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Obtaining nitrophos fertilizers with copper, zinc, molybdenum

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Abstract. The purpose of the research is to obtain micronutrient fertilizers by applying micronutrient salts and their complex forms to phosphorus fertilizers obtained in incomplete standards of nitric acid or their salt mixtures. Central Kyzylkum phosphorites (low-grade P_2O_5-12.38%, CaO-43.68%, CO_2-13.48%, non-enriched P_2O_5-16.38%, CaO-45.93%, CO_2-18, 15%, SO_3-1.86%, thermoconcentrate P_2O_5-27.40%; CaO-54.68%; CO_2-4.52%) were decomposed with nitric acid at different rates and phosphorus fertilizers were obtained. Copper and zinc sulfate and ammonium molybdate (Cu, Zn, Mo) salts and their complex forms were applied to the obtained fertilizers and their chemical composition was studied. Central Kyzylkum phosphorite flour was processed in different concentrations of nitric acid. The process of granulation of recycled phosphorus fertilizers in different standards was carried out with the participation of micronutrient salts and their complex solutions, and it was possible to obtain micronutrient phosphorus fertilizers. Along with nitrogen, phosphorus, potassium fertilizers, the demand for micronutrient fertilizers is also increasing in our republic. Currently, microfertilizers are used in practice in very small quantities. In the production of mineral fertilizers, the use of various wastes and products of non-ferrous metallurgy containing microelements, sour effluents, spent catalysts and other types of secondary products is one of the solutions for the production of microelement fertilizers.

1 Introduction

80% of the elements in D.I. Mendeleev's periodic system are trace elements, which do not exceed 0.01% in the earth's crust, plant and animal organisms (on a dry matter basis). Some heavy metals (such as mercury, lead) are harmful to plants and living organisms, while other elements have a positive effect on their development. Mineral fertilizers whose main nutrient is trace elements are called microfertilizers. In the following literature [1; 239-241-b, 2; p. 8-9] for adequate development of organisms, living organisms absorb micronutrients from food, and plants from soil. Microorganisms enter the soil through soil-forming rocks, natural waters, and cosmic dust. In addition, microfertilizers are given artificially.

Research scientists E.V. Vobko, M.A. Belousov, O.F. Tueva, M.Ya. Shkolnik, U.E. Brenchley and K. Warringtons studied the properties of trace elements important for plants in 1927-1934. Brenchley and Warrington found that micronutrients have specific effects on

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plants. M. Ya. Shkolnik and E.V. Vobko noted that trace elements not only have a catalytic effect on biochemical processes in the body, but also have an effect on the physico-chemical properties of plasma, surface tension, diffusion, osmosis, the increase of colloids, absorption, and the formation of a redox potential difference in metabolic processes.

14 vitally important trace elements have been identified. They include B, Mo, Cu, Zn, Mn, Co, etc. Microelements, together with enzymes, vitamins, hormones, pigments, etc. in the body, affect the vital processes of organisms. They participate in biochemical changes and affect the physiological functions that occur in the plant organism through enzyme systems. Increases the use of light in the process of photosynthesis, accelerates protein synthesis. Some microelements activate the useful properties of plants, that is, they strengthen their properties, such as drought and cold resistance, acceleration of seed germination and development, and disease resistance. Their lack leads to disruption of metabolic processes, diseases of plants and living organisms [1].

Microfertilizers are fertilizers that are applied in small amounts (grams and kilograms per hectare). Boric acid, copper (II) sulfate, ammonium molybdate and other technical salts are used in the composition. Coal ash, manganese sludge (slurry), precipitated magnesium borate and other micronutrient wastes are insoluble in water. They are processed into a water-soluble form or used directly as fertilizer. Water-soluble and insoluble microfertilizers are used in agriculture. Complex fertilizers are fertilizers containing at least two nutrients. Secondary complex fertilizers (for example, nitrogen-phosphorus, nitrogen-potassium, phosphorus-potassium) and tertiary complex fertilizers (for example, nitrogen-phosphorus-potassium) are divided into types. Tertiary fertilizers are called complete fertilizers. Complex fertilizers may also contain micronutrients, pesticides, and plant growth additives [2].

In the works of A.I. Fateev, it is mentioned that a certain amount of microelements leave the soil as a result of assimilation by plants or for other reasons, and they are not replenished at the moment, which is due to the fact that organic fertilizers, which are the main source of replenishment of absorbable forms of microelements, are not introduced into the soil [3, 4] It should be noted that it is the presence and absorption of microelements by plants that synthesize all the enzymes that allow them to efficiently use soil energy, water, fertilizers and soil nutrients.

