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Microelement (Cu) Salts Application to Superphosphate Fertilizer Technology

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Abstract

When the elements nitrogen, phosphorus, potassium, which are considered macronutrients, are replenished by adding mineral fertilizers to plants, it is not possible to meet the demand for plants due to the lack of production of micronutrients. Despite the great importance of micronutrients in the life of plants, micronutrients are almost not produced in the country. The following are the reasons that delay the production of micronutrient fertilizers. Cheap and usable copper, zinc, cobalt, nickel, molybdenum, manganese, barium and other micronutrient raw materials are not fully studied, lack of annual data on the mobile forms of trace elements in the soil, insufficient scientific and technological developments, Insufficient recommendations on the rational application of micronutrients in the soil and information on the status of micronutrients in the production of complex fertilizers. Fertilizers based on Central Kyzylkum phosphorites meet 25-30% of agricultural demand for phosphorus fertilizers. Due to the lack of micronutrients, plants suffer from various diseases. As a result, the absorption of 25-30% of the phosphorus nutrient or other nutrients used by plants is reduced and the nutrients remain in the soil. Therefore, a decrease in productivity is observed in plants. At a time when the demand for phosphorus fertilizers in the country is high, their waste is one of the factors delaying the development of agriculture in our country. Along with nitrogen, phosphorus and potassium fertilizers, the demand for micronutrient fertilizers is growing in the country. At present, micronutrients are used in very small quantities in practice. Various wastes and products of non-ferrous metallurgy, which contain trace elements in the production of mineral fertilizers, the use of sour effluents, depleted catalysts and other types of by-products is one of the solutions for the production of micronutrient fertilizers.

Keywords: Superphosphate, micronutrient salts, micronutrient secondary raw materials of industry, monoethanolamine, citric acid, micronutrients in complex form

Introduction

The rapid growth of the world's population, the depletion of arable land resources and water resources are exacerbating the problem of new types of fertilizer production worldwide. Therefore, one of the important tasks of the fertilizer industry and agriculture is to fully meet the demand of the population for quality products. Therefore, it is important to implement the production of micronutrient fertilizers, expand their range and reduce the cost of manufactured products.

Globally A.I.Fateev, A.S.Zarishnyak, B.A.Yagodin, S.P.Torshin, S.P.Polyanchikov, G.Gospadarenko, I.V.Jerdetskiy, E.A.Krylov, I.A.Gaysin A number of scientists have conducted research on the chemical composition and brand properties of micronutrient fertilizers, as well as the development of technologies for improving their quality.

In this regard, a large scientific school on the technology of micronutrient fertilizers under the leadership of Academician M.N. Nabiev has been established in Uzbekistan and is still operating. M.R.Adilova, E.K.Badalova, A.M.Amirova, M.T.Saibova, T.X.Taksanova, V.K.Khakimova, S.To'xtaev, S.M.Tadjiev, I.I. Usmanov, Z. Turaev and others are conducting large-scale research on the technology of micronutrient fertilizers, the scientific development of this field. Scientific sources on the production of micronutrient mineral fertilizers with high absorption properties by the above-mentioned scientists have not yet been studied.

Fertilizers based on Central Kyzylkum phosphorites meet 25-30% of agricultural demand for phosphorus fertilizers. Due to the lack of micronutrients, plants suffer from various diseases. As a result, the absorption of 25-30% of the phosphorus nutrient or other nutrients used by plants is reduced and the nutrients remain in the soil. Therefore, a decrease in productivity is observed in plants. At a time when the demand for phosphorus fertilizers in the country is high, their waste is one of the factors delaying the development of agriculture in our country. Along with nitrogen, phosphorus and potassium fertilizers, the demand for micronutrient fertilizers is growing in the country. At present, micronutrients are used in very small quantities in practice.

