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Using active sludges of cleaning facilities for obtaining the organomineral nitrophosphoric polymicroelement fertilizer enhanced by phosphorus

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INTRODUCTION

The world experience of many countries shows that the rise in the national economy begins with agriculture. Only the systematic introduction of mineral fertilizers, and primarily phosphorus-containing, allows increasing the productivity of agricultural plants more than two times.

Uzbekistan, being an agro-industrial country, occupies 3.73 million hectares of irrigated arable land. About 39.8 kg of P_2O_5 is applied per hectare of irrigated arable land, whereas it is necessary to introduce P_2O_5 , cotton 145-165 kg/ha P_2O_5 , 100-110 kg/ha P_2O_5 , 140 rice -145 kg/ha P_2O_5 , maize for grain 120-140 kg/ha P_2O_5 . This circumstance is evidence of a large deficit in phosphorus-containing fertilizers.

Deficiency of phosphorus fertilizers is further aggravated by the fact that a large number of nutrient components are removed from the soil with the crop. It is known that one ton of raw cotton takes out of the soil annually 45 kg of nitrogen, 15 kg of P_2O_5 and 45 kg of K_2O . One ton of wheat takes out 35 kg of nitrogen, 10 kg of P_2O_5 and 24 kg of K_2O from the soil every year. With a gross harvest of 3 million tons of raw cotton and 6.1 million tons of wheat, only 348,500 tons of nitrogen, 106,000 tons of phosphorus and 281,400 tons of potassium are lost annually from these two crops. But other cultures also carry a large amount of nutrients out of the soil. Their quantity in the soil must be systematically replenished[1].

It should be noted that the production of phosphorus-containing fertilizers in the Republic is limited by the quality of our phosphorite of the Central Kyzylykum field. It is a phosphorus-poor raw material, which also contains a large amount of undesirable impurities, in particular, carbonates and chlorine. To get high-quality phosphorus fertilizer from such raw materials, it must be previously enriched. Therefore, the Kyzylykum Phosphorite Plant (KPP) carried out multi-stage enrichment: crushing, dry enrichment to obtain ordinary phosphorite flour, washing away from chlorine, roasting to remove CO_2 [2].

There is another, unconventional biotechnological way of enriching low-grade phosphorites, when the disintegration of calcites with the release of CO_2 takes place with the participation of microorganisms. The method of biotechnological enrichment of phosphorites is based on the ability of certain types of microorganisms under certain conditions to use mineral compounds as sources for nutrition and reproduction. A lot of microorganisms that make up the active sludge of the biological treatment plant, being in the waste liquid, absorb the pollutants into the cell where they undergo the biochemical transformations under the influence of enzymes. In this case, organic and some types of inorganic pollutants are used by the bacterial cell in two ways:

1. Biological oxidation in the presence of oxygen to harmless products of carbon dioxide and water:
$$\text{Organic matter} + O_2 \text{ (in the presence of enzymes)} \Rightarrow CO_2 + H_2O + Q.$$
2. The energy released in this case is used by the cell to ensure its vital activity (movement, breathing, reproduction, etc.).
3. Synthesis of a new cell (multiplication):
$$\text{Organic substance} + N + P + Q \text{ (in the presence of enzymes)} \Rightarrow \text{a new cell}.$$

The intensity and depth of the processes depends on the qualitative composition of the activated sludge, the variety of forms and species of microorganisms, their adaptability (adaptation) to the specific composition of the pollutants of the waste fluid and the conditions of the process. The active sludge looks like various flakes floating in the water or septic tank fixed on the load. Purification of effluents is due to the absorption of organic constituents by the simplest microorganisms, as well as biochemical oxidation and biosorption.

In the aerobic purification of effluents, two basic microbiological processes occur: oxidation of organic carbon and nitrification with the participation of filamentous, flocculating microorganisms and nitrifying bacteria.

