to 90.8 % is observed at a sulfuric acid concentration of 64%.

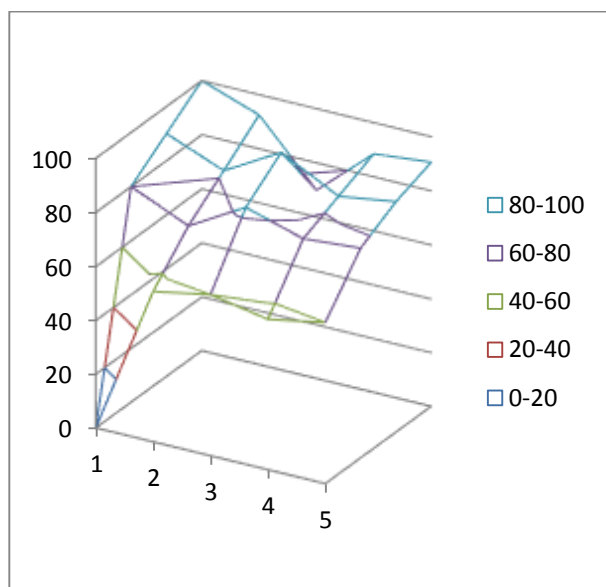


Fig. 1. Influence of concentration and rate of sulfuric acid on decomposition

The results show that during the decomposition of the thermoconcentrate, the concentration of sulfuric acid in the range studied by us has little effect on the coefficient of  $\alpha_{\text{evil}}$ . When the acid concentration changed from 56% to 64%, the degree of decomposition of the thermoconcentrate varied from 86.7% to 87.1%, reaching a maximum (87.7 %) at a sulfuric acid concentration of 62 % and 100 % normal.

This character of the curves is explained by the influence of two factors on the rate of decomposition of phosphorite: the activity of the acid (concentration of free hydrogen ions) on which the initial rate of decomposition of phosphorites and the resistance to diffusion of the acid to the surface of the unreacted mineral depend.

All other things being equal, the rate of the decomposition process depends on the crystallization conditions of calcium sulfate. In the region of low  $\text{H}_2\text{SO}_4$  concentration (56 %),

the increase in acid activity with an increase in its concentration is small, compared with an increase in the resistance of the layer of calcium sulfate crystals on the phosphate particles. With an increase in the concentration of sulfuric acid up to a certain limit, the increase in the activity of the acid outstrips the increase in the resistance to diffusion to the mineral.

Table 1. Influence of concentration and rate of sulfuric acid on decomposition  
(in fresh sour superphosphate)

H <sub>2</sub> O <sub>4</sub> concentration wt. %	H <sub>2</sub> O <sub>4</sub> norm from stoichiometry	P <sub>2</sub> O <sub>5</sub> content wt. %				P <sub>2</sub> O <sub>5</sub> ass.	P <sub>2</sub> O <sub>5</sub> water.	P <sub>2</sub> O <sub>5</sub> free	Moisture wt. %
		general	assim.	water	free.	P <sub>2</sub> O <sub>5</sub> total % rel.	P <sub>2</sub> O <sub>5</sub> total % rel.	P <sub>2</sub> O <sub>5</sub> total % rel.	
1	2	3	4	5	6	7	8	9	10
				Raw ore					
	80	11,60	8,20	7,08	1,01	70,7	60,5	8,71	6,50
56	90	10,42	8,50	7,73	3,09	81,5	74,2	29,65	10,01
	100	9,53	8,81	7,92	4,21	92,5	83,1	44,20	16,42
	80	11,65	8,53	7,08	1,05	73,2	60,8	9,01	6,01
58	90	10,55	8,73	7,71	3,19	82,8	73,1	29,24	9,76
	100	9,63	9,02	8,09	4,43	93,7	84,0	46,00	15,30
	80	11,62	8,25	6,98	1,03	71,0	60,1	8,90	5,75
60	90	10,38	8,59	7,66	3,17	82,7	73,8	30,54	9,56
	100	9,73	9,13	8,43	4,65	93,4	86,6	47,79	14,13
	80	11,56	8,08	7,05	0,88	69,9	60,9	7,60	5,60
62	90	10,36	8,53	7,67	2,86	82,3	74,0	27,61	9,58
	100	9,93	9,21	8,50	5,72	92,7	86,0	57,40	12,15
	80	11,76	8,15	7,17	0,96	69,3	60,9	8,16	5,46
64	90	10,75	8,70	8,26	2,86	80,9	76,8	26,60	7,37
	100	9,73	8,84	8,66	5,98	90,8	89,0	61,46	14,35
				Washed concentrate					
	80	16,63	12,71	11,85	1,71	76,3	71,2	10,27	6,75
56	90	13,47	11,10	11,04	3,20	82,4	81,9	23,76	18,80
	100	12,60	11,16	10,09	5,08	88,5	87,2	40,32	20,92
	80	16,16	12,85	11,85	1,72	77,4	71,4	10,36	6,10
58	90	13,55	11,27	11,10	3,46	83,1	81,8	28,22	18,06
	100	12,66	11,40	11,05	5,24	90,4	87,3	44,94	20,34
	80	16,40	12,79	11,87	1,70	78,0	72,4	10,37	5,75
60	90	14,02	12,00	11,47	3,88	85,6	81,8	27,67	14,39
	100	13,00	11,77	11,76	6,21	90,5	90,4	47,77	16,93
	80	16,32	12,52	11,72	1,75	76,7	71,7	10,72	6,06
62	90	15,37	12,15	11,65	5,08	79,1	75,8	33,05	10,32
	100	14,57	12,73	12,49	7,42	87,4	85,7	50,93	10,05
	80	15,93	11,74	11,24	1,80	83,7	70,5	11,32	6,49
64	90	15,50	12,18	12,03	3,73	78,55	77,6	24,08	7,42
	100	14,34	12,40	12,03	7,81	86,5	83,9	54,48	10,07
				Thermoconcentrate					
56	90	13,98	11,27	11,16	4,01	80,6	79,8	28,68	14,85
	100	13,40	11,61	11,29	6,57	86,7	84,3	49,03	16,11
58	90	14,16	11,49	11,43	4,39	81,1	80,7	31,00	13,45
	100	13,56	11,79	11,70	6,45	86,9	86,2	47,57	15,83
60	100	13,79	12,62	11,88	5,50	87,1	86,7	39,88	13,70
62	100	13,90	12,18	12,09	4,64	87,6	86,9	33,38	10,51
64	100	13,91	12,11	11,98	2,73	87,1	86,2	19,63	10,79

