NEWS

OF THE NATIONAL ACADEMY OF SCIENCES OF THE REPUBLIC OF KAZAKHSTAN SERIES OF GEOLOGY AND TECHNICAL SCIENCES

ISSN 2224-5278

Volume 1, Number 433 (2019), 80 – 89

https://doi.org/10.32014/2019.2518-170X.10

UDC 547.992.2

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OBTAINING AND RESEARCH OF PHYSICAL AND CHEMICAL PROPERTIES OF CHELATED POLYMER-CONTAINING MICROFERTILIZERS ON THE BASIS OF TECHNOGENIC WASTE FOR RICE SEED BIOFORTIFICATION

Abstract. The way of obtaining the chelated polymer-containing microfertilizers on the basis of technogenic waste is developed. The optimum conditions of their obtaining with the use of water-soluble polyelectrolytes are established. Element composition of the cottrell dust and brown coal was studied by mean of scanning electron microscope ISM-6490-LV (JEOL, Japan). The mechanism of formation of chelated polymer-containing microfertilizers by elemental analysis and electron microscopy was studied. It was found that mainly in microfertilizers, structure formation occurs in the form of an amorphous structure with a small inclusion of metals. The characteristics of microelements and their physiological significance before and after biofortification in the cultivation of rice grain have also been studied. At the same time it is established that the resulting microfertilizer, along with potassium and sodium humates, also contains Ba, Fe, Mn, Ti and Mg. These trace elements are involved in the formation of the crop and determine its qualitative and quantitative components, i.e., are rich in trace elements that activate the action of enzymes, hormones and vitamins.

The received microfertilizers are characterized by the high content of humic substances which participate in structurization of the soil layer which is around the biofortified of seeds, accumulation of nutritious elements and minerals in a form, available to plants, promote regulation of geochemical streams of metals in water and soil ecosystems. In the end, all this provides a living organism with mineral substances and vitamins, which affects the protective functions of a living organism, to a large extent, activates its immune properties.

Keywords: chelate polymer-containing microfertilizers, technogenic waste, cottrell dust, humic acid, esterified derivatives of the hydrolyzed polyacrylonitrile.

Introduction. In accordance with the norms, which developed by the Kazakh Academy of Nutrition, the annual demand of the Republic of Kazakhstan for rice is 132.6 thousand tons per year (8.5 kg/year for 1 person). The statistics for 2008-2013 shows growth tendency in consumption of rice, and the percentage ratio of production of the peeled rice annually decreases in the ratio with the imported volumes. Therefore, in the near future not only remain, but the deficiency of this major food product even more will amplify [1-4]. It is also necessary to note that the satisfaction of the growing need of the population of the globe for food is the main problem of the present. This problem gains more and more acuity in developing countries where the increase in population advances a production gain. Due to small opportunities of expansion of acreage of crops now by further increase in agricultural production his comprehensive intensification is only. Today search of new nontoxical and highly effective mineral fertilizers and the chelate of microfertilizers which can be used successfully at cultivation of agricultural raw materials and also for the solution of narrower, but very important problem - receiving materials with the increased contents the essential of minerals is extremely relevant.

Along with this, some plants show the increased need for certain minerals, and microfertilizers provide a balanced set of minerals for requirements of various cultures [1, 2].

Nutrition of modern man is characterized by an increased content of cereals, potatoes, confectionery, and insufficient intake of milk, meat, fish, fresh vegetables, fruits. This indicates an insufficient supply of the body with vitamins and minerals. Mineral compounds affect the protective functions of a living organism, largely providing its immune properties. Microelements, primarily activate the action of enzymes, hormones, vitamins and thus participate in all types of metabolism [5-7].

From positions of the classical theories stated by Frenkel Ya.I. and Landau L.D., [8, 9] the absence of anions in solution have to change qualitatively mobility of cations and it is essential to change processes of formation of interfaces of a liquid and firm phase in respect of essential decrease in coefficient of a superficial tension that in turn will create thicker adsorptive layer of the dissolved connections on an interface (the surface of the parenchymal tissues of the leaf plate, the meristem membrane, the seed coat, etc.). In this regard, use of the amphoteric polyelectrolytes with complex-forming groups i.e. hydrolyzed and the modified polyacrylonitriles promote the formation of stable helated complex compounds with metal ions [10].

The presence of water-soluble polyelectrolytes in the system leads to the formation of thick films at the interface, causing an increase in the chemical potential gradient, which in turn forms directed flows into plant tissues and then through the membranes of the plasmolemma into the cytoplasm.

