



Section 3. Technical sciences in general

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CONCENTRATION OF EXTRACTIVE PHOSPHORIC ACID WITH HOT AIR

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Abstract

This article substantiates methods for concentrating extraction phosphoric acid (EPA) with a concentration of $22 \div 28\%$ P_2O_5 , obtained on the basis of phosphorites of the Central Kyzylkum to 55% P_2O_5 . As a result of evaporation using heated air under laboratory conditions, the amount of fluorine in it is much less than during evaporation under normal conditions. Initially, EPA was obtained from local Kyzylkum phosphorites under normal laboratory conditions by the dihydrate method. For this, a simple glass reactor was used, with a moving stirrer with an electric motor and vacuum filtering devices. The initial composition of the resulting EPA was analyzed by chemical and physicochemical methods and evaporated in three ways to a concentration of 55% P_2O_5 . Laboratory work carried out by these methods was carried out under strict temperature control. The experiment was repeated several times and the results were compared. Experiments have shown that when EPA is evaporated to a concentration of $39 \div 46\%$ P_2O_5 , a precipitate is formed, which is convenient to separate. After separating the precipitates, it turned out that the resulting highly concentrated EPA does not lose its fluidity.

Keywords: Local phosphorite, reactor, extractive phosphoric acid (EPA), concentration, evaporation, vacuum, heated air, thermal phosphoric acid (TPA)

1. Introduction

Increasing the amount of agricultural products is important for the survival of people on the globe. Because the most important part of food security is the amount of food products. In agriculture, increasing crop yields due to salinization of crop areas and the spread of drought is one of the main and most important issues.

To do this, it is necessary to increase the amount of substances and elements in the soil that are absorbed by the plant. In practice, there is a deficit of natural organic fertilizers, so to compensate for this, it is necessary to develop the mineral fertilizer industry. That is why these issues are included in the main plan of our country.

The problem of fertilizers is in the first place in the development of agriculture. To solve these problems, the development of their main types and the development of new technologies are among the main tasks facing the scientists-chemists of our republic. In our country, the industry for the production of mineral fertilizers is widely developed. However, it is necessary to continue scientific work in order to increase the level of absorption by plants, use raw materials economically and ensure the quality of fertilizer.

Currently, organic, mineral and organomineral types of fertilizers are used in agriculture, among which the leading place is occupied by complex fertilizers containing assimilable phosphorus. The necessary raw material for this is the phosphorite of the Central Kyzylkum, which is considered the poorest among phosphorus-containing phosphorites. There are many scientific works on their processing and concentration of phosphorus acids obtained from them in order to obtain high-quality phosphorus fertilizers (Awwad N. S., El-Nadi Y. A., Hamed M. M. 2013; Jiahui Men, Yiming Li, Peifeng Cheng, Zhanming Zhang. 2022; Khurramov N. I., Nurmurodov T. I., Erkaev A. U. 2021; Melikulova G. E. [and others]. 2019). Most importantly, in addition, due to the simplicity of production, the production of ammophos, simple and double superphosphate has been widely established (Bakhriddinov, N.S., & Turgunov, A.A., 2022). Their quality, firstly, it is necessary to increase the amount of phosphorus in the composition, and secondly, to increase the level of assimilation of the available phosphorus by plants.

Central Kyzylkum phosphorites used for the production of phosphorus fertilizers are local, which is convenient for production. However, the phosphorus content in it is $14-16\%~P_2O_5$, the amount of phosphorus is increased in relation to the total mass of this phosphorite, using the enrichment method

by reducing the content of excess substances through additional processing. Due to the use of this method, it is possible to obtain from them a concentration of extraction phosphoric acid (EPA) up to 22-28% P_2O_5 already at the primary stage.

In the process of producing phosphorus fertilizers, along with the enrichment of phosphorites, EFA is also obtained in low concentration during the production process. As a result, the evaporation process is widely used to reduce the water content of this acid in order to obtain high-quality, i.e. high-phosphorus EPA. The amount of energy consumed is also high, which leads to an increase in the cost of the produced mineral phosphorus fertilizer. Taking this into account, it is necessary to use an economical method of concentration. Another property of mineral fertilizers is that the level of absorption by plants is very low after their introduction into the soil and after watering. In particular, the rate of application of nitrogen fertilizers is based on the ease of evaporation and solubility of nitrogen in water. To eliminate these shortcomings, it is necessary to use gradually dissolving types of fertilizers, i.e. types in the form of polyforms.

