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ХАРАКТЕРИСТИКА ЖЕЛВАКОВЫХ ФОСФОРИТОВ КАРАКАЛПАКСТАНА И ИХ ПЕРЕРАБОТКА В ГРАНУЛИРОВАННЫЙ ПРОСТОЙ СУПЕРФОСФАТ

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В условиях дефицита фосфорных удобрений необходимо привлечение низкосортного фосфоритного сырья в сельскохозяйственное производство, и каракалпакский фосфорит может служить примером. Отличие каракалпакского фосфорита от других типов фосфорного сырья заключается, во-первых, в высоком содержании кварца; во-вторых, в том, что фосфорный минерал в нем представлен курскитом; в-третьих, в каракалпакском фосфорите курскит представляет усвояемую форму P_2O_5 в количестве 35-40%. Гранулометрический анализ порлытауской фосфоритной муки показал, что класс крупности (-5+3)-(-2+1) мм составляет половину количества фосфорной муки (48,73%). Класс крупности (-0,5+0,25)-(-0,25+0,16) мм также имеет высокое содержание фосфорной муки (30,65%). Результаты показали, что сырье имело следующие параметры: P - 6,45%, O - 46,21%, C - 8,08%, Si - 10,05%, Fe - 1,92%, Al - 1,30%, K - 0,85%, Mg - 0,22%, Ca - 18,61%, F - 3,20%, S - 0,49%. Минеральный состав сырья включает 45,62% курскита, 2,79% гипса, 2,86% магнезита, 5,24% полевого шпата, 2,41% лимонита, 38,77% кварца и другие. Норма серной кислоты для получения простого суперфосфата была рассчитана. Сырье обрабатывали 93%-ной серной кислотой в зависимости от ее нормы и концентрации при температуре 70 °С в течение 30 мин. Показано, что по содержанию свободной H_3PO_4 продукты с нормой 90 и 100% не соответствуют требованиям сельского хозяйства. При концентрациях серной кислоты 68 и 80% не обеспечивается гранулирование массы суперфосфата, при этих концентрациях образуются текучие и мажущие массы. При концентрации 93% образуется легко перемешиваемая масса, подходящая для гранулирования в шнековом смесителе. При оптимальных условиях активации (норма 75% и концентрация H_2SO_4 - 93%) продукт содержит свободный P_2O_5 - 2,49%; общий P_2O_5 - 12,43%; $P_2O_{5раствор.}$: $P_2O_{5\ общий}$ = 93,72%; $P_2O_{5водн.}$: $P_2O_{5общий}$ = 62,51%. Водорастворимая форма P_2O_5 свидетельствует о содержании монокальцийфосфата и частично свободной H_3PO_4 в продукте. Усвояемая форма P_2O_5 указывает на присутствие дикальцийфосфата. Относительное содержание усвояемой формы P_2O_5 менее 100% свидетельствует о наличии некоторого количества неразложенного, но активированного фосфорного минерала в продукте. Продукт содержит большое количество дигидрата и полугидрата сульфата кальция.

Ключевые слова: желваковый фосфорит, серная кислота, разложение, химический и минералогический составы

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CHARACTERISTICS OF NODULAR PHOSPHORITES OF KARAKALPAKSTAN AND THEIR PROCESSING INTO GRANULAR SIMPLE SUPERPHOSPHATE

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In conditions of deficit of phosphate fertilisers it is necessary to involve low-grade phosphate rock into agricultural production, and yolk phosphate rock can serve as an example. The difference of yolk phosphorite from other types of phosphate raw materials is that firstly: it contains a large amount of quartz, and secondly: the phosphate mineral in it is represented by kurskite; thirdly, in yolk phosphorite - kurskite relative content of assimilable form of P_2O_5 is 35-40%. The sieve analysis of Porlytau jelly phosphate flour showed that the coarseness class (-5+3)-(-2+1) mm accounts for half of the amount of phosmucus (48.73%). Meanwhile, the coarseness class (-0.5+0.25)-(-0.25+0.16) mm also has the highest amount of phosmucus (30.65%). The results showed that the raw material had the following parameters: P - 6.45%, O -46.21%, C - 8.08%, Si - 10.05%, Fe -1.92%, Al - 1.30%, K - 0.85%, Mg - 0.22%, Ca - 18.61%, F - 3.20%, S - 0.49%. The mineral composition of raw materials contains 45.62% kurskite, 2.79% gypsum, 2.86% magnesite, 5.24% feldspar, 2.41% limonite, 38.77% quartz and others The rate of sulphuric acid for obtaining simple superphosphate was calculated. Raw materials were treated with 93 % sulphuric acid depending on its rate and concentration at the temperature of 70 °C for 30 min. It is shown that by the content of free H_3PO_4, the products at the rates of 90 and 100% do not meet the requirements of agriculture. And at concentrations of sulphuric acid 68 and 80% the granulation of superphosphate mass is not provided, at them liquefied and smearing masses are formed. And at concentration of 93% a doughy easily movable mass is formed, quite suitable for granulation in a screw mixer. Under the optimum activation condition (75% rate and 93% concentration of H_2SO_4) the product contains $P_2O_{5free.}$ - 2.49; P_2O_{5total} - 12.43%; P_2O_{5accer} : P_2O_{5total} = 93.72%; $P_2O_{5water.}$: P_2O_{5total} = = 62.51%. The water-soluble form of P_2O_5 gives the content of monocalcium phosphate and partially free H_3PO_4 in the product. The digestible form of P_2O_5 means the presence of dicalcium phosphate. The relative content of the digestible form of P_2O_5 less than 100% indicates that a certain amount of undecomposed but activated phosphate mineral remains in the product. The product contains a large amount of calcium sulphate di- and semi-hydrate.