Methods. The way of obtaining the chelated polymer-containing microfertilizers of the Helafos series [11, 12] on the basis of technogenic waste is developed for the cardinal solution of the above-stated tasks: cottrell dust and brown coal of the Lenger field.

Element composition of the cottrell dust and brown coal was studied by mean of scanning electron microscope ISM-6490-LV (JEOL, Japan).

Chemical composition was calculated on the basis of elemental composition which was obtained from the spectra of scanning electron microscopy.

Results. The chemical composition of the cottrell dust is shown in table 1.

Composition, %								Specific		
The substances which are a part of cottrel dust	sq.m/g	CaO	MgO	SiO ₂	Al ₂ O ₃	F	Na ₂ O K ₂ O	Fe ₂ O ₃	Σ	surface, sq.m/g
Rich slime	37,5	8,0	3,0	26,0	10,1	2,1	10	1,52	98,22	

Table 1 – Chemical composition of cottrell dust

Element and mineralogical composition of withdrawal of brown coal of the Lenger field are given in figure 1, in table 2.

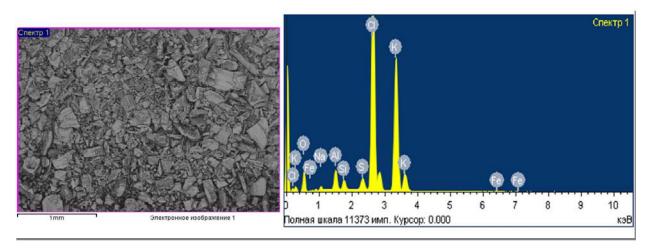


Figure 1 – Elemental composition and microstructure of a sample of brown coal of the Lenger field

Element	Weight, %	Oxides	Elemental composition, converted to oxides, %
O	55,17		
Na	0,23	Na ₂ O	0,31
Mg	0,36	MgO	0,59
Al	10,60	Al ₂ O ₃	20,03
Si	21,11	SiO ₂	45,15
S	3,34	SO ₃	8,35
K	1,31	K ₂ O	1,58
Ca	1,85	CaO	2,59
Ti	0,67	TiO ₂	1,12
Fe	5,39	Fe ₂ O ₃	7,41

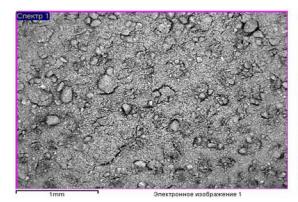
Table 2 – Element and mineralogical structure of a sample of brown coal of the Lenger field

From the figure 1 and from table 2 it is visible what in element structure of a sample of brown coal of the Lenger field contains in %: Al - 10,6, Si - 21,11, Fe - 5,39, Mg - 0,36, Ti - 0,67, etc. Such maintenance of elements as a part of brown coal is enough for his use as initial raw materials for receiving humic acids and on their basis of microfertilizers. Receiving humates is carried out from dumps of brown coal of the Lenger field by oxidation by 1% KOH or NaOH solution (environment 12,0 pn) [13, 14]. The oxidation of brown coal is carried out at a reaction mixture temperature of 80 °C for 2 hours, the weight ratio of alkali to crushed coal being $0.125 \div 0.150$: 1. To produce humic acid, humates were precipitated with a 5% solution of hydrochloric acid, then filtered in a nutch filter (the pH of the filtrate was 0.85).

As a part of the emitted humic acid, besides organic compounds, contain as well mineral substances. For definition of an inorganic component the received humic acid was exposed to calcination at 500 °C. The element and mineralogical structure of the received cindery rest was analyzed on a raster electronic microscope. Results of researches are given in table 3 and in figure 2.

Element	Weight, %	Elemental composition, converted to oxides, %	Weight, %
О	23,68	-	-
Na	1,06	Na ₂ O	1,43
Al	2,70	$\mathrm{Al_2O_3}$	5,1
Si	1,28	SiO ₂	2,74
S	1,51	SO_3	3,77
C1	34,52	-	_
K	34,97	K ₂ O	42,14
Fe	0,28	Fe_2O_3	0,40

Table 3 – Mineralogical composition of a sample of solution of the evaporated humic acid



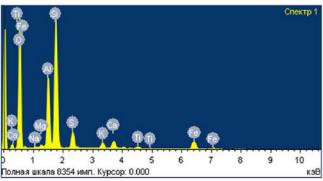


Figure 2 – Elemental composition and microstructure of a sample of the evaporated humic acid

