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New Organic-Mineral Fertilizer Based on Low-Grade Phosphorites and Microflora of Activated Sludge

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The possibility of transforming a phosphorite rock containing less than 10% P_2O_5 into a full-fledged organic-mineral nitrogen–phosphorus multielement fertilizer with subsequent disinfection of the obtained product is determined. X-ray fluorescent analysis of phosphorites shows that its microelement composition in quantitative terms coincides with the plant one, and therefore, applying this fertilizer, the plant can get all the necessary minor nutrient elements. As revealed, for the samples with the liquid phase, microorganisms have the high activity in comparison with the control variants that is associated with the impact of a biogenic factor only. The ability of microorganisms of activated sludge to degrade insoluble compounds of phosphorite ore and slime and to transform their insoluble form into soluble one is shown. The process flow diagram and points of the addition of low-grade phosphorites and phosphorite slime to the primary and secondary sedimentation tanks tested at the operating biochemical treatment station are given. Thus, the obtained solid condensed deposits from low-grade phosphorites and sludge will contain up to 18–22% P_2O_5 , 12–14% nitrates and nitrites, 4–5% K_2O , and CaO and MgO, which are digestible together with the accompanying set of all trace elements by plants.

Визначено можливість перетворення фосфоритової породи, що містить менше 10% P_2O_5 , у повноцінне органо-мінеральне азотно-фосфорне багатоеlementне добриво з подальшою дезінфекцією одержаного продукту. Рентгенофлюоресцентна аналіза фосфоритів показала, що його мікроелементний склад в кількісному вираженні подібний до рослинного, а отже, застосовуючи це добриво, рослина може отримувати всі необхідні поживні мікроелементи. Виявлено, що для зразків із рідкою фазою мікроорганізми мають високу активність порівняно з контрольними варіантами, що пов'язане лише з впливом біогенного чинника. По-

казано здатність мікроорганізмів активного мулу піддавати деградації нерозчинні сполуки руди і шламу та трансформувати їх з нерозчинної форми в розчинну. Наведено технологічну схему та точки внесення низькосортних фосфоритів і шламу в первинні та вторинні відстійники, апробовані на діючій станції біохемічної очистки. Таким чином, одержані тверді конденсовані відкладення з низькосортних фосфоритів і шламу матимуть до 18–22% P_2O_5 , 12–14% нітратів і нітритів, 4–5% K_2O , а також CaO та MgO , що засвоюються рослинами разом із супутнім набором всіх мікроелементів.

Key words: activated sludge, low-grade phosphorite, slime, fertilizer, microflora.

Ключові слова: активний мул, низькосортні фосфорити, шлам, добриво, мікрофлора.

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1. INTRODUCTION

The Republic of Uzbekistan is a country of intensive consumption of phosphorus fertilizers. The problem of creating the new types of complex organic fertilizer with increased agro-ecological value and efficiency is currently relevant. The use of complex fertilizers containing a complete set of both macro- and micronutrients will significantly improve the productivity of agricultural production and product quality. On the other hand, this will significantly reduce the dose of fertilizer, balance the ratio of nutrients, and reduce the technological costs of their application and storage.

Many naturally occurring wastes can be used as secondary raw materials for the production of fertilizers, as well as means to increase of land productivity and crop yields. Among such types of organic waste, livestock waste (cattle manure, bird droppings), crop production, as well as industrial production (low-grade phosphorite of Central Kyzyl-Kum) can be highlighted. It should be noted that, at the Kyzyl-Kum Phosphoritic Plant (KPP) of Navoi Mining and metallurgical Combine (NMMC), during the enrichment of highly carbonated phosphorites of the Central Kyzyl-Kum, waste is formed in the form of off-balance ore with a content of 13–15% P_2O_5 and sludge phosphorites with a content of 8–12% P_2O_5 [1–7]. The total volume of accumulated waste phosphorites has already reached about 13 million tons.

At present, there is an acute question of finding new rational ways of processing low-grade phosphorites of the Central Kyzyl-Kum region into high-quality phosphorus-containing fertilizers.

