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CHEMICAL COMPOSITION OF SINGLE-SIDED PHOSPHORIC FERTILIZERS OBTAINED FROM BALANCED PHOSPHORITE ORE OF CENTRAL KYZYLKUM BY PHOSPHORIC ACID ACTIVATION WITH SULFURIC ACID ADDITION

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ABSTRACT

In this study, the process of activation of off-balance phosphorite ore of the Central Kyzylkum desert (14,33% P_2O_5*) by extraction phosphoric acid (14,32%* P_2O_5*) with the addition of sulfuric acid (93%) at 75 ℃, reaction time 30 min. at different mass ratios* P_2O_{5PAE}: P_2O_{5FC} and P_2O_{5PAE} : H_2SO_{4mng}*. It is shown that the addition of* H_2SO_4 *significantly intensifies the process of phosphoric acid activation of the phosphate mineral of the raw material. The water-insoluble part of the products of phosphoric acid activation with the addition of sulfuric acid has been studied. It has been shown that both the fertilizer itself and it's water-insoluble part contain a significant amount of the* P_2O_5 *form assimilable for plants.*

KEYWORDS: *Off-Balance Phosphorite Ore, Phosphoric And Sulfuric Acids, Activation, Single Phosphorus Fertilizers, Water-Insoluble Part.*

INTRODUCTION

Phosphorites of the Central Kyzyl Kum deposits are the main phosphate raw materials for the factories of Uzbekistan that produce phosphorus-containing fertilizers. But this is a raw material poor in phosphorus, containing a large number of undesirable impurities, in particular carbonates and chlorine, which is not subject to flotation enrichment [1].

To obtain high-quality phosphorus-containing fertilizers from such raw materials, the Kyzylkum Phosphate Plant (KPP), with the participation of the German company Engineering Dobersek GmbH, carried out a multi-stage enrichment: crushing, dry enrichment to obtain ordinary phosphorite flour, washing from chlorine, roasting to remove CO_2. Since 2007, this plant has put into operation capacities for the production of 400 thousand tons per year of washed calcined phosphoconcentrate (WCPC) with a content of up to 30% P_2O_5, 200 thousand tons per year of washed dried phosphoconcentrate (MSA with a content of 18-19% P_2O_5) and 200 thousand tons per year of ordinary phosphorite flour (OPF with a content of 16-18% P_2O_5). Of these, WCPC is the only phosphate raw material suitable for the production of ammophos at Ammophos-Maxam JSC. There is a lack of phosphate raw materials. In order to increase the production of ammophos in 2014 within the framework of the project "Expansion of the existing products for the enrichment of phosphorite raw materials", on the basis of the KPP according to the new enrichment scheme, the production capacity of WCPC was increased from 400 to 716 thousand tons per year with an average content of 26% P_2O_5 (picture).

At the same time, the volume of extracted phosphate ore with an average grade of 17.58% P_2O_5 is 1,874.6 thousand tons or 320.93 thousand tons of P_2O_5.

The investment project, although it will significantly expand in the future the resource base for WCPC (1.8 times) of ammophos production, but the volume produced (186.16 thousand tons of P_2O_5) cannot radically change the situation, since the planned volumes do not allow to cover the needs of agriculture in phosphate fertilizers (525.21 thousand tons of P_2O_5). In addition, the enrichment scheme currently functioning in KLF cannot be considered rational.

Since the new enrichment scheme does not provide for incidental production of both MSA and OPF. And an increase in the concentration of P_2O_5 in WCPC (26%) compared to its concentration in the original raw ore (17.58%) by only 8.42% occurs against the background of a significant loss of P_2O_5 (134.77 thousand tons of P_2O_5 or 42% of the initial P_2O_5 in ore) with dressing tailings with the status "Off-balance ore". These, 9.6% occurs during dry sorting at the site of the ore control station, 28.3% during hydro-sorting and 4.1% at the stages of drying and roasting. These are, respectively, a mineralized mass (12-14% P_2O_5), slurry phosphorite (10-12% P_2O_5) and a pulverized fraction (18-20% P_2O_5).

