

STUDY OF OBTAINING WET-PROCESSING PHOSPHORIC ACID PROCESSES BY CLINKER METHOD ON BASE OF MINERALIZED MASS

Abstract.

The article presents the results of the study of extractive phosphoric acid extraction processes from the mineralized mass (MM) - mineralized mass (MM) produced during the thermal processing of Central Kyzylkum (CK) phosphorite. Effects of sulfuric acid concentration on the quality parameters of the obtained wet-processing phosphoric acid (WPPA), processes of WPPA extraction through different concentrations of phosphoric acid solution from phosphoric acid-gypsum porridge were studied. The optimal sizes of these processes have been determined. In the composition of WPPA received in acceptable sizes 19,30-19,59% P_2O_5 , 0,08-0,09% F will be. Filtration rates of phosphoric-acid-gypsum porridges during extraction with different concentrations of WPPA are equal to 1096-1192 $kg/m^2 \cdot h$. It was also determined that the main composition of the resulting phosphogypsum is as follows: 2,44-2,59% $P_2O_{5total(t)}$, 29,93-30,09% $CaO_{total(t)}$, 41,29-41,50% SO_3 and F 1,16-1,21%.

Key words: mineralized mass, sulfuric acid, wet-processing phosphoric acid, decomposition coefficient, separation coefficient, filtration rate.

INTRODUCTION.

Currently, there are two methods of producing phosphoric acid from phosphate raw materials (PRM) in the world industry: the first is the dry (electrofurnace) method, the phosphorus contained in PRM is returned in free form, the obtained phosphorus is oxidized, and phosphoric acid is received from the P_2O_5 formed. The phosphoric acid produced by the electrofurnace method from phosphate raw materials is called thermal phosphoric acid (TPA). The second method is called the extraction method, in which phosphoric acid is extracted from PRM using mineral acids (nitric, hydrochloric, and sulfuric acids), and the extracted phosphoric acid is called wet-processing phosphoric acid (WPPA). Despite the purity and high concentration of TPA, its cost is much more expensive than WPPA. But still, TPA is used to obtain phosphorus compounds needed by the food and pharmaceutical industries. WPPA is mainly used for the production of simple and complex fertilizers containing phosphorus.

Nowadays there are two methods the production of WPPA, which are the first decomposition of phosphate raw materials with WPPA and then sulfuric acid extraction (wet method) and the first treatment of phosphate raw materials with 93-98% sulfuric acid and then the extraction of phosphoric acid from it with water, is to get (clinker method). The first method, the wet method, is very common around the world. This method is used in countries with almost have phosphate raw materials. In these processes, calcium sulfate is formed as a precipitate and it is separated from the main suspension. Depending on the temperature and the amount of P_2O_5 and SO_4^{2-} in the solution, calcium sulfate is formed in the form of dihydrate ($CaSO_4 \cdot 2H_2O$), hemihydrate ($CaSO_4 \cdot 0,5H_2O$) and anhydride ($CaSO_4$). The main method of production of WPPA, which is relatively simple and reliable both in our country and in other countries, is the dihydrate method. [1].

A lot of scientific studies have been conducted in our country on obtaining WPPA from PRM by sulfuric acid method. N.V. Volynskova and others studied in detail the processing of washed and burned phosphorite concentrate (WBPC) thermally enriched from phosphorites of the Central Kyzylkum (CK) with sulfuric acid to obtain WPPA. [2-6]. In this study, the main technological variables of WPPA extraction (process temperature, ratio of S:L, circulating WPPA concentration and the amount of free SO_3 in WPPA) and the effect of these parameters on the values of $K_{decomposition}$, $K_{leaching}$ and K_{yield} in the sulfuric acid extraction of washed calcined phosphorite

concentrate (WBPC) were studied. The optimal parameters for sulfuric acid extraction have been determined: process temperature - 85-90°C, S : L ratio - 1 : (2,5-3,5), concentration of circulating WPPA - 12-15% and amount of free SO₃ in WPPA – 1,5-2,5%. In addition, the negative effect of free CaO in WBPC on sulfuric acid extraction was also studied. In order to eliminate this negative effect, a vacuum cooling system of sulfuric-phosphate acid slurry was used, and regular studies were carried out on the extraction of sulfuric acid WPPA from WBPC in the presence of ammonium sulfate, and an increase in $K_{\text{decomposition}}$, K_{leaching} and K_{yield} values was achieved.