The aim of this work is the usage of microflora of activated

sludge from biochemical treatment plants for municipal waste as an alternative for the recycling of low-grade phosphorite ores. For this, low-grade phosphorites and waste from the Kyzyl-Kum Phosphoritic Plant (KPP) of NMMC were taken as initial materials for obtaining a new type of organic-mineral fertilizer enriched with phosphorus and activated sludge. This makes it possible to transform phosphorite rock containing less than 10% P_2O_5 into a full-fledged organic-mineral nitrogen-phosphorus multielement fertilizer with subsequent disinfection of the obtained product.

An achievement of such a result is possible by transforming the indigestible P_2O_5 form to a form that is digestible for plants using the microflora of sewage treatment plants [8–12].

Thus, the objective of the present study was to study the chemical composition and microflora of activated sludge from the biochemical cleaning plant of JSC Navoiyazot and low-grade phosphorite rock from KPP NMMC, resulting from the addition of both low-grade ore and phosphorite wastes into the aerotanks of the station.

2. MATERIALS AND METHODS

Laboratory studies on the destruction of various types of minerals from low-grade phosphate of KPP NMMC using aerobic types of neutrophilic microorganisms of AS of the biochemical cleaning plant of JSC Navoiyazot were carried out at the Navoi State Mining Institute.

The object of the study was low-grade phosphorite ore and sludge from KPP NMMC with the following chemical composition (Tables 1, 2) similar to [13].

Analyses of the chemical composition of activated sludge and variants with a mineralized mass and phosphorite slime were performed at the Research Institute of General and Inorganic Chemistry of the Academy of Sciences of the Uzbekistan Republic.

For the cultivation of aerobic microorganisms of activated sludge

TABLE 1. The chemical composition of phosphorites of the Central Kyzyl-Kum.

No.	Compound name	Elements content, %	No.	Compound name	Elements content, %
1	P_2O_5	8–12.2	7	CO_2	8–15
2	Al_2O_3	1.5–3.0	8	Fluorine	1.8–3.2
3	SiO_2	6.0–8.0	9	SO_3	2.5–3.5
4	CaO	42–48.1	10	H_2O	10.0
5	MgO	2.5–3.5	11	Organic substance	2.8–3.0
6	Fe_2O_3	0.6–0.8	12	Insoluble residue	8.0–8.2

TABLE 2. Chemical composition of activated sludge from the shop of biochemical cleaning of JSC Navoiazot.

Composition of activated sludge, %								
Moisture	Organic substances	Nitrogen	P ₂ O ₅	K ₂ O	CaO	MgO	Ash	Dry weight
95.38	2.99	0.12	0.14	0.01	0.17	0.02	1.41	4.67
In conversion to oven-dry basis, %								
10.05	28.54	11.44	13.35	0.95	16.21	1.90	13.44	44.26

involved in the processing ore and slime of phosphorites, reactors were created, resembling in their design aerotanks. 500 grams of phosphorite ore or slime per 2 litres of the liquid phase or solid precipitate of activated sludge were loaded into these reactors with the same ratio. In other words, the ratio of solid (*S*) to liquid (*L*) *S:L* = 1:4 was completed.

The following variants for ore and sludge are drawn up (Table 3).

The reactor, created in the laboratory, simulated the processes occurring in the aeration tanks. Variants for the interaction of activated sludge with phosphorites were kept for 14 days.

In variants of the samples, the total content of P₂O₅, digestible by Trilon B and in 2% citric acid solution was determined according to a known method described in detail [14]. Studies were carried out according to GOSTs: ash content in line with GOST 26714-85, humidity in line with GOST 26712-94, organic substance content in line with GOST 27980-80 [15–17]. The solid phase (after the separation of alkaline-soluble organic substances from it) is the final decomposition product of organic substances. It was thoroughly washed with distilled water and then dried to a constant weight, and the yield of organic compound was determined.

After bacterial leaching out for 14 days, samples of the liquid and solid phases were sent to the State Unitary Enterprise ‘Uzbek Research and Development and Survey Institute of Geology and Nonferrous Metallurgy’ (‘UzGEORANGMETLITI’) for study-

TABLE 3. Variants of experiments on phosphorite ore and slime.