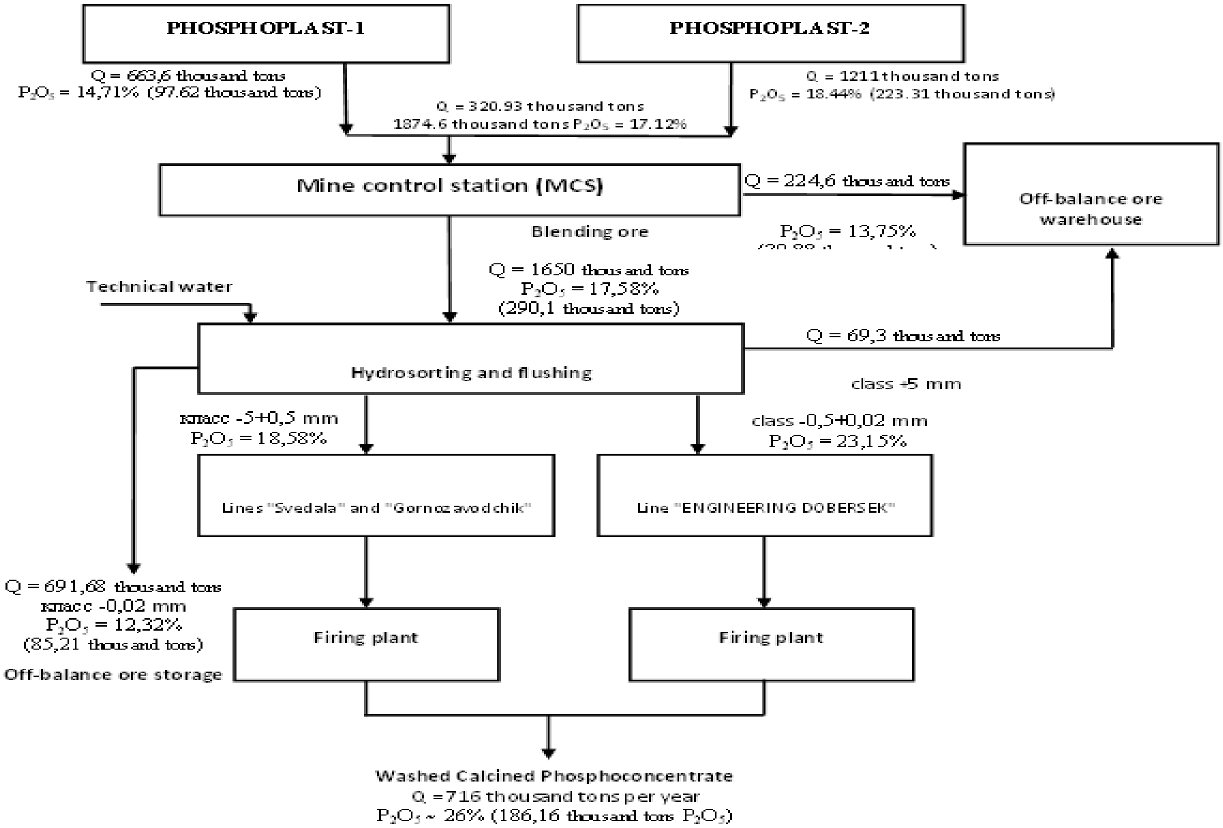

Fig. 1. New technological scheme of thermal enrichment of phosphorites of Central Kyzyl Kum

In this waste, we see a reserve for increasing the production of phosphorus-containing fertilizers. Traditional methods of processing phosphate raw materials, such as nitric and sulfuric acid, are not acceptable for them. Therefore, it requires an unconventional approach to the search for an alternative method for processing poor phosphorites into phosphorus-containing fertilizers. Therefore, we stopped at their phosphoric acid activation [2].