Currently, 716000 tons of WBPC containing 26% P₂O₅ are being produced according to the new scheme, and there was a need to study its properties and determine the optimal conditions for obtaining WPPA, ammophos and diamphos from it. Sh.S. Namazov, A.R. By Seytnazarov and others, the physical-mechanical and physical-chemical properties of this WBPC and the optimal conditions for obtaining WPPA from it were determined. [7-8].

It has also been shown by us that it is possible to obtain WPPA from the chemically enriched phosphorite concentrate (CEPC), which is formed during the nitric acid enrichment of MK phosphorites. [9].

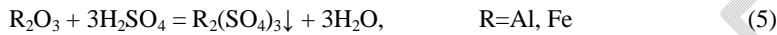
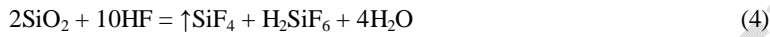
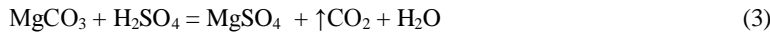
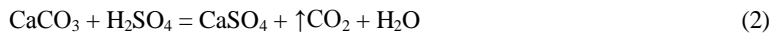
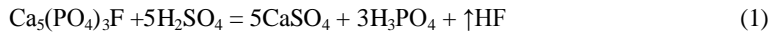
The second method of WPPA production, the clinker method, is currently not used in practice around the world. However, this method has also been studied in foreign countries. In the invention presented by the USA scientists [10], phosphate raw materials are mixed with 98% sulfuric acid and granulated, the grains are heated at 200-240°C or 350-400°C for the purpose of fluorine removal, and phosphoric acid is extracted from the resulting mixture using water. Production of WPPA in this way has a number of advantages: high concentration of P₂O₅ in the received WPPA and low amount of fluorine. Scientists of our country have not carried out scientific research on the extraction of WPPA from phosphate raw materials in this way. However, in recent times, we have conducted preliminary scientific studies on the clinker method of obtaining WPPA from phosphorites of Central Kyzylkum (MK) with low grade and high carbonate [11,12]. Extraction of phosphoric acid from ordinary phosphorite flour (OPF), which is Central Kyzylkum (CK) phosphorite, by the clinker method, and the effect of OPF decomposition time on its quality indicators, as well as the processes of extracting WPPA from phosphate-acid-gypsum porridge through water and phosphoric acid solution were studied. It was shown that 8,19-9,37% P₂O₅-containing WPPA (extracted with water) and 16,57-17,39% P₂O₅-containing WPPA (extracted with 10% WPPA) were formed at the given decomposition times. The filtration rates of phosphatic-acid-gypsum porridges in extraction with water and WPPA have been studied [11]. In [12], the possibility of obtaining WPPA from washed-burned phosphorite concentrate (WBPC) by clinker method was studied, and the optimal sizes of WPPA extraction processes with water were determined. 16,01-16,93% of P₂O₅ is contained in the WPPA composition obtained in acceptable sizes. [12] In contrast to this, in [13], the effect of different concentrations and standards of sulfuric acid on the extraction of from WBPC, and the containing 10% P₂O₅ was used in the extraction of from phosphoric acid gypsum porridge. 25,02-25,93% P₂O₅ is present in WPPA compositions received in acceptable sizes.

In this scientific research, scientific studies were carried out to study the processes of obtaining WPPA from mineralized mass (MM) by the clinker method from the phosphate waste generated during the thermal enrichment of PRM in our country.

RAW MATERIALS AND EXPERIMENTAL PROCEDURE.

For the experiments were used mineralized mass containing chemical composition (mass.%): 14,60% $P_2O_{5total(t)}$; 3,07% $P_2O_{5acceptable}$ by citric acid (ac.c.a.); 43,99% CaO; 1,01% MgO; 14,11% CO_2 ; 1,04% Al_2O_3 ; 0,89% Fe_2O_3 ; 1,58% SO_3 ; 1,30% F; 1,00% H_2O ; CaO: P_2O_5 – 3,01 and 10,32% insoluble residue.