Variants title	Ratio <i>S:L</i>	Variant 1	Variant 2	Variant 3	Variant 4	Variant 5
Phosphorite ore (PhO)	1:4	Water + PhO	AS + PhO _(l.ph.)	AS _(s.res.) + PhO	AS _(l.ph.) + PhO + O ₂	AS _(s.res.) + PhO + O ₂
		Variant 6	Variant 7	Variant 8	Variant 9	Variant 10
Phosphorite slime (Sl)	1:4	Water + Sl	AS + Sl _(l.ph.)	AS _(s.res.) + Sl	AS _(l.ph.) + Sl + O ₂	AS _(s.res.) + Sl + O ₂

Note: l.ph.—liquid phase, s.res.—solid residue.

ing by an universal x-ray fluorescent energy-dispersive spectrometer BRA-135F, which allows us to determine simultaneously more than two dozen chemical elements during a time not exceeding 300 seconds in a wide range of concentrations ranging from hundreds to ppb. BRA-135F analyses samples in the solid, powdery, and liquid states, as well as deposited on the surface or the filters.

3. RESULTS AND DISCUSSION

Kyzyl-Kum phosphorites consist mainly of phosphoritized faunal residues bonded with fine-grained calcite cement [18, 19]. Foraminifera with shell sizes from 0.04 to 0.5 mm predominate among these remains of fauna. Isotropic and slightly crystallized phosphorite with point inclusions of calcite fills the internal cavities of their shells. Relic calcite preserved from phosphorite substitution sometimes forms the shell and internal partitions of sinks as well. In the scientific and technical literature, such a kind of calcite, which is located inside phosphate formations, is called 'endo calcite' and composing the cement of the rock 'exo calcite' [20, 21]. The third form of calcium carbonate is found in the form of a phosphate mineral isomorphically entering the crystal lattice. Phosphatized residues of other organisms play the subordinate role; they are the sections and nuclei of pelecites, gastropod shells up to 5 mm in size constituting up to 5–10% of the breed, elongated conical Pteropods up to 4–5 mm, sea urchin needles, fish scales, *etc.* Cementation is often fragile; therefore, many phosphorites easily disintegrate under mechanical action, especially after soaking in water. Among organic residues, primary phosphate materials, namely, shark teeth, vertebrae and small (of a few millimetres) fragments of the marine animals' bones, are present in a small amount. Cement for phosphate is fine-grained calcite mixed with clay and phosphate-clay material. In the bottom part of the second stratum, the cement is strong crystalline; it is represented by calcite and gypsum, sometimes with a siliceous component. The results of the mineralogical study of granular phosphorite ores indicate the monotony of their composition [22–24].

The main phosphate minerals are francolite (fluorocarbonate) and calcite included in the ore by 80–90%. Francolite contains on average of about 42.1% P_2O_5 , 55.4% CaO, 1.2% F, 2.3% Cl, 0.6% H_2O ; the sum of rare elements (TR) reaches 0.03%. Such ores have the following average mineral composition (weight, %): francolite—56.0; calcite—26.5; quartz—7.5; hydromica minerals and feldspars—4.0–4.5; gypsum—3.5; goethite—1.0; zeolite < 1.0; organic matter—about 0.5 [13, 22, 24].

The characteristics of the material composition of options with phosphate ores and slime with activated sludge are given in Table 4.

X-ray fluorescent spectral analysis of samples treated with activated sludge showed the presence in the solid phase of 25 elements, including those related to rare and scattered metals. For all samples, phosphorus is converted to a water-soluble form. The amount of calcium compounds in the solid phase ranged from 19.5085 to 16.32335 mg/l that indicates a partial transition of calcium compounds to the soluble form. In addition, the calcium content in the ore was higher (18.8715 mg/l) than in the sludge (17.8540 mg/l). With this fact, a partial dissolution of calcium compounds by water is evident. The amount of Fe compounds in the ore composition is minimal of 0.3398 mg/l, and in the slime composition, it increases to 1.4644 mg/l.

Adding a solid residue of activated sludge, the amount of Fe in variants 5 and 10 increases and is of 1.9544 and 2.5010 mg/l, respectively, *i.e.*, Fe is extracted from active sludge. For the remaining trace elements involved in the physiological reactions of living organisms, the contents of Ti, Mn, Cu, Zn and partially Mo elements increase. Some decrease in the number of elements in the solid phase is noted in the variants with vanadium. Here, it is observed an ambiguous picture. If the amount of vanadium in phosphate ore increased from 0.0040 to 0.0045 mg/l, for the variants with phosphate slime, its amount decreased from 0.0045 to 0.0040 mg/l, *i.e.*, under the influence of microflora of activated sludge, partial dissolution of vanadium occurs in the slimes, but in the composition of the ore vanadium, perhaps, it is in insoluble form. Similar results are noted for Ni, Ag, Sb, Ba, and Pb. For the others elements (Cr, Co, Ga, Ge, Ni and W) significant changes were not noted.

