DOI: 10.24412/2181-144X-2020-2-59-67 Temirov U.Sh., Doniyarov N.A., Usanbaev N.Kh., Tagaev I.A., Juraqulov B.A.

ORGANOMINERAL FERTILIZERS BASED ON SEDIMENTS OF WASTE WATERS AND PHOSPHORITES OF CENTRAL KYSILKUM

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Annotation. This paper presents the results of incomplete decomposition of Central Kyzylkum non-conditioned phosphorites with sulfuric acid (30-70% stoichiometric norm relative to CaO) and its subsequent processing into phosphorus-humus fertilizers with active turbidity. Optimal ratios of starting materials in the production of phosphorus-humus fertilizers on the basis of active turbid and non-conditioned phosphorites and the rate of sulfuric acid for the decomposition of Central Kyzylkum non-conditioned phosphorite were determined.
Keywords: Active sludge, simple phosphorite flour, sulfuric acid, phosphorus, calcium, organic mineral fertilizer.

ОҚАВА СУВЛАР ЧЎКИНДИЛАРИ ВА МАРКАЗИЙ ҚИЗИЛҚУМ ФОСФОРИТЛАР АСОСИДА ОРГАНИК МИНЕРАЛ ЎҒИТЛАР

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Аннотатсия. Ушбу мақолада Марказий Қизилқум фосфоритларни сульфат кислота билан тўлиқсиз меъёрда парчалаш (СаО нисбатан 30-70% стехиометрик меъёрда) ва сўнгра уни фаол лойқа билан фосфор-гумусли ўғитларга қайта ишлаш натижалари келтирилган. Фаол лойқа ва фосфоритлар асосида фосфор-гумусли ўғитлар олишда дастлабки моддаларнинг мақбул нисбаталари ва Марказий Қизилқум фосфоритни парчалаш учун сульфат кислотанинг меъёри аниқланган.
Калит сўзлар. Фаол лойқа, оддий фосфорит уни, сульфат кислота, фосфор,калтций, органоминерал ўғитлар.

ОРГАНОМИНЕРАЛЬНЫЕ УДОБРЕНИЯ НА ОСНОВЕ ОТЛОЖЕНИЙ СТОЧНЫХ ВОД И ФОСФОРИТОВ ЦЕНТРАЛЬНОГО КИСИЛКУМА

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Аннотация. В данной работе представлены результаты неполного разложения фосфоритов Центральных Кызылкумов серной кислотой (стехиометрическая норма 30-70% по CaO) и их последующая переработка в фосфорно-гумусовые удобрения с активным помутнением. Определены оптимальные соотношения исходных веществ при производстве фосфорно-гумусовых удобрений на основе активной мутности и фосфоритов и нормы серной кислоты для разложения фосфоритов Центральных Кызылкумов.
Ключевые слова: активный ил, простая фосфоритная мука, серная кислота, фосфор, кальций, органо-минеральные удобрения.