According to I.B. Karmazin, B.A. Yagodin, S.P. Polyanchikov, only nitrogen, phosphorus and potassium are not enough for the normal functioning of the plant organism. Micronutrients play an important role in plant nutrition. They participate in the synthesis of proteins, carbohydrates, vitamins. Under their influence, photosynthesis improves, resistance to drought increases, immunity against disease-causing agents increases, as a result, productivity increases and their quality improves [5].

G. Gospodarenko and others' studies show that plants without microelements suffer from various diseases and productivity decreases by themselves, and even if plants are supplied with nitrogen, phosphorus, and potassium fertilizers as needed, due to the lack of microelements, plants do not fully absorb the nitrogen, phosphorus, and potassium compounds in the fertilizer or get diseases [6] mentioned.

Many scientific researches are devoted to the addition of micronutrient compounds to ordinary fertilizers of microfertilizers.

In the works of T.S. Bauatdinov, complex fertilizers containing macro and microelements were obtained by activating phosphate raw materials with Karakalpak glauconite decomposed products in mineral acids [7].

In the territory of Karakalpakstan, there is a large area of sands containing glauconite, which makes it possible to use them widely as raw materials in the production of local fertilizers. Glauconites contain a large amount of microelements, therefore, one of the urgent problems is to create micronutrient fertilizers with sorption, ion exchange and complexing properties necessary for the growth of plants in the soil [8].

In the studies of I.A. Gaysin, R.N. Sagitova, R.R. Habibulins, copper, molybdenum, as well as various proportions of copper and molybdenum microelements were obtained in complex forms with the presence of monoethanolamine and their positive effect on increasing productivity and plant development was studied by applying them to grain crops [9]. About 15 author's certificates and patents were obtained for the invention on the basis of these preparations. Currently, in the Republic of Tatarstan, microelements in complex form are used in large-scale fields. These microelements in a complex form allow to reduce the application of pesticides to plants by 3-4 times, together with the rapid development of plants, increase in productivity.

In the works of Z. Toraev and others, the conditions of adding CuO and $CuSO_4$ to thermal phosphoric acid (32% P_2O_5) as part of the main components used in the production of micronutrient fertilizers were studied. When copper oxides and copper sulfates are added to thermal phosphoric acid, copper dihydrophosphate is formed as a result of the reaction: CuO $+2H_3PO_4 = Cu(H_2PO_4)_2 + H_2O$ $CuSO_4 + 2H_3PO_4 = Cu(H_2PO_4)_2 + H_2SO_4$ The resulting solution is adjusted to a value of rN of 1.6 with gaseous ammonia. when it is neutralized, the phase composition of the solution remains unchanged. At values of 1.6-2 pH, formation of a weak airy precipitate occurs. Chemical analysis of the resulting precipitate revealed the absence of nitrogen and the presence of 35.5-36.2% Cu^2+ and 54.0-54.5% HPO_4^2 -, this result is theoretically obtained composition (Cu^2+ 35.8%, HPO_4^{2-} 54.1%) fully corresponds to [10].

Shamshitdinov I.T. of [11] in the research works, methods of purification and activation of extractable phosphoric acid (EFA) obtained from magnesium phosphorites from additives, as well as technologies for processing high-magnesium phosphorites into EFA and processing secondary phosphates into concentrated phosphorous and micronutrient fertilizers by decomposing secondary phosphates in activated EFA were developed.

The process of obtaining double superphosphate micronutrient fertilizers based on low-grade Central Kyzylkum phosphorites and micronutrients of copper and zinc has been studied. The experimental results of dissolving the microelements trapped in the electrofilters from the liquefaction of metals in an oxygen-flame furnace and the dust formed from the spirit and copper dry grinding with the help of phosphoric acid solution have been studied [12].

In the works M.N. Nabiev, I.I. Usmanov and S. Tukhtaev enrichment of superphosphate with trace elements [13] and in order to transfer them to a state that is easily absorbed by plants, and in order to improve the physico-chemical properties of the fertilizer and the mechanical properties of the finished product, granulation of the fertilizer was carried out with the participation of ammonia complexes of trace elements. According to the proposed method, the introduction of sulfate salt into trace elements at the initial stage of superphosphate production accelerates the decomposition of phosphorite and shortens the ripening process [14] is defined.