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

Researchers EV Vobko, M.A. Belousov, O.F. Tueva, M.Ya. Shkolnik, U.E. Brenchley and K. Warington studied the properties of micronutrients that are important for plants in 1927-1934. Brenchley and Warington found that micronutrients have a specific effect on plants. M.Ya. Shkolnik and E.V. Vobko noted that micronutrients not only have a catalytic effect on biochemical processes in the body, but also have an effect on the physicochemical properties of plasma, surface tension, diffusion, osmosis, proliferation of colloids, absorption and oxidation-reduction potentials in metabolic processes. 14 micronutrients of vital importance have been identified. These include B, Mo, Cu, Zn, Mn, Co, and others. Micronutrients, along with enzymes, vitamins, hormones, pigments, etc. in the body, affect the vital processes of organisms. They are involved in biochemical changes and affect physiological functions that take place in the plant organism through enzyme systems. Increases the use of light in the process of photosynthesis, accelerates protein synthesis. Some micronutrients activate the beneficial properties of plants, i.e., enhance their properties such as drought and frost resistance, accelerate seed germination and development, increase disease resistance. Their deficiency leads to disruption of metabolic processes, disease of plants and living organisms [1; 239- 241-p.].

Micro-fertilizers are fertilizers applied in small quantities (grams and kilograms per hectare). Boric acid, copper (II) sulfate, ammonium molybdate and other technical salts containing trace elements are used. Magnesium borate and other micronutrient wastes insoluble in manganese sludge (sludge) are insoluble in water. They are processed into a water-soluble state or used directly as fertilizer. Water-soluble and insoluble micronutrients are used in agriculture. Complex fertilizers are fertilizers that contain at least two nutrients. Secondary complex fertilizers (e.g., nitrogen-phosphorus, nitrogen-potassium, phosphorus-potassium) and tertiary complex fertilizers (e.g., nitrogen-phosphorus-potassium) are divided into types. Tertiary fertilizers are called complete fertilizers. Complex fertilizers may also contain trace elements, pesticides and additives [2; 8-9 p.].

In the works of A.I.Fateev it is stated that a certain amount of micronutrients is released from the soil as a result of assimilation by plants or for other reasons, they are not replenished at present, which is due to the lack of organic fertilizers in the soil. [3; 115-118 p, 4; 17-19 p]. It should be noted that the presence of micronutrients and their assimilation by plants synthesize all the enzymes that allow them to effectively use soil energy, water, fertilizers and nutrients in the soil.

Gospodarenko G. and in the work of others, plants without micronutrients suffer from various diseases, resulting in spontaneous decline in productivity and lack of micronutrients, even in the case of adequate nitrogen, phosphorus, potassium fertilizers. [5; 26-29 p, 6; 28-30 p].

In conclusion, the main obstacles in the production of superphosphate or other types of phosphorous, nitrogenous micronutrients containing micronutrients are incomplete study of micronutrient industry by-products and lack of cheap raw materials.

The use of non-ferrous metallurgy secondary products as a raw material base is one of the most pressing issues today. However, the literature does not adequately cover the physicochemical basis of micronutrient production and micronutrient production technologies. Also, the methods of complex transformation of microelement salts and secondary raw materials and their attachment to macronutrients are not widely covered.

Object and Methods of Research

The aim of the research is to introduce micro-elemental salts and microelement fertilizers using microelement salts and microelement-containing secondary raw materials of hydrometallurgy to the technologies of obtaining new varieties of phosphorus fertilizers on the basis of incomplete norms of mineral fertilizers and acids or their salty mixtures, which are widely used in agriculture.

Despite the above-mentioned advantages of micro-fertilizers, micro-fertilizers are almost not used in practice in our country. One of the main reasons for this is the lack of research on the availability of cheap and usable local micronutrient raw materials, the lack of science and technology-based micronutrient production technology and developments. In order to theoretically substantiate the production of micronutrients and to develop their production technology, it is necessary to conduct in-depth physicochemical studies to study the interaction of micronutrients with nitrogen-phosphorus fertilizers, fertilization technology and interactions in the addition of micronutrients to fertilizers.

Research Results

Central Kyzylkum phosphorites (low grade P_2O_5-12.38%, CaO-43.68%, CO_2-13.48%, unenriched P_2O_5-16.38%, CaO-45.93%, CO_2-18, for the purpose of obtaining micronutrient fertilizers 15%, SO_3-1.86%, thermoconcentrate P_2O_5-27.40%; CaO-54.68%; CO_2-4.52%) were decomposed to different rates with sulfuric and nitric acids to obtain superphosphate and nitrophos fertilizers. Copper and zinc sulfate and ammonium molybdate (Cu, Zn, Mo) salts, microelement secondary raw materials of industry, as well as their complex forms and their chemical composition were applied to the obtained superphosphate and nitrophos fertilizers and their chemical composition was studied. [7; 92-95 p, 8; 79-82 p, 9; 41-44 p, 10; 974-979 p, 11; 53-57 p,12; 95-99 p, 13; 173-175 p, 14; 427- 430 p, 15; 65-66 p, 16; 65-66 p, 17; 13-14 p, 18; 282-286 p, 19; 179-182 p, 20; 128-130 p, 21; 131-132 p, 22; 288-290 p.].