Laboratory experiments on the decomposition of MM with sulfuric acid were carried out in a tubular glass reactor equipped with an electric motor-driven screw stirrer. This reactor was placed in a thermostat to keep the temperature constant. A pre-weighed phosphorite sample (MM) was placed in the reactor, and then sulfuric acid with different concentrations of 103 and 105% standards was slowly added. Sulfuric acid concentrations were obtained in the range of 70-93% min. Phosphorite decay time is 30 minutes. The temperature during the decomposition of phosphorite varies in the range of 80-100°C, depending on the standards and concentrations of the acid. The temperature of the reaction mass is kept at 80°C. When MM is decomposed with sulfuric acid, the following chemical reactions occur:



Due to the decomposition of MM with high concentration of sulfuric acid, the formation of large foams is not observed. The resulting phosphoric acid and gypsum clinker was mixed with 10, 12 and 15% WPPA at a ratio of 1,0:2,5 to the original MM for 5-10 minutes and filtered under vacuum. The coefficient of decomposition of MM ($K_{decom.}$), the coefficient of leaching P_2O_5 into solution ($K_{leaching}$) and the filtration rates of phosphoric acid gypsum pulps (porridge) were calculated [14]. The substances in phosphogypsum and extracted WPPA were analyzed by precise methods [15,16].

RESULTS AND DISCUSSION.

The results of the laboratory experiments are presented in Table 1. As can be seen from these received results, when the ratio of sulfuric acid is 103% and MM: H_2O =1.0:2.5, when its concentration increases from 70 to 93%, the amount of P_2O_5 in the obtained WPPA increases from 13,22 to 15,60%. $K_{decom.}$ and K_{yield} coefficients increase from 96,18 to 97,01% and from 93,02 to 93,35%, respectively. It is observed that the filtering speed of gypsum porridges with phosphate acid decreases from 1284 to 1105 kg/ m^2 ·h. In addition, it can be observed that the values of CaO, SO_3 , and F in the received WPPA samples increase from 0,31 to 0,41%, from 3,05 to 3,23%, and from 0,03 to 0,07%, respectively. The presence of such laws can be explained by the increase in the concentration of sulfuric acid. $P_2O_{5st.}$ in phosphogypsum samples formed in these sizes. and CaO_t amounts are observed to increase from 2,35 to 2,56% and from 29,11 to 30,01%, respectively. The values of SO_3 and F in its content increase from 40,55 to 41,42% and from 1,09 to 1,22%, respectively. A slight increase in the amount of these substances in phosphogypsum can be explained by a decrease in the filtration rate of phosphogypsum. When sulfuric acid is 105%, all the laws mentioned above are repeated. The amount of P_2O_5 in the received WPPA is slightly higher, up to from 13,32 to 15,93%. But it is observed that the values of CaO in its content increase from 0,28 to 0,35% and at the same time it is slightly less than that obtained in the norms of 103%. One of the main reasons for this is that the amount of Ca^{2+} ion passes into the solution due to the large amount of acid. All the laws observed in the 103% standard of sulfuric acid are also observed in the 105% standard. It may not be economically viable to process WPPA of this composition into simple and complex fertilizers, but these are preliminary results. Despite this situation, it is possible to obtain fertilizer and nutrient phosphates from this acid solution.

In our further studies, we focused on increasing the amount of P_2O_5 in WPPA solutions. For this purpose, we extracted phosphoric acid from the resulting phosphoric acid clinker through a 12%-WPPA solution. The procedure for performing experiments is the same as above. The received results are presented in Table 2. As can be seen from these results, the above-mentioned regularities are also repeated here. But in this case, all indicators are

higher than when extracted with 10% WPPA. For example, with H_2SO_4 -103% and in ratio MM:12%WPPA =1.0:2.5, when its concentration increases from 70 to 93%, the amount of P_2O_5 in the obtained WPPA content increases from 15,17 to 17,55%. $K_{\text{decom.}}$ and K_{yield} coefficients increase from 96,16 to 97,00% and from 93,00 to 93,34%, respectively. It is observed that the filtering speed of gypsum porridges with phosphate acid decreases from