In the works of E.K. Badalova, A.M. Amirova, M.T. Saibova, $MnSO_4$ was added to thermal phosphoric acid and neutralized with NH_3 to obtain amphos containing Mn. In this case, it was observed that trace element Mn became insoluble in water [15].

In B.A. Yagodin's research, the main focus is on the study of environmental problems in the use of fertilizers, in particular, the use of micronutrient industrial and household waste in plant science [16].

It is known from experiments that the efficiency of mineral salts of trace elements is lower than that of their chelated compounds. It has been found out from many experiments that micronutrient complexes (chelates) can increase the productivity of agricultural crops even when used 2-10 times less than the amount of micronutrient salts.

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Currently, OEDFK (hydroxyethylenediphosphonic acid), NTF (nitriletrimethylphosphonic acid), DTPA (diethylenetriaminepentaacetic acid), EDDYAK (ethylenediaminediacetic acid), EDTA (ethylenediaminetetraacetic acid), DBTA (dihydroxybutylenediaminetetraacetic acid), DTPA (diethylenetriaminepentaacetic acid) and others are used as chelating agents. . Chelates of microelements are easily absorbed by plants compared to inorganic salts (sulfates, carbonates, etc.), chelates based on lignins are 4 times easier, and those based on citrates are 6-8 times easier. Plants absorb them much better and efficiently, because the simple salts of trace elements react with each other in the soil and form hard-to-be-absorbed compounds. Chelates do not enter into such a reaction, and also do not combine with the soil. As a result, 30-40% of simple micronutrients are absorbed by plants, while chelated micronutrients are absorbed by 90% and more [17].

B.A. Yagodin, Yu.P. Zhukov, V.I. Kobzarenko and others [18] the results of practical and fundamental research on complex micronutrient nutrition of agricultural products for many years are presented.

In the works of E.A. Krylov [19] microbiometalls referring to d-elements, they have 2 to 10 d-electrons in their outer electron shell. They are characterized by their oxidation state from +1 to +3. Octahedral shelling is typical for them, due to which stability forces (energy) are formed in the formation of complexes under the influence of the field of ligands, depending on the structure of the outer electronic layer. Usually, microbiometal ions are present in plants in the form of complexes with proteins.

In the scientific works of I.A. Gaysin and others [20] to stimulate plant growth, a solution obtained by adding boric acid and copper sulfate salt to an aqueous solution of monoethanolamine has been shown to be more effective than using the ingredients alone when applied to plants.

The use of secondary products of non-ferrous metallurgy as a base of raw materials is one of the most urgent issues of today. However, the physico-chemical basis of microfertilizer production and microfertilizer production technologies are not sufficiently covered in the cited literature. Also, methods of converting micronutrient salts and secondary raw materials into a complex form and attaching them to macrofertilizers have not been widely explained.

2 Materials and methods

The purpose of the research is to implement the technologies of obtaining new types of phosphoric fertilizers based on the incomplete standards of mineral fertilizers and acids widely used in agriculture or their salt mixtures, using microelement salts and secondary raw materials containing microelements of hydrometallurgy.

Despite the above-mentioned positive aspects of microfertilizers, microfertilizers are practically not used in our Republic. One of the main reasons for this is explained by the lack of study of cheap and usable local micronutrient raw materials, the lack of scientific and technological microfertilizer production technology and development. In order to theoretically justify the production of micro-fertilizers and create their production technology, it is necessary to conduct in-depth physico-chemical studies to study the interaction of micro-elements with nitrogen-phosphorus fertilizers, the technology of production of fertilizers and the effects that occur during the process of adding micro-elements to fertilizers as additives.

3 Results and discussions

Micronutrients were applied to nitrophos fertilizers in order to develop the scientific basis of obtaining nitrophos fertilizers containing copper, zinc, and molybdenum salts at the same time. Copper, zinc, molybdenum salts were added to nitrophos fertilizers obtained at different levels of nitric acid in the proportions 1:0.007, 1:0.01, 1:0.02, and their chemical composition (Table 1) was analyzed based on standard methods.

Analyzing the chemical composition of microelement fertilizers, the nutrient elements in nitrophos fertilizers are almost the same as those of nitrophos, zinc nitrophos and molybdenum nitrophos fertilizers. It differs from other nitrophos fertilizers by the presence of 3 different micronutrient salts in one fertilizer. Nitrophos fertilizers, rich in microelements, compared to other nitrophos fertilizers, reduce excess costs when applied to plants and fully satisfy the plant's need for microelements.