In terms of chemical composition, the precipitates formed during the treatment of urban wastewater are classified as raw materials for the production of organic fertilizers. These sediments contain a wide range of various organic and inorganic substances of biogenic and abiogenic origin, including toxic elements, pathogenic microorganisms, helminth eggs, etc. Despite the presence of pollutants in sediments, they are characterized by a high content of many elements for plant nutrition - up to 50% organic matter, 1-2% of total nitrogen, 3-5% of phosphorus, as well as other macro- and microelements that can be converted into an accessible and safe form for plants. Agricultural use of sewage sludge (SS) is limited by the presence of heavy metals in them, the amount of which can exceed the permissible concentration. However, in recent years, due to the improvement of wastewater treatment technologies and a decrease in emissions due to the reduction of a number of industries, the newly formed sludge has a lower content of heavy metals. Therefore, many research institutions are developing efficient, environmentally friendly technologies for the production and use of fertilizers based on sewage sludge and their application, taking into account the doses, timing of application and the selection of appropriate crops. The use of SS after rational processing for fertilizers will reduce the deficit of mineral fertilizers and humus in soils [1,2]. Technological schemes of urban wastewater treatment operating in cities of different countries have a similar structure. However, the methods of disposal and recycling of waste generated in the process of water treatment are very diverse. The work [2] presents the results of an experimental study of the process of anaerobic microbiological destruction of sludge sediments with the reduction and removal of phosphorus compounds from them. The regularities and mechanisms of biochemical transformations of phosphorus compounds in silt sediments during processing have been determined. Shown is a process flow diagram for the extraction of phosphorus compounds from sludge sediments and waste streams In works [3,4], an analysis of existing methods of wastewater sludge disposal is given, according to which the most effective are thermal methods of SS utilization with the subsequent use of secondary waste in the production of building materials, and the most undesirable waste management method is the method of wastewater sludge disposal on sludge ponds or polygons. The popularity of this wastewater sludge management method is due to both its ease of use and its low cost. Sewage sludge deposition facilities cause great damage to the environment and occupy large areas. Therefore, a number of European countries are legally prohibited from depositing precipitation in areas. The work [5] also indicates the advantages of wastewater sludge processing in order to obtain products used in industrial production and heat power engineering, due to the absence of strict sanitary restrictions on the presence of toxic compounds of the resulting products. The work [2] shows the state of disposal and recycling as well as use by country (table 1). The table clearly shows that the main amount of precipitation is used in agriculture, although many works indicate that incineration is considered a more rational method for processing SS.

In works [6, 7] practically all possibilities of utilization and processing are given, as well as their use as fertilizer in liquid, dehydrated, dry forms, obtaining feed products based on excess sludge, obtaining commercial products by pyrolysis, (pyrocarbonate, resin, gasoline , kerosene, wax), production of commercial products (soap and fats), production of materials for the construction industry (ash for cement additives), production and utilization of digester gas (biogas) in installations for obtaining thermal, mechanical and electrical energy, preparation of fuel briquettes from raw precipitation and activated sludge. It is also shown that a large amount of excess activated sludge is formed during biological wastewater treatment. Since the bacterial nature of activated sludge determines the high content of protein substances, amino acids, microelements, B vitamins, including B_{12}, this product is highly effective in feeding animals, birds, fish, fur animals. In many countries of the world, one of the areas of wastewater sludge use is their processing into fertilizers with the addition of lime, peat, manure, sawdust by composting [8-10]. In [11], in order to study the effect of sewage sludge on the yield of agricultural crops and their quality, a field experiment was laid. In the experiments, two types of sediments were studied - long-term storage and fresh, used against the background of liming and without it. The introduction of sewage sludge provided a reliable increase in yield in almost all variants of the experiment. SS contributed to an increase in the yield of hay by 1.3-1.6 times

Table 1.

The main methods of disposal of sewage sludge (in%) in modern conditions

Country	Used in agriculture	Landfill disposal	Incineration	Discharge into the sea, ocean
England	53	16	7	24
Austria	20	49	31	-
Germany	25	55	15	5
Denmark	45	28	18	9
USA	25	25	35	15
Italy	20	60	-	20
Finland	40	41	-	19
Switzerland	50	30	20	-
Sweden	60	30	-	10
France	23	46	31	-

