Physico-chemical Properties of Evaporated Extraction Phosphate Acid (40÷56% ) from Phosphorites of the Central Kyzylkum

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**Abstract:** This article describes the technology for obtaining high-quality phosphorus fertilizer by increasing the concentration of extraction phosphate acid (EPA) for the production of phosphorus fertilizers required by modern agriculture. The technology for obtaining high-quality phosphorus fertilizers from poor phosphates of the Central Kyzylkum Desert requires studying the composition of the obtained phosphorus fertilizers. The main reason for this is that impurities contained in phosphorite ore pass into the EPA, which leads to the fact that the amount of these impurities in the fertilizer obtained from it exceeds the established norm. If it is possible to increase the concentration of EPA by purifying it from additional impurities using appropriate purification methods, then, firstly, the resulting fertilizer will be highly concentrated, and secondly, its chemical composition will contain impurities that do not exceed the maximum permissible concentrations. To achieve the set goal, an EPA with a concentration of 25,13 to 27,5% , was obtained, while a two-stage concentration method was chosen compared to previous works. In the process of concentrating the EPA using the two-stage evaporation method, 4 samples of the EPA were selected, and 16 samples were selected from them, and changes in the properties and composition of the EPA during the concentration process were described. The main aspect of this is that the thickening of the EPA during evaporation makes their further processing difficult. To do this, a magnesium-rich precipitate with an average content of 40–46% is separated by filtration. It is substantiated that the characteristic changes in the obtained concentrated acids in the range from 45% to an average of 55% , which are used to obtain high-quality fertilizers, make it possible to obtain mineral-quality fertilizers.

**Keywords:** phosphorite, sulfuric acid, extraction phosphoric acid (EPA), EPA purification, fluorine, viscosity, electrical conductivity, acid schedule, complex fertilizer, phosphorus fertilizer, defluorination

**INTRODUCTION**

It is known that the development of agriculture depends on fertilizers. Phosphorus is an important component of nutrition for all agricultural crops. Thanks to it, the quality of respiration, photosynthesis and metabolic processes of the plant improves. Thus, a large amount of phosphorus allows it to better absorb potassium, nitrogen, magnesium and other nutrients.

Phosphorus fertilizers for plants improve the effect of other fertilizers, increasing the quality of fruits and yield. In the article we will tell you why fertilizers containing phosphorus are needed, to obtain the same fertilizer, what methods are used to process phosphorites, phosphoric acids obtained by the extraction method of phosphorite, what they are and why it is important to use them.

The number of types of phosphorus fertilizers is smaller than others. This is compensated by the fact that this component is present in many other fertilizers, consisting of a large number of mineral elements. Let's take a closer look at the main groups of fertilizers used by farmers, gardeners and summer residents. Usually there are Simple and Double Superphosphate. Simple superphosphate contains about 16-20% phosphorus. It is a powder fertilizer that easily dissolves in water or acids. In addition, it contains nitrogen, magnesium, calcium and sulfur. Due to its form, such fertilizer does not cake, having a low moisture level [1, 2, 3].

Suitable for any soil. But it is worth noting that on acidic soil such feeding will show low efficiency. Double superphosphate Also has a powder form, due to which it is easily dissolved in water. Refers to concentrated fertilizers, therefore it contains 42-50% phosphorus. Due to this, it has a serious effect and can be used on any type of soil. It is worth noting that both types of superphosphates are considered useful for plant feeding. However, in practice, double superphosphate and similar phosphorus-rich types of ammonium phosphates are considered more important.

To improve the quality of phosphorus fertilizers, it is important to ensure a high phosphorus content in phosphate rock and the absence of deterioration of its quality indicators by other impurities. It is also technologically possible to process EPA and obtain high-quality EPA from them.

The rapid growth of the Earth's population requires an increase in the quantitative indicators of its food supply. This, of course, requires an increase in the quantity and improvement of the quality of fertilizers, which are the basis for plant development. Based on this, the development of phosphorus-containing fertilizers is considered a priority [4, 5].