In order to develop a rational technology for obtaining micronutrient fertilizers, Central Kyzylkum phosphorite samples were processed in an accelerated manner with concentrated (93% H_2SO_4) sulfuric acid at 60, 80, 100% stoichiometric norms. Experiments have shown that the interaction between phosphorite flour and acid occurs quickly and easily. Because the reaction is exothermic, a large amount of heat is released and it is used to dry the reaction product.

Table 1: Chemical composition of superphosphate fertilizers based on Central Kyzylkum phosphorites,%

H_2SO_4 norm	P_2O_5		CaO		$K_{fragment}$		H_2O	CO_2	SO_3	pH
	general	nouris	genera	nouris	P_2O_5	CaO				

		hment	l	hment						
Based on low-grade phosphorite flour										
60	10,01	7,13	35,41	25,71	71,23	72,61	4,64	4,36	19,79	5,76
80	9,41	7,86	33,29	23,49	83,53	70,56	5,82	2,05	24,81	5,13
100	8,88	8,29	31,41	21,99	93,36	70,01	7,53	0,0	29,26	4,88
Unenriched phosphorite is based on it										
60	12,45	9,28	34,92	25,60	74,54	73,31	5,86	5,51	24,88	5,65
80	11,53	9,43	32,34	23,12	81,79	71,49	7,23	2,55	30,71	5,14
100	10,74	10,37	30,11	21,36	96,55	70,94	8,42	0	35,75	4,98
Thermoconcentrate based on it										
60	18,87	12,65	37,66	20,14	67,04	53,48	3,40	1,10	19,76	5,28
80	17,10	14,37	34,12	18,01	84,04	52,78	4,10	0,38	23,87	5,01
100	16,15	15,83	32,23	16,41	98,02	50,92	4,76	0,0	26,07	4,68

Studies have shown that in the processing of phosphorite flour, the rate of their decomposition increases with increasing levels of sulfuric acid. Low-grade phosphorite flour and 10.01% of the total phosphorus (P_2O_5) in the processed product with a 60% acid content were found to be in a form that could be assimilated by 71.23% of the plant.

When the acid rate increases from 80% to 100%, the decomposition rate increases from 83.53% to 93.36%. It can be seen that the total calcium (CaO) in the fertilizer is decompose to 72.61%, 70.56%, 70.01% in accordance with the acid norm.

When studying the composition of superphosphate fertilizers based on unenriched phosphorite flour, the content of fertilizers obtained at 60% of acid content is 12.45% of total phosphorus, 74.54%, 11.53% of total phosphorus, 81.79% of total phosphorus, and 10.74% of 100%, it was found that 96.55% of the total phosphorus was decompose. Also, the decomposition of total calcium is 73.31%, 71.49%, 70.94%, respectively, in accordance with the acid norm. In the superphosphate obtained from thermoconcentrate, 67.04% of the total phosphorus (P_2O_5) at 60% of the acid content, with an increase in the acid content, ie at 80% of the total 17.10% of the total phosphorus, 84.04%, and at 100% of the acid content, a total of 16.15% phosphorus was observed to be decomposed by 98.02%. It was also found that the decomposition of calcium was 53.48%, 52.78%, 50.92%, respectively, in accordance with the acid norm. It was observed that the pH of all superphosphate fertilizers obtained on the basis of phosphorite samples was in a neutral state.

The process of granulation of superphosphate powder processed at different rates of sulfuric acid to obtain copper micronutrient superphosphate fertilizers was carried out in the presence of a solution of copper sulfate salt. The ratio of P_2O_5: Cu in the obtained micronutrient superphosphate fertilizers (Table 2) is 1: 0.007, 1: 0.01, 1: 0.02. The granulated fertilizers were dried at a temperature of 90-105 ^{0}C and their chemical composition was analyzed.