This allows, firstly, to involve poor phosphate raw materials in the production of concentrated fertilizers, and secondly, to obtain single phosphorus fertilizers, which are so necessary for applying for autumn ploughing. The essence of the activation process is the treatment of raw materials with phosphoric acid, but in much smaller quantities than is required for the complete decomposition of phosphorites. In this case, the so-called undecomposed or partially decomposed phosphates are formed, agrochemical tests of which have shown their high efficiency [3, 4].

Earlier [5], the mineralized mass was activated with the following composition (wt.%): 14,33 P_2O_5; 43,02 CaO; 1,19 MgO; 1,38 Fe_2O_3; 1,18 Al_2O_3; 2,22 SO_3; 14,70 CO_2; 13,23 н.о.; CaO: P_2O_5 =3,0 n.d.; CaO: P_2O_5 =3,0 by treating it with extraction phosphoric acid of three concentrations (14.32; 18.55; 24.23% P_2O_5) at a temperature of 75 °C, a time of 30 minutes and at various rates of phosphoric acid (in the range of weight ratios P_2O_5 in acid to P_2O_5 in raw materials from 1: 0.66 to 1: 0.17, that is, in the range of phosphoric acid rates from 20 to 75% of the stoichiometric rate for the formation of monocalcium phosphate). At the request of agriculture, a single phosphorus fertilizer must have a high content of the total and assimilable forms of P_2O_5, and the relative content of the water-soluble form of P_2O_5 must be at least 50%. Based on this requirement, the optimal rate of phosphoric acid for processing the mineralized mass is 55% of the stoichiometry (P_2O_{5PAE} : $P_2O_{5ФС}$ = 1: 0,24). In this case, the product contains $P_2O_{5tot.}$ - 40,98%; $P_2O_{5усв.}$ - 30,06%; $P_2O_{5wate.}$ - 21,28%; $P_2O_{5усв.}$: $P_2O_{5tot.}$ = 73,35%; $P_2O_{5wate.}$: $P_2O_{5tot.}$ = 51,93%. It is calcium dimonophosphate. The main component of calcium dimonophosphate is a mixture of salts of water-soluble monocalcium phosphate $Ca(H_2PO_4)_2 \cdot H_2O$ and citrate-soluble dicalcium phosphate $CaHPO_4 \cdot 2H_2O$. The advantage of this fertilizer is that it contains phosphorus in both fast- and slow-acting forms. The fast-acting form of P_2O_5 in the fertilizer is monocalcium phosphate, and the slow-acting form is dicalcium phosphate and undecomposed but activated phosphorite. Therefore, this fertilizer has a prolonged action. The results of this work showed that an increase in the concentration of phosphoric acid has practically no effect on the composition of the fertilizer.

From the practice of obtaining double superphosphate, when phosphoric acid for the decomposition of raw materials is taken in an amount of 5-10% more than the stoichiometric norm for the formation of monocalcium phosphate, it is known that the rate of decomposition of natural phosphates by phosphoric acid at the initial stage of the process is relatively high, but over time it sharply decays and becomes so small that in practice the reaction is stopped when the degree of decomposition of phosphate reaches 80-90% [6]. The reaction of the interaction of phosphates with extraction phosphoric acid from poor phosphorites proceeds especially slowly, as a result of which this acid is considered unsuitable for the production of double superphosphate [7]. The process of decomposition of phosphate raw materials with phosphoric acid can be intensified by adding to it a small amount of nitric, hydrochloric and sulfuric acids [8-10]. The use of sulfuric acid was undertaken in this work. The chemistry of the interaction of phosphate raw materials with phosphoric acid is as follows. The phosphate mineral reacts with phosphoric acid by the reaction:

$$Ca_5F(PO_4)_3 + 7H_3PO_4 + 5H_2O \longrightarrow 5Ca(H_2PO_4)_2 \cdot H_2O + HF$$

