

e-ISSN:2582-7219



INTERNATIONAL JOURNAL OF MULTIDISCIPLINARY RESEARCH

IN SCIENCE, ENGINEERING AND TECHNOLOGY

Volume 6, Issue 11, November 2023



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INTERNATIONAL STANDARD SERIAL NUMBER INDIA

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Impact Factor: 7.54

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| ISSN: 2582-7219 | www.ijmrset.com | Impact Factor: 7.54 | Monthly Peer Reviewed & Referred Journal |



Volume 6, Issue 11, November 2023

| DOI:10.15680/IJMRSET.2023.0611007 |

Two-Stage Decomposition of Phosphorite Flour into Granulated Simple Superphosphate

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ABSTRACT: The methods for producing simple superphosphate by two-stage sulfuric acid decomposition of natural phosphates are analyzed. For the first stage, the process of decomposition of high-carbonate powdered phosphorite is studied depending on the rate of sulfuric acid from stoichiometry to the formation of H₃PO₄, its concentration and the reaction time of the starting components. For the second stage, the process of neutralization of phosphoric acid is studied, depending on the norm of phosphate rock for the formation of Ca (H₂O₄)₂. The drying process of superphosphate is carried out, in which granular superphosphate is obtained, and P₂O_{5total}. - 12%, P₂O_{5free}. - 4.6%, P₂O₅ : P₂O_{5total} = 90%, P₂O_{5 water} : P₂O_{5total} = 79%.

KEYWORDS: powdered phosphorite, sulfuric acid, decomposition, phosphoric acid, neutralization, mono basic calcium phosphate, simple superphosphate.

I. INTRODUCTION

In Uzbekistan, three joint-stock companies (JSC): "Ammofos-Maxam", "Samarkandkimyo" and "Kokand superfosfat zavodi" produce phosphoric fertilizers, the assortment of which consists of ammophos (10% N; 46% P₂O₅), suprefos-NS (8-15% N; 20-24% P₂O₅), ammonium sulfate phosphate (15-19% N; 4-23% P₂O₅), PS-Agro (4-6% N; 34-41% P₂O₅), feed ammonium phosphate (12% N; 53 -55% P₂O₅), nitrocalcium phosphate (6% N; 16% P₂O₅), simple (1.5% N; 13.5% P₂O₅) and enriched superphosphate (2.5% N; 18-26% P₂O₅). All three companies are provided by Kyzyl Kum phosphorite complex in phosphate raw materials.

From above the assortments of phosphorous-containing fertilizers it can be seen that the most of them are complex, nitrogen-phosphorous, with exception of simple and enriched superphosphates, which are as unary phosphoric fertilizers. However, their amount is a little; in kind it does not 100-120 thousand tons per year.

Complex fertilizer is not applied to the soil under autumn tillage. Since in winter there is a loss of nitrogen and, unlike phosphorus, it does not accumulate in the soil and is simply washed out with rain and melt water. Under cotton, 60-70% of the annual norm of phosphorus should be brought under the chill, under vegetable crops to be 75% and under cereal crops to be 100% [1]. Therefore, it is necessary to increase the volume of single phosphorus fertilizers [2].

Simple superphosphate is one of the cheapest and most affordable phosphate fertilizers [3]. This fertilizer is a product consisting of a mixture of mono basic calcium phosphate (calcium dihydrogen phosphate) and gypsum. Simple superphosphate is produced by the sulfuric acid decomposition of natural phosphates by chamber, stream or chamber-stream methods.

Its production is based on the reaction of sulfuric acid with phosphate raw materials and subsequently phosphoric acid with an undecomposed part of the raw material according to:

$$Ca_{5}(PO_{4})_{3}F + 5H_{2}SO_{4} + 2.5H_{2}O = 3H_{3}PO + 5CaSO_{4} \cdot 0, 5H_{2}O + HF$$
(1)
$$Ca_{5}(PO_{4})_{3}F + 7H_{3}PO_{4} + 5H_{2}O = 5Ca(H_{2}PO_{4})_{2} \cdot H_{2}O + HF$$
(2)

They are summarized as follows:

$$2Ca_{5}(PO_{4})_{3}F + 7H_{2}SO_{4} + H_{2}O = Ca(H_{2}PO_{4})_{2} \cdot H_{2}O + 7CaSO_{4} + 7H_{2}O + 2HF$$
(3)