To form phosphorus in the form of polyphosphate in phosphorus fertilizer, its concentration is increased by evaporating the EPA intended for mineral fertilizer. It is known that the use of thermal phosphoric acid (TPA) is convenient for obtaining highly concentrated phosphoric acid, easily absorbed in mineral fertilizers. However, due to the high cost in Uzbekistan, such TPA is not produced for the purpose of obtaining phosphorus mineral fertilizers, and EPA is used instead. To obtain high-quality ammonium phosphates from EPA, it is necessary to carry out a process of their purification from heavy metals, arsenic, calcium, magnesium and fluorine. This must be done in an economical way. Such methods are divided into evaporation, precipitation, the use of organic solvents, ion exchange, crystallization (Najmiddinov, R., Shamshidinov, I., Qodirova, G., Nishonov, A., & Sayfiddinov, O. 2022; Bakhriddinov, N.S., 2022; Bakhriddinov, N.S., 2021). One of the convenient methods currently being implemented is evaporation, which reduces the amount of water contained in the original EPA. Laboratory chemical and physicochemical analyses have shown that the original EPA is completely composed of orthophosphoric acid P_2O_5 . To increase the amount of phosphorus pentoxide in phosphorus P_2O_5 fertilizer, it is necessary to evaporate the primary EPA obtained from phosphorites (Kodirova, G. K., Shamshidinov, I.T., Turaev, Z., & Najmiddinov, R. Yu. U., 2020).

2. Main body

Laboratory work was carried out in a laboratory setup with a simple glass and also a metal reactor made of stainless steel, a water cooler from the evaporation of water as a result of the reaction and a stirrer driven by an electric motor, by gradually adding sulfuric acid to the phosphorite of the specified sample. Thermal concentrate of phosphorite from Central Kyzylkum (composition: P₂O₅-25.68%; CaO --53.28%; CO₂-2.68%; MgO -0.52%; F -2.76, $R_2O_3-3.58\%$; $SO_3-5.01\%$) and 93% sulfuric acid H₂SO₄ were used for laboratory work. The stoichiometric rate of sulfuric acid was set equal to 100% of the amount required for the decomposition of calcium in phosphorite, and was brought to an aqueous solution of the corresponding concentration. When it interacts with the sulfuric acid H₂SO₄ - phosphorite Ca₅F(PO₄)₃ taken for the reaction, the following process is observed:

$$Ca_{5}F(PO_{4})_{3}+5H_{2}SO_{4}=3H_{3}PO_{4}+5CaSO_{4}+HF$$

It is known that in addition to the basic phosphoric acid H₃PO₄ formed during the acidic decomposition of phosphorites, calcium dihydro- and hydrophosphates are formed:

$$2Ca_5F(PO_4)_3+7H_2SO_4=3Ca(H_2PO_4)_2+7CaSO_4+2HF$$

$$2Ca_5F(PO_4)_3 + 4H_2SO_4 = 6CaHPO_4 + 4CaSO_4 + 2HF.$$

The process lasted 4 hours, and the EPC was isolated by filtration. The chemical composition of the EPA was obtained by repeating this process 3 times, the EPA was first taken separately, then three EPAs were put together and mixed until one mass was formed, and the total mass composition was determined analytically using the established method (Table 1).

It is known that extracted EPA is used in the production of double superphosphate or ammonium phosphates. If we take into account the requirements of the present time, then the abundance of additional substances and elements in its composition, especially excess fluorine, negatively affects the quality of the resulting fertilizer. Taking this into account, the process of concentrating EPA is carried out at high temperature, which gives a positive result. For this purpose, it has been experimentally established that when the original EPA is evaporated by heated air, the rate of volatilization of the fluorine contained in it increases.

Table 1. Chemical composition of EPA obtained from phosphorite

Nº	P_2O_5	H ₂ SO ₄	CaO	MgO	Al_2O_3	Fe ₂ O ₃	F		
Composition of separately received EPA									
1	27.76	0.20	0.48	1.25	1.90	1.46	2.21		
2	27.61	0.24	0.51	1.22	1.95	1.39	2.27		
3	27.92	0.27	0.47	1.18	1.89	1.43	2.23		
With mixed EPAs									
4	27.76	0.25	0.49	1.22	1.91	1.43	2.24		

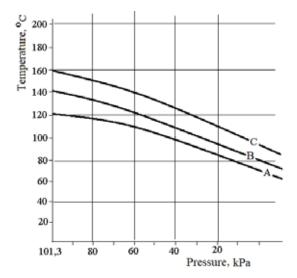
During the evaporation process of the EPA, as its concentration increases, the boiling point also increases accordingly. When the obtained concentration of the EPA reaches 45-46% P₂O₅, the boiling point reaches 120 °C, 140 °C at 50% and 160 °C at 55% (Fig. 1).