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Table 1. The main composition of WPPA and phosphogypsum (MM : 10%-ли WPPA= 1.0 : 2.5)

$N_{H_2SO_4}, \%$	$C_{H_2SO_4}, \%$	Amount of substances, %										$K_{decom.}, \%$	$K_{yield}, \%$	Filtration speed, $kg/m^2 \cdot h$
		WPPA				Phosphogypsum								
		P_2O_5	CaO	SO_3	F	P_2O_5 total	P_2O_5 w.s.	CaO total	SO_3	F	H_2O			
103	70	13,22	0,31	3,05	0,03	2,35	0,51	29,11	40,55	1,09	19,02	96,18	93,02	1284
	75	13,93	0,34	3,08	0,03	2,38	0,53	29,30	40,60	1,12	19,24	96,29	93,08	1265
	80	14,55	0,36	3,12	0,04	2,41	0,55	29,50	40,74	1,16	19,47	96,45	93,15	1239
	85	14,79	0,38	3,16	0,05	2,43	0,58	29,73	40,92	1,19	19,77	96,61	93,21	1220
	90	15,03	0,40	3,20	0,06	2,48	0,60	29,96	41,30	1,20	19,86	96,85	93,26	1192
	93	15,60	0,41	3,23	0,07	2,56	0,68	30,01	41,42	1,22	19,96	97,01	93,35	1105
105	70	13,32	0,28	3,15	0,04	2,31	0,49	29,16	40,59	1,06	19,05	97,18	93,12	1295
	75	14,63	0,29	3,18	0,04	2,35	0,51	29,35	40,62	1,10	19,29	97,26	93,28	1280
	80	14,95	0,30	3,22	0,05	2,38	0,53	29,54	40,78	1,14	19,52	97,35	93,35	1268
	85	15,41	0,31	3,26	0,06	2,41	0,56	29,79	41,02	1,16	19,82	97,41	93,41	1243
	90	15,73	0,33	3,30	0,07	2,44	0,58	30,02	41,47	1,18	19,92	97,52	93,45	1202
	93	15,93	0,35	3,35	0,08	2,53	0,64	30,09	41,50	1,20	19,98	97,66	93,49	1135

Table 2. The main composition of WPPA and phosphogypsum (MM : 12%-WPPA = 1.0 : 2.5)

$N_{H_2SO_4}, \%$	$C_{H_2SO_4}, \%$	Amount of substances, %										$K_{decom.}, \%$	$K_{yield}, \%$	Filtration speed, $kg/m^2 \cdot h$
		WPPA				Phosphogypsum								
		P_2O_5	CaO	SO_3	F	P_2O_5 total	P_2O_5 w.s.	CaO total	SO_3	F	H_2O			
103	70	15,17	0,32	3,07	0,04	2,38	0,52	29,09	40,53	1,08	19,00	96,16	93,00	1280
	75	15,90	0,36	3,10	0,04	2,41	0,54	29,28	40,59	1,11	19,22	96,27	93,06	1261
	80	16,50	0,38	3,14	0,05	2,43	0,56	29,49	40,73	1,15	19,45	96,43	93,13	1235
	85	16,74	0,39	3,18	0,06	2,45	0,59	29,71	40,91	1,18	19,75	96,60	93,20	1216
	90	17,00	0,42	3,22	0,07	2,50	0,61	29,95	41,29	1,19	19,84	96,83	93,25	1190
	93	17,55	0,43	3,25	0,08	2,58	0,69	30,00	41,40	1,21	19,94	97,00	93,34	1101
105	70	15,27	0,30	3,17	0,05	2,33	0,50	29,15	40,58	1,05	19,03	97,17	93,11	1290
	75	15,59	0,31	3,20	0,05	2,38	0,52	29,34	40,60	1,09	19,27	97,25	93,26	1275
	80	16,90	0,33	3,24	0,06	2,40	0,54	29,53	40,76	1,13	19,50	97,34	93,33	1265
	85	17,21	0,34	3,28	0,07	2,43	0,57	29,77	41,01	1,15	19,80	97,40	93,40	1239
	90	17,43	0,35	3,32	0,08	2,46	0,59	30,01	41,45	1,17	19,90	97,50	93,43	1198
	93	17,63	0,37	3,38	0,09	2,56	0,65	30,08	41,49	1,19	19,96	97,64	93,47	1131