Keywords: nodular phosphorite, sulfuric acid, decomposition, chemical and mineralogical compositions

INTRODUCTION

The world's population continues to increase. It is expected that by 2050 the current population (7 billion people) will increase to about 9 billion people [1]. In order to improve the food situation, increase food security and achieve success in combating malnutrition, it is necessary that the growth of agricultural production outpaces population growth. This should utilise mainly existing agricultural land. In this aspect, issues of chemicalisation of agricultural production are a priority. The main prospect of agricultural development is related to the increase in yields through the introduction of the latest technologies of soil cultivation, the use of high-yielding varieties of technical crops, and, of course, the integrated use of mineral fertilisers and plant protection products.

In 2019, the chemical industry of Uzbekistan produced 944.7 thousand tonnes of nitrogen, 143.0 thousand t of phosphorus and 138.0 thousand tonnes of potassium fertilizers per 100% nutrients. And the need of agriculture of the republic in 2020 was 1075.8 thousand tonnes of N, 759.3 thousand tonnes of P_2O_5 and 373.9 thousand tonnes of K_2O [2]. That is, the industry meets the need of agriculture in nitrogen fertilisers only by 83.5%, and in phosphate fertilisers only by 15.8% and in potash fertilisers only by 53%.

The main problem faced by phosphate-based fertiliser producers is the lack of high-quality domestic raw materials. In order to supply phosphate raw materials to the plants, Kyzylkum Phosphorite Complex LLC carried out thermal enrichment of phosphate rock from the Kyzylkum deposit (16-18% P_2O_5), including crushing, screening, grinding to produce phosphate flour, chlorine washing and roasting to remove CO_2. According to the scheme, the annual production capacity of washed burnt concentrate is 716,000 tonnes with an average content of 26% P_2O_5. Ammofos-Maxam JSC receives ammophos (10% N; 46% P_2O_5), suprephos-NS (8-15% N; 20-24% P_2O_5), enriched superphosphate (2.5% N; 18-26% P_2O_5) and partially feed ammonium phosphate (12% N; 53-55% P_2O_5). At the same time, Qo'qon superposfat zavodi JSC produces simple ammoniated superphosphate (1.5% N; 11.5% P_2O_5) from phosphate meal (17-18% P_2O_5, 44-45% CaO and 15-17% CO_2) The volume of simple superphosphate in physical terms does not exceed 100 thousand tonnes per year.

Given the shortage of quality phosphate rock, it is necessary to start developing local agronomic ores, which are not yet developed on an industrial scale. This can be exemplified by yolk phosphate rock in Karakalpakstan, where there are dozens of deposits. These are Khudjakul, Sultan-Uizdag, Khodjeyli, Nazarkhan, Chukai-Tukai and others. [3]. In mineralogical composition they are close to phosphorites of the Egorievsky, Vyatsko-Kamsky (Russia) and Chilisaysky (Kazakhstan) deposits [4-6]. However, they do not meet the requirements for sulfuric acid extraction without enrichment stage.

And their use in finely milled form as a direct fertiliser without any chemical processing would provide agriculture with cheap phosphate fertiliser [7]. However, phosphate flour is not effective for neutral and alkaline (carbonate) soils of Uzbekistan.

As shown by a number of studies, replacing the chemical energy of mineral acids used in conventional technologies with mechanical and mechanochemical activation leads to the conversion of unabsorbable phosphorus into an absorbable form. There are several ways to apply the mechanochemical method for processing phosphate ores – reagentless mechanical activation and mechanochemical treatment of phosphate ores in the presence of mineral salts [8-13].

The Institute of Solid State Chemistry and Mechanochemistry of the Siberian Branch of the Russian Academy of Sciences investigated the properties of mechanically activated ores from 40 deposits of Russia, CIS countries, Mongolia, Morocco, etc. [14]. The influence of apatite type, mineral composition of phosphate ores and conditions of mechanical activation on the content of citrate- and citrate-soluble forms of phosphate was studied in laboratory conditions. The data obtained indicate that deposits with small reserves of phosphate ores can be involved in processing for local use without long-distance transport. These advantages of the mechanochemical method of phosphate ore processing over conventional milling make it promising for covering the deficit of phosphate fertilisers in the country.