X-ray fluorescent analysis of phosphorites showed that its microelement composition in quantitative terms coincides with the plant one, which contains of about 75 elements of the D. I. Mendeleev periodic system, and therefore, applying this fertilizer, the plant can get all the necessary minor nutrient elements. Among the chemical elements necessary for plants, 16 ones are following as major *organogenesis* highlighted: carbon, oxygen, hydrogen, and nitrogen; ash elements: phosphorus, potassium, calcium, magnesium, and sulphur; trace elements: boron, molybdenum, copper, zinc, cobalt, manganese, and iron. The place of one element cannot be filled by another one, because each of them performs its own specific physiological function.

Trace elements are not random ingredients of tissues and fluids of living organisms, but components of a naturally existing ancient and very complicated physiological system that is involved in regulation of the vital functions of organisms at all development stages. Among the vital elements, there 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+}).

TABLE 5. Chemical composition and characteristics of promising options for the production offering.

Chemical composition of samples	Chemical composition of phosphorite ore at interaction with liquid phase and solid residue (%)			Chemical composition of slime at interaction liquid phase and solid residue (%)		
	PhO + H ₂ O	PhO + AS _(l,ph.) + H ₂ O + O ₂	PhO + AS _(s,res) + H ₂ O + O ₂	Sl + H ₂ O	Sl + AS _(l,ph.) + H ₂ O + O ₂	Sl + AS _(s,res.) + H ₂ O + O ₂
Moisture	75.68	70.73	71.94	75.56	74.71	71.68
Organic substance	0.12	4.31	5.18	0.08	2.24	5.05
Ash	23.5	23.96	22.88	24.08	23.05	23.27
Nitrogen total	0.03	0.12	0.14	0.02	0.13	0.15
P ₂ O ₅ total	3.54	3.58	3.76	2.16	2.46	2.62
P ₂ O ₅ dig. by trilon B	1.53	1.79	2.06	0.25	1.19	1.16
P ₂ O ₅ dig./P ₂ O ₅ total	36.26	50.12	56.38	26.25	48.31	44.35
CaOtotal	10.65	10.66	10.89	10.01	10.36	10.83
CaO _{dir.} per 2% citric acid	8.14	8.33	9.33	1.47	6.43	8.51
CaO _{dir.} /CaOtotal	76.52	78.22	85.66	74.35	81.32	78.62
MgO	0.31	0.33	0.36	0.31	0.34	0.38
Dry weight	24.03	26.27	25.06	26.33	25.29	28.32

Note: *l.ph.—liquid phase; dig.—digestible; s.res.—solid residue.

Microorganism excretions contain of a whole complex of organic compounds consisting of vitamin-like substances, humid, abscisic, gibberellic and other acids, which can be as the growth and development stimulants of plant [25, 26].

Thus, along with the enrichment of low-grade phosphorites with microorganisms of activated sludge and their secretions, they will be enriched with additional organic compounds and trace elements, which stimulate growth and development of plants. The results of chemical analysis of phosphorite and slime samples treated with activated sludge are presented in Table 5. Samples were taken directly from the reactors. Therefore, they have a high humidity in the range of 70–75%. As we can see from Table 5, the most optimal are 4th and 5th variants. The amount of organic substance, nitrogen, phosphorus, calcium and magnesium in the variants with the use of a solid residue significantly exceeds the amount of these compounds in a liquid phase. However, the most interesting fact is following: in the samples with the liquid phase, there is the high activity of microorganisms in comparison with the control variants that is associated with the impact of a biogenic factor only. As a result, there was a transformation of mineral compounds of ore and slime, and these compounds turned from the insoluble form to the soluble one.