According to this equation, for 1 part by weight of P_2O_5 in the phosphate mineral, 2.33 parts by weight of P_2O_5 in acid is required. Carbonates present in the highly carbonated phosphorites of the Central Kyzyl Kum are also decomposed by phosphoric acid:

$$CaCO_3 + 2H_3PO_4 \longrightarrow Ca(H_2PO_4)_2 \cdot H_2O + CO_2$$

$$Ca_5F(PO_4)_3 + 2H_3PO_4 \longrightarrow 5CaHPO_4 + HF$$

According to this equation, for 1 weight part of CO_2 in the feed, 3.23 weight parts of P2O5 in acid is required. Therefore, for the complete decomposition of the phosphate raw material with phosphoric acid with the formation of monocalcium phosphate per one weight part ($P_2O_5 + CO_2$) in the raw material, 5.56 weight parts of P_2O_5 in acid are required. The reaction of the interaction of a phosphate mineral with phosphoric acid with the formation of dicalcium phosphate is unlikely:

The product containing $CaHPO_4$ can be obtained by direct interaction of phosphorite, phosphoric acid and water in an autoclave under a pressure of 7 atm. at 200 °C. In our case, dicalcium phosphate can be formed as a result of hydrolysis of monocalcium phosphate [6]:

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

The sulfuric acid present in PAE, as a result of reaction with phosphorite, forms gypsum and phosphoric acid:

$$Ca_5(PO_4)_3F + 5H_2SO_4 + 10H_2O = 5[CaSO_4 \cdot 2H_2O] + 3H_3PO_4 + HF$$

It can be seen from the above reaction equations that phosphoric acid is consumed not only for the decomposition of the phosphate part of the raw material but also for the decomposition of the impurity minerals contained in it.

The activation of the mineralized mass (14.33% P_2O_5) with phosphoric acid in a mixture with sulfuric acid was carried out in the range of weight ratios P_2O_5 in acid to P_2O_5 in raw materials from 1: 0.29 to 1: 0.24 and P_2O_5 in acid to $H_2SO_{4(monohydrate)}$ from 1: 0.25 to 1: 0.5. For processing of phosphate raw materials used extraction phosphoric acid composition (wt. %): 14,32 P_2O_5; 0,86 CaO; 0,29 MgO; 1,32 Fe_2O_3; 0,32 Al_2O_3; 0,38 SO_3 and sulfuric acid of 93% concentration. The experiments were carried out as follows: a mixture of phosphoric and sulfuric acid was slowly poured into a thermostated glass beaker, in which a sample of phosphate raw material was located, at a temperature of 65 °C. The mixture was thoroughly mixed. The duration of contacting the components was 30 min. at a temperature of 75 °C. Then the mass was dried, first at 60 °C for a day, and then at 105 °C until constant weight. And granulation of samples of wet phosphate masses was carried out in the process of drying by the method of intensive stirring and rolling. The moisture content of the pulp was determined by drying to constant weight. The dried product was analyzed for the content of various forms of phosphorus and calcium according to generally accepted methods [11]. The strength of the granules was determined using a MIP-10-1 device [12]. The experimental results are shown in Table 1.

TABLE 1 COMPOSITION OF SINGLE PHOSPHATE FERTILIZERS OBTAINED IN THE PROCESS OF PHOSPHORIC ACID ACTIVATION WITH THE ADDITION OF SULFURIC ACID OF THE OFF-BALANCE ORE OF THE CENTRAL KYZYL KUM