Sulphuric acid processing to obtain water- and citric acid-soluble phosphate fertilisers from poor phosphate raw materials to produce simple superphosphate is suitable [15-17]. Superphosphate is a universal phosphate fertiliser, it can be effectively used for any crops and on any soils. Especially, the effect of superphosphate is better on alkaline, carbonate soils. The main nutrient elements for plants are nitrogen, phosphorus, potassium, sulphur, calcium and magnesium, and simple superphosphate contains four of them.

If we look at the global consumption of superphosphate in 2020, we can see that out of 51 million tonnes of phosphate-containing fertilizers in global production, 11.7% (about 5.95 million tonnes) is the fertilizer, superphosphate class (TSP) [18].

The main consumers of simple superphosphate were Central and South America, followed by China and Southwest Asia. For example, India, with a phosphate fertiliser requirement of about 4.5 million tonnes, imports about 3 million tonnes of MAP and DAP annually. Part of the demand is met by domestically produced simple superphosphate. There are more than 56 large-scale plants in India producing nitrogen, phosphate or potash fertilisers. In addition, there are another 72 small-scale enterprises producing simple superphosphate [19]. In China, there are 65 large and medium-sized enterprises producing simple and ammoniated, granulated superphosphate [20].

In our opinion the demand for simple superphosphate can be listed by the following circumstances:

- Availability of phosphate rock for phosphate production, including the suitability of low-grade natural phosphate rock for this purpose;
- simplicity of its production technology and absence of waste;
- low capital investment;
- low energy intensity of production and cheapness of products;
- possibility of use in any soil and climatic zones and for any agricultural crops;
- can be used together with nitrogen and potassium fertilisers and organic composts;
- the fertiliser has a long-lasting effect, it can be fertilised 1-2 times in one season, excess phosphorus is not washed out, it is stored in the soil by absorption by the structure and microorganisms;
- ideal for auxiliary farms, best results are achieved with potatoes, vegetables, fruit crops and flowers;
- due to its gypsum content, it is a good soil structure builder and chemical ameliorant.

The objective of the present work is to develop a rational and economical technical solution to ensure the processing of poor phosphate raw materials and reduce the consumption of sulphuric acid.

METHODS AND MATERIALS

In laboratory conditions phosphate meal (PM) of Porlytau deposit, composition (weight %) was used for production of simple superphosphate: P_2O_5 – 15.76; CaO – 27.19; Fe_2O_3 – 2.66; Al_2O_3 – 1.03; CO_2 – 44.10; SO_3 – 1.30; F – 1.88; SiO_2 – 38.77; insoluble residue – 0.74; CaO_{total} : $P_2O_{5total.}$ = 1.73.

The dispersed composition is characterised as follows (weight.%): sample-1: class (-5+3 mm) – 8.78; (-3+2) – 23.65; (-2+1 mm) – 16.30; (-1+0.5 mm) – 1.82; (-0.5+0.25 mm) – 18.02%; (-0.25+0.16 mm) – 12.63; (-0.16+0.1 mm) – 6.57; (-0.1+0.05 mm) – 7.64; (-0.05 mm) – 4.59%. Sieve analysis of the raw material showed that the coarseness class (-5+3) – (-2+1) mm accounted for half the amount of phosmucus (48.73%). The coarseness class (-0.5+0.25) – (-0.25+0.16) mm also has the highest amount of phosmucus (30.65%). Prior to chemical and physicochemical studies, the raw material was ground on a porcelain mortar to a particle size of 0.25 mm.

For approximate determination of mineralogical composition of flour we took its X-ray diffractogram on diffractometer XRD-6100 (Shimadzu, Japan). CuK_α-radiation (β-filter, Ni, tube current and voltage mode 30 mA, 30 kV) and a constant detector rotation speed of 4 deg/min were used, and the scanning angle was varied from 4 to 80°. Qualitative phase identification of the presented sample was performed using the MATCH! Phase identification from Powder Diffraction programme (Crystal Impact, GbR, Bonn, Germany, 2015).

Process methodology for the production of activated simple superphosphate

Chemical analysis of products for the content of various forms of P_2O_5 and CaO was carried out according to known methods [21]. The content of P_2O_{5free}. in was determined by titration with 0.1n NaOH using methyl orange and phenolphthalein indicators. The digestible form of P_2O_5 was determined by solubility in both 2% citric acid and 0.2M Trilon B solution. The digestible form of CaO was determined only by 2% citric acid. pH of the product was determined after one hour shaking of its 10% aqueous suspension.

RESULTS AND DISCUSSION

The radiograph of the phosphate meal is shown in Fig. 1.