The number of microorganisms in the active sludge is also called biomass. It may include: protozoa; actinomycetes (microscopic fungi); bacteria; infusoria; amoeba; worms (nematodes); rotifers, etc. [3]

Domestic and foreign experience in the use of sludge in biological wastewater treatment facilities testifies to the prospects of the method of its utilization as a fertilizer in the absence of toxic impurities, in particular, heavy metal compounds [4].

In Germany, for example, about 30% of the annually formed precipitation is used as fertilizer, it is deposited to 60% and burns no more than 10%.

In the Netherlands, with an annual quantity of 5.5 million tons of sludge, up to 70% is used as a fertilizer. A certain experience of recycling is available in Switzerland, India and other countries. The application of the dried sludge was approved in the Stavropol Territory. As an optimum dose, for example, for barley, it is suggested to use 60 kg per 1 hectare, which corresponds to 3 tons per hectare of sludge with a moisture content of 35% or 6.5 tons per hectare at a humidity of 80%. Analysis of samples of the fermented sludge of wood chemical plants, carried out using conventional methods of agrochemistry, showed the presence of 18-20% of ash elements and 80-82% of organic substances in dry matter [5].

Table 1.
Chemical composition of the phosphorites of Central Kyzylkum

No.	Name of connections	Contents of elements,%	No.	Name of connections	Contents of elements,%
1.	P ₂ O ₅	8-12,2	8.	CO ₂	8-15
2.	Al ₂ O ₃	1,5-3,0	9.	Fluorine	1,8-3,2
3.	SiO ₂	6,0-8,0	10.	SO ₃	2,5- 3,5
4.	CaO	42-48,1	11.	U	0,003-0,008
5.	MgO	2,5-3,5	12.	The summary of REE	0,04-0,089
6.	Fe ₂ O ₃	0,6-0,8	13.	H ₂ O	10,0
7.	SO ₃	2,8-3,0	14.	Insoluble residue	8,0-8,2

Sewage sludge, used as fertilizer, must have a complex of properties, of which the main ones are moisture, phosphorus, nitrogen, potassium and heavy metals. Precise criteria for the classification and normalization of the application of precipitation as fertilizers are not present, since the composition of precipitation, in particular, the content of biogenic elements of phosphorus, nitrogen, and potassium in them. They vary greatly depending on the type and origin of the sediments [6].

The undoubted advantage of active sludge (AS) is a high content of organic matter up to 75%. A high estimate of organic matter is also given in the work in which it is noted that organic matter largely determines the direction of the soil formation process, the biological, chemical and physical properties of the soil environment.

During the research, the possibility of using the liquid phase and active sludge directly on the low-grade phosphorites of the Central Kyzylkum to study the possibility of microorganisms of activated silt for their growth and development to use carbonate carbonate in the calcite was considered.

In connection with the foregoing, the Navoi State Mining Institute conducted laboratory studies on the leaching of various elements from low-grade phosphorites of the Jeroy-Sardara deposit using aerobic species of neutrophil microorganisms in the active sludge of the biochemical purification plant of JSC "Navoiazot".

The object of the study was a low-grade phosphorite ore from the Jeroy-Sardara deposit with the following chemical composition (Table 1).

Phosphorites of the Kyzylkum are composed mainly of phosphatized faunal residues, bound with fine-grained calcite cement [7].

Among phosphatized remains of fauna prevail foraminifera with sizes of shells from 0.04 to 0.5 mm. Isotropic and weakly crystallized phosphate with point inclusions of calcite fills the internal cavities of their shells. Relic calcite, preserved from phosphate substitution, sometimes also forms a shell and internal septa shells. In the scientific and