From figure 2, it is visible that the investigated ashes have difficult and crystal mineral structure. Colourless detrital and fragmental crystals of a rhombic and cubic gabitus are characteristic of potassium and sodium chlorites. The intermediate fine-grained structure with some isometricity of tabletless colorless crystals is characteristic of aluminosilicate potassium compounds. Fine granular clusters of crystals characterize the presence of insignificant inclusions of aluminum silicates in the microstructure of the test sample, and dark, fossil, diffuse structures are characteristic of insignificant inclusions of iron silicates. Availability of sulfur up to 1.2% is obligatory for all humic acids that is confirmed in this case. The lack of phosphorus which content usually reaches 0,5%, is explained by the fact that initial raw materials for receiving humic acids were coal mining waste. Rather large amount of potassium and chlorine is explained by the fact that when leaching initial raw materials potassium hydroxide was used, and the humates received at the same time were acidified by hydrochloric acid.

To increase in content of the general phosphates and enrichment of finished product minerals are added to initial raw materials 1% of boric acid, 1% of sulfate of copper, 1% of sulfate of iron, 0,5% of sulfate of manganese, 1% of ammonium molybdate. Process is carried out in the temperature-controlled reactor from stainless steel with the mixer and a shirt at a temperature of 60 °C within 60 minutes. The received mix is filtered in the Nutsche filter and for receiving the chelating microfertilizers it is used a liquid component. Then add 0.2% of esterified derivatives of the hydrolyzed polyacrylonitrile (EPPAN) to the received solution of 25 cm³ (pH = 4.16).

To increase the content of total phosphates and to enrich the finished product with microelements, 1% of boric acid, 1% of copper sulfate, 1% of ferrous sulfate, 0.5% of manganese sulfate, 1% of ammonium molybdate are added to the feed. The process is carried out in a thermostated stainless steel reactor with a stirrer and jacket at a temperature of 60°C for 60 minutes. The resulting mixture is filtered in a nutch filter and a liquid component is used to prepare the chelating microfertilizers. Then 0.2% of the ethylenated derivatives of hydrolyzed polyacrylonitrile (EPPAN) are added to the resulting 25 cm³ solution (pH = 4.16).

The chemism of process of decomposition of cottrel dust in acidic environment of water humic acid can be described by the following equation:

$$Ca_5(PO_4)_3F + 8 \text{ humic acid} + 4H_2O = H_3PO_4 + 4Ca(humate)_2 + Ca(H_2PO_4)_2 + HF$$

Use of humic acid for decomposition of mix of cottrel dust and also EPPAN will allow to exclude sulfuric acid from process and to reach contents of assimilable phosphates in a finished liquid product to 10%, in a firm product up to 12,5%. Element and mineralogical compositions of chelate polymer-containing microfertilizers are presented in table 4.

Element	Weight, %	Oxides	Weight, %
С	28,75	_	_
О	36,64	_	_
F	1,89	_	_
Na	1,23	Na ₂ O	1,66
Mg	0,74	MgO	1,22
A1	0,76	$\mathrm{Al_2O_3}$	1,44
Si	3,58	SiO_2	7,66
P	13,34	P_2O_5	30,56
S	0,22	SO_3	-
C1	8,65	-	10,42
K	3,40	K ₂ O	4,76
Ca	0,11	CaO	0,14
Mn	0,42	MnO	0,60
Fe	0,28	Fe_2O_3	0,35

Table 4 – Elemental and mineralogical composition of a sample of chelate polymer-containing microfertilizer

From table 4 it is visible that the developed polymer-containing chelate microfertilizer also has in structure minerals and minerals necessary for the normal growth and development of plant ($K_2O - 4.76\%$, $P_2O_5 - 30.56\%$). The element composition and a microstructure of a sample of chelated polymer-containing microfertilizer are presented in figure 3.

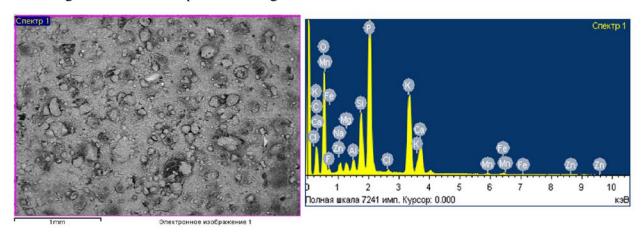


Figure 3 - Element composition and microstructure of a sample of chelated polymer-containing microfertilizer

Results of microscopic pictures (figure 3) reflect the results received when carrying out the element analysis. At the same time presence of the hydrolyzed polyacrylonitrile provides formation of granular crystal structure the chelated of microfertilizers.