1280 to 1101 kg/m²·h. In addition, it can be observed that the values of CaO, SO₃, and F in the received WPPA samples increase from 0,32 to 0,43%, from 3,07 to 3,25%, and from 0,03 to 0,07%, respectively. The presence of such laws can be explained by the increase in the concentration of sulfuric acid. P₂O₅ in phosphogypsum samples formed in these sizes and CaO_{umum} amounts are observed to increase from 2,38 to 2,58% and from 29,09 to 30,00%, respectively. The values of SO₃ and F in its content increase from 40,53 to 41,40% and from 1,08 to 1,21%, respectively. Here, too, the slightly increased amount of these substances in phosphogypsum can be explained by a decrease in the filtration rate of phosphogypsum. When sulfuric acid is 105%, all the laws mentioned above are repeated. The amount of P₂O₅ in the received WPPA is slightly higher, from 15,27 to 17,63%. But it is observed that the values of CaO in its content increase from 0,30 to 0,37% and at the same time it is slightly less than that received in the norms of 103%. As above one of the main reasons for this is that the Ca²⁺ ion passes into the solution in a smaller amount due to the large acid ratio. From the results obtained above, it is possible to take the 103% norm of sulfuric acid and the concentrations of 90 and 93% as acceptable values. The maximum amount of P₂O₅ in WPPA from which these acceptable values are received K_{decom.} and K_{yield} based on the fact that the coefficients have the largest values. The amount of P₂O₅ in WPPA received in acceptable sizes is in the range of 17,00-17,63%. These amounts are close to, but less than, the amounts of P₂O₅ in WPPA produced in the industry of our country. Therefore, the amount of P₂O₅ in the circulating solution was increased to 15% to obtain WPPA containing P₂O₅ in amounts equal to and greater than these values. The procedure for performing experiments is the same as above. The obtained results are presented in Table 3. From the received results, it can be seen that the amount of P₂O₅ in the formed acid increases significantly when phosphoric acid is extracted from phosphatic gypsum porridge with 15% P₂O₅. For example, when the concentration of sulfuric acid increases from 70 to 93% at the rate of 103%, the amount of P₂O₅ in the produced WPPA is from 17,40 to 19,59%. At the rate of 105% of sulfuric acid, the amount of P₂O₅ in the above concentrations is from 18,22 to 19,65%. Here too, 103% standard of sulfuric acid, its 90 and 93% concentrations can be considered acceptable values. The resulting WPPA contains 19,30-19,65% P₂O₅. As mentioned above, these quantities are distinguished by the large amount of P₂O₅ from the WPPA currently produced in our country. In general, this type of WPPA is of a lower quality than the WPPA produced in the world industry, but it is possible to produce various simple and complex fertilizers from this WPPA, but the cost of the received fertilizers will be much higher. In fig. 1 showed the effect of the rate and concentration of sulfuric acid and the concentration of circulating WPPA used in the extraction on the amount of P₂O₅ in WPPA. As can be seen from the given results, the amount of P₂O₅ in the obtained WPPA is significantly affected by the concentration of sulfuric acid and the concentration of circulating WPPA used in the extraction.