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technical literature, such calcite, which is located inside the phosphate formations, is called the "endocalcite", and the cement composing the rock is "exocalcite". The third form of calcium carbonate is found in the form of a phosphate mineral isomorphically entering the crystal lattice. The subordinate role is played by the phosphatized remains of other groups of organisms: valves and cores of pelecites, gastropod shells up to 5 mm in size, up to 5-10% in places; elongated cone-shaped pteropods up to 4-5 mm in length and up to 1.5 mm in diameter, needles of sea urchins, fish scales, etc. Cementation is often unstable, many phosphorites easily disintegrate under mechanical action, especially after soaking in water. Among organic residues, a small amount of primary phosphate material is present - teeth of sharks, vertebrae and small (several millimeters) fragments of bones of marine animals. The cement of phosphorites is fine-grained calcite with an admixture of clay and phosphate-clay material. In the later part of the second layer the cement is strong crystalline, it is represented by calcite and gypsum, sometimes with a siliceous component. The results of mineralogical study of granular phosphorite ores indicate a monotony of their composition.

The main phosphate mineral - francolite (fluorocarbonateapatite) and calcite - make up 80-90% of the ore. Francolite contains on average about 42,1% P_2O_5 , 55,4% CaO, 1,2% F, 2,3% Cl, 0,6% H_2O , the sum of rare elements reaches 0,03%. The ore of the deposit has the following average mineral composition, (weight,%): Francolite – 56,0; calcite – 26,5; quartz – 7,5 – 8,0; hydromica minerals and feldspars – 4,0 – 4,5; gypsum – 3,5; goethite – 1,0; zeolite <1,0; organic matter is about 0,5 [8].

The active sludge in the form of a liquid phase (L) was mixed with phosphorite in the ratio F: T = 4: 1. The experiments were carried out in several versions in reactors using water, the liquid phase of activated sludge, a solid sediment of activated sludge with compression air supply with continuous stirring. After bacterial leaching for 14 days, the samples of the liquid and solid phases were subjected to qualitative and quantitative analysis, and also sent for analysis in the State Unitary Enterprise "Uzgeorangmetliti" for X-ray fluorescence energy dispersive spectral analysis.

To determine the change in the morphological structure of phosphorite grains and calcite crystals, a conventional optical microscope was used, where not only conventional but also stereoscopic images were obtained by increasing 150 times, changing the illumination angle (illumination of the sample surface).

The results obtained and their discussion.

A significant part of the field crop production (directly or indirectly) is sent to human food. Consequently, the release of the human body should contain large amounts of nitrogen and ash constituents taken from plants from the soil. In comparison with the excrement of herbivores, the garbage of the human body should be more richly (counting on dry matter) nitrogen and phosphoric acid, firstly, because human food is richer in proteins than feed for herbivores.

If, for example, 1.5% of nitrogen is contained in animal food, counting for dry matter, then in human food it is from 2-3% (grain bread) to 15% (meat). Secondly, people's food is better digested, so most of it is oxidized, giving water and carbon dioxide, and then the remaining fraction is even more enriched with gas than in the herbivorous organism. The first experiments to study the fertilizer value of sewage sludge showed that active silt can be equated with manure and mineral fertilizers. Similar conclusions were drawn by other authors. According to the data, in the sewage sludge the content of total nitrogen and phosphorus is 1.5-2 times higher than in cattle manure, namely these elements determine the value of any type of fertilizer.

The high content of activated sludge batteries confirms that silt from urban wastewater treatment plants contained a percentage of the wet weight: N total - 0.8; P_2O_5 - 0.9; K_2O - 0.4; nitrate nitrogen - 6.4 mg/100; Ammonium nitrogen - 457 mg/100; mobile phosphorus - 542 mg/100 g of weight.

