

**DESULFATION OF WET-PROCESS PHOSPHORIC ACID USING POTASSIUM CARBONATE ( $K_2CO_3$ )**

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**Abstract.** JSC “Ammophos-Maxam” is well known for the production of ammophos fertilizers based on concentrated phosphorites. In the production process, extraction phosphoric acid (EPA) obtained by the dihydrate process is used as a raw material; its composition contains 18–19% phosphorus pentoxide ( $P_2O_5$ ). The low  $P_2O_5$  concentration in EPA significantly increases heat and energy consumption per unit of final product during the stages of evaporation, purification, and granulation in the production of high-phosphorus fertilizers. In the Republic, extensive efforts are currently being undertaken in this area. This article presents recent research results on the purification of dihydrate-process phosphoric acid from impurities carried out at JSC “Ammophos-Maxam”.

**Keywords:** phosphorus, extraction phosphoric acid (EPA), Central Kyzylkum, ammophos, sunflower ash.

**Аннотация.** АО «Амморфос-Махам» известно производством аммофосных удобрений на основе концентрированных фосфоритов. В технологическом процессе в качестве сырья используется экстракционная фосфорная кислота (ЭФК), полученная ди-гидратным методом, содержащая 18–19% пятиокси фосфора ( $P_2O_5$ ). Низкая концентрация  $P_2O_5$  в ЭФК приводит к значительному увеличению удельных затрат тепла и энергии на единицу готовой продукции на стадиях выпаривания, очистки и гранулирования при производстве высокофосфорных удобрений. В республике в настоящее время проводятся широкомасштабные работы в данном направлении. В статье представлены последние результаты исследований по процессу очистки экстракционной фосфорной кислоты, полученной ди-гидратным методом, от примесей, выполненных на АО «Амморфос-Махам».

**Ключевые слова:** фосфор, экстракционная фосфорная кислота (ЭФК), Центральные Кызылкумы, аммофос, зола подсолнечника.

**Annotatsiya.** “Ammophos-Maxam” AJ konsentrlangan fosforitlar asosida ammosfos o‘g‘itlar ishlab chiqarishi bilan tanilgan. Ishlab chiqarish jarayonida xomashyo sifatida digidrat usulda olingan ekstraksion fosfat kislotasi (EFK), uning tarkibida 18–19% fosfor beshksidi ( $P_2O_5$ ) mavjud. Konsentratsiyasi past  $P_2O_5$  tarkibli EFK ni bug‘latish, tozalash va donadorlash bosqichlarida yuqori fosforli o‘g‘itlarga yakuniy mahsulot birligiga to‘g‘ri keladigan issiqlik va energiya sarfini sezilarli darajada oshirish. qurilish ko‘rinishida mavjud bo‘lgan respublikada keng ko‘lamli ishlar olib borilmoqda. Ush maqolada “Ammophos-Maxam” AJ da digidrat usulda olingan fosfat kislotasini tarkibidagi qo‘shimchalardan tozalash jarayoni bo‘yicha olib borilgan so‘nggi material bayon qilingan.

**Kalit so‘zlar:** fosfor, ekstraksion fosfat kislotasi (EFK), Markaziy Qizilqum, ammosfos, kungaboqar kuli.

### **Introduction**

The production of high-concentration phosphorus fertilizers has long remained a topical issue associated with the search for innovative solutions, particularly in the field of nutrient supplements for agricultural crops. This process clearly reflects the adaptability of the agricultural sector to modern production requirements and challenges. In particular, in order to ensure food security and the production of high-quality agricultural products, the research and practical implementation of advanced nutrient additives are of great importance. These consistent efforts demonstrate the willingness of farms

to adopt new technologies and approaches and contribute to ensuring the sustainability and competitiveness of global agriculture under modern agro-industrial conditions.