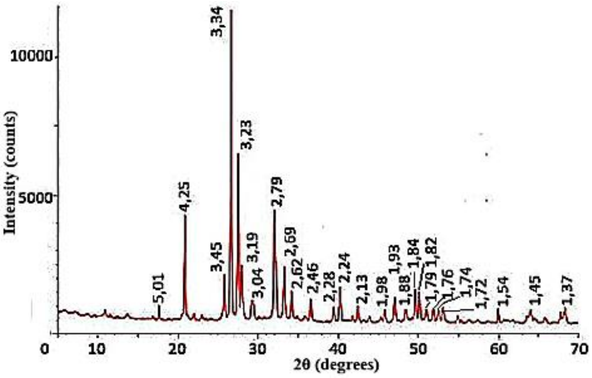


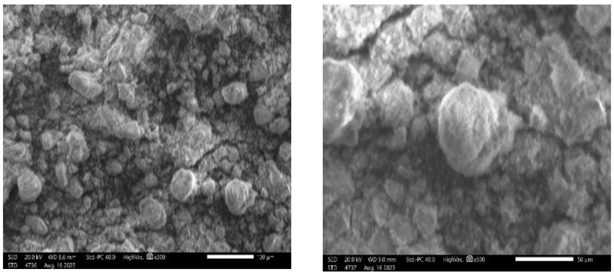
Fig. 1. XRD of phosphorite powder of Porlytau
Рис. 1. Рентгенодифракционный анализ фосфоритного порошка Порлытау

The diffraction bands 1.72; 1.74; 1.79; 1.84; 1.93; 2.24; 2.62; 2.69; 2.79; 3.19; 3.45 A° belong to fluorocarbonapatite. Interplanar distances of 1.88; 2.28; 3.04 A° characterise calcite. Bands 1.37; 1.54 A° belong to dolomite. The most intense peaks 1.82; 1.98; 2.13; 2.46; 3.23; 3.34; 4.25 A° indicate the presence of large amounts of silicon oxide in the phosphate raw material. Bands 1.37 and 1.93 A° are also attributed to calcium fluoride, while 2.46 and 3.23 A° can be attributed to $CaSiO_3$.

The scanning electron microscope with elemental dispersive analysis (SEM-EDX) – JSM-IT200 (JEOL, Japan) was used to take images of the jelly powder. SEM-EDX elemental analysis with electron beams excites the atoms of the sample under study and emits X-ray radiation characteristic of each chemical element. By studying the energy spectrum of such radiation, it is possible to draw conclusions about the qualitative and quantitative composition of the sample.

Images of the surface of phosphorite grains samples at different magnification (200 and 500 times), show the shape and structure of the surface of glauconite grains (Fig. 2). At the same time, various structures of chemical corrosion were revealed on the surface of grains, which were accompanied by removal of quartz, goethite, limonite.

From the micrograph it can be assumed that phosphorite is represented in the form of aggregate grains, associating with quartz. The size of glauconite grains varies from 0.02 to 0.25 mm. The colour of fresh phosphorite meal varies from orange-green to black-green.

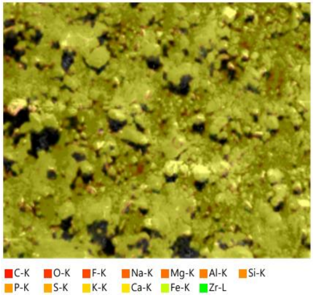


x 200 times · x 500 times

в 200 раз · в 500 раз

Electron microscopic photography

Электронно-микроскопическая фотография



Elemental dispersion analysis

Элементный дисперсионный анализ

Fig. 2. Electron microscopic photographs and micrograph of phosphate flour

Рис. 2. Электронно-микроскопические фотографии и микрофотография фосфатной муки

Using a microscope and an energy dispersion analyser, the elemental compositions of this sample were simultaneously determined. The results showed that it has the following values: P – 6.45%, O – 46.21%, C – 8.08%, Si – 10.05%, Fe -1.92%, Al – 1.30%, K – 0.85%, Mg – 0.22%, Ca – 18.61%, F – 3.20%, S – 0.49% (Fig. 3).

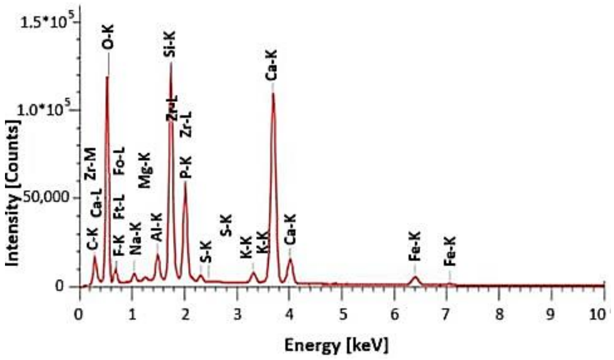


Fig. 3. Diffractogram of phosphorite meal

Рис. 3. Дифрактограмма фосфоритной муки

Calculation of mineralogical composition of Porlytau phosphate rock flour

The content of phosphorus and fluorine in the taken sample confirms that Porlytau phosphorite flour is represented only by kurskite. For example, in francolite $F/P_2O_5 = 3.44/37.14 = 0.09262$ and in kurskite $F/P_2O_5 = 3.85/34.32 = 0.11152$, and in our case $F/P_2O_5 = = 1.88/1576 = 0.11929$.