In the technological cycle of wastewater treatment, different types of precipitation are obtained, which, due to their fertilizing qualities, can differ sharply from each other. Fluctuations in the content of the basic elements of nutrition in the activated sludge are: for nitrogen 0.8 ... 6%, phosphorus 0.6 ... 5.6%, potassium 0.1 ... 0.5%. Approximately the same data lead scientists of the USA and Canada: nitrogen 1.1 ... 7.6%, phosphorus 1.3 ... 8.0%, potassium 0.1 ... 0.3%. The undoubted advantage of activated sludge is the high content of organic matter up to 75%. A high estimate of organic matter is also given in the work in which it is noted that organic matter largely determines the direction of the soil formation process, the biological, chemical and physical properties of the soil environment. A number of other foreign researchers pay attention to this. They come to the conclusion that, after many years of treatment, the soil begins to lack organic substances, as cultivation speeds up its destruction, and the return from stubble residue is insufficient to compensate for the losses. Organic matter forms aggregates of soil particles, between which there are large pores through which air can penetrate to the roots, and excess water evaporates. With a lack of organic substances, soil aggregates lose their strength and decay. The soil becomes denser, air access stops and as a result, root growth occurs abnormally. Sand and silty soils are subjected to the greatest degree to such structural changes. The introduction of organic fertilizers into such soils improves their quality, as a result of which the yield will be higher than when

applying the optimal amount of conventional fertilizers, but without the addition of organic matter. Precipitation solids are more effective than equivalent amounts of litter.

Table 2.
Chemical composition of used activated sludge

Content of metals, mg/kg	Cu	Zn	Mn	Ni	Cr	Pb	Cd
By the state standards 17.4.3.07-2001	750	1750	1000	200	500	56	15
In the active sludge	76	218	794	29	38	39	5
In an aqueous extract, mg/l	PO ₄ ⁻³	NO ₂ ⁻	NO ₃ ⁻	Ammonia nitrogen	K ₂ O	Acidity	Organic matter, %
	0,7	0,71	0,08	3,1	0,4	1,4	50

With increasing soil porosity, the rate of water infiltration increases and water losses as well as erosion of soil as a result of surface runoff decrease. Where liquid sediments are deposited on the surface of the soil, the pores are temporarily closed, and for a few days the infiltration of water slows down. As soon as the layer of precipitation begins to dry out, it cracks, and water easily penetrates between the particles. For some time these particles protect the soil underneath themselves from structural destruction and blockage of pores after rain. Thus, a longer exposure results in an increase in the rate of infiltration of water. The solids of precipitation as a result of grinding penetrate into the movements of earthworms, which accelerates the absorption of soil by the water entering the surface. Extracellular enzymes in active silt contain proteases, hydrolases, cellulases, peroxidases. Extracellular enzymes catalyze the oxidation of substrates with the participation of hydrogen peroxide and catalase, decomposing H₂O₂ [9].

In Navoi region, the industrial-developed infrastructure of the Republic in Navoiyazot JSC operates a large biochemical treatment station for domestic and industrial sewage, capable of processing daily up to 120,000 m³ of sewage. At present, solid wastes from communal and industrial wastewater have been studied in more or less detail, as can be seen from Table 2. The amount of cationic elements in the sewage composition is lower than the accepted state standards, and therefore metals in the solid sludge sediments can serve as microelements for plant nutrition. There are more than 75 such biogenic elements in the composition of plants, including macro-, micro- and ultramicroelements. With a small amount of water-soluble ammonium in the sediment, some of it, connected with organic matter, is a source that slowly supplies nitrogen, which can use plants with a longer growing season more fully.

Table 3.
Density of phosphorite ore and slimes after treatment with activated sludge

Variants	Density g/l	pH	Variants	Density g/l	pH
Control phosphorite ore + water	1184,62	6,83	Control phosphorite sludge + water	1170,58	7,00
Phosphorite ore + activated sludge (liquid phase)	1132,82	6,82	Phosphorite sludge + activated sludge (liquid phase)	1164,7	6,88
Phosphorite ore + activated sludge (solid residue)	1131,37	6,60	Phosphorite sludge + activated sludge (solid residue)	1150,61	6,69
Phosphorite ore + activated sludge (liquid phase) with oxygen	1105,54	7,05	Phosphorite sludge + activated sludge (liquid phase) with oxygen	1091,9	7,14
Phosphorite ore + activated sludge (solid residue) with oxygen	1099,01	6,86	Phosphorite sludge + activated sludge (solid residue) with oxygen	1092,2	6,86