compared with the control option, and the combination of lime with the introduction of sewage sludge was more effective. From the above, it can be seen that one of the main ways of using SS is their processing for fertilizers. It should be noted that at present in many countries of the world there is a decrease in the resources of phosphate raw materials and the content of humic substances in soils cultivated by agricultural crops. In 2018, the enterprises of Uzkimyosanoat JSC of the Republic of Uzbekistan produced 153.8 thousand tons of phosphate fertilizers (in terms of 100% P_2O_5). And the need for agriculture is 691.7 thousand tons of P_2O_5. These figures indicate that the provision of agriculture with phosphate fertilizers is insufficient. At present, at the Kyzylkum phosphorite plant, during the enrichment of highly carbonated phosphorites of the Central Kyzylkum, waste is generated 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. The total volume of accumulated waste phosphorites already reaches 15 million tons. In the conditions of an acute shortage of high-quality phosphate raw materials, the most affordable way to use substandard phosphorites is its combined processing with sewage sludge. Combined processing of sewage sludge and substandard phosphorites allows using the capabilities of activated sludge microorganisms to convert indigestible forms of phosphorus of substandard phosphorites into an assimilable form for plants, since microorganisms contained in sewage sludge are able to use many minerals, including phosphate, for their growth and development, in addition to this, sewage sludge contains a significant amount of carboxylic acids capable of binding calcium ions contained in partially degraded phosphorites with mineral acids. That is, under the influence of organic acids formed during the decomposition of sewage sludge, phosphorus, which is part of substandard phosphorites, passes from an indigestible form to a form assimilable for plants and thereby will exhibit its fertilizing properties. In addition, activated phosphorite with mineral acids binds $(NH_4)_2CO_3$, free NH_3 and other organic volatile organic substances into non-volatile forms. To study the processes of obtaining organomineral fertilizers, wastewater sludge from the city of Navoi was used with the following composition (wt.%): Moisture - 65.43; ash - 9.74; organic substances - 24.83; humic acids - 3.05; fulvic acids - 7.47; water-soluble organic substances - 2.13; P_2O_5 - 1.39; N 1.17; K_2O - 0.44; CaO - 4.14 [12-15].

Table 2 shows the results of mass spectrometric analysis (ICP – MS) of sewage sludge ash. It follows from the table that the sewage sludge contains in its composition a number of trace elements necessary for the growth and development of plants.

Table 2

Results of mass spectrometric analysis of ash from sewage sludge in the city of Navoi

Name and content of elements t/year									
Poultry manure ash composition									
Li 600	Si 100000	B 3000	Na 40000	Mg 30000	Al 80000	P 62310	K 32000	Ca 303608	Sr 2000
Mn 1000	Fe 11312	Co 80	Ni 130	Cu 1000	Zn 30	Mo 1,46	Ag 3,01	Hg 2,0	Ti 500

Simple phosphate rock (SPR) was used as a phosphate raw material. It was ground to a particle size of 0.25 mm before use. The composition of the SPR is given in table. 3.

Table 3

Chemical composition of phosphorite

Content of components, weight. %								
P_2O_5	CaO	Al_2O_3	Fe_2O_3	MgO	F	CO_2	SO_3	H.O.
16,6	48,62	0,98	0,78	1,42	1,48	13,02	1,09	4,85