Table 2: Chemical composition of copper microelement superphosphate fertilizers,%

H_2SO_4 norm	P_2O_5		Cu			ME consumption degree %		H_2O	pH
	general	consumption	general	consumption	soluble in water	consumption	soluble in water		
Low-grade phosphorite is a copper superphosphate obtained on the basis of flour									
60	10,24	7,30	0,06	0,023	0,010	39,12	16,03	2,23	5,97
60	10,23	7,29	0,11	0,045	0,021	41,03	18,93	2,21	5,99
60	10,21	7,27	0,16	0,071	0,034	44,07	21,33	2,19	6,01
80	9,84	8,23	0,05	0,017	0,007	33,08	13,04	1,45	5,18
80	9,81	8,19	0,10	0,037	0,014	36,65	14,43	1,43	5,21
80	9,78	8,16	0,15	0,060	0,024	39,81	15,99	1,41	5,27
100	9,47	8,85	0,04	0,011	0,004	27,98	10,69	1,34	4,84
100	9,45	8,83	0,09	0,027	0,010	29,45	11,38	1,32	4,91
100	9,42	8,80	0,14	0,045	0,017	32,04	11,97	1,30	4,99
Copper superphosphate obtained on the basis of unenriched phosphorite flour									
60	12,99	9,69	0,07	0,027	0,010	37,88	14,31	1,47	5,61
60	12,96	9,66	0,13	0,052	0,021	39,69	15,89	1,46	5,65
60	12,93	9,63	0,18	0,074	0,033	41,01	18,51	1,45	5,68
80	12,15	9,95	0,06	0,022	0,007	31,66	11,18	1,95	5,12
80	12,13	9,93	0,11	0,036	0,014	33,14	13,04	1,94	5,14
80	12,11	9,90	0,14	0,050	0,021	35,96	14,99	1,93	5,16
100	11,43	11,04	0,04	0,004	0,004	24,85	9,83	2,42	5,01
100	11,40	11,01	0,09	0,024	0,010	27,18	10,78	2,39	4,98
100	11,37	10,98	0,13	0,039	0,015	29,94	11,31	2,36	4,93
Copper superphosphate obtained on the basis of thermoconcentrate flour									
60	19,20	13,15	0,08	0,028	0,010	35,31	12,95	1,23	5,22
60	19,17	13,11	0,17	0,064	0,025	37,64	14,62	1,22	5,28
60	19,13	13,06	0,25	0,099	0,040	39,62	16,16	1,21	5,32
80	17,62	14,93	0,07	0,021	0,007	29,86	10,21	0,79	4,97
80	17,58	14,88	0,15	0,047	0,018	31,53	11,93	0,78	5,01
80	17,53	14,82	0,22	0,074	0,030	33,76	13,76	0,77	5,13

100	16,78	16,48	0,06	0,015	0,006	24,18	9,29	0,86	4,61
100	16,72	16,40	0,13	0,033	0,012	25,35	10,13	0,85	4,68
100	16,65	16,32	0,19	0,053	0,020	27,98	10,68	0,84	4,73

When analyzing the composition of low-grade phosphorite flour and copper superphosphate fertilizers obtained at a rate of 60% of acid, the content of fertilizer in the ratio P_2O_5: Cu = 1: 0.007 was 0.06% Cu, the plant assimilation form was 39.12%, water-soluble form was 16.03%. is formed. As the amount of copper in the fertilizer increased, ie at 0.11 and 0.16%, 41.03 and 44.07%, respectively, were found to be plant-soluble, and 18.93 and 21.33% were soluble in water.

The same pattern was observed in micronutrient fertilizers based on unenriched phosphorite flour and thermoconcentrate as in low-grade phosphorite flour-based fertilizers. As the acid content increases, the plant-assimilated form of phosphorus in superphosphate increases, and the copper sulfate salt reacts with the phosphates to form copper phosphate salts. As a result, micronutrients are converted into a form that is difficult for plants to assimilate. The content of copper in superphosphate fertilizers obtained on the basis of thermoconcentrate was 1.5-3.0% lower than that of enriched phosphorite and 2.0-5.5% lower than that of low-grade phosphorite flour. In order to convert the copper microelement in superphosphate fertilizers into a fully plant-absorbing form, their granulation process was processed in the presence of a solution of copper sulfate complex salts of monoethanolamine and citric acid. The granulated fertilizers were dried at a temperature of 90-105 ^{0}C and their chemical composition was analyzed.