Table 3. The main composition of WPPA and phosphogypsum (MM : 15%- WPPA = 1.0 : 2.5)

$N_{H_2SO_4, \%}$	$C_{H_2SO_4, \%}$	Amount of substances, %										$K_{decom.}, \%$	$K_{yield.}, \%$	Filtration speed, $kg/m^2 \cdot h$
		WPPA				Phosphogypsum								
		P_2O_5	CaO	SO_3	F	P_2O_5 total	P_2O_5 w.s.	CaO total	SO_3	F	H_2O			
103	70	17,40	0,33	3,10	0,05	2,40	0,53	29,07	40,51	1,07	18,98	96,14	92,98	1274
	75	18,12	0,37	3,12	0,05	2,43	0,55	29,26	40,58	1,10	19,20	96,25	93,04	1255
	80	18,65	0,39	3,16	0,06	2,45	0,57	29,47	40,71	1,14	19,43	96,41	93,11	1230
	85	18,89	0,40	3,20	0,07	2,47	0,60	29,70	40,90	1,17	19,73	96,58	93,18	1211
	90	19,30	0,43	3,24	0,08	2,51	0,62	29,93	41,27	1,18	19,82	96,81	93,24	1185
	93	19,59	0,44	3,26	0,09	2,59	0,70	29,98	41,39	1,20	19,92	96,98	93,33	1096
105	70	18,22	0,31	3,18	0,06	2,35	0,52	29,13	40,57	1,04	19,01	97,15	93,10	1285
	75	18,65	0,32	3,22	0,06	2,40	0,53	29,32	40,59	1,08	19,25	97,23	93,25	1269
	80	18,80	0,35	3,26	0,07	2,41	0,55	29,51	40,75	1,12	19,48	97,32	93,31	1260
	85	19,20	0,36	3,30	0,08	2,44	0,58	29,75	40,98	1,14	19,78	97,38	93,38	1233
	90	19,40	0,37	3,34	0,09	2,47	0,60	30,00	41,42	1,16	19,88	97,48	93,41	1190
	93	19,65	0,38	3,40	0,10	2,58	0,66	30,07	41,48	1,18	19,94	97,62	93,45	1125