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For example, in a fertilizer made on the basis of unenriched phosphorite flour, with an acid level of 40%, with a composition of P_2O5:Cu, P_2O_5:Zn 1:0.007, the total 0.06% of copper absorbed by plants is 38.75%, and the water-soluble form is 16.21%, the total 38.88% of 0.06% zinc is in plant-absorbable form, and 16.38% is in water-soluble form. It was also found that the total 0.02% of molybdenum was 37.38% plant-absorbable, and 14.22% water-soluble form. As the ratio of trace elements to phosphorus increases, their plant-absorbable and water-soluble form increases. 40.73% of the plant-absorbable form of copper in the complex fertilizer (P_2O_5:ME 1:0.02), obtained at the rate of 40% of the acid, and 19.28% of the water-soluble form, 40.77% of the trace element zinc is in the plant-absorbable form, 19.47 And % i is soluble in water. The plant-absorbable form of molybdenum is 41.43%, and the water-soluble form is 18.61%. This pattern is also observed in fertilizers with 60 and 80% acidity.

Since microelements are in ionic state in complex nitrophos fertilizers with copper, zinc, molybdenum, they combine with various salts contained in fertilizers or in the soil, and it is observed that they change to a hard-to-dissolve form. For this reason, the complex combinations of microelements listed above were used to increase the plant-absorbable form of microelements.

In complex nitrophos fertilizers containing non-enriched phosphorite flour at the rate of 40% of acid (P_2O_5:Cu, P_2O_5:Zn 1:0.01), the plant-absorbable state of trace elements is 86.56% and 86.40%, respectively, and the water-soluble form is 77.46%. and is 77.60%. It can be seen that in the fertilizer with an acid content of 80%, the plant-absorbable form of copper is 81.33%, and the water-soluble form is 70.31%, and this indicator is 81.75% and 70.47%, respectively, in the zinc trace element. This pattern was also observed in fertilizers with ratios of copper and zinc to phosphorus of 1:0.007 and 1:0.02. In the fertilizer with P_2O_5:Cu, P_2O_5:Zn 1:0.02, the plant-absorbable form of copper microelement is 87.39%, and the water-soluble form is 79.57%, the plant-absorbable form of zinc microelement is 87.53%, and the water-soluble form is 79.48 is %.

Table 1. Copper, zinc, molybdenum nitrophos fertilizer based on unenriched phosphorite flour, %.

HNO_3 %	N	P_2O_5		Cu			ME consumption degree %		Zn			ME consumption degree %		Mo			ME consumption degree %		H_2O	pH
		Total	Digestible	Total	Digestible	Soluble in water	Digestible	Soluble in water	Total	Digestible	Soluble in water	Digestible	Soluble in water	Total	Digestible	Soluble in water	Digestible	Soluble in water		
Nitrophos fertilizers containing simple trace elements																				
40	6.58	14.05	9.74	0.06	0.023	0.010	38.75	16.21	0.06	0.023	0.010	38.88	16.38	0.02	0.007	0.002	37.38	14.22	2.04	6.44
40	6.53	14.00	9.68	0.12	0.048	0.022	39.85	18.35	0.12	0.048	0.022	39.91	18.42	0.03	0.012	0.005	39.52	15.35	1.97	6.39
40	6.48	13.95	9.62	0.18	0.073	0.035	40.73	19.28	0.18	0.073	0.035	40.77	19.47	0.05	0.021	0.009	41.43	18.61	1.88	6.28
60	8.22	11.74	8.86	0.04	0.015	0.006	36.54	15.72	0.04	0.015	0.006	36.67	15.63	0.02	0.006	0.002	31.49	11.71	2.82	5.92
60	8.17	11.69	8.80	0.08	0.030	0.013	37.72	16.64	0.08	0.030	0.012	37.84	16.54	0.03	0.010	0.004	33.67	13.62	2.73	5.83
60	8.12	11.64	8.74	0.12	0.046	0.022	38.57	17.98	0.12	0.046	0.021	38.73	17.89	0.05	0.018	0.007	35.39	14.38	2.64	5.75
80	10.14	10.87	9.19	0.03	0.008	0.004	28.15	13.39	0.03	0.009	0.004	28.61	13.67	0.01	0.002	0.001	24.71	9.91	2.33	5.25
80	10.09	10.82	9.13	0.07	0.021	0.010	30.14	14.41	0.07	0.021	0.010	30.49	14.29	0.01	0.003	0.001	27.34	10.43	2.26	5.14
80	10.04	10.77	9.07	0.10	0.032	0.016	31.54	15.57	0.10	0.032	0.015	31.83	15.37	0.02	0.006	0.002	29.83	11.71	2.18	5.06
Nitrophos fertilizers containing the citrate form of trace elements																				
40	6.00	12.81	8.95	0.05	0.043	0.038	85.55	76.45	0.05	0.043	0.038	85.59	76.52	0.02	0.007	0.003	35.39	14.16	7.79	6.13
40	5.95	12.76	8.89	0.11	0.095	0.085	86.52	77.61	0.11	0.095	0.085	86.58	77.54	0.03	0.015	0.006	37.61	14.65	1.72	6.05