Rice seed biofortification process before crops on fields of LLP "I.Zhahaev Kazakh Research Institute of Rice" is carried out and also process of influence of chelated microfertilizer throughout a stage of growth of a plant and its blossoming (rice) on the subsequent productivity of culture has been investigated. For biofortification of seed of rice connections complex the chelated polymer-containing of microfertilizers are chosen.

The following operational stage the chelated polymer-containing of microfertilizers is spraying of plants during growth and blossoming. Results of field tests on identification of efficiency complex the chelated polymer-containing of microfertilizers received on the basis of phosphoric slime, cottrell dust have been received. Results of field tests have shown that the productivity of seed of rice increases by 3 times, i.e. from 25 to 75 grain/vessel.

The element composition and the microscopic picture of samples of rice before introduction of microfertilizers are given in figure 4 and in table 5.

In table 6 and also in figures 4 there are presented element composition of grains before crops of rice after biofortification with application the chelate of microfertilizers. From figure 4 it is visible that after biofortification of seed rice is dispersed and as a result becomes more small granular with increase in an amorphous part.

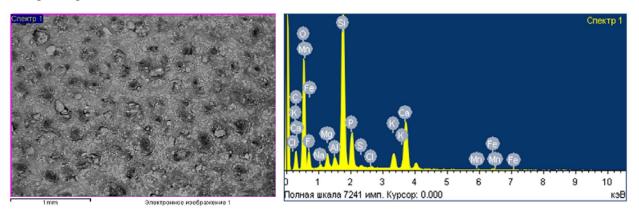


Figure 4 - Element composition and microstructure of grains before crops of rice after biofortification

Element Weight, % Oxides Weight, % O 51,81 0,62 Na₂O 0,83 Na 1,56 2,59 Mg MgO 32,19 68,85 Si SiO₂ P 6,00 P_2O_5 13,75 0,44 0,48 S SO_3 K K₂O 7,45 6,18 Ca 1,18 CaO 1,65

Table 5 – Element composition of a sample of rice before biofortification

Table 6 – Element composition of grain before crops of rice after biofortification

Element	Weight, %	Oxides	Weight, %
О	46,79		
Na	0,22	Na ₂ O	0,29
Mg	4,16	MgO	6,9
A1	3,31	Al_2O_3	6,25
Si	11,57	SiO ₂	24,75
P	5,96	P ₂ O ₅	13,65
S	0,29	SO ₃	0,72
K	1,84	K ₂ O	2,22
Ca	19,30	CaO	27,00
Ti	0,47	TiO ₂	0,78
Mn	0,24	MnO	0,31
Fe	5,69	Fe ₂ O ₃	8,14
Ва	0,14	ВаО	0,16

The element and mineralogical composition and also rice seed sample microstructure after harvesting with use of the chelated polymer-containing microfertilizer is presented in table 7 and in figure 5. From figure 5 it is visible that the rice seed sample microstructure after harvesting is condensed and as a result of which gets a crystal form with reduction of an amorphous part. It is explained by the fact that probably the large role is played by presence at composition of seed of rice of various minerals: Na - 0.40, Mg - 1.76, Al - 0.14, Si - 31.80, P - 5.29, S - 0.84, K - 6.77, Ca - 0.93, Mn - 0.17, Fe - 0.09.

Table 7 – Elemental and mineralogical composition of rice grain after harvest with the use of chelated polymer-containing microfertilizer

Element	Weight, %	Oxides	Weight, %
О	51,80	-	
Na	0,40	Na ₂ O	0,54
Mg	1,76	MgO	2,92
Al	0,14	Al_2O_3	0,26
Si	31,80	SiO ₂	68,02
P	5,29	P_2O_5	12,12
S	0,84	SO ₃	2,1
K	6,77	K ₂ O	8,16
Ca	0,93	CaO	1,3
Mn	0,17	MnO	0,22
Fe	0,09	Fe_2O_3	0,13