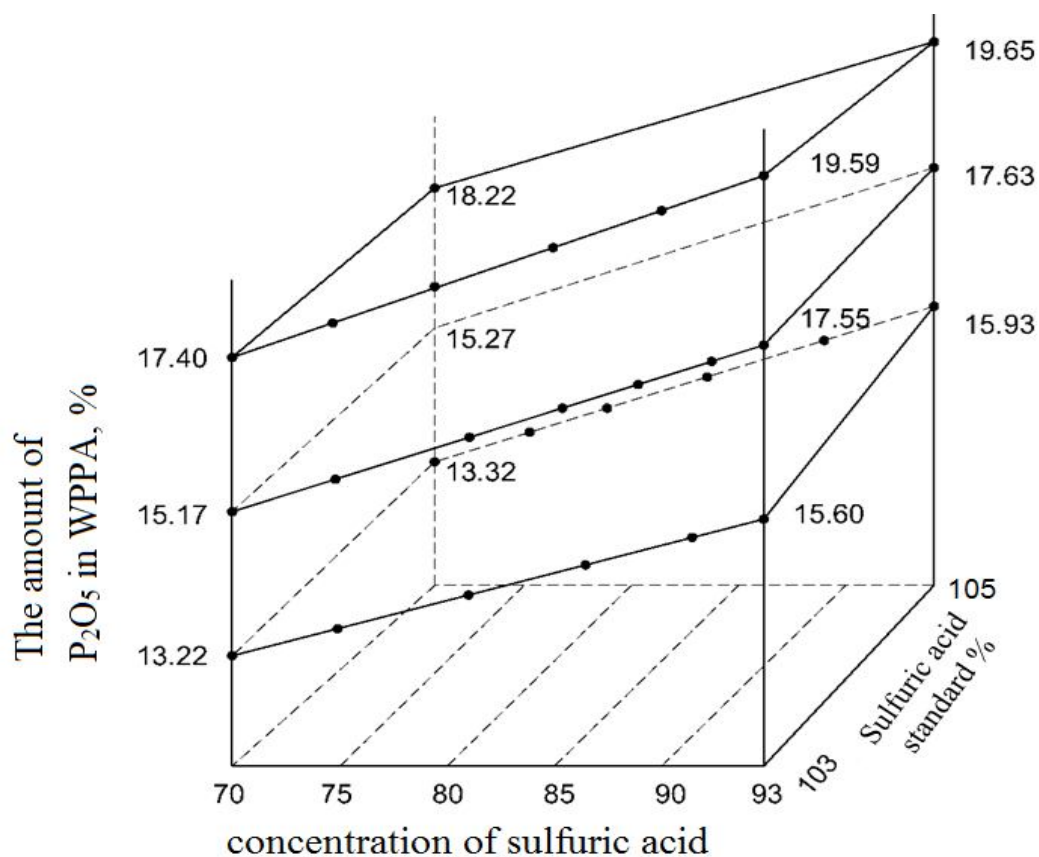


Figure 1. The effect of sulfuric acid rate, concentration and concentration of circulating WPPA solution on the amount of P_2O_5 in WPPA: 1-10% WPPA; 2-12% WPPA and 3-15% WPPA.

It is known that the interphase distribution of fluorine also plays an important role in obtaining WPPA from phosphate raw materials. Figure 2 shows the interphase distribution of fluorine in the extraction of sulfuric acid from MM at 103% standard and 93% concentration of WPPA. As can be seen from the given results, the main part of fluorine is distributed in the gas phase (HF, SiF_4), and the remaining parts are distributed in liquid (WPPA) and solid phases (phosphogypsum).

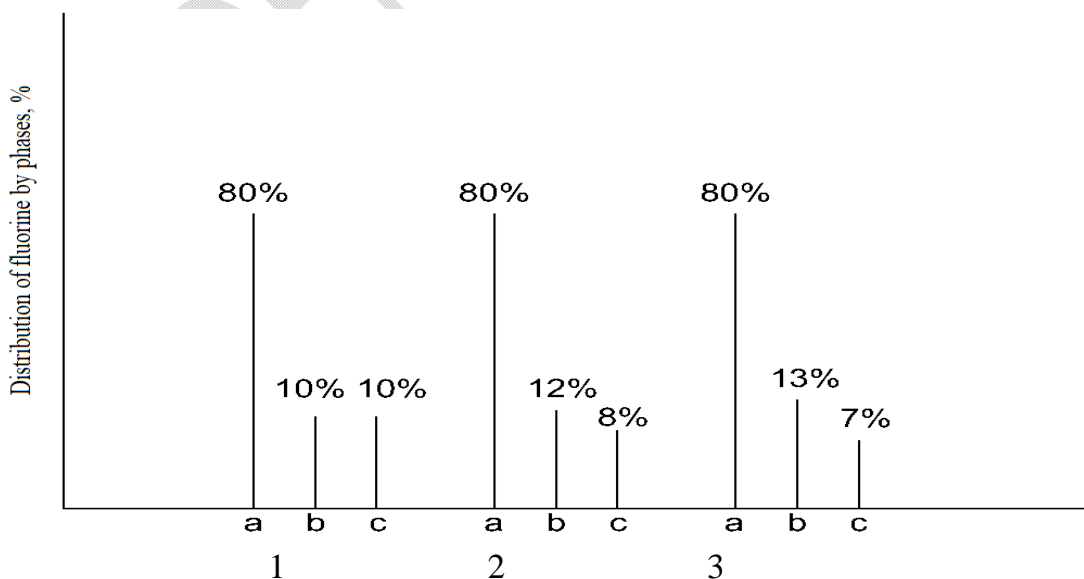


Figure 2. Distribution of fluorine by phases. a-gas phase; b-liquid phase; c-solid phase. 1-10% WPPA; 2- 12% WPPA and 3-15% WPPA.

It is known that the amount of fluorine should not exceed 0,1% in the WPPA used in the production of nutritional phosphate. These values are less than 0,1% in WPPA samples received by us. Therefore, the obtained WPPA are suitable for obtaining nutritional phosphates according to the requirements for fluorine. But even so, it will be necessary to analyze WPPA according to all requirements (heavy metals, non-toxic metals).

Conclusion

The following can be concluded from the above information:

1. Due to the use of high-concentration sulfuric acid, a large amount of foaming is not formed when breaking down MM;
2. The amount of P_2O_5 in the resulting WPPA is higher than that of the traditional method.
3. The emergence of a possibility to involve the thermal enrichment of phosphorites in the direct extraction of MM, which is a phosphate waste.
4. Low amount of fluorine in the obtained WPPA.

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