From table. 3, it can be seen that SHF is characterized by a low phosphorus content (16.6% P_2O_5), a high carbonate content (13.02% CO_2) and a high calcium modulus (CaO : P_2O_5 = 2.93). Chemical analysis of sewage sludge from the city of Navoi, sludge phosphorite and products of their processing was carried out by the methods below. Humidity was determined according to GOST 26712-85, ash content according to GOST 26714-85 and organic matter according to GOST 27980-80. The amount of the water-soluble fraction of organic substances extracted from the products with water was determined by filtration and evaporation in a water bath, drying the solid residue to constant weight, followed by its combustion to determine the ash content and subtract it. Humic acids were isolated by treating the products with 0.1 N alkali solution followed by acidification of the solution with mineral acid [16]. The solid phase after separation of alkali-soluble organic substances from it contains residual organic matter. It was thoroughly washed with disc water, dried to constant weight, and the content of organic matter was determined. The difference between the amounts of alkali-soluble organic substances and humic acids gives us the fulvic acid content. The determination of all forms of P_2O_5 was carried out by the gravimetric method by precipitation of a phosphate ion with a magnesian mixture in the form of magnesium ammonium phosphate, followed by calcining the precipitate at 1000-1050 ° C in accordance with GOST 20851.2-75. The assimilable forms of P_2O_5 were determined by their solubility both in 2% citric acid and in 0.2 M Trilon B. The determination of the CaO content was carried out complexometrically: by titration with 0.05 N solution of Trilon B in the presence of the fluorexon indicator. For the decomposition of slurry phosphorite, sulfuric acid with a concentration of 92% was used. The rate of sulfuric acid was varied in the range of 30-70% of the stoichiometry for the decomposition of CaO of phosphate raw materials. The experiments were carried out in the following way, sulfuric acid was slowly poured into a glass reactor, in which a sample of phosphate raw materials was located. The duration of the interaction of the components was 30 minutes, after which the waste water sludge was added to the pulp, and stirring was continued for 60 minutes. Drying was carried out at 80 ° C to a moisture content of 10-15% in the finished product. The processing of the products of sulfuric acid decomposition by sewage sludge was carried out in the range of weight ratios of sewage sludge to sludge phosphorite from 100: 10 to 100: 40. The results are shown in Tables 4-6 4. From Tables 4-6, it can be seen that the higher the rate of sulfuric acid and the more wastewater sludge is taken, the less P_2O_5total in the product, but the greater the relative content of the assimilable form of P_2O_5, the water-soluble form of CaO, organic substances and humic substances. As you can see from the table. 4, when the ratio of SS: SPR = 100: 10 and the rate of sulfuric acid is 30% of the stoichiometry, an organomineral fertilizer containing P_2O_5total is obtained. – 5.17%; P_2O_5 ass. by lim. to-those – 2.24%, P_2O_5 ass. : P_2O_5total. – 39.25 %; P_2O_5 water : P_2O_5total. - 4.78%; CaOtotal. - 14.99%; CaOwater. - 1.73%, CaOass. : CaOtotal. – 60.59%; CaOwater. : CaOtotal. – 11.53%, organic matter – 42.83%; humic substances - 23.70%; humic acids – 5,63%, fulvo acid - 13,78%, water soluble organic matter - 3,93%, SO3общ. – 3,63%, SO3вод. – 1,11%, азот – 2,16%. With the same ratio of SS to mineralized mass, but at a rate of 70% acid, a fertilizer containing P_2O_5total. – 5.43%; P_2O_5 ass. by lim. to-those – 4.66%; P_2O_5 ass. : P_2O_5total. – 85.78%; P_2O_5 water : P_2O_5total. – 40.28%; CaOtotal. - 13.39%; CaOwater. - 6.07%, CaO ass. : CaOtotal. – 40.28%; CaOwater. : CaOtotal. – 50.47%, organic matter - 40.72%; humic substances - 22.53%; humic acids – 5,32%, fulvo acid – 13,04%, water soluble organic matter– 3,80%, SO3total. – 7,73%, SO3water. – 2,01%, nitrogen – 2,04%.

At the request of agriculture, phosphate fertilizer must have a high content of the total and assimilable forms of P_2O_5, and the relative content of the water-soluble form of P_2O_5 must be at least 50%. Based on this requirement, the optimal rate of sulfuric acid for decomposition non-codic phosphorites is 40% of stoichiometry, and the optimal ratio of SS: substandard phosphorite can be considered 100 : 30, at which the relative content of P_2O_5ass. is 51.36%. In this case, organomineral fertilizer using SHF has a composition (wt%): P_2O_5total. – 7.76; P_2O_5 ass. – 3.65; CaOtotal. – 21.59; CaOwater. – 5.95; organic substances - 27.85; humic substances - 15.41. Thus, the studies carried out convincingly show that on the basis of sewage sludge after dehydration, or without it, using a small amount of sulfuric acid or other mineral acids used in the production of mineral fertilizers, it is possible to intensively process sewage sludge and sludge phosphorite into organomineral fertilizers.