As is known, phosphoric acid is used to produce phosphate fertilizers. There are two types of phosphoric acid: thermal and extraction phosphoric acid. Thermal production of these acids is expensive and, of course, requires the extraction of phosphoric acid. Therefore, various impurities in phosphate rock, especially those that are not needed for fertilizer, cause problems in the fertilizer industry. That is why phosphoric acid is obtained by extraction and used for production.

If we consider the entire world industry of phosphorus fertilizer production, it is convenient to obtain high-quality fertilizer by purification from the initially obtained phosphorite and the EPA obtained from it. It is known that scientists from all over the world have achieved many achievements in this direction. The cost of this is determined by the cost of the reagents used [6].

Therefore, in order to provide agricultural production with high-quality phosphorus and complex nitrogen-phosphorus, and, if necessary, potassium fertilizers, special attention is paid to the following areas: development of effective methods for simplifying the process of purification of EPA using inexpensive and cheap calcium salts and magnesium, including mineral substances as additives. It is important to develop an appropriate technology for obtaining improved quality phosphate ore and complex nitrogen-phosphorus fertilizers.

To date, our republic has achieved significant results in modernizing and improving the chemical industry based on innovation, building enterprises to produce new types of high-quality products, and localizing the raw material base. Large-scale measures have been implemented to develop the chemical industry. Both scientific and practical results have been achieved in the production of new types of import-substituting products [7, 8].

In this regard, the issue of developing a technology for obtaining high-quality phosphorus and complex nitrogen-phosphorus fertilizers free from unnecessary impurities by purifying extracted phosphoric acid (EPA) from fluorides, sulfates, etc. during the extraction process is relevant. It is very important that the technological efficiency of the processes of obtaining EPA, its concentration, obtaining solid and liquid phosphorus fertilizers and feed phosphates largely depends on the physicochemical properties of the solutions used and formed and, it should be noted, on the technology of obtaining their products [9, 10, 11, 12, 13].

Such properties of EPA solutions as density, viscosity and electrical conductivity are necessary for assessing the possibility of transporting solutions, analyzing the processes of decomposition of phosphate raw materials and crystallization, selecting flow meters, pumps and other chemical equipment, and carrying out EPA evaporation processes [14, 15, 16, 17].

An analysis of scientific and technical literature shows that the density ρ, viscosity µ and electrical conductivity *w* of extraction phosphoric acid are mainly determined by its chemical composition, which depends on the type and quality of the phosphate raw material and the content of various impurities in it [18, 19].

For example, the viscosity of the EPA from Karatau phosphorites, all other things being equal, is 1.5÷3.2 times higher than the viscosity of the acid from the apatite concentrate. Moreover, as the acids evaporate, the difference increases sharply [20-24].

The viscosity of evaporated EPA from Chilisai phosphorite, containing 47.7% at 70 °C is 3 times higher than the viscosity of acid from Karatau phosphorites [22]. Thus, the type and quality of natural phosphate raw materials has a significant impact on the rheological characteristics of phosphoric acid solutions.

In the literature, we did not find any information about the physicochemical properties of extractive phosphoric acid from phosphorites of the Central Kyzylkum Desert. The latter are characterized by a fairly high content of carbonates, mainly in the form of calcite, a relatively low content of impurities of and , which creates certain prerequisites for obtaining high-quality extraction phosphoric acid and its concentration.

**METHODS**

In this regard, we conducted studies to determine the density, viscosity and electrical conductivity of evaporated EPA from phosphorites of the Central Kyzylkum. Evaporation of the initial EPA of the following composition, wt.% (Table 1):

**TABLE 1.** Contents of components in the composition of EPA

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| EPA at the rate of  from stoichiometry, % | Content of components, mass% | | | | | | |
|  |  |  |  |  |  |  |
| 95 | 25.13 | 0.41 | 0.56 | 0.57 | 1.09 | 0.362 | 0.014 |
| 100 | 2.53 | 0.34 | 0.55 | 0.58 | 1.08 | 0.364 | 0.015 |
| 101 | 27.39 | 0.24 | 0.53 | 0.58 | 1.07 | 0.377 | 0.015 |
| 103 | 27.42 | 0.21 | 0.49 | 0.59 | 1.07 | 0.387 | 0.016 |

The procedure was carried out in the same way as described in the previous section, with the concentration of EPA reaching 40÷56,10% . Preliminary experiments have shown that with single-stage evaporation of acid, without intermediate purification of the EPA from impurities, as the content increases above 57-58% by weight at room temperature, the acid quickly crystallizes and loses fluidity. In this regard, concentration was carried out to a content in the EPA, depending on the sulfuric acid standard, of 54,88 and 56,11% .