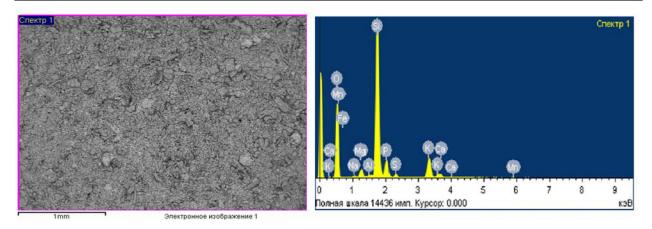


Figure 5 – Element composition and microstructure of sample of rice grain after harvest with the use of chelated polymer-containing microfertilizer

Besides the presence of humic acid and polyelectrolytes leads to the formation of crystal structure of more condensed type and confirms the previous microstructures (figures 3, 4). Thus, colourless detrital and fragmental crystals of a rhombic and cubic gabitus are formed, are characteristic of chlorites of potassium and sodium. The intermediate fine-grained structure with isometricity of tabletless colorless crystals is characteristic of aluminosilicate potassium compounds. Small granular clusters of crystals characterize the presence of insignificant inclusions of aluminum silicates in the microstructure of the test sample, and dark, fossil, diffuse structures are characteristic of insignificant inclusions of iron silicates.

Element composition of rice seed before biofortification (figure 4, table 5) following (%): Na – 0.62, Mg – 1.56, Si – 32.19, P-6.0, K – 6.18, Ca – 1.18.

Mineralogical composition of seed of rice after biofortification (figure 5, table 6) the following: Na -46,79, Mg -4,16, Al -3,31, Si -11,57, P -5,96, S -0,29, K -1,84, Ca -19,30, Ti -0,47, Mn -0,24, Fe -5,69, Ba -0,14. From figure 6, table 7 it is visible that the maintenance of elements of seed of rice after assembly of a harvest with use of changes a little Na -0.40, Mg -1.76, Al -0.14, Si -31.80, P -5.29, S -0.84, K -6.77, Ca -0.93, Mn -0.17, Fe -0.09.

It is visible that the developed microfertilizer contains humates of potassium and sodium with additive Fe, Mn, Si, Mg, K, Sa, Al, P. These minerals participate in formation of a harvest and define its qualitative and quantitative components that are rich with the minerals intensifying effect of enzymes, hormones and vitamins. Finally, all this provides with mineral substances and vitamins a live organism that influences his protective functions and substantially makes active its immune properties.

Data of vegetative experiment on rice of a grade of Marzhane on production crops and the experimental site of LLP "I.Zhahaev Kazakh Research Institute of Rice" are presented in table 8. At the same time the productivity of rice increases within 65-74 g/a vessel.

#	Options	Productivity (g/vessel) on repetitions				S-sum	M-average	М	D from
	experience	I	II	III	IV	harvests	harvest	101	helofos-1
1	Without fertilizers	33	31	20	20	104	26	_	-
2	N-Na	54	53	50	43	200	50	_	1
3	NP-Na, Pc- background	68	65	55	50	240	60	1	
4	Biofortified seeds of Chelafos-1 (100 ml)	76	76	74	70	296	74	74	14
5	Biofortified seeds of Chelafos -2 (500 ml)	76	74	70	72	292	73	73	13
The sum of harvests on repetitions		307	299	269	269	1132	56,6]	Mπ1~57

Table 8 – Data of vegetative experiment on rice of a grade of Marzhane

On the basis of the above, the developed technology of receiving the chelated polymer-containing microfertilizer can be recommended at cultivation of grain crops, in particular, of rice.

Discussion. The way of receiving the chelated polymer-containing microfertilizer on the basis of technogenic waste - cottrell dust, brown coal of the Lenger field is developed and optimum conditions of their receiving with use of water-soluble polyelectrolytes are established.

Rice seed biofortification by chelated polymer-containing microfertilizer before crops on the experimental site of LLP "I.Zhahaev Kazakh Research Institute of Rice" is carried out.

The mechanism of formation of chelated polymer-containing microfertilizers by elemental analysis and electron microscopy was studied. It was found that mainly in the presence of chelated polymer-containing microfertilizers, structure formation occurs in the form of an amorphous structure with a partial appearance of sections of the crystallization structure. The characteristic of minerals and their physiological value before biofortified at rice seed cultivation is studied.

The received microfertilizers are characterized by the high content of humic substances which participate in structurization of the soil layer around the biofortified of seeds, accumulation of nutritious elements and minerals in a form, available to plants, promote regulation of geochemical streams of metals in water and soil ecosystems.