Mass ratio P_2O_{5PAE}: P_2O_{5FS}	Pulp moisture after decomposition,%	product pH	The chemical composition of the dried product, wt. %							$P_2O_{5usv.}$: $P_2O_{5tot.}$ by lim. to-those,%	$P_2O_{5usv.}$: $P_2O_{5tot.}$ по тр. Б, %	$P_2O_{5wate.}$: $P_2O_{5tot.}$ %	$CaO_{усв.}$: $CaO_{tot.}$ by lim. to-those,%	$CaO_{wate.}$: $CaO_{tot.}$ %	Strength, MPa
			P_2O_{5tot}	$P_2O_{5усв.}$ by lim. to those	$P_2O_{5усв.}$ по 0,2 М трил.Б	P_2O_{5wate}	$CaO_{tot.}$	$CaO_{усвby\ lim.\ to\ those}$	$CaO_{wate.}$						
With the addition of H_2SO_4 at a ratio of P_2O_{5PAE} : H_2SO_{4mng} = 1: 0.25															
1:0,29	57,33	2,60	36,75	25,56	24,61	16,95	26,61	17,70	10,24	69,55	66,96	46,12	66,52	38,48	3,18
1:0,26	56,54	2,58	37.98	27,19	26,09	19,32	25,51	18,06	11,56	71,59	68,69	50,87	70,79	45,32	2,96

1:0,24	55,06	2,44	38,99	28,80	27,68	21,83	24,43	18,27	12,11	73,86	70,99	55,99	74,79	49,57	2,38
With the addition of H_2SO_4 at a ratio of P_2O_{5PAE} : H_2SO_{4mng} =1 : 0,375															
1:0,29	56,53	2,50	35,28	24,63	24,17	19,47	25,52	18,45	13,43	69,81	68,51	55,19	72,30	52,63	2,78
1:0,26	55,72	2,48	36,16	26,04	25,36	21,74	24,40	18,64	14,54	72,01	70,13	60,12	76,39	59,59	2,61
1:0,24	54,17	2,33	37,15	27,50	26,64	24,09	23,24	18,62	14,72	74,02	71,71	64,85	80,12	63,34	2,21
With the addition of H_2SO_4 at a ratio of P_2O_{5PAE} : H_2SO_{4mng} =1 : 0,5															
1:0,29	55,76	2,37	34,98	24,86	24,19	19,82	25,25	19,58	14,54	71,07	69,15	56,67	77,54	57,58	2,23
1:0,26	54,88	2,35	35,85	26,31	25,35	22,43	24,07	19,74	14,29	73,39	70,71	62,57	82,01	63,52	2,13
1:0,24	53,33	2,21	36,82	27,59	26,58	24,38	23,02	19,92	15,44	74,93	72,19	66,21	86,53	67,07	1,46

It can be seen from it that the more phosphate raw materials are taken for activation, the lower the content in the products of the general, assimilable and water-soluble form of P_2O_5, the assailable and water-soluble forms of CaO, the more the total form of CaO. The addition of sulfuric acid increases the content of water-soluble forms of P_2O_5 and CaO in products and decreases the content of the general form of P_2O_5. So, with the ratio P_2O_{5PAE} : H_2SO_{4mng} = 1: 0.25, an increase in the proportion of phosphate raw materials in the form of P_2O_5 relative to P_2O_5 in acid from 1: 0.24 to 1: 0.29 leads to a decrease in $P_2O_{5tot.}$ in the product. From 38.99 to 36.75%, P_2O_5 ass. for citric acid from 28.80 to 25.56%, P_2O_5 water. from 21.83 to 16.95%, $SaO_{usv.}$ from 18.27 to 17.70%, $CaO_{wq.}$ from 12.11 to 10.24% and an increase in $CaO_{total.}$ from 24.43 to 26.61%. Agrochemists and soil scientists consider the best phosphorus fertilizer to be one in which the content of the water-soluble form of P_2O_5 is at least 50% of the total form of P_2O_5. When the mineralized mass is activated with phosphoric acid at P_2O_{5PAE} : H_2SO_{4mng} = 1: 0.25, the optimal P_2O_{5PAE} : H_2SO_{4FS} ratio is 1: 0.26. The product has $P_2O_{5total.}$ = 37.98%, P_2O_5 ass. : P_2O_{5total}. = 71.59% and P_2O_5 water. : $P_2O_{5total.}$ - 50.87%. An increase in the amount of sulfuric acid additive (P_2O_{5PAE} : H_2SO_{4mng} from 1: 0.25 to 1: 0.5) allows to shift this limit towards increasing the amount of phosphate raw material to be processed. That is, the more sulfuric acid is introduced, the greater the amount of phosphate raw material can be involved in processing into concentrated single phosphate fertilizers. So, when adding sulfuric acid in the ratio P_2O_{5PAE} : H_2SO_{4mng} = 1: 0.375, the optimal ratio of P_2O_{5PAE} : H_2SO_{4FS} is not 1: 0.26, but becomes already 1: 0.29. In this case, the product contains P_2O_{5total}. = 35.28%, $P_2O_{5ass.}$: P_2O_{5total}. = 69.81% and $P_2O_{5water.}$: P_2O_{5total}. - 55.19%.