Above the specified limit of sulfuric acid concentration (58-62 %), the process of decomposition of phosphorites slows down, since the initial decomposition rate at high concentrations of sulfuric acid is very high, as a result of which a large saturation of the phase complex of the system with calcium sulfate, formed under these conditions in the form of microdispersed crystals enveloping the phosphorite particles with a film that is not permeable to acid.

In the sulfuric acid decomposition of phosphorite ore in the studied range, the concentration of sulfuric acid, the interaction of the components proceeds very quickly with an abundant release of carbon dioxide, which promotes loosening of the film formed by calcium sulfate and phosphate, thereby improving the diffusion of the acid to the original mineral. This suggests that the higher the content of carbohydrate dioxide in phosphorite, the lower the concentration of  $H_2SO_4$  will be required for decomposition. This phenomenon is slower during the decomposition of the washed concentrate, and is practically unnoticeable for the thermoconcentrate.

The effect of the degree of saturation of the system with calcium sulfate on the resistance value and on the decomposition rate is clearly seen from the data obtained for different acid norms. The practical rate of sulfuric acid for apatite and phosphorites is recommended to be taken 3-5% higher than the calculated one. It is known that with an increase in the rate of sulfuric acid, the rate and degree of decomposition of phosphorite in the production of superphosphate increases. However, with an increase in the rate of sulfuric acid, the moisture content and the content of free acid in superphosphates increase, which leads to an increase in the hygroscopicity of the finished fertilizer.

The results of experiments on the influence of the acid rate on the degree of decomposition of phosphorite are in good agreement with the literature data. An increase in the rate of sulfuric acid from 80 to 100% in the entire studied range of phosphorite decomposition leads to an increased the amount of the liquid phase in the system and, naturally, an increase in the rate of interaction of the components and the decomposition coefficient of phosphate raw materials from 73 to 84 %.

The decomposition of the original phosphorite ore at 80 % sulfuric acid rate in the initial stage proceeds very quickly, then sharply slows down, due to the formation of a difficult-to-mix mass. This is due to the fact that a decrease in the amount of the liquid phase creates a very high saturation of the liquid phase with calcium sulfate, leading to the formation of a precipitate, the resistance of which exceeds the increase in acid activity. With an increase in the concentration of sulfuric acid from 56 to 64 % at 80 %, the rate, due to a decrease in the liquid phase in the reaction mass, becomes more complicated, the course of the ore decomposition process.

And the process of decomposition of the washed concentrate (at a rate of 80-90 %) under the same conditions proceeds gently, a well-mixed pulp is obtained, as a result of which favorable conditions are created for the interaction of components and the diffusion of hydrogen ions to the surface of the phosphate mineral. As a result, the Cr of the washed concentrate is 3-5 % higher than that of the original ore.

Studies have shown that the process of decomposition of the thermoconcentrate at 80 % acid rate and a concentration of 56-64 % is practically impossible due to the formation of a fast-setting mass. This phenomenon at a rate of 90 % occurs starting with a sulfuric acid concentration of 60 %. Not only the rate and

degree of decomposition of phosphate raw materials depends on the rate of sulfuric acid, but it also has a significant effect on the physicochemical properties of the finished superphosphate. At a reduced rate of sulfuric acid for the decomposition of phosphorite (80-90 % of stoichiometry) in freshly prepared acidic superphosphate, the content of free  $P_2O_5$  and moisture fluctuates in the range of 10-30 %  $H_2SO_4$  at all rates of sulfuric acid used, a decrease in the degree of decomposition by 3 is observed %. With a decrease in the concentration of sulfuric acid to 58 % for ore and up to 60 % for washed concentrate, it is possible to achieve decomposition of phosphorite already in the chamber product at rates of sulfuric acid 90-100 % of stoichiometry 82.7-93, 7 % and 85.6-90, 6 % respectively. At the same time, at a sulfuric acid rate of 80 % of stoichiometry, the degree of decomposition in the chamber superphosphate barely reaches 73.2 % for ore and 78.0 % for washed concentrate. An increase in the rate of sulfuric acid during the decomposition of phosphorite for concentrations (56-64 %) leads to an increase in the content of the water-soluble form of  $P_2O_5$  in the acidic superphosphate.

Thus, in the production of superphosphate, the rate of sulfuric acid for the decomposition of phosphate raw materials is of great importance. the physicochemical and mechanical properties of the finished fertilizers depend on it.

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