On the basis of chemical analysis and physico-chemical studies, the calculation for mineral components of phosphorite with some additions of the differences in the content of components was performed. The calculation was performed for 100g of phosphate flour.

The chemical formula of kurskite in phosmuk is as follows:

$$Ca_{10}P_{4.8}C_{1.2}O_{22.8}F_2(OH)_{1.2} \rightarrow$$
$$\rightarrow 2.4P_2O_5 + 9CaO + 1.2CO_2 + CaF_2 + 0.6H_2O$$

Distribution of components by kurskite in phosphate rock flour

First, based on the P_2O_5 CaO. F and CO_2, the amount of kurskite is calculated:

$$Ca_{10}P_{4.8}C_{1.2}O_{22.8}F_2(OH)_{1.2} =$$
$$986.4 \text{ g/mol}$$
$$\begin{array}{lllll} = 2.4P_2O_5 + & 9CaO + & 1.2CO_2 + & CaF_2 + 0.6H_2O \\ 340.8 \text{ g/mol} & 504 \text{ g/mol} & 52.8 \text{ g/mol} & 78 \text{ g/mol} \end{array}$$

$$m(cursite) = \frac{15.76 \cdot 986.4}{340.8} = 45.62g$$
$$m(CaO) = \frac{15.76 \cdot 504}{340.8} = 23.31g$$
$$m(CO_2) = \frac{15.76 \cdot 52.8}{340.8} = 2.44\ g$$
$$m(CaF_2) = \frac{15.76 \cdot 78}{340.8} = 3.61g$$

Determination of the amount of fluorite in the mineral:

$$\begin{array}{ccccc} CaF_2 & = & CaO & + & 2F^- \\ 78 \text{ g/mol} & & 56/ \text{ g/mol} & & 38\text{g/mol} \end{array}$$

$$m(F) = \frac{3.61 \cdot 38}{78} = 1.76g$$

$$m(CaO) = \frac{3.61 \cdot 56}{78} = 2.59g$$

By the excess fluorine (1.88 - 1.76 = 0.12) the mass of fluorite is found:

$$m(CaF_2) = \frac{0.12 \cdot 78}{38} = 0.25g$$

$$m(CaO) = \frac{0.12 \cdot 56}{38} = 0.18g$$

Determining the amount of gypsum:

$CaSO_4 \cdot 2H_2O$ = CaO + SO_3 + $2H_2O$

172 g/mol 56 g/mol 80 g/mol

$$m(gypsum) = \frac{1.30 \cdot 172}{80} = 2.795g$$

$$m(CaO) = \frac{1.30 \cdot 56}{80} = 0.91g$$

Determination of the amount of calcite:

$CaCO_3$ = CaO + CO_2

100 g/mol 56 g/mol 44 g/mol

The total amount of CaO in kurskite, fluorite and gypsum (23.31+0.18+0.91+2.59) is 26.99. And the amount of CaO in calcite is equal by 27.19 – 26.99 = 0.2.

$$m(calcite) = \frac{0.2 \cdot 100}{56} = 0.36g$$

$$m(CO_2) = \frac{0.2 \cdot 44}{56} = 0.16g$$

Determination of magnesite mass:

$MgCO_3$ = MgO + CO_2

84 g/mol 40 g/mol 44 g/mol

Amount of CO_2 carbonate in kurskite and calcite (2.44 + 0.16 = 2.6). The amount of CO_2 in magnesite is equal to per 4.10 – 2.6 = 1.5.

$$m(magnesite) = \frac{1.5 \cdot 84}{44} = 2.86g$$

$$m(MgO) = \frac{1.5 \cdot 40}{44} = 1.36g$$

Determination of the amount of magnesium silicate.

Mg_2SiO_4 = 2MgO + SiO_2

140 g/mol 80 g/mol

The amount of magnesium oxide in magnesium silicate is per 1.57-1.36=0.21.

$$m(Mg_2SiO_4) = \frac{0.21 \cdot 140}{80} = 0.36g$$

Determination of the amount of feldspar and mica:

$2NaAlSi_3O_8 \rightarrow Na_2O + Al_2O_3 + 6SiO_2$

2·262 g/mol 102 g/mol

$$m(feldspar) = \frac{524 \cdot 1.02}{102} = 5.24g$$

The excess aluminium oxide (1.03 - 1.02 = 0.01) will be related to the composition of the mica:

$(OH)_4K_2(Si_6Al_2)Al_4O_{20} \rightarrow K_2O + 3Al_2O_3 + 6SiO_2 + 2H_2O$

796 g/mol 3·102 g/mol

$$m(micaceous) = \frac{796 \cdot 0.01}{306} = 0.02g$$

Determination of the amount of goethite and limonite:

$2FeO(OH) \rightarrow Fe_2O_3 + H_2O$

2·89g/mol 160 g/mol

$$m(\text{goethite}) = \frac{178 \cdot 0.5}{100} = 0.55g$$

$2Fe(OH)_3 \cdot Fe_2O_3 \rightarrow 3Fe_2O_3 + 3H_2O$

2·267 g/mol 3·160 g/mol

$$m(\text{limonite}) = \frac{534 \cdot 2.16}{480} = 2.41g$$

On the basis of the obtained data, Table 1, reflecting the mineral composition of Porlytau jelly phosphate rock flour, was compiled.