Table 4

The composition of organomineral fertilizer obtained by activation of phosphorite with sulfuric acid and sewage sludge from the city of Navoi

Mass ratio of SS to phosphorite	P_2O_5 total%	P_2O_5 усв. %	P_2O_5 water.%	CaO total%	CaO усв. %	CaO water.%
H_2SO_4 norm from stoichiometry for decomposition CaO , 30 %						
100 : 10	5,71	2,24	0,27	14,99	9,08	1,73
100 : 20	7,06	2,72	0,39	19,29	11,29	2,49
100 : 25	7,55	2,85	0,48	20,85	11,80	2,81
100 : 30	7,96	2,97	0,57	22,16	12,23	3,20
100 : 40	8,61	3,11	0,70	24,21	12,90	3,74
H_2SO_4 norm from stoichiometry for decomposition CaO , 40 %						
100 : 10	5,64	2,85	0,42	14,80	8,14	3,40
100 : 20	6,92	3,39	0,59	18,90	10,24	4,77
100 : 25	7,38	3,55	0,70	20,37	10,86	5,37
100 : 30	7,76	3,65	0,80	21,59	11,26	5,95
100 : 40	8,36	3,82	0,94	23,51	11,90	6,91
H_2SO_4 norm from stoichiometry for decomposition CaO , 50 %						
100 : 10	5,57	3,55	0,95	14,61	7,22	4,78
100 : 20	6,78	4,15	1,30	18,52	8,90	6,48
100 : 25	7,21	4,31	1,47	19,92	9,45	7,25
100 : 30	7,57	4,39	1,65	21,07	9,83	7,87
100 : 40	8,12	4,61	1,97	22,85	10,28	9,02
H_2SO_4 norm from stoichiometry for decomposition CaO , 60 %						
100 : 10	5,50	4,15	1,53	14,43	6,40	6,10
100 : 20	6,65	4,86	2,02	18,16	7,62	8,02
100 : 25	7,06	5,03	2,24	19,48	7,97	8,71
100 : 30	7,39	5,08	2,42	20,56	8,26	9,47
100 : 40	7,90	5,32	2,77	22,23	8,49	10,86
H_2SO_4 norm from stoichiometry for decomposition CaO , 70 %						
100 : 10	5,43	4,66	2,09	14,25	5,74	7,19
100 : 20	6,52	5,44	2,68	17,81	6,88	9,40
100 : 25	6,90	5,63	2,90	19,06	7,21	10,31
100 : 30	7,21	5,85	3,15	20,08	7,43	11,15
100 : 40	7,69	6,03	3,52	21,64	7,72	12,40

Table 5

The composition of organomineral fertilizer obtained by activation of phosphorite with sulfuric acid and sewage sludge from the city of Navoi

Mass ratio of SS to phosphorite	$P_2O_{5ass.}$ / $P_2O_{5total.}$ *100%	P_2O_5 water. / P_2O_5total. *100%	CaO ass / CaO total *100%	CaO water. / CaO total *100%	organic substances	Humic substances
H_2SO_4 norm from stoichiometry for decomposition CaO , 30 %						
100 : 10	39,25	4,78	60,59	11,53	42,83	23,70
100 : 20	38,51	5,47	58,54	12,89	34,28	18,97
100 : 25	37,68	6,38	56,59	13,49	31,17	17,25
100 : 30	37,25	7,18	55,21	14,42	28,58	15,81
100 : 40	36,18	8,14	53,29	15,45	24,50	13,56
H_2SO_4 norm from stoichiometry for decomposition CaO , 40 %						
100 : 10	50,56	7,45	55,02	22,98	42,28	23,40
100 : 20	48,99	8,54	54,18	25,22	33,58	18,58
100 : 25	48,05	9,47	53,29	26,37	30,45	16,85
100 : 30	47,03	10,34	52,14	27,54	27,85	15,41
100 : 40	45,66	11,26	50,63	29,38	23,79	13,16
H_2SO_4norm from stoichiometry for decomposition CaO , 50 %						
100 : 10	63,78	17,10	49,39	32,69	41,75	23,10
100 : 20	61,25	19,22	48,04	34,97	32,91	18,21
100 : 25	59,72	20,41	47,47	36,42	29,77	16,47
100 : 30	57,98	21,77	46,68	37,36	27,17	15,03
100 : 40	56,80	24,25	44,99	39,47	23,13	12,80
H_2SO_4 norm from stoichiometry for decomposition CaO , 60 %						
100 : 10	75,41	27,85	44,34	42,29	41,23	22,81
100 : 20	73,13	30,41	41,98	44,15	32,27	17,86
100 : 25	71,26	31,81	40,94	44,71	29,11	16,11
100 : 30	68,81	32,82	40,19	46,05	26,51	14,67
100 : 40	67,31	35,09	38,21	48,86	22,50	12,45
H_2SO_4 norm from stoichiometry for decomposition CaO , 70 %						
100 : 10	85,78	38,54	40,28	50,47	40,72	22,53
100 : 20	83,44	41,03	38,60	52,76	31,65	17,52
100 : 25	81,53	42,01	37,81	54,09	28,48	15,76
100 : 30	81,02	43,70	36,99	55,55	25,89	14,33
100 : 40	78,33	45,80	35,67	57,32	21,90	12,12