Table 6 shows the results of measuring the density of phosphorite ore and slime after treatment with activated sludge and pH value at the ratio of solid (*S*) to liquid (*L*) 1:4. The decrease in density of the medium from 1184.62 g/l to 1092.2 g/l is apparently due to a decrease in carbonates amount in the calcite mineral composition, in which microorganisms for their growth and development use the carbonium. Particularly intense decrease in density was noted in 4th and 5th variants. This circumstance allows under production conditions to use low-grade phosphorites by direct addition into aerotanks. Due to their low density, they are capable to be carried by a water stream into thickeners to separate the liquid and solid phases. The pH of the medium in all the studied variants was mostly neutral and changed insignificantly within 6.60–7.14. This fact confirms that the neutral and alkaline phosphorites, calcium phosphate are present in the predominant amount. Therefore, in the biochemical processing of the household and industrial effluents in the amount of 55–60 thousand m³/day (Fig. 1), phosphorite slimes can be loaded into primary clarifiers, because, due to their low density and fine dispersion, they can freely transfer to sludge accumulators.

Thus, we carried out the pilot test of phosphorite slime at the treatment plant in Zarafshan city, where 4 tons of phosphorite slime were loaded into the primary clarifiers.

TABLE 6. The change in the density of phosphorite ore and slime after treatment with activated sludge.

Variants	Density g/l	pH	Variants	Density, g/l	pH
Control			Control		
Phosphorite ore + Water	1184.62	6.83	Phosphorite slime + Water	1170.58	7.00
Phosphorite ore + activated sludge (l.ph.)	1132.82	6.82	Phosphorite slime + activated sludge (l.ph.)	1164.7	6.88
Phosphorite ore + activated sludge (s.res.)	1131.37	6.60	Phosphorite slime + activated sludge (s.res.)	1150.61	6.69
Phosphorite ore + activated sludge (l.ph.) + O ₂	1105.54	7.05	Phosphorite slime + activated sludge (l.ph.) + O ₂	1091.9	7.14
Phosphorite ore + activated sludge (s.res.) + O ₂	1099.01	6.86	Phosphorite slime + activated sludge (s.res.) + O ₂	1092.2	6.86

Note: l.ph.—liquid phase; dig.—digestible; s.res.—solid residue.

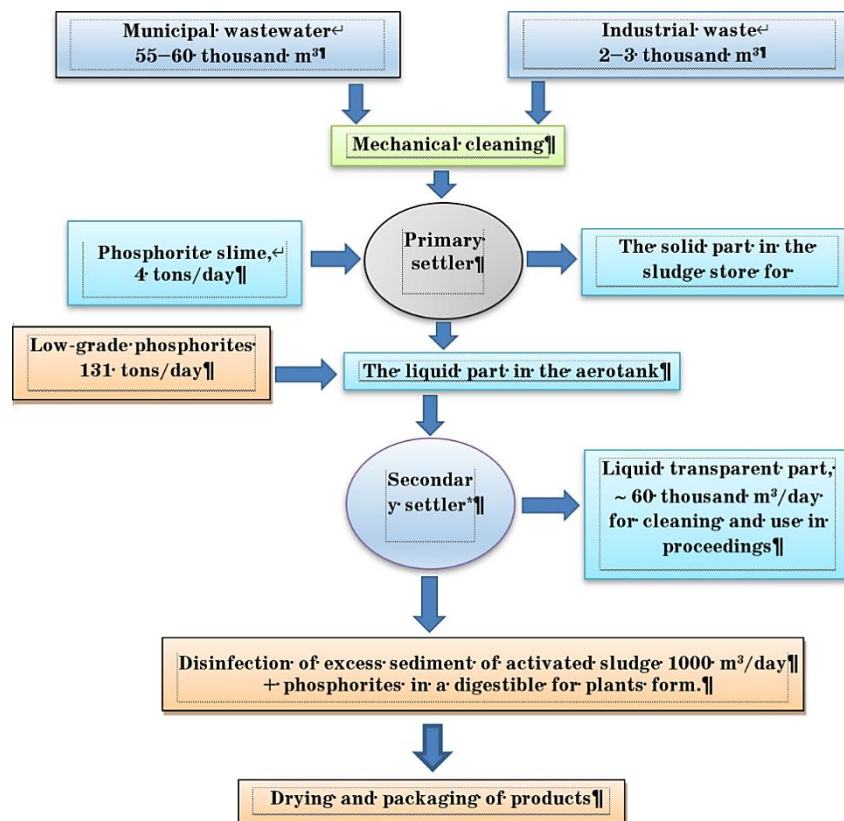


Fig. 1. The basic process flow diagram of obtaining an organic fertilizer (*coagulants can be added).