When concentrating EPA in the established order, as their concentration increases, the fluidity naturally decreases due to the increase in the viscosity level. It is necessary to know two basic concepts, firstly, in the process of concentration, as the concentration of sediment in EPA increases, even the fluidity of acids disappears, secondly, the presence of excess impurities in the resulting concentrated EPA leads to a decrease in its quality indicators.

In fact, to obtain a high-quality phosphorus fertilizer, we need high-quality EPA. In order to have a high-quality EPA, it is necessary to reduce the number of prime numbers in its composition. Therefore, at the first stage of concentration, it is best to separate the impurities by filtering the entire mass until 45-46% is reached.

**RESULTS AND DISCUSSION**

Samples of evaporated EPA were analyzed for the content of , , , , ,. For this, modern chemical analysis methods were used. The chemical composition of evaporated EPA from phosphorites of the Central Kyzyl Kum is given in Table 2.

**TABLE 2**. Chemical composition of evaporated EPA from phosphorites of the Central Kyzylkum

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| No of acid samples | Norm of when obtaining EFA, % of stoichiometry | Content of components, mass% | | | | | |
|  |  |  |  |  |  |
| 1 | 95 | 38,0 | 0,85 | 0,35 | 0,77 | 0,79 | 1,50 |
| 2 | 45,5 | 0,98 | 0,32 | 0,81 | 0,91 | 1,64 |
| 3 | 48,8 | 1,30 | 0,34 | 0,96 | 1,01 | 1,80 |
| 4 | 53,9 | 1,46 | 0,37 | 0,11 | 1,12 | 1,96 |
| 5 | 100 | 40,0 | 1,31 | 0,25 | 0,73 | 0,88 | 1,60 |
| 6 | 46,1 | 1,50 | 0,29 | 0,84 | 1,01 | 1,85 |
| 7 | 49,8 | 1,71 | 0,31 | 0,91 | 1,09 | 2,00 |
| 8 | 54,9 | 1,76 | 0,34 | 0,99 | 1,20 | 2,20 |
| 9 | 101 | 41,7 | 2,20 | 0,26 | 0,73 | 0,90 | 1,73 |
| 10 | 46,6 | 2,37 | 0,29 | 0,82 | 1,01 | 1,94 |
| 11 | 51,2 | 2,62 | 0,32 | 0,90 | 1,10 | 2,13 |
| 12 | 56,1 | 2,91 | 0,35 | 0,99 | 1,21 | 2,33 |
| 13 | 103 | 41,9 | 2,25 | 0,27 | 0,76 | 0,93 | 1,78 |
| 14 | 46,2 | 2,47 | 0,30 | 0,83 | 1,04 | 2,04 |
| 15 | 50,9 | 2,71 | 0,38 | 0,92 | 1,12 | 2,19 |
| 16 | 56,7 | 2,98 | 0,42 | 1,09 | 1,27 | 2,41 |

The determination of the density , viscosity μ and electrical conductivity *w* of the evaporated EPA from the phosphorites of the Central Kyzylkum was carried out in the temperature range of 20÷120 °C with a thermostatting accuracy of ±0.1 °C, coinciding with the methods. The results of the determinations are presented in **Tables 3-5**. Analysis of the data presented in Table 3 shows that the density of the solutions of evaporated EPA depends significantly on the concentration of the acid and the temperature.

An increase in temperature leads to a linear decrease in the acid density. At the same time, the slope tangent of the straight lines is practically constant for all studied concentrations of evaporated EPA from phosphorites of the Central Kyzylkum. This indicates that, at a constant rate of sulfuric acid in the process of obtaining EPA, the density of the evaporated EPA (in the studied concentration range) is directly proportional to the content of . Accurate analyses were achieved by performing the experiment in triplicate.

From the data conducted it can be shown that 100% and 101% sulfuric acid standards coincide almost closely with production. Therefore, the data below shows data on these standards.