The global demand for mono- ( $\text{KH}_2\text{PO}_4$ ) and dipotassium phosphates ( $\text{K}_2\text{HPO}_4 \cdot 3\text{H}_2\text{O}$ ) (MKP and DKP) [1,2], as well as potassium–phosphorus (KP) and nitrogen–phosphorus–potassium (NPK) fertilizers, clearly demonstrates their decisive role in modern agriculture. The production of these fertilizers is entirely based on concentrated phosphoric acid. Therefore, the development of technologies for concentrating phosphoric acid obtained by the dihydrate process (extraction phosphoric acid, EPA) and the production of high-quality phosphorus fertilizers constitute an urgent and important task.

At present, domestically produced products are increasingly competing with foreign analogues. Addressing these challenges requires the development of well-founded solutions across several key scientific and technical directions, which constitutes one of the most pressing tasks today. First and foremost, determining optimal conditions for the desulfation of low-concentration wet-process phosphoric acid (WPA) is among the most important objectives. At the next stage, it is necessary to improve the concentration process of desulfated extraction phosphoric acid (EPA) through evaporation. At the same time, particular attention is paid to improving the rheological properties of the evaporated EPA. At the final stage, it is necessary to develop technologies for producing various grades of potassium phosphates and complex fertilizers based on concentrated EPA. This integrated approach not only addresses current challenges but also enables the domestic fertilizer industry to achieve a competitive level with foreign manufacturers.

In chemical enterprises, concentrated extraction phosphoric acid (EPA) obtained by the dihydrate process plays a crucial role in the production of double superphosphate, ammophos, and liquid fertilizers. The concentration of this solution is determined by the type of fertilizer being produced. In particular, for the production of double superphosphate and ammophos, the  $\text{P}_2\text{O}_5$  content in EPA should be in the range of 45–55%, whereas for potassium polyphosphates and liquid fertilizers, a  $\text{P}_2\text{O}_5$  concentration of 72–83% is required [3–5].

To achieve such high concentrations, EPA is evaporated to reduce its water content, thereby increasing the concentration of phosphorus pentoxide ( $\text{P}_2\text{O}_5$ ). From a theoretical standpoint, the evaporation process makes it possible to obtain solutions with very high  $\text{P}_2\text{O}_5$  concentrations, since the vapor released from pure  $\text{H}_3\text{PO}_4$  consists entirely of water. This circumstance reflects the complexity of advanced technological processes used in the production of various phosphorus fertilizers and their capability to yield products with different concentrations tailored to agricultural needs [6].

Scientific and technical literature extensively discusses the production of evaporated EPA based on thermal concentrates derived from Kyzylykum phosphorites, as well as the possibility of producing liquid complex fertilizers from such acid. However, it should be emphasized that the composition of EPA obtained from thermal concentrates differs significantly from that of EPA produced from washed and calcined phosphoconcentrate (WKPC–26), which is used as the primary raw material at JSC “Ammophos-Maxam”. In particular, the presence of impurities such as  $\text{MgO}$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{CaO}$ , and  $\text{SO}_3$  has a substantial effect on the chemical composition of phosphoric acid. These impurities can significantly influence both the evaporation process and the rheological properties of the evaporated phosphoric acid. Therefore, a detailed study of these differences, which are inherent to various phosphorite sources and processing technologies, is of great importance for optimizing production processes and ensuring the required rheological properties of evaporated phosphoric acid.

Since sulfate ions constitute the main impurity in EPA, the primary purification stage should be focused on desulfation. In the studies described, the authors carried out desulfation of EPA with the specified composition using  $\text{Ca}(\text{OH})_2$  and  $\text{CaCO}_3$  in the presence of WKPC–26 containing up to 20%  $\text{CaO}_{\text{dig}}$ . Subsequently, for additional purification of the desulfated EPA, acetone (99.5%  $\text{C}_3\text{H}_6\text{O}$ ) was used in a threefold excess. However, despite the use of calcium-based reagents and WKPC–26, complete removal of sulfate ions could not be achieved, and the degree of sulfate removal did not exceed 85%.

To overcome these limitations, the authors propose the use of  $\text{K}_2\text{CO}_3$  salts as an effective method capable of providing deeper desulfation. The main technological advantage of potassium salts lies in their ability to reduce the residual  $\text{SO}_4^{2-}$  ion content in EPA solutions by a factor of 25–30. This approach eliminates the drawbacks inherent in conventional calcium-based methods and represents one of the most promising solutions for more complete and efficient removal of sulfate ions during the processing of EPA.