In the production of simple superphosphate, these two stages proceed sequentially. This is because the coexistence of H_2SO_4 and Ca $(H_2PO_4)_2$ in aqueous solutions is impossible due to the exchange reaction with the release of sparingly soluble calcium sulfate:

$$H_2SO_4 + Ca(H_2PO_4)_2 = CaSO_4 + 2H_3PO_4$$
 (4)

| ISSN: 2582-7219 | www.ijmrset.com | Impact Factor: 7.54 | Monthly Peer Reviewed & Referred Journal |



Volume 6, Issue 11, November 2023

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This means that at each stage there is only one chemical reaction. At the end of the reaction, the resulting superphosphate mass is fed to storage ripening, where the decomposition of the phosphate feedstock with phosphoric acid is completed within a few days.

Such a long process of decomposition of phosphate raw materials is explained by the fact that, upon reaction with H₂SO₄, a surface crust of CaSO₄ crystals forms on the surface of phosphorite grains [4]. In the course of the reaction with H3PO4, another layer of crystals of calcium phosphate is formed. Thus, in both sulfuric and phosphoric acids, it is necessary to overcome the layer of crystals deposited on the surface of the grains in order to react with the phosphate grain itself. Since at the stages of chamber and warehouse ripening, the limiting stage of the process is the rate of acid diffusion through the formed salt crust [5]. Currently, JSC "Kokand superfosfat zavodi" produces simple ammonized superphosphate by the chamber method [6]. This method includes the following stages: decomposition of the washed dried Kyzylkum concentrate (18-19% P₂O₅) with sulfuric acid at a concentration of 60% at its rate of 100% and 70-75 °C; chamber maturation of superphosphate mass for 1-1.5 hours at 115-120 °C; storage ripening for 6 days with 3 single shoveling; granulation and ammonization, drying and sieving of the product. In this case, the finished product contains $12\% P_2O_{5total}$, 1.5% N, P_2O_{5water} : $P_2O_{5total} = 50\%$ and granule strength of 1.5 MPa.

The disadvantages of chamber (conventional) technology for the production of simple superphosphate are as follows:

a) six-day ripening at the warehouse and three-time shoveling, passivation of the active centers of all spent fluorocarbonate apatite and incomplete extraction of H_3PO_4 into the liquid phase;

b) diffusion inhibition of the reaction of formation of Ca $(H_2PO_4)_2$ and a high content of free H_3PO_4 in the chamber product;

c) unsatisfactory granulation of superphosphate mass and high reture (tiny particles of finished product using for granulation);

d) unorganized emission of harmful substances and high dustiness of industrial premises, as warehouse ripening is a powerful source of emission of fluoride compounds into the atmosphere.

In [7], an analysis was made for the working conditions of workers, the main professions engaged in the production of superphosphate. At the same time, workplaces were exceeded the maximum permissible concentrations of industrial aerosols, worsened microclimate parameters, and exceeded vibration level indicators. With a general hygienic assessment of the classes of working conditions of the driver-scraper and the operator-loader corresponds to the 3rd harmful and 2nd degree; crane operator and packaging-packing machines, apparatchik - 3rd harmful and 3rd degree.

A variant of exclusion from the scheme of chamber maturation and warehouse maturation, as well as ammonization, is shown by the example of the production of simple superphosphate from Karatau phosphorites [8], where it is proposed to process phosphorite in two stages: in the first stage, phosphorite is decomposed with diluted 55% sulfuric acid using for this purpose, a three or four-chamber superphosphate mixer (residence time - 5-7 minutes). Next, water is supplied to the pulp at solid and liquid - S: L = 1: 1.5, and the obtained non-thickening pulp is kept for 1-1.5 hours with stirring. Before drying and granulation, the non-thickening pulp is mixed with a part of the fine dried product (with a size of less than 1 mm) at a ratio of 1: 1.5 (second stage). A positive point is the complete exclusion of the stage of warehouse aging. However, the hardware design is complex, high retouring, and in addition, the technology is energy-intensive due to the need to evaporate a large amount of water from diluted pulp at the stages of drying and granulation.

The proposed two-stage method of processing phosphate raw materials into superphosphate, where in the first stage the main amount of raw materials is treated with sulfuric acid to form phosphoric acid, and the resulting acid mass, consisting of phosphoric acid and calcium sulfate, is neutralized in the second stage of the process using reactive phosphorite or other neutralizing agents can be encouraging, both in terms of the intensification of individual stages, and the creation on their basis of a rational from the classical version of the production flow technology.