## TABLE 3. Density , () of evaporated EPA from phosphorites of the Central Kyzylkum

|  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| № | Temperature, оС | | | | | | | |
| 20 | 30 | 40 | 50 | 70 | 80 | 100 | 120 |
| 1 | 1602,9 | 1596,1 | 1589,7 | 1582,9 | 1567,3 | 1558,6 | 1544,6 | 1529,9 |
| 2 | 1713,6 | 1706,1 | 1698,3 | 1691,9 | 1675,9 | 1668,1 | 1652,7 | 1637,3 |
| 3 | 1802,8 | 1793,3 | 1786,0 | 1778,4 | 1763,1 | 1754,4 | 1739,2 | 1725,1 |
| 4 | 1888,8 | 1879,4 | 1871,1 | 1864,4 | 1849,3 | 1840,4 | 1825,6 | 1810,8 |
| 9 | 1541,7 | 1535,2 | 1528,2 | 1521,3 | 1506,9 | 1499,7 | 1485,8 | 1471,3 |
| 10 | 1642,1 | 1634,4 | 1627,7 | 1620,4 | 1604,7 | 1597,3 | 1582,1 | 1567,2 |
| 11 | 1752,6 | 1745,5 | 1738,3 | 1730,9 | 1715,1 | 1707,8 | 1691,8 | 1676,3 |
| 12 | 1853,2 | 1746,6 | 1839,0 | 1832,2 | 1816,3 | 1808,3 | 1792,4 | 1777,1 |

Increasing the rate of sulfuric acid in the process of obtaining EPA from 100 to 101% of stoichiometry leads to a significant change in the physicochemical properties of the solutions. With the same content of in the evaporated EPA, the acid ρ in the second case is 60-70 lower than in the first. As the concentration of EFA increases, this difference decreases somewhat, but insignificantly. The concentration of acid and temperature have a very significant effect on the viscosity of the evaporated EPA (Table 4).

**TABLE 4**. Viscosity μ, () of evaporated EPA from phosphorites of the Central Kyzylkum

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| № | Temperature, оС | | | | | |
| 20 | 40 | 60 | 80 | 100 | 120 |
| 1 | 30,25 | 27,66 | 8,28 | 5,58 | 4,11 | 2,84 |
| 2 | 66,15 | 30,10 | 14,98 | 10,23 | 6,78 | 3,33 |
| 3 | 202.30 | 77,26 | 37,46 | 21,23 | 13,16 | 8,88 |
| 4 | 829,40 | 254,20 | 17,40 | 53,26 | 30,96 | 19,66 |
| 5 | 26,52 | 21,45 | 7,98 | 5,25 | 5,69 | 2,13 |
| 6 | 61,32 | 27,87 | 15,29 | 9,38 | 6,19 | 3,08 |
| 7 | 192,64 | 73,15 | 35,56 | 19,96 | 12,43 | 8,28 |
| 8 | 676,40 | 213,00 | 89,43 | 44,97 | 25,91 | 16,31 |

Such a sharp change in viscosity in solutions with an increase in the concentration of phosphoric anhydride is due to a significant decrease in the content of free water in the system and a corresponding increase in the salt mass of the solutions. Such a sharp change in viscosity in solutions with an increase in the concentration of phosphoric anhydride is due to a significant decrease in the content of free water in the system and a corresponding increase in the salt mass of the solutions. A decrease in the temperature of concentrated (50÷56% ) leads to the formation of stable solutions supersaturated with phosphates of sesquioxides, which have high viscosity. For example, in EPA 40,0% the viscosity at 20 °C is 30,25, and at 120 °C 2,84 , respectively, i.e. the ratio 20 / 120  10,65..

Some reduction in the viscosity of EPA is facilitated by an increase in the content of free sulfuric acid in solutions, achieved by using a slight excess of sulfuric acid in the process of obtaining EPA. Apparently, this is due to an increase in the solubility of salts and an increase in the proportion of a component that is less viscous than phosphoric acid.