The phosphorus contained in it corresponds, according to the effect on plant growth, to phosphorus extracted from mineral fats by lemon acid extract. The lack of potassium in the sediment requires its addition in the form of mineral

fertilizer. The sewage sludge contains basic plant nutrients, especially N and P, trace elements (Zn, Cu, Mo, Mn), improves the physical properties of the soil, structure, water retention, moisture transfer [10].

As a result of sorting phosphorite ore with P₂O₅ content below 12-15%, which is carried out directly in the process of extraction, a large amount of phosphorite ore is stored as a waste, unsuitable for fertilizer production. To date, the volume of such waste is more than 6 million tons, and the number of such unused substandard phosphorites is growing from year to year.

Laboratory experiments on the enrichment of low-grade phosphorite ore and phosphorite waste in the form of slurries in reactors simulating the aeration tanks of treatment facilities showed that at a ratio of T: H = 1: 4, the density of introduced phosphorites in the variants from 1184.62 g / l to 1092, 2 g / l (Table 2).

The decrease in density is apparently due to a decrease in the carbonate content of the calcite mineral CaCO₃. This circumstance makes it possible to use low-grade phosphorites in production conditions by direct introduction into aerotanks, since at their low density they can be carried out by the current of water into thickeners to separate the liquid and solid phases.

The pH of the medium in all the studied variants was basically neutral and varied insignificantly, varying within 7,0 and 6,7. This fact confirms the thesis that in the neutral and alkaline phosphorites, calcium phosphate is predominantly present. Therefore, when biochemical processing of domestic and industrial wastewater in an amount of 55-60 thousand m³/day and yield in the form of condensed precipitation of about 1 thousand m³, 275 tons of low-grade phosphorites or sludges can be added directly to the aeration tanks, and a year is possible utilization of up to 90 thousand tons of phosphorites with a content of 8-10% P₂O₅ (Figure 1) with subsequent release of finished products.

Table 4.
Distribution of alkaline earth and radioactive metals in solid and liquid phases

N o.	Variants	Solid phase								Liquid phase		
		Mo	Ni	Cu	Zn	As	Sr	Th	U	Sr	Th	U
1	Initial dry	0,0001 9	-0,0052	0,0026	0,0122 8	0,000918	510,52 1	3,248	1,906	-	-	-
2	Init.+H ₂ O	0,0002 4	-0,0038	-0,0008	0,0178 1	0,001421	683,44 8	7,206	14,32	13,0 4	0,54 7	4,452
3	Alkaline init.+ H ₂ O	0,0002 4	-0,0050	-0,0012	0,0144 4	0,001547	544,35 3	3,516	0,555	21,2 0	1,09 0	5,964
4	Alkal. init.+O ₂	0,0002 4	-0,0053	0,0057	0,0143	0,001296	545,48 2	4,468	5,309	11,6 3	0,42 1	7,971

Solid thickened precipitates obtained after thickening with the addition of low grade phosphorites will contain up to 16-18% P₂O₅, 12-14% nitrates and nitrites, and 4-5% K₂O. Thus, qualitatively new fertilizers, referred to as organomineral nitrogen-phosphorus polymicroelement fertilizers [4], will be obtained. The resulting liquid complex fertilizer is disinfected to destroy possible sources of disease and is packaged as a ready-to-eat product.

It should be noted that the distinctive feature of the Kyzylkum phosphorites is their high degree of carbonate content, the concentration of CO₂ in some formations reaches 27% or more.