The article presents the purification of impurities using  $\text{K}_2\text{CO}_3$  salt during the investigation of  $\text{MgO}$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{CaO}$ , and  $\text{SO}_3$  precipitates formed from initially desulfated and evaporated extraction phosphoric acid (EPA) obtained by the dihydrate process.

### **Experimental section.**

The evaporation process of wet-process phosphoric acid (WPA) obtained by the dihydrate process from the operating production of JSC “Ammophos-Maxam” was systematically investigated under laboratory conditions. The mass-based chemical composition of the WPA used as the object of study was as follows: P<sub>2</sub>O<sub>5</sub> – 18.97; CaO – 0.28; MgO – 0.27; Fe<sub>2</sub>O<sub>3</sub> – 0.42; Al<sub>2</sub>O<sub>3</sub> – 0.57; F – 0.47; SO<sub>3</sub> (total) – 2.97; and SO<sub>3</sub> (dissolved) – 0.16.

At the desulfation stage, an approach significantly different from the conventional method was applied, namely, potassium salts were used instead of calcium-based minerals. For the desulfation process, potassium carbonate contained in *sunflower ash* with a K<sub>2</sub>CO<sub>3</sub> content of at least 62% was selected. This substitution is justified by the high efficiency of potassium salts in removing sulfate ions from phosphoric acid.

Furthermore, during the subsequent evaporation of the desulfated phosphoric acid, when the concentration of phosphorus (V) oxide reached 40–62%, a pronounced precipitation of other impurity components present in the acid was also observed. The combination of using potassium salts for desulfation followed by evaporation defined the distinctive features of the experimental approach. This innovative methodology, applied in laboratory studies, is aimed at improving the purity and quality of phosphoric acid and contributes to the enhancement of production technologies at JSC “Ammophos-Maxam”.

Experiments on the purification of WPA using K<sub>2</sub>CO<sub>3</sub> were carried out in triplicate, and average values were calculated. The degrees of precipitation of MgO, Fe<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub>, CaO, and SO<sub>3</sub> were determined accordingly. All calculations were performed using MS Excel (Windows 10) with a confidence level of 95% (P = 95%).

Concentrated solutions were obtained by evaporating the desulfated acid, and their chemical composition was analyzed in detail using various analytical methods. The P<sub>2</sub>O<sub>5</sub> content was determined by the photolorimetric method, SO<sub>3</sub> by the gravimetric method, and fluorine by the potentiometric method. The contents of calcium and magnesium were determined by the complexometric method, in which titration with Trilon B was carried out, using a change in indicator color as the endpoint criterion (fluorexon for calcium and a dark blue chrome indicator for magnesium).

Sulfate ions were determined by a gravimetric method based on the precipitation of sulfates by the addition of barium chloride in an acidic medium, followed by weighing of the resulting precipitate. The contents of iron and aluminum oxides were also determined using the complexometric method. In particular, a 0.0125 M iron solution was titrated with Trilon B in the presence of sulfosalicylic acid, and the excess amount of Trilon B was subsequently determined by back-titration with a 0.0125 M zinc sulfate solution. This multistep procedure enabled the determination of aluminum content using xylene orange as an indicator.

### **Results and Discussion.**

It was determined that the optimal condition for the formation of potassium sulfate corresponds to 100% of the stoichiometric amount of potassium carbonate. During the experiment, the initial dihydrate-process extraction phosphoric acid (EPA) was charged into a reactor equipped with a screw stirrer and gradually heated to 80 °C using a water bath. Subsequently, the calculated amount of potassium carbonate powder was slowly added to the extraction phosphoric acid over a period of 30 minutes under continuous stirring.

After completion of this stage, the reactor contents were allowed to settle at 60–65 °C for 60 minutes. Following sedimentation, the desulfated extraction phosphoric acid was separated from the precipitate by filtration; the precipitate was dried and weighed. Both the clear phase (desulfated extraction phosphoric acid) and the resulting precipitate were analyzed for total SO<sub>3</sub> and P<sub>2</sub>O<sub>5</sub> contents in accordance with established analytical procedures.