A description of a more simplified approach to a two-stage scheme, which eliminates the stages of chamber and warehouse maturation of superphosphate mass, is stated in the patent. [9]. According to it, at the first stage, the main part of poor dolomotized phosphorite (50-70%) is decomposed with sulfuric acid with a concentration of 25-35%. The residence time of the pulp in the reactor of the extractor type is 6-8 hours. In the second reactor of the same type, the remaining portion (30-50%) of phosphorite is added to the pulp with constant stirring to completely neutralize H_3PO_4 with the formation of mono basic calcium phosphate, where the residence time of the reaction pulp is 2-4 hours. The pulp is dried and granulated in a drum-type apparatus (DGD). The disadvantage is the same need for evaporation of diluted pulp and a long decomposition process.

In the patent [10], an embodiment of the first stage, similar to the sulfuric acid extraction mode of phosphoric acid, is described for a two-stage processing of natural phosphates. At the first stage, 70-90% of phosphate raw materials of the total amount are used, decomposition with sulfuric acid is carried out in the mode corresponding to the dihydrate mode of wet process phosphoric acid (WPA), maintaining the ratio S: L equal to 1: (1.7-2.5). In the second

| ISSN: 2582-7219 | www.ijmrset.com | Impact Factor: 7.54 | Monthly Peer Reviewed & Referred Journal |



Volume 6, Issue 11, November 2023

| DOI:10.15680/IJMRSET.2023.0611007 |

stage, the rest of the feedstock is introduced into the suspension, phosphorites being used as this. Neutralization is carried out to NH₃: H_3PO_4 equal to 0.95-1.3. The resulting product contains mainly ammonium monophosphate (ammonium dihydrogen phosphate) and calcium sulfate. The residence time of the phosphoric acid suspension in the reactor at 80 ° C is 1.5 hours (first stage). The duration of the 2nd stage of neutralization at 60-80 ° C is 20 minutes. In both process steps, the temperature is maintained by the heat of the reactions.

According to the above method, Kyzylkum phosphorites are processed at the Chardzhev Chemical Plant named after S. Niyazova (Turkmenistan) into granular superphosphate, so called "ammonium superphosphate" [11].

Organization of production according to the proposed scheme requires the investment of large capital costs. In addition, the neutralized pulp contains about 50% of water, which leads to increased energy consumption.

A brief review shows that the choice of modes of crystallization of calcium sulfate from phosphoric acid solutions in the initial stage of decomposition of raw materials with sulfuric acid plays an important role, not only in classical schemes of production technology of WPA, where the main emphasis is on the production of phosphoric acid with an increased concentration of P_2O_5 and crystallization from it of $CaSO_4$ in large forms, but also in organizing the initial stage of the production technology of simple superphosphate, although the latter does not set the goal of removing $CaSO_4$ from the reaction medium.

To intensify the initial stage of a two-stage scheme for processing natural phosphates, some methods [10, 12-14] considered options for regulating the conditions of the sulfuric acid extraction regime.

Depending on the concentration of phosphoric acid and the temperature, the formed calcium sulfate according to reaction (1) can precipitate in the form of anhydrite (n = 0), hemihydrate (n = 0.5) and dihydrate (n = 2). Three variants of the extraction method for the production of WPA are based on them: anhydrite, hemihydrate, and dihydrate [15].

Thus, in the dihydrate method, calcium sulfate precipitates in the form of $CaSO_4 \cdot 2H_2O$ (at 70-80 ° C and an acid concentration in the reaction mixture of 20-32% P_2O_5), in the hemihydrate method, in the form of $CaSO_4 \cdot 0.5H_2O$ (at 90-100 °C and an acid concentration of 35- 42%). The anhydrite regime (CaSO₄) corresponds to crystallization fields above 110 °C and P_2O_5 concentrations above 50% [16]: Under real conditions of sulfuric acid extraction (temperature 80-90 °C, concentration 20-38% P_2O_5), gypsum (dihydrate) is a stable form, where phase transformations occurs according to the scheme:

$$CaSO_4 \cdot 0.5H_2O \rightarrow CaSO_4 \rightarrow CaSO_4 \cdot 2H_2O \leftarrow CaSO_4 \cdot 0.5H_2O$$
 (5)