40	5.90	12.71	8.83	0.16	0.140	0.127	87.40	79.29	0.16	0.140	0.127	87.49	79.61	0.05	0.031	0.013	38.80	15.67	1.63	5.93
60	8.16	11.67	8.86	0.04	0.033	0.029	83.48	71.38	0.04	0.033	0.028	83.62	71.48	0.02	0.006	0.002	30.16	10.63	2.49	5.50
60	8.11	11.62	8.80	0.08	0.068	0.058	84.43	72.47	0.08	0.068	0.059	84.58	72.38	0.03	0.013	0.005	32.21	13.11	2.42	5.41
60	8.06	11.57	8.74	0.12	0.103	0.088	85.57	73.36	0.12	0.103	0.088	85.47	73.57	0.05	0.028	0.011	34.58	14.26	2.33	5.34
80	10.08	10.80	9.19	0.03	0.024	0.021	79.51	69.19	0.03	0.024	0.021	79.55	69.59	0.01	0.002	0.001	22.14	9.13	2.07	4.80
80	10.03	10.75	9.13	0.07	0.057	0.049	81.49	70.24	0.07	0.057	0.049	81.62	70.48	0.01	0.005	0.002	26.21	10.22	1.99	4.72
80	9.98	10.70	9.07	0.10	0.083	0.072	83.44	71.52	0.10	0.084	0.071	83.54	71.43	0.02	0.011	0.005	27.67	11.27	1.90	4.63

These indicators are 2.15 times higher than the plant-absorbable and water-soluble form of ordinary copper and zinc trace elements contained in nitrophos fertilizers, and 4.08 times higher than the water-soluble form. The plant-absorbable form of molybdenum in nitrophos fertilizers ranged from 22.14% to 38.80%, and the water-soluble form ranged from 9.13% to 15.67%.

If nitrogen, phosphorus, and potassium, which are considered macroelements, are replenished by applying mineral fertilizers to plants, it is not possible to meet the demand for plants due to the lack of production of microelements. Despite the great importance of microelements in plant life, microfertilizers are hardly produced in our Republic. The reasons for delaying the production of micronutrient fertilizers include: Copper, zinc, cobalt, nickel, molybdenum, manganese, boron and other micronutrient raw materials, which are inexpensive and have opportunities for use, have not been fully studied, there is a lack of annual data on the mobile forms of micronutrients in the soil, Insufficient scientific and technological developments, recommendations on the rational application of micronutrients to the soil and complex insufficient information on the status of micronutrients in the production of fertilizers.

Fertilizers produced on the basis of phosphorites of Central Kyzylkum meet the demand of agriculture for phosphorus fertilizers by 25-30%. Due to the lack of microelements, plants suffer from various diseases. As a result, the uptake of 25-30% of the applied phosphorus nutrient element or other nutrient elements by plants decreases, and the nutrient elements remain in the soil. Due to this, there is a decrease in productivity in plants. At a time when the demand for phosphorus fertilizers is high in our republic, allowing them to be wasted is one of the factors that cause the development of agriculture in our country to be delayed. Along with nitrogen, phosphorus, potassium fertilizers, the demand for micronutrient fertilizers is also increasing in our republic. Currently, microfertilizers are used in practice in very small quantities. In the production of mineral fertilizers, the use of various wastes and products of non-ferrous metallurgy containing microelements, sour effluents, spent catalysts and other types of secondary products is one of the solutions for the production of microelement fertilizers.

4 Conclusion

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In the production of mineral fertilizers, the use of various wastes and products of non-ferrous metallurgy containing trace elements, sour effluents, spent catalysts and other types of secondary products is one of the important problems.

It can be said that the main obstacles in the production of nitrophos or other types of phosphoric, nitrogen micro fertilizers containing microelements are the incomplete study of industrial secondary products with microelements and the lack of cheap raw materials.

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