

МЕДИЦИНА, ПЕДАГОГИКА И ТЕХНОЛОГИЯ: ТЕОРИЯ И ПРАКТИКА

ISSN: 3030-3001

SJIF 2023: 3.019, 2024: 5.444 ResearchBib IF: 13.14/ 2024

Том 3, Выпуск 1

STATE OF STUDY OF THE PROCESSES OF OBTAINING MONOCALCIUM AND MONOPOTASIIUM PHOSPHATE.

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Abstract. Currently, due to the rapidly growing population of the planet, with the reduction of arable and irrigated land, providing the population with food and drinking water is becoming increasingly urgent. Despite the enormous achievements in agriculture and livestock farming, this problem remains unresolved at the beginning of the 21st century. One of the most effective ways to solve this problem is to further increase the yield of agricultural crops and the productivity of livestock, poultry, and fish farming. In the world, in almost all developed countries, there is a tendency to increase the production and range of chlorine-free potassium and NPK fertilizers, and feed calcium phosphates. To provide agriculture with chlorine-free potassium and, on their basis, chlorine-free, completely water-soluble NPK fertilizers for drip irrigation and foliar feeding of plants, livestock in feed-grade calcium phosphates, it is necessary to justify a number of decisions: the development of effective methods for producing chlorine-free potassium fertilizers and feed-grade monocalcium phosphate; study of the content of fluorine and other impurities during the concentration of defluorinated and desulfated extraction phosphoric acid (EPA) from phosphorites of the Central Kyzylkum (CK); establishing optimal technological parameters for the conversion process of monosodium phosphate and potassium chloride.

Keywords: Central Kyzylkum, monocalcium, monopotassium phosphate, NPK fertilizers.

Application, demand, scale of production of calcium and potassium phosphates.

In the life activity of all living organisms and flora, along with carbon, hydrogen and oxygen, an important role belongs to phosphorus and its compounds [1]. Phosphorus occupies a special place among chemical elements. It is part of many minerals, primarily calcium phosphates. In living nature it forms organophosphorus compounds, which serve as carriers of high-energy reactions that ensure the vital activity of living organisms [2]. The role of phosphorus in living nature is unique. You

can find a replacement for coal, oil or iron, but there is no replacement for phosphorus [3].

Phosphorus is the most important component of feed rations for livestock, poultry, and fish [4]. It is part of nucleic acids, phosphates, phosphoproteins and other compounds, and is a necessary component for building bone tissue. A lack of phosphorus in the diets of farm animals reduces meat and dairy productivity, leads to the occurrence of bone diseases and impaired reproductive function. The global range of basic mineral supplements includes more than 10 items. Phosphorus-containing mineral fertilizers based on calcium, sodium, ammonium phosphates and other chemical components are widely used in animal husbandry, poultry farming, and fish farming [5].

The most valuable are calcium phosphates [6]. In feeds where there is a significant amount of calcium and insufficient phosphorus, sodium phosphorus additives are used. To compensate for the lack of protein in the diets of cattle and sheep, non-protein nitrogen-containing compounds – ammonium phosphates – are used.

Phosphorus and calcium participate in the body's metabolic processes and determine the high efficiency of feed mineral additives. The quality of feed calcium phosphates is assessed by the content of digestible forms of nutrients in them with a minimum concentration of harmful impurities, such as fluorine, lead, arsenic, cadmium, and mercury. The biological digestibility of phosphorus from feed calcium phosphates-monocalcium phosphate, dicalcium phosphate, tricalcium phosphate is at least 80% [7,8].

Currently, global consumption of feed calcium phosphates is more than six million tons per year and continues to increase annually. Calcium phosphates are produced in powder and granular form, and the share of granular products is constantly increasing and has already exceeded 70% [9]. This is due to their use in the production of premixes and mixed feed.

Uzbekistan's need for feed additives (feed ammonium, calcium, sodium phosphates, etc.) exceeds more than 100 thousand tons per year and also continues to increase [10].

The increase in demand for feed phosphates from mineral raw materials is associated with the widespread absolute refusal to use cheaper feed additives obtained from bone meal, which is associated with the danger of infection with the "mad cow disease" virus. The danger of infection by this virus has led to an unpredictable increase in demand for feed phosphates from mineral raw materials [11].