Table 1

Mineralogical composition of Porlytau phosphate flour samples

***Таблица 1.* Минералогический состав образцов фосфатной муки из Порлытау**

Mineral name	Content, wt.%	Mineral name	Content, wt.%
Curskite ($Ca_{10}P_{4.8}C_{1.2}O_{22.8}F_2(OH)_{1.2}$)	45.62	Feldspar (K, Na, Ca (Al_2Si_2 or $AlSi_3$)O_8)	5.24
Fluorite (CaF_2)	0.25	Mica ($(OH)_4K_2(Si_6Al_2)Al_4O_{20}$)	0.02
Gypsum ($CaSO_4 \cdot 2H_2O$)	2.79	Limonite ($Fe_2O_3 \cdot nH_2O$)	2.41
Calcite ($CaCO_3$)	0.36	Goethite (FeO(OH))	0.55
Magnezit ($MgCO_3$)	2.86	Acid-soluble quartz (SiO_2)	38.77
Magnesium silicate (Mg_2SiO_4)	0.36	Insoluble residue	0.74

Thus, this raw material belongs to the jelly type, the main minerals of which are kurskite. From it it is quite possible to obtain simple superphosphate for local application.

Calculation of sulphuric acid rate for production of simple superphosphate

To study the process of obtaining simple superphosphate, the total consumption of sulphuric acid was calculated for the treatment of yolk phosphorite with the formation of monocalcium phosphate and gypsum. The main impurities of phosphate raw material were taken into account.

The production of simple superphosphate is based on the process of phosphate raw material decomposition by sulphuric acid, which can be represented by the following total reaction equation:

$$2Ca_5(PO_4)_3F + 7H_2SO_4 + 6.5H_2O =$$
$$= 3Ca(H_2PO_4)_2 \cdot H_2O + 7CaSO_4 \cdot 0.5H_2O + HF$$

Calculate sulphuric acid consumption for decomposition of calcium oxide of phosphate rock to form monocalcium phosphate:

$$CaO + P_2O_5 \rightarrow Ca(H_2PO_4)_2$$

$$m(CaO) = (15{,}76 \cdot 56)/142 = 6{,}215g$$

where, 15.76 – amount of P_2O_5 in 100g of phosphate rock, g; 56 – M(CaO), g/mol; 142 – M(P_2O_5), g/mol.

$$CaO + SO_3 \rightarrow CaSO_4$$

$$m(CaO) = \frac{56 \cdot 1{,}30}{80} = 0{,}91g$$

where, 0.91 – the amount of CaO in 100g of phosphate rock bound as $CaSO_4$, g; 56 – M(CaO), g/mol; 80 – M(SO_3), g/mol.

Thus, the amount of CaO available for acid decomposition is as follows:

27.19 – 6.215 -0.91 = 20.065 g.

Hence the required sulphuric acid consumption for the total amount of CaO:

$$CaO + H_2SO_4 \rightarrow CaSO_4 + H_2O$$

$$m(H_2SO_4) = \frac{98 \cdot 20{,}065}{56} = 35{,}11 \text{ г}$$

where, 56 – M(CaO), g/mol; 98 – M(H_2SO_4), g/mol.

For the decomposition of magnesium oxide, the amount of acid consumed is:

$$MgO + H_2SO_4 \rightarrow MgSO_4 + H_2O$$

$$m(MgO) = \frac{98 \cdot 1{,}57}{40} = 3{,}85\ g$$

where, 1.57 – amount of MgO in 100g of phosphate rock, g; 24 – M(MgO), g/mol; 98 – M(H_2SO_4), g/mol.

Sulphuric acid is used for the decomposition of semisulphuric oxides:

$$Al_2O_3 + 3H_2SO_4 \rightarrow Al_2(SO_4)_3 + 3H_2O$$

$$m(Al_2O_3) = \frac{294 \cdot 1{,}03}{102} = 2{,}969\ g$$

where, 1.03 – amount of Al_2O_3 in 100g of phosphate rock, g; 102 – M(Al_2O_3), g/mol; 98 – M(H_2SO_4), g/mol.

$$Fe_2O_3 + 3H_2SO_4 \rightarrow Fe_2(SO_4)_3 + 3H_2O$$

$$m(Fe_2O_3) = \frac{294 \cdot 2{,}66}{160} = 4{,}888\ g$$

where, 2.66 – amount of Fe_2O_3 in 100g of phosphate rock, g; 160 – M(Fe_2O_3), g/mol; 98 – M(H_2SO_4), g/mol.