The results obtained by X-ray fluorescence energy dispersive spectral analysis confirmed the assumptions of bacterial leaching of various elements, in particular, strontium, thorium and uranium, into the solution. In the variant with active silt, the amount of strontium and thorium leached into the solution was 21.2 and 1.09 mg / l, respectively. Therefore REEs and radioactive elements transferred to the liquid phase are recommended to be extracted by existing methods. After extraction of cationic elements from clarified water, it is recommended that it be subjected to fine chemical purification and sent to production. Thus, it is possible to create a closed production mode.

Spectral analysis of samples treated with active silt showed that not only radioactive but also rare-earth metals were released into the liquid phase. In the solid phase, the amount of radioactive and rare-earth metals varies within different limits, proceeding from partial dissolution and isolation into the liquid phase. The greatest amount of uranium - 7.97 mg / l was leached in the version with the use of activated sludge with air supply and using nitrogen fertilizing in the form of urea. One should especially note the behavior of arsenic, which also underwent oxidation and passed into solution, especially in the third variant (Table 4).

Analysis of the morphological structure of minerals in the composition of phosphorite ore and sludges under a microscope showed selective crushing of phosphorite ore. The obtained results show that, apparently, the

microorganisms underwent destruction of the organic component of phosphorites, which is about 0.5%. In addition, phosphorite grains were used as a source of nutrition as a food source, which in microscopic photos decreased in size and acquired a round or spherical shape.