It is known from the literature that with increasing acid concentration, its density and viscosity increase. Electrical conductivity decreases. Most importantly, the presence of excess free acids is also important for ensuring readability. In this case, readability can be improved by adding excess sulfuric acid. If we take into account the composition of modern mineral fertilizers, it can be shown that it is important in the production of sulfate-ion fertilizers.

Thus, the analysis of the results of the study of the rheological characteristics of evaporated EPA from phosphorites of the Central Kyzylkum shows that it retains sufficient fluidity for technological purposes up to a content of 50-56% Increasing the temperature of acids to 40-60 °C leads to a sharp decrease in the viscosity of evaporated EPA.

The study of the electrical conductivity of evaporated EPA has established (Table 5) that w depends significantly on the acid concentration and temperature, and fluctuates in the range of . An increase in the content and a decrease in temperature leads to a decrease in the specific electrical conductivity of the evaporated EPA.

Increasing the temperature from 20 to 120 °C increases the electrical conductivity of concentrated (55÷56% ) EPA by 9÷14 times, while in solutions containing 40÷42% only by 3,2÷3,3 times. Moreover, with an increase in the content of free sulfuric acid in the evaporated EFC, the electrical conductivity of the latter increases (Table 5), which is due to an increase in the concentration of hydrogen ions in the system.

## TABLE 5. Specific electrical conductivity ℵ, () of evaporated EPA from phosphorites of the Central Kyzylkum

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| № | Temperature, оС | | | | | |
| 20 | 40 | 60 | 80 | 100 | 120 |
| 1 | 6,97 | 10,60 | 14,44 | 18,16 | 20,76 | 22,77 |
| 2 | 4,04 | 6,85 | 9,75 | 13,32 | 17,87 | 19,68 |
| 3 | 2,35 | 4,32 | 7,03 | 11,07 | 15,10 | 19,54 |
| 4 | 1,04 | 2,50 | 4,71 | 7,13 | 11,29 | 14,86 |
| 5 | 9,49 | 14,05 | 18,34 | 23,24 | 25,90 | 30,04 |
| 6 | 6,33 | 10,34 | 14,71 | 17,11 | 22,41 | 26,39 |
| 7 | 3,63 | 6,55 | 10,19 | 14,40 | 17,87 | 22,23 |
| 8 | 1,93 | 3,87 | 6,76 | 10,12 | 13,51 | 17,43 |

Based on the conducted research, it can be concluded that extraction phosphoric acid from phosphorites of the Central Kyzylkum can be quite easily evaporated without preliminary purification from impurities to a concentration of 50÷55% . The content of impurities in the evaporated acid varies within the range, wt.%: – 0,7 ÷ 1,0; – 0,9 ÷ 1,2; – 1,6 ÷ 2,2. Evaporated extraction phosphoric acid has satisfactory rheological characteristics, which creates technological prerequisites for its use in the production of liquid complex fertilizers, polyphosphate fertilizers, double superphosphate and feed phosphates.

In obtaining phosphorus fertilizers, such compositions of EPA are considered effective. Since improving their composition and rheological properties by pre-treatment with acids is important for improving the quality of fertilizers. From this we can conclude that ordinary superphosphate and ammonium phosphate are compared with each other.

**CONCLUSION**

To produce high-quality phosphorus fertilizers based on modern technologies for the production of mineral fertilizers, the phosphorus fertilizer obtained from phosphorite, which is its main raw material, must be of high quality. The main problem is that the high content of fluorine, which is an impurity in phosphate rock, relative to the total mass makes it difficult to obtain high-quality fertilizer. Therefore, an important aspect of the process is heating the initially obtained low-concentration EPA in order to concentrate it and remove excess fluorine from it. It has been proven that complex fertilizer and, in addition, livestock feed can be obtained on the basis of the obtained concentrated EPA.

One of the main possibilities of animal feed production is that the amount of fluorine in the EPA is below the permissible concentration. One of the most important experimental methods to achieve this was the cncentration of the original EPA by heating it under normal conditions, while the second method was carried out by evaporation under conditions of sharply reduced atmospheric pressure. The effective side of this is also the saving of thermal energy and the increase in fluorine gas emissions due to the sudden boiling of the EPA. It is important to note that the result is concentrated EPA with a reduced fluorine content. This guarantees the efficiency and safety of using concentrated EPA.

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