that is, gypsum can crystallize by flooding both anhydrite and hemihydrate. Then, under temperature (> 110 °C) and concentration (P_2O_5 > 50%) conditions for the production of simple superphosphate, anhydrite will become a stable form, where these transformations occur according to the scheme:

$$CaSO_4 \cdot 0, 5H_2O \rightarrow CaSO_4 \cdot 2H_2O \rightarrow CaSO_4 \leftarrow CaSO_4 \cdot 2H_2O$$
(6)

that is, hemihydrate turns into anhydrite not direct, but through watering it into gypsum.

This region is an unexplored part of the CaSO₄ crystallization field; under temperature and concentration conditions, they can display themselves if natural phosphate is decomposed by concentrated sulfuric acid (93-98% H₂SO₄), without introducing water from outside. Due to the high temperature of the decomposition reaction of phosphate with concentrated sulfuric acid, water introduced by its solution (2-7% H₂O) instantly evaporates at the very beginning of the process, and molecular water released by the reactions can serve as a source for the formation of CaSO₄ \cdot 0.5H₂O crystalline hydrate (1, 3) for its crystallization.

If it speaks of the sizes of crystals of hemi- and anhydrite, which are precipitated from concentrated solutions of phosphoric acid at elevated temperatures, then anhydrite is precipitated in the form of larger crystals, forming plates of irregular shape in the case when the solution has a concentration of 45-55% P_2O_5 in superphosphate mass containing some excess of sulfuric acid (3-5% SO₃).

II. METHODS AND MATERIALS

A feature of our proposed method for producing granular superphosphate from carbonate phosphorites in comparison with the classical scheme is that the technological process is carried out in two stages:

1) The main part (70-80% of the total mass) of phosphorite decomposes with 92-93% H_2SO_4 taken at 100-105% of the stoichiometry for the formation of 45-50% P_2O_5 phosphoric acid, where calcium sulfate is crystallized as anhydrite;

2) The acid reaction mixture obtained in the first stage containing concentrated phosphoric acid (45-50% P_2O_5) and anhydrite is treated with the remaining part (20-30%) of phosphorite, as a result of which the process of neutralization of phosphoric acid with the formation of mono basic calcium phosphate occurs.

| ISSN: 2582-7219 | www.ijmrset.com | Impact Factor: 7.54 | Monthly Peer Reviewed & Referred Journal |



Volume 6, Issue 11, November 2023

| DOI:10.15680/IJMRSET.2023.0611007 |

The duration of the first stage is 15-20 minutes at 120-125 $^{\circ}$ C. The processes of neutralization and obtaining a granular product by rolling are combined, and they are combined in one apparatus. Duration is 25-30 minutes. Moreover, the stage of drying the product is excluded from the scheme.

To determine the mechanism of decomposition of phosphate raw materials in two stages, the present study was carried out.

For experiments, phosphorite powder of the composition (wt.%): (wt.%) $P_2O_5 - 17.54$; CaO - 47.75; MgO - 1.79; CO₂ - 16.5; $Fe_2O_3 - 0.73$; Al₂O₃ - 0.95; SO₃ - 4.06; F - 1.7; SiO₂ - 1.24; insoluble residue - 4.03; CaO: $P_2O_5 - 2.72$ and 93% H_2SO_4 are taken as raw materials.

Decomposition experiments of phosphorite are carried out in a thermostated reactor equipped with a paddle stirrer at 122 °C (The required temperature for the anhydrite regime was maintained due to the reaction heat). The norm of sulfuric acid was taken 100% of the stoichiometry for the formation of H_3PO_4 (reaction 1), taking into account the decomposition of the impurity components of the phosphate feedstock. The content of P_2O_{5free} in reaction masses and products is determined by titration of 0.1 n NaOH using indicators of methyl orange and phenolphthalein.

III. RESULTS AND ITS DISCUSSION

At the beginning, we studied the kinetics of the decomposition of phosphate raw materials. The contact time of the components ranged to be 2; 5; 10; 20; 40 and 60 minutes. The results are shown in Table 1.