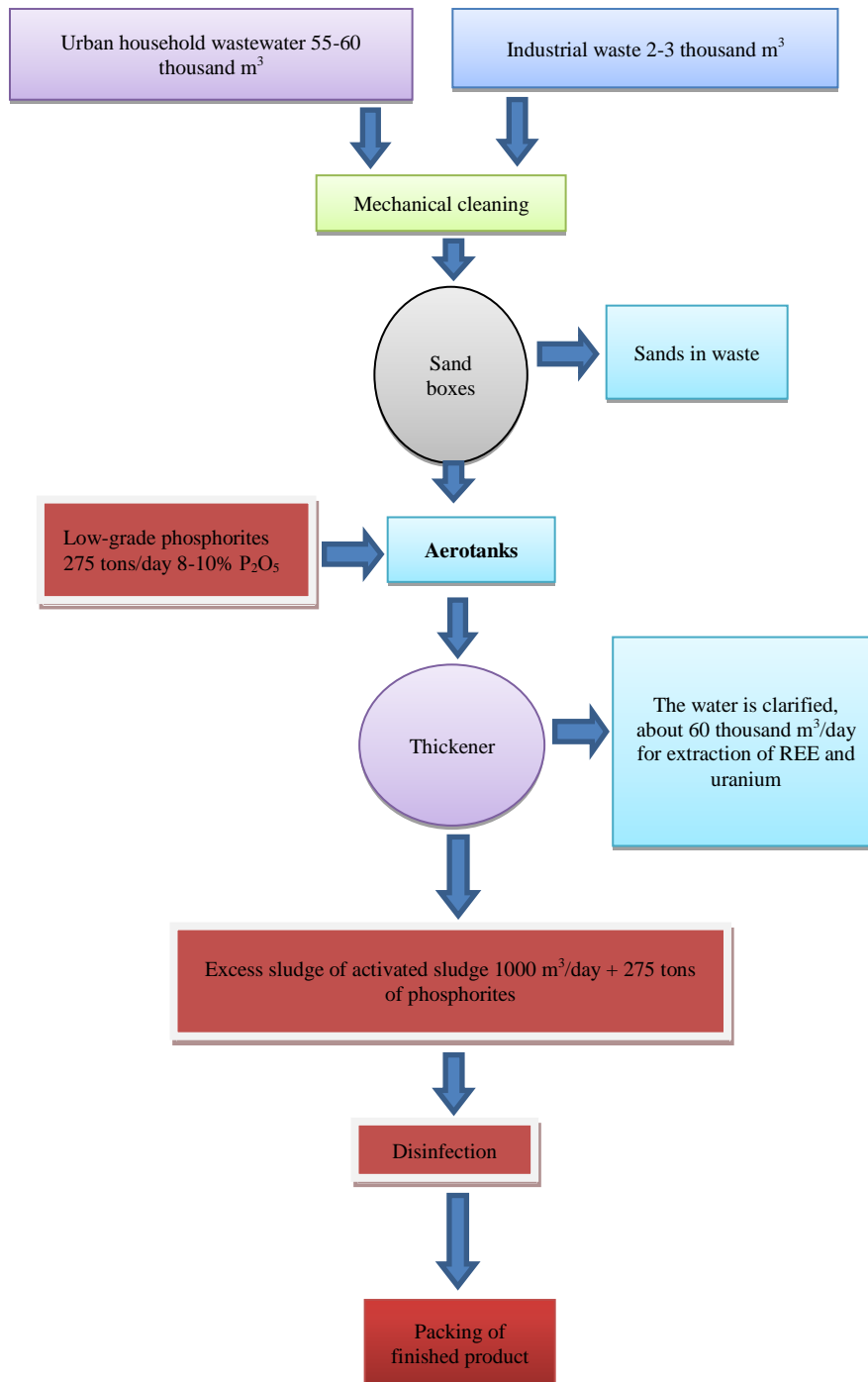


Fig. 1. Principal technological scheme for obtaining organomineral fertilizer.

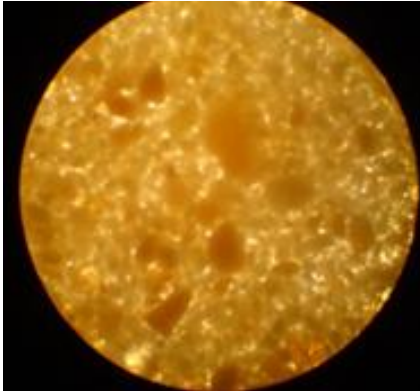


Fig. 2. Wrong angular form of phosphorite grains in the control variant (an increase of 150 times).

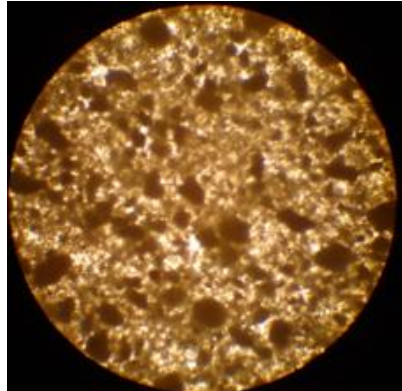


Fig. 3. Sample treated with activated sludge. Calcite particles are crushed and make up a matte background. Phosphorite grains look like rounded grains.

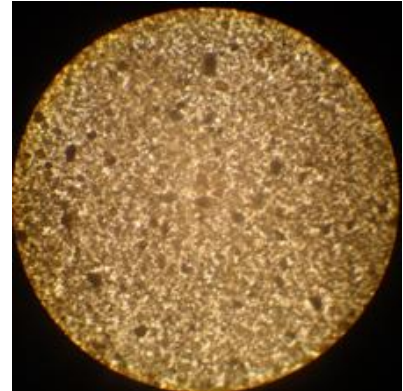


Fig. 4. Option treated with active silt, carbamide and air. The amount of phosphorite grains (2) increased with a parallel decrease in their size.

From Fig. 2, 3, 4, it can be seen that the particles of phosphorite grains also underwent destruction and turned from an irregular angular shape to round ones with a smaller size. Therefore, there is a real possibility of finding out phosphorites and an additional increase in the specific mass of P_2O_5 in the total fertilizer mass.

Analysis of stereoscopic microphotos (Figures 5-7) of calcite crystals showed a change in the morphological structure of calcite, where the calcite particles underwent a strong grinding and formed the basic powdered white bloom of the background (Fig. 7.). apparently, microorganisms for the lack of carbon organic compounds began to use calcite carbonates.



Fig. 5. Large pieces of calcite are noted in the control variant (an increase of 150 times).