The resulting fertilizers contain calcium dihydrogen phosphate, calcium hydrogen phosphate, iron and aluminium phosphates, calcium sulfate, activated and undecomposed phosphorite. The presence of sulfur in the form of soluble calcium sulfate in the fertilizer promotes better utilization of basic plant nutrients. Sulfur is a part of amino acids - cysteine and methionine is of great importance in protein metabolism and in redox processes. In terms of its physiological role in plant nutrition, sulfur should be ranked third after nitrogen and phosphorus. It has a positive effect on the formation of chlorophyll, promotes the formation of nodules on the roots of legumes and nodule bacteria that assimilate atmospheric nitrogen. It increases the efficiency of nitrogen fertilizers and increases the mobility of soil phosphates. The productivity of cotton increases from 2.1 to 16.3%, and the oil content of seeds up to 3.8%. The optimal sulfur rate is 20-40 kg/ha [13].

In the resulting products, the relative content of the assimilable form of P_2O_5 less than 100% indicates that some amount of undecomposed phosphate mineral remains in the fertilizer. We checked what is the undecomposed part of the phosphate mineral in the mineralized mass when activated with a mixture of phosphoric and sulfuric acids. For this, product samples obtained by activation were dissolved in a 20-fold amount of distilled water in relation to the weight of the fertilizer. Then the aqueous suspension was filtered under a vacuum of 550-600 mm Hg. on a Buchner funnel using one layer of filter paper. The precipitate remaining on the filter was washed with acetone. The washed precipitate was dried at 105 °C together with filter paper. The dried precipitate was weighed and analyzed. The degree of P_2O_5 transition from the product to the liquid phase was calculated as the ratio of the amount of P_2O_5 that passed into the solution to the initial amount of P_2O_5 in the product. The results are shown in the table. 2.

TABLE 2 THE COMPOSITION OF THE WATER-INSOLUBLE PART OF SINGLE PHOSPHORUS FERTILIZERS OBTAINED IN THE PROCESS OF PHOSPHORIC ACID ACTIVATION WITH THE ADDITION OF SULFURIC ACID OF OFF-BALANCE PHOSPHORITE ORE OF CENTRAL KYZYL KUM