The average annual growth in consumption of feed phosphates in the world is 6%, which is approximately 2.5 times higher than for phosphorus-containing fertilizers [12]. The American continent consumes the most feed phosphates - 50%, Asia - 18%, Western Europe accounts for 21% and only 9% for Central and Eastern Europe. The largest average annual growth in feed phosphate consumption is observed in Latin

America (Brazil +14%) and Asia (China +10%). The consumption of feed phosphates is also expected to increase in Central Asia. It should be noted here that the largest producer of feed phosphates in Asia, Lomon (China), which has a comprehensive product range and produces up to 600 thousand tons per year of feed calcium phosphates, is completely focused on the domestic market. Brazilian producers of feed phosphates almost completely sell their products on the domestic market [13].

Phosphoric acid and its salts are widely used in the production of mineral fertilizers, in the food industry, medicine, pharmaceuticals, electronics, chemical, textile, glass, aviation, and engineering industries. The main amount of phosphate raw materials is used for the production of mineral fertilizers (about 80%), 12% - for the production of detergents, 5% - for the production of feed phosphates, 3% - for the production of special-purpose products [14]

Pure phosphorus-containing calcium salts are used in the food industry, in the baking powder system, in medicine, and in the perfume industry [15]. They are used in the production of bone tissue in dentistry and as a filler in the production of toothpastes.

The most important components of feed rations for livestock, poultry, and fish are calcium and phosphorus [16]. In this regard, calcium phosphates are a universal mineral supplement for farm animals of all types with a lack of phosphorus and calcium in their diets.

Modern industrial methods of producing livestock products are characterized by the widespread use of mineral feed additives, which help increase productivity, safety of livestock, and reduce feed costs [17].

Monocalcium phosphate (molecular weight 252) is a monosubstituted calcium salt of orthophosphoric acid. Pure monocalcium phosphate in anhydrous form $\text{Ca}(\text{H}_2\text{PO}_4)_2$ contains 60.65% P_2O_5 and 23.96% CaO , and monohydrate $\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O}$ – 56.31% P_2O_5 and 22.25% CaO . According to GOST 23999-80, feed monocalcium phosphate of the 1st and 2nd grades must contain, respectively, at least 55 and 50% P_2O_5 soluble in a 0.4% solution of hydrochloric acid. The product of both grades must contain no more than 18% calcium, 0.2% fluorine, 0.006% arsenic, 0.002% lead, 4.0% water; The pH of a 0.01 M aqueous solution must be at least 3 [18]. Mono- and dicalcium phosphates dissolve incongruently in water. Their dissolution in water is accompanied by the reaction:



In the presence of excess water, monocalcium phosphate dissociates to form dicalcium phosphate and free phosphoric acid.

Application, demand, production scale of potassium phosphate

In recent years, the problem of providing the world's population with food and drinking water has become increasingly acute. Despite the enormous achievements in

agriculture and livestock raising, the supply of food to the planet by the beginning of the 21st century remains unsatisfactory due to the rapid rate of population growth, the reduction of arable and irrigated land, and the sharp rise in prices of energy and fuel sources.

According to the UN, by 2050 the world's population will exceed 9 billion people, while currently more than 1 billion people do not receive the minimum amount of food and half of them are chronically malnourished, more than 20% do not have sources of fresh drinking water [26,27]. This is due to a sharp reduction in land for grain crops. Thus, since the mid-nineties, the area in the world has halved from 0.24 to 0.12 hectares per capita [19]. By 2050, according to the UN, it will decrease to 0.08 hectares per person. In this regard, an acute problem arises in supplying humanity with food. This problem also concerns Uzbekistan.

The drying up of the Aral Sea and the lack of fresh water caused serious damage to the agriculture of the Republic. Due to a shortage of water resources, irrigated arable land per capita decreased from 0.22 hectares to 0.13 hectares [20]. The President and the government of the country pay great attention to the restoration of arable and irrigated lands, the development of rain-fed areas, the use of water-saving, advanced agrotechnical and agrochemical technologies, the intensification of agricultural production by increasing productivity through the creation of new, high-yielding varieties, the use of drip irrigation, and foliar feeding of plants [21].

The introduction of modern, advanced technologies for growing crops in greenhouses and the use of drip irrigation impose more stringent requirements on the range and quality of mineral fertilizers: absence of chlorine, complete solubility in water, content of basic macronutrients - NPK in various proportions [22]. One of the types of complex RA fertilizers and the main component of water-soluble, chlorine-free NPK fertilizers is potassium dihydrogen phosphate, which is suitable for use on any soil and in any ratio with nitrogen and phosphorus fertilizers [23].