The decomposition coefficient of phosphorite powder by 93% sulfuric acid against the interaction time								
Rate H ₂ SO ₄ from		The content of						
stoichiometry for	Interaction time,				$\Gamma_2 O_{5 \text{water}} / \Gamma_2$			
the formation of	min	P_2O_{5total}	P ₂ O _{5water}	Free acid	O_{5total} · 100,			
H ₃ PO ₄ ,%					70			
100	2	9.42	8.48	16.68	90.02			
	5	9.55	8.60	16.55	90.05			
	10	9.71	8.76	16.30	90.22			
	20	9.87	9.09	15.84	92.10			
	40	9.89	9.13	15.45	92.31			
	60	9.90	9.14	15.39	92.32			

Table 1
The decomposition coefficient of phosphorite powder by 93% sulfuric acid against the interaction time

*The content of free acid was determined as the sum of free sulfuric and phosphoric acids.

As can be obviously seen from that with an increase in the duration of the interaction of phosphorite powder with sulfuric acid from 2 to 60 minutes, the decomposition coefficient of phosphate raw materials increases, that is, the relative content of the water-soluble form of P_2O_5 relative to its total form is 90.02 to 92.32%. Moreover, in the reaction mass, the total content of P_2O_5 rises from 9.42 to 9.90%, and free acid, on the contrary, it decreases from 16.68 to 15.39%. From the data we can conclude that from a technological point of view, the decomposition time of phosphate raw materials is enough 20-30 minutes. A further increase in time does not lead to a significant increase in the decomposition coefficient. And with a processing time of less than 20-30 minutes, the formation of a thixotropic mass necessary for further processing of the reaction mass does not occur.

To clarify the effect of the amount of sulfuric acid on the decomposition of raw materials, phosphate raw materials were processed using various standards of sulfuric acid. The experimental results are given in Table 2.

Table 2
The decomposition coefficient of phosphorite powder, depending on the norm of 93% sulfuric acid with a
reaction time of 20 minutes

Rate of H_2SO_4 fromTemperature of P_2O_5 content in the reaction mass, wt. %					P ₂ O _{5water} /			
stoichio-metry for the formation of H ₃ PO ₄ ,%	decomposition products, °C	P ₂ O _{5total}	P_2O_{5water}	Free acid	P_2O_{5total} ·100, %			
70	115	11.52	8.54	3.99	74.13			
80	116	10.92	8.55	7.59	78.30			
90	120	10.39	8.79	11.01	84.60			
100	122	9.87	9.09	15.84	92.10			
105	123	9.67	9.18	17.56	94.93			
110	125	9.47	9.46	21.03	99.89			

*The content of free acid was determined as the sum of free sulfuric and phosphoric acids.

| ISSN: 2582-7219 | www.ijmrset.com | Impact Factor: 7.54 | Monthly Peer Reviewed & Referred Journal |



Volume 6, Issue 11, November 2023

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The results show that with an increase in the acid norm from 70 to 110%, the temperature of the reaction mixture rises from 115 to 125°C. The higher the norm of sulfuric acid, the greater the content of P_2O_5 free in the reaction mass. So, if at a rate of H_2SO_4 of 70% the content of free acid makes up 3.99%, then at the norms of 100 and 110% it increases to 15.84 to 21.03%, respectively. At a 100% rate of H_2SO_4 , the ratio of P_2O_{5water} . : $P_2O_{5total} = 92.10\%$, which indicates a sufficient degree of decomposition of phosphate raw materials.

The choice of a 100% norm of H_2SO_4 is due to the fact that its further increase leads to an increase in excess H_2SO_{4free} and a sharp deterioration in the physical and mechanical properties of the intermediate. A decrease in the rate of acid causes a decrease in the water-soluble form of phosphorus.

Next, the effect of the concentration of H_2SO_4 on the chemical composition and physical properties of the reaction mixture was studied. For this, the acid concentration varied from 70 to 94.5%, while its norm was 90 and 100% of the stoichiometry for the formation of H_3PO_4 with duration of decomposition of phosphorite powder of 20 minutes. The results are shown in Table 3.

Table 3 shows that the higher the concentration of H_2SO_4 , the higher the temperature of the decomposition products. The use of 70 and 80% acid does not provide the anhydrite mode of formation of H_3PO_4 . The most acceptable concentration can be considered 90 and 93%, at which a wet granular mass is formed and suitable for the 2nd stage of processing.