Массовое соотношение $P_2O_{5ЭФК}$: $P_2O_{5ФС}$	Масса готового удобрения, г	Масса сухого осадка, г	Химический состав высушенного						$P_2O_{5\,усв.}$: $P_2O_{5\,общ.}$ по лим. к-те, %	$P_2O_{5\,усв.}$: $P_2O_{5\,общ.}$ по тр. Б, %	$CaO_{усв.}$: $CaO_{общ.}$ по лим. к-те, %	Степень перехода P_2O_5 из продукта в жидкую фазу, %
			$P_2O_{5\,общ.}$	$P_2O_{5\,усв.}$ по лим. к-те	$P_2O_{5\,усв.}$ по 0,2 М трил .Б	$CaO_{общ.}$	$CaO_{усв.}$ по лим. к-	$SO_{3\,общ.}$				
С добавкой H_2SO_4 при соотношении $P_2O_{5ЭФК}$: $H_2SO_{4мнг}$ =1 : 0,25												
1:0,29	10	5,85	29,93	11,93	10,24	29,93	11,80	5,11	39,86	34,21	39,42	52,35
1:0,26	10	5,62	29,03	10,06	8,38	27,20	12,84	5,72	34,65	28,83	47,20	57,04
1:0,24	10	5,31	28,77	8,63	6,65	25,16	13,61	8,97	29,99	23,11	54,09	60,82
С добавкой H_2SO_4 при соотношении $P_2O_{5ЭФК}$: $H_2SO_{4мнг}$ =1 : 0,375												
1:0,29	10	5,81	27,81	10,67	9,33	27,33	12,68	7,45	38,37	33,55	46,39	54,20
1:0,26	10	5,51	27,36	9,01	7,56	24,82	13,35	8,96	32,93	27,63	53,79	58,31

1:0,24	10	4,96	27,05	7,93	6,06	22,89	13,72	9,14	29,32	22,40	59,94	63,88
С добавкой H_2SO_4 при соотношении $P_2O_{5ЭФК}$: $H_2SO_{4мнг}$ =1 : 0,5												
1:0,29	10	5,76	25,58	9,37	8,23	25,85	16,61	11,11	36,63	32,17	64,26	57,87
1:0,26	10	5,40	24,91	7,73	6,66	24,48	17,16	13,83	31,03	26,74	70,09	62,47
1:0,24	10	4,61	24,68	7,08	5,23	20,99	17,53	14,00	28,69	21,19	83,52	69,09

From the data presented in the table, it can be seen that the greater the amount of the mixture of acids taken to activate the mineralized mass, the greater the degree of P_2O_5 transition from the product to the liquid phase. Thus, with a weight ratio of P_2O_{5PAE} : H_2SO_{4mng} = 1: 0.25, a decrease in the P_2O_{5PAE} : H_2SO_{4PS} ratio from 1: 0.29 to 1: 0.24 leads to an increase in the degree of P_2O_5 transition from 52.35 to 60.82%, with P_2O_{5PAE} : H_2SO_{4mng} = 1: 0.375 from 54.20 to 63.88% and with P_2O_{5PAE} : H_2SO_{4mng} = 1: 0.5 from 57.87 to 69.09%.

Comparison of the degree of transition of P_2O_5 into the liquid phase of the water-insoluble part of the products obtained only by phosphoric acid activation ranged from 38.69 to 52.06% [5], and when using a mixture of phosphoric and sulfuric acids, this indicator increased from 52.35 to 69.09%. Such a change in the degree of transition of P_2O_5 into the liquid phase suggests that the use of sulfuric acid as an additive to phosphoric acid intensifies the process of decomposition of the phosphate mineral. The water-insoluble precipitate obtained at optimal ratios P_2O_{5PAE}: P_2O_{5FS} = 1: 0.29 and P_2O_{5PAE} : H_2SO_{4mng} = 1: 0.375 contains 27.81% P2O5total, 27.33% CaO_{total}, and the ratio in it $P_2O_{5usv.}$: $P_2O_{5total.}$ = 38.37%, $CaO_{usv.}$: SaO_{bsch}. = 46.39%. The high relative content of the assimilable form confirms the activation of the phosphate raw material during its phosphoric acid activation. In this case, the degree of P_2O_5 transition from the product to the liquid phase is 54.20%. This fertilizer sediment is assimilated by plants evenly during the period of their growth and maturation. Thus, the conducted studies convincingly show that the phosphate raw material base of Uzbekistan can be significantly expanded by involving local off-balance phosphorites of the Kyzyl Kum in the processing of concentrated single phosphorus fertilizers using the method of phosphoric acid activation. Establishing the production of these types of fertilizers is very promising, also in terms of reducing the consumption of acid reagent in the processing of poor phosphate raw materials.

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