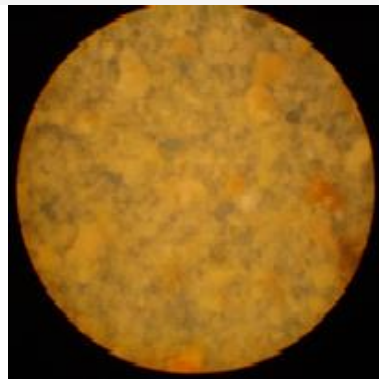


Fig. 6. Reduction in the size of calcite crystals in phosphorites when treated with activated sludge.

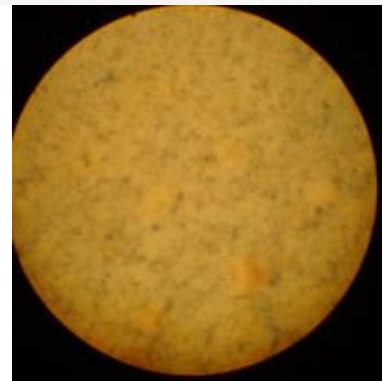


Fig. 7. Option treated with activated sludge, carbamide and air. Crystals of calcite crumbled.

Phosphorite grains in the control variant had a larger size and irregular shape. Depending on the duration of bacterial treatment, the form of phosphorite grains began to decrease (Figures) and take a spherical shape.

Crystals of calcite began to disintegrate and, under stereoscopic observation under a microscope, they resembled the kind of powdered phosphorite grains (Figures 5-7).

One of the options for enriching phosphorites, taking into account the grinding of calcite crystals and the change in the dimensions of phosphorite grains, is the possibility of their washing away from each other by a gravitational method. Fine calcite calcite can be separated from larger phosphorite grains.

Of the chemical elements necessary for plants, 16 main organonic organisms are distinguished: - carbon, oxygen, hydrogen, nitrogen; ash elements - phosphorus, potassium, calcium, magnesium and sulfur; trace elements - boron, molybdenum, copper, zinc, cobalt, manganese and iron. The place of one element can not be replenished by another, because each of them fulfills the specific physiological function assigned to it.



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Trace elements are not accidental ingredients of tissues and fluids of living organisms, but components of the naturally existing very ancient and complex physiological system involved in regulating the vital functions of organisms at all stages of development. Among the 15 vital elements, nine are cations-calcium (Ca^{2+}), sodium (Na^+), potassium (K^+), magnesium (Mg^{2+}), manganese (Mn^{2+}), zinc (Zn^{2+}), iron (Fe^{2+}), copper (Cu^{2+}) and cobalt (Co^{2+}). Six others are anions or are contained in complex anionic groups, chloride (Cl^-), iodide (I^-), phosphate (PO_4^{3-}), sulfate (SO_4^{2-}), molybdate (MoO_4^{2-}) and selenite (SeO_3^{2-}) [9]. Isolations of microorganisms contain a whole complex of organic compounds, consisting of vitamin-like substances, humic, abscisic, gibberellic and other acids in the form of growth stimulators and plant development. Thus, along with the enrichment of low-grade phosphorites with active silt microorganisms and their excreta, they will be enriched by additional stimulating compounds and microelements stimulating growth and development of plants [11].

Along with the proposed goal of obtaining a new azotofosforogo polimikroelementnogo organomineral fertilizer, given the results obtained with an additional amount of trace elements and allocated microorganisms organic growth stimulants, it is proposed a second embodiment - wherein the liquid phase separation may thickeners REE and uranium with thorium.

Thus, the development of a biotechnological method for destruction of low-grade phosphorites and their wastes is possible not only for the creation of a fundamentally new technology, but also economically promising for the recovery of phosphorites by gravity or flotation, with the parallel extraction of radioactive, rare and rare earth elements, but it also appears feasible for obtaining complex organomineral fertilizers.

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