Table 6

The composition of organomineral fertilizer obtained by activation of phosphorite with sulfuric acid and sewage sludge from the city of Navoi

Mass ratio of SS to phosphorite	fulvo acid	humic acids	water soluble organic matter	SO3total.	SO3water.	SO3water. / SO3total. *100 %	nitrogen
H_2SO_4 norm from stoichiometry for decomposition CaO, 30 %							
100 : 10	13,78	5,63	3,93	3,63	1,11	30,62	2,16
100 : 20	11,02	4,50	3,14	5,81	1,88	32,42	1,73
100 : 25	10,02	4,09	2,86	6,61	2,18	33,05	1,57
100 : 30	9,19	3,75	2,62	7,27	2,49	34,28	1,44
100 : 40	7,87	3,21	2,25	8,30	3,00	36,15	1,23
H_2SO_4 norm from stoichiometry for decomposition CaO, 40 %							
100 : 10	13,58	5,55	3,87	4,70	1,38	29,35	2,13
100 : 20	10,78	4,40	3,07	7,46	2,35	31,47	1,69
100 : 25	9,77	3,99	2,79	8,45	2,75	32,57	1,53
100 : 30	8,93	3,65	2,55	9,27	3,09	33,29	1,40
100 : 40	7,63	3,11	2,17	10,55	3,74	35,44	1,19
H_2SO_4 norm from stoichiometry for decomposition CaO, 50 %							
100 : 10	13,40	5,47	3,82	5,74	1,57	27,35	2,10
100 : 20	10,55	4,31	3,01	9,03	2,66	29,41	1,65
100 : 25	9,53	3,89	2,72	10,20	3,12	30,54	1,49
100 : 30	8,69	3,55	2,48	11,17	3,54	31,72	1,36
100 : 40	7,40	3,02	2,11	12,67	4,26	33,61	1,16
H_2SO_4 norm from stoichiometry for decomposition CaO, 60 %							
100 : 10	13,22	5,40	3,77	6,75	1,81	26,78	2,07
100 : 20	10,32	4,21	2,94	10,54	2,99	28,35	1,62
100 : 25	9,30	3,80	2,65	11,87	3,50	29,47	1,46
100 : 30	8,47	3,46	2,41	12,97	3,95	30,43	1,33
100 : 40	7,18	2,93	2,05	14,66	4,80	32,78	1,12
H_2SO_4 norm from stoichiometry for decomposition CaO, 70 %							
100 : 10	13,04	5,32	3,72	7,73	2,01	25,95	2,04
100 : 20	10,11	4,13	2,88	11,98	3,24	27,02	1,58
100 : 25	9,09	3,71	2,59	13,47	3,87	28,71	1,42
100 : 30	8,25	3,37	2,35	14,68	4,29	29,24	1,29
100 : 40	6,97	2,85	1,99	16,53	5,21	31,52	1,09

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