The total 0.06% of the complex form of superphosphate obtained at the rate of 60% of low-grade phosphorite flour and sulfuric acid is 89.79% of the plant assimilation form of water, 81.38% of the water-soluble form. When the content of complex copper microelement in superphosphate increases from 0.11 to 0.16%, the total copper content of the fertilizer is 91.18-93.32%, and the water-soluble form is 83.88-85.39%. is formed.

Copper salt in the form of citrate in superphosphate fertilizers with an acid content of 80% and plant-soluble and water-soluble form of ordinary copper were analyzed and compared.

Table 3: Of granulated superphosphate based on copper citrate solution chemical composition, %

H_2SO_4 norm	P_2O_5		Cu			ME consumption degree %		H_2O	pH
	general	consumption	general	consumption	soluble in water	consumption	soluble in water		
Low-grade phosphorite is a copper superphosphate obtained on the basis of flour									
60	10,22	7,37	0,06	0,054	0,049	89,79	81,38	2,18	6,05
60	10,17	7,32	0,11	0,100	0,092	91,18	83,62	2,10	5,83
60	10,12	7,26	0,16	0,149	0,137	93,32	85,39	2,03	5,75
80	9,81	8,28	0,05	0,043	0,039	85,51	77,67	1,41	5,31
80	9,76	8,22	0,10	0,087	0,080	87,47	79,81	1,33	5,22
80	9,71	8,16	0,15	0,134	0,122	89,38	81,22	1,24	5,16
100	9,45	8,91	0,05	0,041	0,037	81,61	74,49	1,30	5,10

100	9,40	8,85	0,09	0,075	0,069	83,77	76,48	1,21	4,95
100	9,35	8,79	0,14	0,120	0,110	85,68	78,44	1,14	4,86
Copper superphosphate obtained on the basis of unenriched phosphorite flour									
60	12,93	9,82	0,07	0,062	0,056	88,52	80,35	1,39	5,68
60	12,88	9,76	0,13	0,118	0,107	90,45	82,51	1,31	5,60
60	12,83	9,70	0,18	0,166	0,152	92,47	84,38	1,24	5,52
80	12,12	10,04	0,06	0,051	0,046	84,58	76,46	1,91	5,23
80	12,07	9,98	0,11	0,095	0,086	86,64	78,19	1,82	5,15
80	12,02	9,92	0,15	0,133	0,120	88,53	80,22	1,76	5,04
100	11,39	11,10	0,04	0,032	0,029	80,38	73,28	2,35	4,93
100	11,34	11,04	0,10	0,083	0,075	82,62	75,38	2,28	4,86
100	11,29	10,98	0,14	0,118	0,108	84,59	77,40	2,20	4,77
Copper superphosphate based on thermoconcentrate									
60	18,83	13,06	0,08	0,070	0,063	87,55	79,29	2,24	5,64
60	18,78	13,00	0,17	0,152	0,139	89,45	81,48	2,15	5,52
60	18,73	12,94	0,25	0,228	0,208	91,36	83,37	2,07	5,38
80	17,30	14,91	0,07	0,058	0,053	83,53	75,65	1,87	5,20
80	17,25	14,85	0,15	0,128	0,116	85,62	77,56	1,79	5,09
80	17,20	14,79	0,22	0,192	0,176	87,49	79,81	1,66	5,00
100	16,48	16,30	0,06	0,048	0,044	79,58	72,51	1,64	4,79
100	16,43	16,24	0,13	0,106	0,096	81,63	74,49	1,55	4,71
100	16,78	16,18	0,19	0,159	0,146	83,52	76,74	1,44	4,62

In the composition of the fertilizer, the plant-absorbing form of copper in the form of citrate ranges from 85.51% to 89.38%, while the water-soluble form ranges from 77.67% to 81.22%. It was found that these values are 2.25-2.58 times higher than the plant-absorbing form of copper sulfate salt in the fertilizer, and 5.08-5.96 times higher than in the water-soluble form.

The above values are also maintained in the composition of low-grade phosphorite flour and superphosphate fertilizers obtained at a rate of 100% sulfuric acid. These values were found to be 2.67-2.92 times higher than the plant-assimilated form of copper sulphate in copper citrate superphosphate fertilizers with an acid content of 100%, and 6.55-6.97 times higher in the water-soluble form.