Modern industrial production requires the use of energy-efficient technologies. Accordingly, as shown in Figure 1, boiling temperatures are reduced under reduced pressure conditions and energy savings are observed.

It is known that the presence of substances and elements in the EPA and its quantity depend on the content of the phosphorite used. Phosphorite contains fluorine, magnesium and similar additives that pass into the EPA during the extraction process. One of the properties of magnesium is that it causes its condensation when the concentration of evaporating EPA reaches 40-45% P_2O_5 . Therefore, the first stage of the evaporation process is up to 40-45% P_2O_5 , after reaching

this concentration, the EPA is purified from magnesium sediment and other by-products (Sadriddinovich, B. N., 2022; Shamshidinov, I. T., & Arislanov, A. S., 2022; Sobirov, M. M., Bakhridinov, N. S., & Rozikova, D. A., 2020; Sadriddinovich, B. N., 2022; Bakhriddinov, N. S., & Turgunov, A. A., 2022).

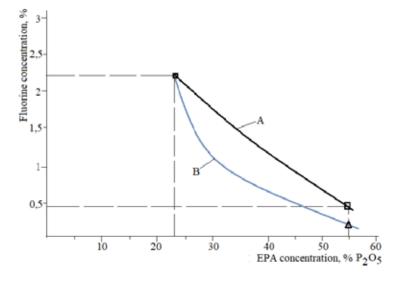
Figure 1. Change in boiling temperature during vacuum evaporation of EPA: A - 45%, B - 50%, C - for EPA with a concentration of 55% P_2O_5



Two methods were used to concentrate the EPA: the evaporation process under normal conditions and under vacuum conditions. Evaporation under normal conditions, i.e. at $P_0 = 101.3$ kPa, with an increase in the content of the EPA from 45% to 55% P_2O_5 , it is clear that its boiling point changes from 120 °C to 160 °C, and with a decrease in pres-

sure to 20 kPa – from 85 °C to 110 °C, respectively (Fig. 1). This, in turn, shows that the vacuum process saves energy for evaporation. Experiments have shown that the amount of volatile substances in the EPA is the same as under normal conditions, based on the corresponding change in saturated vapor pressure with concentration.

Figure 2. Dependence of fluorine release in EPA on the type of vaporization: A – normal and vacuum-heating evaporation; B – evaporation with heated air



If we look at the example of fluorine, we can see that the amount of fluorine in the EPA with a concentration of 27.67% P_2O_5 is 2.26%, but as a result of evaporation to 55% P_2O_5 , the amount of fluorine decreases to an average of 0.47%. This can be explained as follows:

- -0.46% under normal conditions;
- -0.47% under vacuum conditions.

In modern technologies, it is expected that any process will be carried out taking into account energy efficiency, cost reduction and environmental requirements. With this in mind, another type of evaporation was used – a method of supplying heated air to the EFC and removing the water contained in it. The most important thing is that the level

of fluorine in this EPA increases as the temperature of the heated air increases. This is shown in Figure 2 below.

The result of chemical analysis shows that the fluorine contained in EPA is released during the evaporation process. 1.5÷2.5% of volatile fluorine relative to the total amount of EPA can also have a negative impact on the environment. That is why it is good to absorb volatile fluorides in a simple water absorbent.

The transformation of the monophosphates contained in it into polyphosphates during evaporation of EPA is also important. According to the type of evaporation process, the amount of polyphosphate during evaporation by simple heating is given in (table 2).

Table 2. Change in the amount of polyphosphates during the process of increasing the concentration of EPA to 55% by the method of thermal evaporation

Concentration of EPA, %	40	45	50	55
Amount of polyphosphate in EPA, %	3-4	9-15	22-28	31-39

The second side of the changes that occur during the evaporation of the EPA obtained for the experiment to $55\%~P_2O_5$ is the transition of phosphoric acid from the orthoform to the polyform. This mainly begins when the concentration of EPA reaches $40\%~P_2O_5$.

Based on the results obtained by the chromatographic method of physicochemical analysis of concentrated acids obtained on the basis of the experiment, it was confirmed that when the concentration reaches $40\% P_2O_5$, the phosphates contained in it are converted into polyforms (Table 2).