After 24 hours, they were pumped out to the silt reservoirs. Then, they were irrigated with excess activated sludge from aerotanks. Considering the daily volume of the biochemical treatment plants, it is possible to adjust the supply of phosphorite slime. In this case, daily up to 12 tons of phosphorite slime can be loaded into the primary clarifiers of the Zarafshan treatment plant.

Low-grade phosphorites are recommended to be loaded into aerotanks, since they have a higher density (Table 7). By a flow of supplied air, phosphorites with a higher density will be easily leached from the aerotanks and get into the secondary clarifiers. Then, the extraction of condensed sediments will be made from secondary clarifiers. Every twenty-four hours, about 275 tons of low-grade phosphorites can be loaded into aerotanks. Thus, up to 90 thousand tons of phosphorites with a content of 8–10% P_2O_5 (Fig. 1) can be recycled per year, followed by the finished-product output.

TABLE 7. Daily load of phosphorites into aerotanks in the Navoi and Zarafshan cities.

Name	Daily waste volume, m ³	PhO and SI for aerotanks, t/day	Received fertilizer, t/year	Discharge of excess AS, t/day	PhO and SI for excess AS, kg	Received fertilizer, t/year
Biochemical treatment plant of JSC Navoiazot	52 500	131.25	47 906	6	1500	5 475
Biochemical treatment plant of Zarafshan city	49 400	123.50	45 077	5	1250	4 562
Sewage treatment on KPP	150	37.50	13 687	0.02	250	91.3
	102 050	292.25	106 670		3 000	10 128

Obtained after primary and secondary clarifiers, solid condensed sediments with low-grade phosphorites and slime added to them will have up to 18–22% P_2O_5 , 12–14% nitrates and nitrites, 4–5% K_2O , and CaO and MgO are digestible by plants, together with the accompanying set of all trace elements.

In this way, it is possible to obtain the high-quality new fertilizer, referred to as organic-mineral nitrogen–phosphorus polymicroelement fertilizer [26].

In all major cities of the Uzbekistan Republic, there are the biochemical treatment plants of municipal waste. It is not known how excess activated sludge is recycled at these plants. Therefore, application of low-grade phosphorites at these plants will allow us to obtain the organic fertilizer. There would be a real opportunity to provide phosphorus fertilizers in all Republic regions, without spending additional funds on the import of mineral phosphorus-containing fertilizers.

For the daily load of aerotanks of municipal waste-treatment plants in the Navoi region, calculations were made (Table 7). It was shown that, for the daily load, with a ratio of phases $S:L = 1:4$, up to 92 thousand tons of phosphorus-containing organic-mineral fertilizers per year can be obtained. At KPP NMMC, slimes are released as waste with a content of up to 10.5% P_2O_5 . Such slime wastes were accumulated in the first slime storage facility (dried out) with 16730 m^3 , in the second one, with 3000 m^3 , in the third one, with about 7000 m^3 . Considering the size of the slime particles and their ability to be in the peptized state for a long period of time, it is proposed to add these slimes into aerotanks to activate by the microflora of biochemical treatment plant, since they will not linger in them.

Thus, the Navoi region with the effective use of low-grade phosphorite ores and slimes at KPP NMMC can satisfy its regional needs for high-quality organic-mineral fertilizers, but also sell the finished product to third-party organizations as well.

4. CONCLUSION

Based on the provision of agricultural production of the Uzbekistan Republic with own phosphate fertilizers by 30–32%, a radically new solution was found for obtaining a new type of phosphorus-containing fertilizer, where the microflora of activated sludge from biochemical treatment plants is used for transforming the insoluble low-grade phosphorite in organic-mineral nitrogen–phosphorus polymicroelement fertilizer. As a result of studying the degree of production load at the biochemical treatment plants of Navoi and Zarafshan cities, it is proposed addition of the low-grade phospho-

rites at the ratio of the solid-to-liquid phases $S:L = 1:4$ and, in this way, to get the new type of fertilizer without prejudice to the sewage treatment plant.

For all agro-industrial regions of the Uzbekistan Republic, it is recommended to use this technology to ensure their own needs in phosphorus-containing organic fertilizers.

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