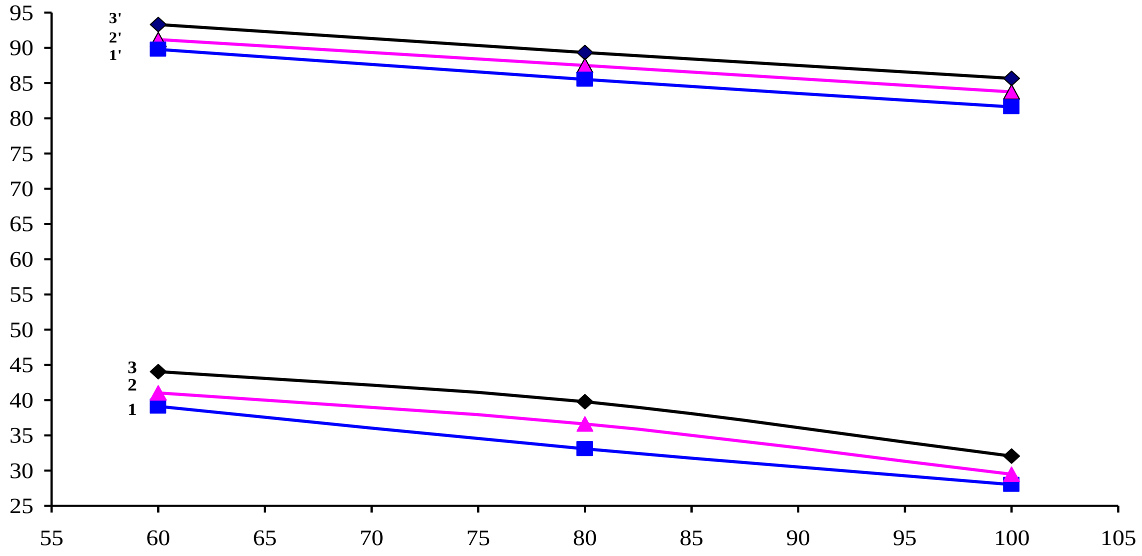

Figure 1. The degree of plant absorption of copper sulfate (1-3) and copper citrate (1'-3 ') in copper microelement superphosphate fertilizers obtained at different rates of sulfuric acid 1) 1, 1' P2O5:Cu 1:0,007, 2) 2, 2' P2O5:Cu 1:0,01, 3) 3, 3' P2O5:Cu 1:0,02

Also, when studying the plant's absorbable and water-soluble form of water in the composition of micronutrient superphosphate fertilizers based on unenriched phosphorite flour and thermoconcentrate, it was observed that in these fertilizers the same patterns as in the composition of micronutrient fertilizers based on low-grade phosphorite flour.

For example, in a superphosphate fertilizer with an acid content of 60% (in the ratio P_2O_5: Cu = 1: 0.007), the plant-absorbing form of copper is 88.52% and the water-soluble form is 80.35%.

With the increase in the ratio of copper to phosphorus, ie in the ratio P_2O_5: Cu = 1: 0.02, it was observed that the plant absorption form of copper reached 92.47%, and the water-soluble form reached 84.38%. It was found that these values are 2.25-2.34 times higher than the plant-absorbing form of ordinary copper microelement in superphosphate fertilizers with an acid content of 60%, and 4.59-5.61 times higher than in the water-soluble form.

When the copper sulfate salt is treated with monoethanolamine or citric acid, it coordinates with the copper ion through oxygen instead of the hydrogen atom in the carboxyl group. The complexly formed copper salt does not combine with phosphates in the fertilizer or other salts in the soil and does not form difficult-to-dissolve salts. Therefore, their plant-absorbing or water-soluble form is several times higher than the normal form in the composition of phosphorus fertilizers.

Based on the results obtained, the material balance of micronutrient superphosphate production was calculated.