Therefore, as a result of the increase in temperature, the precipitation of fluorine contained in the evaporating acid in the form of K_2SiF_6 , $KNaSiF_6$, MgF_2 , K_3SiF_6 accelerates and flies out of the system. The composition of the sediment isolated from 40% EPA was studied based on infrared-IR and X-ray structural analysis data.

Therefore, measures are taken to collect sediments formed during the evaporation of EPA, with the aim of processing and preventing pollution of the atmospheric air by the released fluorine using absorbers, i.e. sorbents.

The above fluorine content in concentrated 55% EPA can be considered acceptable for the resulting concentrated EPA from 0,5 to 1%, and these acids can be used

not only as mineral fertilizers, but also as animal feed. However, when preparing livestock feed, the amount of fluorine should be as small as possible.

The chemical composition of the Central Kyzylkum phosphorites may vary depending on the location of the mineral and the amount of substances and elements in it. For example, a magnesium content of 0.52% allows increasing the concentration of the first stage of the evaporation process of such EPA to 45% P_2O_5 , while the magnesium content does not reach 1% when extracting EPA from this phosphorite. In addition, the evaporation temperatures of EFA with the same magnesium content may also be different.

A second look at the experience:

When observing the evaporation process by spraying air at a temperature of 150–160 °C through a tube made of acid-resistant alloy steel, the growth of the concentration of the obtained EPA, as well as the amount of polyphosphates in it, accelerated. A further increase in the temperature of the heated air leads to an increase in the rate of fluorine evaporation. For this purpose, a chromeplated electric heater with an asbestos coating is used on the surface of this tube.

This was done by spraying the acid into the reactor using an air atomizing pump. The result obtained showed the formation of concentrated EPAs with high polyphosphate content. When this resulting acid was also subjected to chemical analysis, the results shown in table 3 were seen.

Table 3. Change in the amount of polyphosphates in the process of increasing the concentration of EPA to $55\% P_2O_5$ by steaming with heated air

Concentration of EPA, %	40	45	50	55
Amount of polyphosphate in EPA, %	7–9	16-19	29-35	38-47

According to the results of the second experiment, the production of mineral fertilizer from this acid by obtaining concentrated acid with an increase in the amount of polyphosphate due to spraying with heated air is good.

3. Conclusion

The obtained initial stage EPA was poured into 3 cups of 100 grams each and each of them was evaporated using separate methods:

- 1. The concentration reached 55% when the temperature of the mass in the reactor increased from 115 °C to 198 °C during its evaporation in the oil heater bath under atmospheric pressure conditions P_0 =96.7 kPa (723 mm Hg). However, the total mass reached a state in which it lost its fluid state.
- 2. Evaporation was carried out in a similar way, reducing the pressure to 20 kPa, and the temperature was changed from 80 °C to 110 °C, respectively. In this case, the mass in the reactor was also in a thick state.
- 3. The temperature was the same as in the first case when using the method of evaporating heated air under atmospheric pressure conditions $P_0 = 96.7$ kPa (723 mm Hg). When the resulting concentrated EPA was cooled and the transparent part was analyzed, it was found that the amount of fluorine it contained was less than that of the acid obtained by concentrating it using the conventional method. It is evident that acids obtained by such methods can be used to produce phosphorus-containing feed for livestock. Experiments have shown that when evaporating EPA to a concentration of $39 \div 46\%$, a precipitate is formed that is easy

to separate. After separating the precipitates, it turned out that the highly concentrated EPA obtained does not lose its fluidity.

The second group cations and fluorine in concentrated EPA have a negative effect on the fluidity. Therefore, the fluidity is maintained when the precipitate formed during the separation of the first stage of evaporation is separated from EPA. Most importantly, evaporation using heated air ensures that the fluorine content of the resulting concentrated EPA is significantly lower.

It can be said that the boiling point changes depending on the amount of magnesium in the EPA. For example, 0,54% MgO is 110 °C, and 1,50% is 154 degrees. The main reason why evaporation can be continued to the initial stage of the evaporation process of 45% with a magnesium content in the evaporated EPA of 0,54% is its high fluidity at high temperature. Accordingly, with its 1,24% MgO, it was found in the experiment that it is forced to separate from the sediment when the EPA concentration reaches 40%.

The most important thing is that, firstly, the amount of magnesium in concentrated EPA precipitates at the first stage of the concentration process, and experience has shown that it does not exceed 1,5%, and secondly, evaporation under vacuum conditions leads to energy savings.

On the other hand, the experiment showed that the amount of polyphosphate in the concentrated EFC obtained by using heated air during heating and steaming and feeding it to the EPA inside the reactor can be 5–7% more than in the previous case.

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