However, potassium dihydrogen phosphate as a mineral fertilizer is currently practically not produced for wide consumption, due to the limited raw material base and the lack of developed, acceptable technologies for its production. Therefore, it belongs to expensive, scarce products [24].

Potassium is one of the three main plant nutrients and is a macronutrient. Its deficiency in the soil leads to a significant decrease in yield and various diseases [25].

For many years, 75 kg of potassium chloride in terms of K_2O were added to the crops of cotton, the main crop of the Republic, on the recommendation of agrochemists. This norm was underestimated by 1.5-2 times. As a result, due to a lack of potassium, its removal with the harvest, and annual leaching of saline soils, arable lands were depleted of potassium [26]. This led to a sharp decline in raw cotton yield, fiber quality, and the efficiency of nitrogen and phosphorus fertilizers. With proper use of NPK fertilizers, the yield on non-saline soils increases by 1.9-3.0 c/ha, on saline soils by 2.5-

7.0 c/ha. This confirms the need to increase the application rates of potash fertilizers, which has led to an increase in demand for potash fertilizers, especially chlorine-free ones.

The main potassium fertilizer in the world is potassium chloride. It is obtained by flotation and halurgical methods in powder and granular form [27]. The presence of a high chlorine content in its composition limits its use on crops, where chlorine has a strong negative effect.

Chlorine-free potassium fertilizers are especially valuable when growing potatoes, beets, sunflowers, grapes and other crops. They have a much more effective effect on the yield and quality of agricultural products if they are used in combination with nitrogen, phosphorus and organic fertilizers [28].

The demand for chlorine-free potassium fertilizers has increased sharply with the development of greenhouses, hydroponics, drip irrigation and foliar feeding of plants when growing vegetables, fruits, grapes and other crops, as completely water-soluble, chlorine-free potassium fertilizers. Among chlorine-free fertilizers, phosphates and potassium sulfate are in great demand, as the main components of complex NPK fertilizers [29].

Monopotassium phosphate KH_2PO_4 or potassium dihydrogen phosphate are colorless, odorless crystals, molecular weight 136.06 g/mol, density 1380 g/cm³, melting point 230-250°C [30]. Potassium dihydrogen phosphate is highly soluble in water without decomposition. At 25°C, the solubility of pure salt, according to various authors, is 19.92-20.09% [31].

Monopotassium phosphate is a completely water-soluble, chlorine-free, ballast-free, complex fertilizer consisting of two main plant nutrients and containing 52.16% P_2O_5 and 34.60% K_2O . It is used for all types of agricultural crops and on various types of soil, as a phosphorus-potassium mineral fertilizer [32]. Monopotassium phosphate is also used in NPK fertilizers as the main component. Good solubility allows nutrients to be delivered directly to the root system in the form of aqueous solutions. This allows you to save not only water resources, but also increases the utilization rate of nutrients [33].

Chlorine-free potassium fertilizers are especially valuable when growing potatoes, beets, sunflowers, grapes and other crops. They have a much more effective effect on the yield and quality of agricultural products if they are used in combination with nitrogen, phosphorus and organic fertilizers [34].

The republic's demand for chlorine-free potassium fertilizers exceeds 20 thousand tons per year. In addition to the needs of the domestic market, there is a great demand for chlorine-free potassium phosphates in the foreign market.

Potassium compounds are widely used in other industries, such as ferrous and non-ferrous metallurgy, textiles, glass, pharmaceuticals, and pulp and paper. In the food industry as an antioxidant and bactericidal agent, in baking risers, in milk powder and

cream - as a stabilizer, together with other additives - in cheese production. Added to soft drinks for athletes, dairy products (ice cream, condensed milk), desserts, sauces, soups, syrups, and dairy products. They are one of the components of detergents (shampoos, soaps), medicines, and a valuable phosphorus-potassium fertilizer. Despite this, only 5-6% of manufactured products are used in other industries [35].

From 2012 to 2015, the production of potash fertilizers increased from 29.1 million tons to 31.5 million tons of K_2O , or by 4.2%. By 2019, their production increased, compared to 2014, by 9 million tons of K_2O or 21%. The production of potash fertilizers is increasing not only due to an increase in the capacity of enterprises, but also due to the commissioning of new production facilities [36,37].

The need to increase the production of potassium phosphate salts is determined not only by the increase in demand for mineral fertilizers from traditional consumers, but also by the expansion of their areas of application [38].