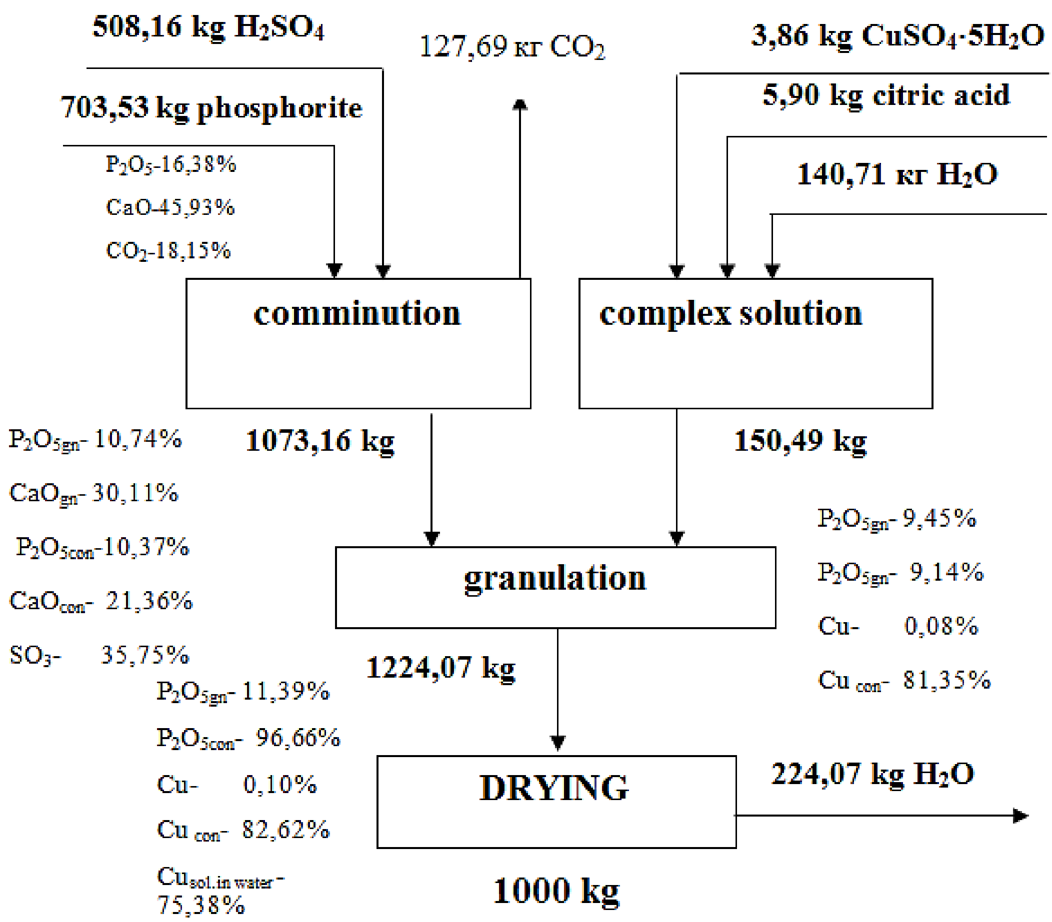

Figure 2. Material balance of copper microelement superphosphate on the basis of unenriched phosphorite flour

Figure 2 shows the material balance of copper micronutrient superphosphate production. To obtain 1 ton of new grade fertilizer, 703.53 kg of unenriched phosphorite was processed in an accelerated manner with 508.16 kg of 92.5% sulfuric acid for 15-20 minutes. The process of granulation of the obtained superphosphate was carried out in the presence of 150.49 kg of copper citrate solution. Copper citrate complex solution is prepared by dissolving 3.86 kg of copper sulfate salt and 5.90 kg of citric acid in 140.71 kg of water. The resulting new variety contains copper microelement superphosphate fertilizer P_2O_{5gn}-11,39%, P_2O_{5con}-96,66%, Cu-0,10%, Cu_{con}- 82,62%, $Cu_{sol.in\ water}$-75,38%, H_2O-2,35% is formed.

Conclusion. In order to increase the ability of microelements to be assimilated by plants, complex compounds with monoethanolamine and citric acid salts were obtained. Micronutrient salts were carried out with monoethanolamine in a ratio of 1: 2 mol, copper sulfate and citric acid in a ratio of 0.65: 1 mol. The results of IR spectral analysis of microelement salts and microelement secondary raw materials converted into complex forms were also studied. The results showed that micronutrient salts and secondary raw materials combined with citric acid to form new bonds. It can be said that it is necessary to obtain complexes of microelement salts and on their basis to develop technologies of micronutrient superphosphate fertilizers and their application in agriculture.

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