Table 3
The decomposition coefficient of phosphorite powder, depending on the concentration and norm of sulfuric acid
at a reaction time of 20 minutes

H_2SO_4 concen- tration, %	Rate of H ₂ SO ₄ from stoichiometry for the formation of	Temperature of decomposition products, °C	P ₂ O ₅ co P ₂ O _{5total}	ntent in the remass, wt. % P_2O_{5water}	Free acid	P ₂ O _{5water} / P ₂ O _{5total} ·100, %	Physical state of the reaction mass, vieually	
	H ₃ PO ₄ ,%	80	9.37	8.62	11.04	92.0		
70	100	83	8.73	8.22	15.37	94.16	Liquefied	
80	90	98	9.93	8.84	13.03	89.02	Smeared	
	100	100	9.07	8.53	17.77	94.04		
00	90	118	10.28	9.53	12.07	92.70	Wet	
90	100	119	9.80	9.27	17.07	94.53	granular	
93 -	90	120	10.39	9.54	11.01	91.82	Wet	
	100	122	9.87	9.09	15.84	92.10	granular	
94.5	90	136	10.61	9.93	11.06	93.59	Hardened	
	100	140	10.33	9.68	15.89	93.71	granules	

*The content of free acid was determined as the sum of free sulfuric and phosphoric acids.

At the final stage, experiments were carried out to obtain the finished superphosphate by neutralizing the acidic reaction mass with phosphorite powder (the second stage of processing). In this case, the rate of phosphorite powder varied from 80 to 200% of the stoichiometry for the formation of mono basic calcium phosphate (reaction 2). Moreover, the general rate of H_2SO_4 (including the 1st and 2nd stages of the decomposition of phosphate feed) ranges from 86 to 102% for the formation of mono basic calcium phosphate. The neutralization time of the reaction mixture with phosphorite powder is 20 minutes at 122°C. Granulation of the neutralized product was carried out by the balling method. The assimilable form of P_2O_5 in the products was determined by solubility in 2% citric acid.

Table 4 shows that with an increase in the rate of phosphorite powder, on the one hand, it reduces the content of P_2O_{5free} , on the other hand, it increases the P_2O_{5total} in the finished product. Taking into account free acidity and the relative content of assimilable and aqueous forms of P_2O_5 in products, the optimum phosphorite norm can be considered 180% (with a total norm of H_2SO_4 - 88.5%). In this case, the content of P_2O_{5free} does not exceed 5%, and P_2O_{5total} - 10.90%, P_2O_5 : P_2O_{5total} = 97.71% and P_2O_{5water} . : P_2O_{5total} = 82.57%. A further increase in the amount significantly reduces the digestible form of phosphorus in the product.

| ISSN: 2582-7219 | www.ijmrset.com | Impact Factor: 7.54 | Monthly Peer Reviewed & Referred Journal |



Volume 6, Issue 11, November 2023

| DOI:10.15680/IJMRSET.2023.0611007 |

Table 4

The composition of the products after neutralization of the reaction mixture (the initial norm of 93% H₂SO₄ is 100% of the stoichiometry for the formation of H₃PO₄) depending on the norm of phosphorite powder for 20 minutes

The norm of	The total norm of	P_2O_5 content in the reaction mass, wt. %					
phosphorite on the formation of Ca(H ₂ O ₄) ₂ ,%	H_2SO_4 on the formation of $Ca(H_2O_4)_2,\%$	P ₂ O _{5total}	P ₂ O _{5assim}	P ₂ O _{5water}	P ₂ O _{5free}	$\begin{array}{c} P_2O_{5assim.} / \\ P_2O_{5total} \\ \cdot 100, \% \end{array}$	$\begin{array}{c} P_2O_{5water} \\ P_2O_{5total} \\ \cdot 100, \% \end{array}$
80	102	10.17	9.80	9.79	13.03*	96.36	96.26
90	100.5	10.37	9.82	9.56	12.27*	94.69	92.19
100	99	10.52	9.84	9.27	10.56*	93.54	88.12
105	98	10.56	9.90	9.21	10.54	93.75	87.21
110	97.5	10.60	10.0	9.15	10.05	94.34	86.32
120	96	10.67	10.12	9.02	9.67	94.84	84.54
150	92	10.79	10.43	9.01	7.88	96.66	83.50
180	88.5	10.90	10.65	9.0	5.09	97.71	82.57
200	86	11.06	10.15	8.76	3.87	91.77	79.20

*The content of free acid was determined as the sum of free sulfuric and phosphoric acids.