Thus, the total consumption of sulphuric acid for decomposition of 100g of jelly phosphorite with formation of monocalcium phosphate and gypsum in the presence of impurity compounds (100% H_2SO_4 rate):

$m(H_2SO_4)$ = 35.11+ 3.85+ 4.888 + 2.969= 46.816g

or in terms of 93% concentration H_2SO_4:

$$m(H_2SO_4) = \frac{46{,}816}{0{,}93} = 50{,}34g$$

Process for the production of activated simple superphosphate

Further the process of obtaining simple superphosphate was studied. The order of carrying out the process of phosphate raw material decomposition by sulfuric acid and obtaining the finished product was as follows: in a glass thermostated beaker, which contained a sample of phosphate flour, slowly poured the calculated amount of sulfuric acid. At the same time, the variable parameters were the acid rate (68; 80; 93% H_2SO_4) its concentration (60; 70; 75; 80; 90; 100% of the stoichiometry for the formation of monocalcium phosphate). The mixture was stirred thoroughly. The duration of interaction between the components was 30 min at 70 °C. Then the obtained mass together with the beaker was placed in a desiccator, where it was dried at 90-100 °C to constant weight. Granulation of the product was carried out by the method of intensive stirring and pelletising.

Table 2 shows the results of chemical analysis of the products depending on the rate of 93 % sulphuric acid.

The data show that with increasing norm of sulphuric acid the content of total P_2O_5 in products decreases from 12.63 to 11.44%, and P_2O_{5free}. on the contrary increases from 1.25 to 5.01%. According to the content of free H_3PO_4, products at the norms of 90 and 100% do not meet the requirements of agriculture.

The effect of H_2SO_4 concentration on the composition of simple superphosphate was also studied. At the same time the rate of acid is constant - 75%. The results are given in Table 3.

It shows that as the acid concentration increases from 68 to 93%, the content of P_2O_{5total} in products increases from 11.98% to 12.43%, P_2O_{5diges}. from 10.95 to 11.65%, P_2O_{5water} from 7.27 to 7.77%, CaO_{total} from 20.67 to 21.45% and CaO_{diges}. from 13.47 to 14.89%. These values are quite acceptable for agriculture (H_3PO_4 content should not exceed 5% or 3.5% P_2O_5).

Table 2

Composition of products obtained by decomposition of Porlytau phosphate flour samples with 93% sulphuric acid depending on its rate

Таблица 2. Состав продуктов, полученных при разложении образцов фосфатной муки Порлытау 93%-ной серной кислотой в зависимости от ее концентрации

Concentration H_2SO_4, %	Component contents, weight %					
	$P_2O_{5free.}$	$P_2O_{5total.}$	$P_2O_{5\ assim.by\ citric.\ acid.}$	$P_2O_{5water.}$	$CaO_{total.}$	$CaO_{assim.by\ citric.\ acid.}$
60	1.25	12.63	11.46	6.93	21.79	14.66
70	1.84	12.54	11.56	7.23	21.64	14.91
75	2.40	12.43	11.64	7.77	21.44	14.89
80	2.83	12.12	11.58	8.47	20.91	14.61
90	3.63	11.62	11.33	9.56	20.04	14.12
100	5.01	11.44	11.33	10.13	19.73	13.93

Table 3

Composition of products obtained by decomposition of Porlytau phosphate flour with 75% sulphuric acid depending on its concentration

***Таблица 3*. Состав продуктов, полученных при разложении фосфатной муки Порлытау 75%-ной серной кислотой, в зависимости от ее концентрации**

Consumption norm of H_2SO_4, %	Component contents, weight %					
	$P_2O_{5free.}$	$P_2O_{5total.}$	P_2O_5 accep.by citric. acid.	$P_2O_{5water.}$	$CaO_{total.}$	CaO assep.by citric. acid.
68	2.15	11.98	10.95	7.27	20.67	13.47
80	2.40	12.26	11.38	7.51	21.15	14.27
93	2.49	12.43	11.65	7.77	21.45	14.89

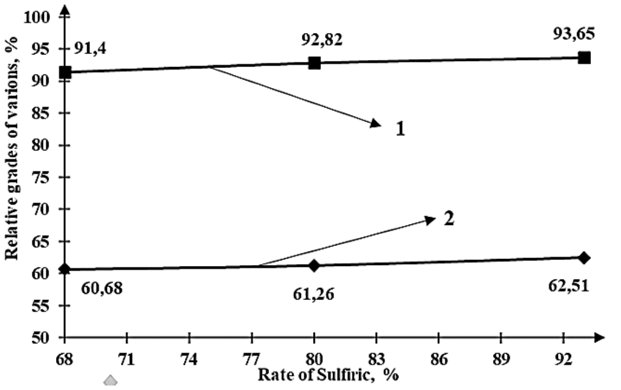


Fig. 4. Relative contents of assimilable and aqueous forms of P_2O_5 in products depending on the concentration of sulphuric acid (H_2SO_4 norm - 75%). 1 - relative form of the digestible form of phosphorus ($P_2O_{5assim.by\ citric.\ acid.}$: P_2O_{5total}), 2 - relative form of the aqueous form of phosphorus ($P_2O_{5water.}$: P_2O_{5total})