The desulfated dihydrate-process extraction phosphoric acid containing 21.17% P<sub>2</sub>O<sub>5</sub>, 0.27% CaO, 0.28% MgO, 0.46% Fe<sub>2</sub>O<sub>3</sub>, 0.61% Al<sub>2</sub>O<sub>3</sub>, and 0.11% SO<sub>3</sub> was subsequently subjected to a controlled evaporation process in order to achieve the predetermined P<sub>2</sub>O<sub>5</sub> concentration. The evaporation experiments were carried out at atmospheric pressure in a tubular quartz reactor externally heated by a specialized electric furnace and equipped with a blade-type quartz stirrer.

As shown in the figure, the degrees of precipitation of MgO, Fe<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub>, CaO, and SO<sub>3</sub> impurities from extraction phosphoric acid using various inorganic reagents are presented. In this study, the purification efficiency of extraction phosphoric acid using KCl, CaCO<sub>3</sub>, and K<sub>2</sub>CO<sub>3</sub> was investigated over a contact time range of 5 to 30 minutes. The results clearly demonstrate that K<sub>2</sub>CO<sub>3</sub> exhibits the

highest purification efficiency. This finding confirms the feasibility of increasing the concentration of extraction phosphoric acid to higher levels following effective desulfation and impurity removal.

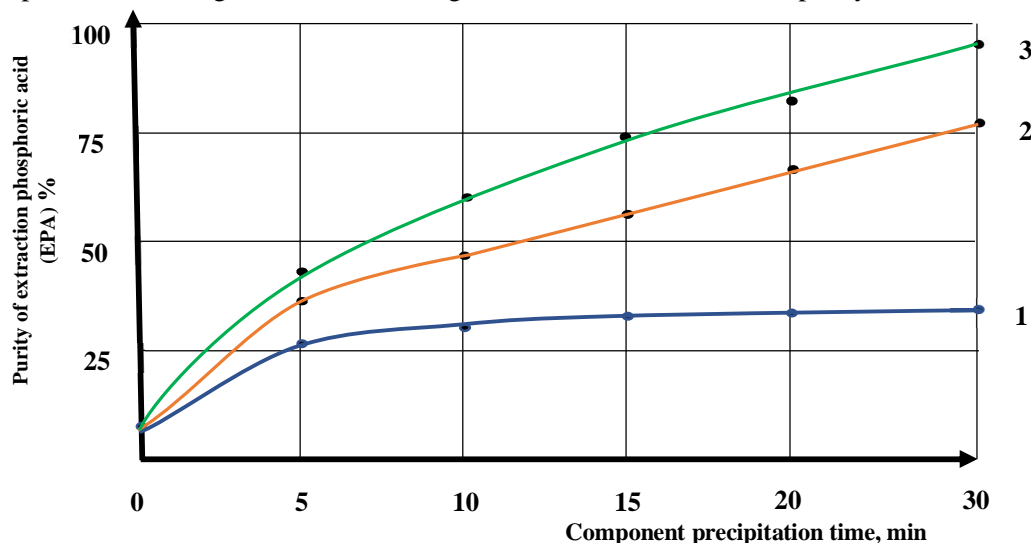


Figure 1. Dependence of the degree of purification of extraction phosphoric acid (EPA) from impurities on time: 1)KCl, 2) CaCO<sub>3</sub>, 3) K<sub>2</sub>CO<sub>3</sub>.

Based on the research results, it can be concluded that purification using K<sub>2</sub>CO<sub>3</sub> achieved an extraction phosphoric acid (EPA) purity of 98.24%. It was established that K<sub>2</sub>CO<sub>3</sub> contained in sunflower ash reduces the impurity content of EPA to a significantly greater extent compared with KCl and CaCO<sub>3</sub>. This, in turn, indicates that the potential for producing high-concentration phosphorus fertilizers is substantially expanded.

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