Existing methods for producing calcium phosphate

The production of feed calcium phosphates can be divided into the following groups:

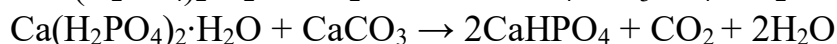
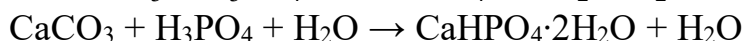
- hydrothermal calcination of natural phosphates and thermal defluorination of double superphosphate.
- interaction of fine limestone, chalk or dicalcium phosphate dihydrate with thermal phosphoric acid or purified extraction phosphoric acid [39].
- conversion of ammonium phosphates with calcium nitrate.

Feed phosphate technologies are divided according to the methods of decomposition of phosphate raw materials (nitrogen, sulfuric, hydrochloric acid), process recycle (return, non-return), thermal, and the component used for precipitation (lime milk, chalk or limestone suspension, quicklime) [40].

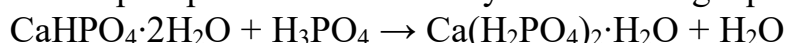
Double superphosphate is a concentrated phosphorus fertilizer, the main component of which is monocalcium phosphate. Double superphosphate is obtained by treating natural phosphates with concentrated phosphoric acid [41].

The presence of a large amount of fluorine in double superphosphate (up to 3% or more) does not allow its use as a feed additive. To obtain a feed product, double superphosphate is subjected to defluorination by heat treatment at a temperature of 150-180°C. This is the cheapest way to obtain feed monocalcium phosphate. However, it is almost impossible to obtain purer calcium phosphate salts from it.

Phosphorus salts of higher qualification are obtained by neutralizing deeply purified thermal phosphoric acid to the corresponding grades "pure", "analytical grade", "reagent grade" with appropriate carbonates or metal hydroxides. The chemistry of producing monocalcium phosphate, dicalcium phosphate and their mixtures can be represented by the following reaction equations:



Another way to obtain feed grade and purer calcium phosphates involves reacting dicalcium phosphate dihydrate with purified phosphoric acid. The process of obtaining monocalcium phosphate is described by the following equation:



Depending on the level of phosphoric acid, you can get a mixture of monocalcium phosphate and dicalcium phosphate with different ratios.

In recent years, research has been carried out on the production of feed and purer calcium phosphates from EPA, the essence of which is to purify the acid from fluorine, sulfates, iron, aluminum and other interfering impurities by introducing alkali metals, partial neutralization with ammonia in the presence of calcium salts, separating precipitated precipitate of compounds and release of dicalcium phosphate [42].

Until recently, practically the only proven and technologically implemented method for processing phosphate raw materials into feed phosphates was the hydrothermal process of high-temperature calcination of natural phosphates [43]. Defluorination of phosphate raw materials is carried out at a temperature of 1400-1450 °C in rotary kilns with small additions of SiO₂ or EPA. The decisive role in this process is played by water vapor, which causes the transition of fluorapatite to hydroxyapatite with the release of fluoride compounds into the gas phase. Hydroxylapatite decomposes under high temperature into tricalcium phosphate and tetracalcium phosphate. This method is associated with high energy costs and allows one to obtain a low-quality product containing 36-38% P₂O₅ [44].

Other disadvantages of this process are difficult sanitary conditions, complex equipment, and low equipment utilization. More concentrated feed calcium phosphates (monocalcium phosphate and dicalcium phosphate) are obtained only from high-quality phosphate raw materials - apatite concentrate, or using expensive and scarce electrothermal phosphoric acid [45].

The method for producing feed calcium phosphate includes the decomposition of phosphate raw materials with an excess of phosphoric acid at elevated temperatures, defluorination of the resulting mass and neutralization with an easily decomposed calcium-containing reagent [46]. Decomposition is carried out at 80-120°C, and before defluorination and neutralization, the resulting mass is filtered and the filtrate is returned to the decomposition stage to achieve a total H₃PO₄ rate of 250-400% of the stoichiometry.

To obtain primary and secondary acid calcium orthophosphates, 10-40% neutralization of the first H⁺ ion of phosphoric acid (which contains 40-55% P₂O₅ and

$\leq 0.3\%$ F) is carried out with phosphorites at 90-130°C. The resulting sludge is treated with calcium carbonate, oxide or hydroxide at 50-100°C to obtain an aqueous solution that has a pH of 3-6 and the resulting product is dried at 80-150°C [47].