It should be noted that, depending on the norm of phosphorite powder in products, the water content is 2.5-3%. Based on this, the product granules were first dried at 75 °C for 24 hours, then at 10 °C to constant weight. The compositions of the dried samples are given in Table 5.

So, if at the norm of sulfuric acid - 88.5% or the norm of phosphorite powder - 180% before drying, the content of P_2O_{5total} is 10.9% after drying, it rises to 11.99%, and P_2O_{5free} vice versa, decreases from 5.09 to 4.61%. At the same time, the relative content of assimilable and water-soluble forms of P_2O_5 decreases from 97.71 to 89.66% and from 82.57 to 78.98%. The decrease in the relative content of water and assimilable forms of P_2O_5 is attributed by the fact that impurities of the phosphate feedstock worsen the passage of the main reaction, that is, they bind some of the useful part of P_2O_5 acid necessary for the complete decomposition of the phosphate feedstock [4].

Table 5
The composition of the products after drying the neutralization products (the initial norm of 93% H ₂ SO ₄ is
110% of the stoichiometry for the formation of H ₃ PO ₄ , the reaction time is 20 minutes) depending on the norm
of phosphorite powder

of phosphorite powder							
The norm of	The total norm of H_2SO_4 on the formation of $Ca(H_2O_4)_2,\%$	P_2O_5 content in the reaction mass, wt. %					
phosphorite on the formation of $Ca(H_2O_4)_2,\%$		P ₂ O _{5total}	P ₂ O _{5assim}	P ₂ O _{5water}	P ₂ O _{5free}	$\begin{array}{c} P_2O_{5assim.} \\ P_2O_{5total} \\ \cdot 100, \% \end{array}$	$\begin{array}{c} P_2O_{5water} \\ P_2O_{5total} \\ \cdot 100, \% \end{array}$
100	99	10.89	10.21	9.48	8.37	93.75	87.05
120	96	11.11	10.29	9.59	7.23	92.62	86.32
135	94	11.49	10.53	9.81	6.68	91.64	85.38
150	92	11.72	10.66	9.72	5.69	90.95	82.93
165	90	11.87	10.71	9.65	4.84	90.23	81.29
180	88.5	11.99	10.75	9.47	4.61	89.66	78.98
190	87.5	12.08	10.61	9.08	4.42	87.83	75.16
200	86	12.12	10.59	8.98	4.24	87.37	74.09

In addition, during the drying process, mono basic calcium phosphate is partially hydrolyzed to dicalcium phosphate (calcium hydrogen phosphate) and phosphoric acid as:

 $Ca(H_2PO_4)_2 \cdot H_2O + H_2O = CaHPO_4 + H_3PO_4 + 2H_2O$ (7)

| ISSN: 2582-7219 | www.ijmrset.com | Impact Factor: 7.54 | Monthly Peer Reviewed & Referred Journal |



Volume 6, Issue 11, November 2023

| DOI:10.15680/IJMRSET.2023.0611007 |

In any case, with the studied norms of sulfuric acid and phosphorite powder, samples of simple superphosphate are obtained, the granule strength of which is at least 2 MPa.

IV. CONCLUSION

In order to eliminate the stage of ripening, ripening, ammonization, and drying, we have studied the process of obtaining granular simple superphosphate, the essence of which is a two-stage decomposition of phosphate raw materials.

With a 20 minute decomposition of phosphorite powder with a 100% norm of concentrated sulfuric acid (93% H_2SO_4) from stoichiometry to the formation of H_3PO_4 , its degree of decomposition is 92.1%. In the reaction mass, the content of P_2O_{5free} is 15.84%. The temperature of the decomposition product rises to 122 C. At the same temperature, the process of neutralizing phosphoric acid of the reaction mass of the remaining part of phosphorite powder was carried out. It was shown that at a 180% norm of phosphorite powder spent on the formation of mono basic calcium phosphate, the obtained simple superphosphate contents 10.9% P_2O_{5total} , $P_2O_{5total} = 97.71\%$ and P_2O_{5water} : $P_2O_{5total} = 82.57\%$, and P_2O_{5free} does not exceed 5%. Additional drying of the reaction mass leads to a certain decrease in the relative content of assimilable and water-soluble forms of P_2O_5 , although the product contains P_2O_{5total} rises to 12%.

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