Рис. 4. Относительное содержание усвояемой и водной форм P_2O_5 в продуктах в зависимости от концентрации серной кислоты (норма H_2SO_4 - 75%). 1- относительная форма усвояемый формы фосфора ($P_2O_{5усв.}$: $P_2O_{5общ.}$), 2- относительная форма водный формы фосфора ($P_2O_{5водн.}$: $P_2O_{5общ.}$)

It should be noted that at concentrations of sulfuric acid 68 and 80% does not provide granulo-formation of superphosphate mass, they are formed liquefied and smearing masses. And at the concentration of 93% a doughy, quite suitable for granulation in the screw mixer easy to move mass is formed. At this concentration, the highest phosphate raw material K-fraction is also achieved.

From Fig. 4 shows that the higher the sulphuric acid rate, the higher the $K_{disl.}$ of phosphate raw materials, i.e. the more $P_2O_{5assim.by\ citric.\ acid}$. : P_2O_{5total} and P_2O_{5water}. : P_2O_{5total} in the products.

The water-soluble form of P_2O_5 means that the products contain monocalcium phosphate and partially free H_3PO_4. The digestible forms of P_2O_5 and CaO indicate the presence of dicalcium phosphate in the products. The relative content of the digestible form of P_2O_5 less than 100% indicates that a certain amount of undecomposed phosphate mineral remains in the composition of the products.

Fig. 5 shows the X-ray radiography of the product obtained by decomposition of phosphate flour with 75% sulphuric acid. The figure shows that the main components of simple superphosphate are monocalcium phosphate characterised by diffraction maxima of 3.90, 3.20, 3.01, 2.86, 2.80, 2.69, 2.09, 1.75, 1.55 A°, two-water dicalcium phosphate having diffraction minima of 3. 80, 2.47, 2.25, 2.14, 1.94, 1.88, 1.82 A°, anhydrous dicalcium phosphate having diffraction maxima of 2.19 A°, calcium sulfate dihydrate having diffraction maxima of 4.29, 3.80, 2.22, 2.14, 1.99 A°, and calcium sulfate semihydrate having a diffraction maximum of 1.64 A°.

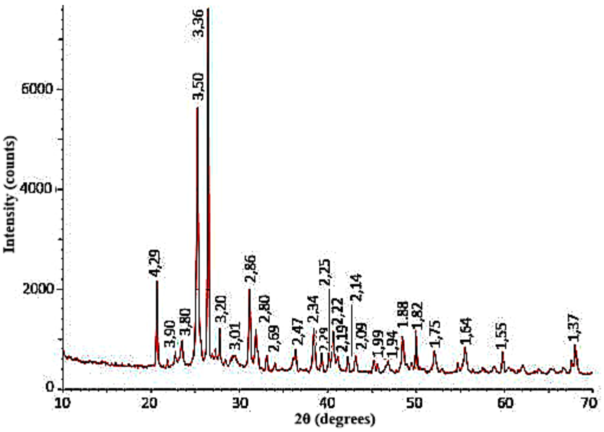


Fig. 5. X-ray radiograph of simple superphosphate
Рис. 5. Рентгенограмма простого суперфосфата

Thus, the involvement of yolk phosphate rock from Karakalpakstan in the production of mineral fertilisers increases the gross amount of phosphate-containing fertilisers, which are in great shortage in the country. The technology eliminates the consumption of the most expensive input component - ammonia, there are no stages of storage ripening and drying. Besides, the method (acid activation) allows to reduce the rate of deficient sulphuric acid by 30-40% when obtaining 1 tonne of P_2O_5.

CONCLUSIONS

Chemical, mineralogical and disperse compositions of jelly phosphorite flour of Porlytau deposit were investigated. It was established that the main phosphate minerals of Porlytau phosphate flour are represented only by kurskite. Calcite, magnesite, glauconite, quartz, feldspar and others are present as impurities.

On the basis of chemical analysis and physicochemical studies, the calculation for mineral components of phosphorite with on the content of components for sulphuric acid was carried out.

The optimum concentration of sulphuric acid (93% H_2SO_4) was found for the production of simple superphosphate, which results in a doughy b easily transportable mass, quite suitable for granulation in a screw mixer. At this concentration, the highest phosphate feedstock K_{dasl} is still relatively achieved.

The authors declare the absence a conflict of interest warranting disclosure in this article.

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