To obtain primary and secondary acid calcium orthophosphates, 10-40% neutralization of the first H^+ ion of phosphoric acid (which contains 40-55% P_2O_5 and $\leq 0.3\%$ F) is carried out with phosphorites at 90-130°C. The resulting sludge is treated with calcium carbonate, oxide or hydroxide at 50-100°C to obtain an aqueous solution that has a pH of 3-6 and the resulting product is dried at 80-150°C [48].

To increase the productivity of livestock, poultry and fish farming, calcium phosphates are used as mineral supplements, either independently, added to feed, or as part of biofeeds. Calcium phosphates - monocalcium phosphate, dicalcium phosphate, tricalcium phosphate contain such important elements for life as phosphorus and calcium [49].

The advantages of calcium phosphates, compared to bone meal and plant-based feeds, are determined by chemical, physical and biological properties. The quality criteria are:

- high biological digestibility of the product;
- stable nutrient content;
- extremely low levels of heavy metals and fluorine;
- grading.

Until the end of the twentieth century, in the CIS countries, defluorinated phosphates, bone meal, feed precipitate, as well as mono- and disodium phosphates were used in large quantities as mineral supplements[50,51].

There is a known method for producing feed calcium phosphates, including the decomposition of tricalcium phosphate EPA and drying the product at a temperature of 110-150°C. EPA is pre-treated with tricalcium phosphate or another salting out component, the precipitate is separated and the acid is taken in the amount necessary to achieve the ratio $P_2O_5EPA:P_2O_5CP = 1:(0.5-2.0)$, the process is carried out at a moisture content of 30-50%, temperature 60- 100°C and granulated at 60-120°C [52].

In recent years, methods for producing monocalcium phosphate using liquid-phase circulation methods have become widespread. The essence of the method is the decomposition of phosphate raw materials with a 3-5 fold excess of concentrated 40-65% P_2O_5 phosphoric acid at temperatures of 60-90°C, crystallization of monocalcium phosphate upon cooling and separation from the mother liquor. The advantage of the cyclic method is the ability to obtain monocalcium phosphate from almost any type of phosphate raw material[53].

In works using solubility diagrams in the $CaO-P_2O_5-H_2O$ and $CaO-P_2O_5-HCl-H_2O$ systems, graphical calculations of the process of obtaining monocalcium phosphate monohydrate were carried out under mother liquor recycle conditions for a temperature of 40°C from phosphorites of Karatau and Central Kyzylkum [54].

The results obtained indicate the feasibility of processing low-grade phosphorites with a 4.5-5 fold excess of phosphoric acid and the possibility of obtaining high-quality monocalcium phosphate.

To establish the optimal conditions for the decomposition of Karatau phosphorites with thermal phosphoric acid containing 40% P_2O_5 under conditions of non-thickening pulps using a recirculation scheme, graphic calculations were carried out and it was shown that at an acid rate of 450-500% of stoichiometry, a contact time of 50-60 minutes, at a temperature of 90- 95°C, followed by filtration and separation of the insoluble residue, cooling the filtrate to 40°C for 90 minutes, you can obtain monocalcium phosphate, in which the decomposition coefficient is 99.0-99.5[55].

In order to involve low-quality substandard phosphate raw materials from the Chilisay deposit into highly concentrated phosphorus fertilizers by decomposing them with a large excess of phosphoric acid, the rates and mechanism of the process were studied [56]. The kinetic parameters of the decomposition process were determined and it was found that the decomposition of Chilisay phosphorites proceeds quite quickly (25-30 min), since the phosphate component of phosphorites is provided in the form of the kurskite mineral and that after the fourth cycle, phosphoric acid can be regenerated and returned to the decomposition stage[57].

EPA standards have been established for RPM and MOFC, which are 400-500% for EPA containing 41.20% P_2O_5 and 400-600% for EPA containing 44.98% P_2O_5 , decomposition time 60 minutes, temperature not higher than 100% [58].

One of the real ways to process low-grade phosphorites with recycle of the mother liquor when producing monocalcium phosphate is the decomposition process using hydrochloric acid. The essence of these processes is that mother solutions, after separation of monocalcium phosphate, and containing calcium chloride are mixed with phosphoric acid and returned to the decomposition stage in the form of a circulating solution.

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ISSN: 3030-3001

SJIF 2023: 3.019, 2024: 5.444 ResearchBib IF: 13.14/ 2024

Том 3, Выпуск 1

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