Influence of the microfertilizers developed the chelated polymer-containing on growth and productivity of a stalk of rice is investigated.

At the same time it is shown that increase in productivity of rice within 65-74 g/vessel demonstrates that in the initial stage the main role is played by oxides of manganese and magnesium as a part of chelated polymer-containing microfertilizers that provides high viability and germination of seeds due to rice seed biofortification. Contents as a part of microfertilizers of iron regulate breath of plants, cellular exchange, photosynthesis and resistance to chlorosis when spraying pesticides.

Thus, on the basis of the above, it can be concluded that the chelated polymer-containing microfertilizer actively participates in biochemical processes, i.e. activates enzymes, exhibits photosynthetic activity, participates in the biosynthesis of chlorophyll, affects carbohydrate and nitrogen metabolism, increases resistance to diseases, accelerates the growth and development of plants. All these processes ultimately contribute to higher yields and, especially, the quality of the rice grain.

The developed microfertilizer containing humates of potassium and sodium with additive Fe, Mn, Si, Mg, K, Sa, Al, P in a chelated form of the improved structure has shown excellent operational properties, and it can be recommended to application at cultivation of rice in the southern regions.

Acknowledgements. The work was financially supported by the Ministry of Education and Science of the Republic of Kazakhstan.

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КҮРІШ БИОФОРТИФИКАЦИЯСЫНА ТЕХНОГЕНДІ ҚАЛДЫҚТАРДАН АЛЫНАТЫН ХЕЛАТТЫ ПОЛИМЕРҚҰРАМДАС МИКРОТЫҢАЙТҚЫШТАРДЫҢ ФИЗИКА-ХИМИЯЛЫҚ ҚАСИЕТТЕРІН ЗЕРТТЕУ

Аннотация. Техногенді қалдықтар негізінде хелатты полимерқұрамдас микротыңайтқыштарды алудың технологиялық әдісі әзірленген және сулы полиэлектролиттерді пайдалану арқылы алудың оңтайлы жағдайлары орнатылған. Бастапқы шикізат болып табылатын қоңыр көмір мен котрельді шаңның элементтік құрамдары ISM-6490-LV (JEOL, Жапония) сериялы электронды микроскоп көмегімен анықталған. Сонымен қатар электронды микроскопиялық және элементтік талдау әдісімен хелатты полимерқұрамдас микротыңайтқыштарды құралу механизмі меңгерілді.

Микротыңайтқыштардың құрылымында аморфты күйде болатын біршама металдарды ілесуі жүреді. Сондай-ақ күрішті өсіру кезінде пайдаланылатын микротыңайтқыштардың қасиеттері мен физиологиялық мәндері биофортификацияға дейін және кейінгі жағдайлары қарастылған. Алынған микротыңайтқыштардың құрамында натрий және калий гуматымен қоса Ва, Fe, Mn, Ti, Mg сияқты элементтер кездеседі. Бұл микро-

элементтер өсімдіктің өсіп, өнуіне және сандық құрамымен өнімділігін анықтайды, яғни бай микротыңайтқыштар ферменттер, гармондар және дәрумендерді белсендендіруге әрекеттендіреді.

Алынған микротыңайтқыштар құрамындағы жоғары мөлшерде болатын гумин қышқылдары топырақ қабатындағы биофортификацияланған дәндердің құрылымдық түзілуіне қатысады да, жинақталған элементтер мен микроэлементтер өсімдіктерге қолайлы күйде болып, сулы және топырақты экожүйедегі металдардың геохимиялық ағынын реттеуге қатысады. Қорыта келгенде барлығы өсімдікті минералды заттармен дәрумендермен толықтырып, тірі өсімдіктің қорғаныс қызметін жағдай жасап, оның иммундық қасиетін белгілі бір дәрежеде белсендіруге әсер етеді.

Түйін сөздер: хелатты полимерқұрамдас микротынайткыш, техногенді қалдықтар, котрельді шаң, гумин қышқылы, полиакрилонитрилдің этерифицирленген гидролизді туынды.

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ПОЛУЧЕНИЕ И ИССЛЕДОВАНИЕ ФИЗИКО-ХИМИЧЕСКИХ СВОЙСТВ ХЕЛАТНЫХ ПОЛИМЕРСОДЕРЖАЩИХ МИКРОУДОБРЕНИЙ НА ОСНОВЕ ТЕХНОГЕННЫХ ОТХОДОВ ДЛЯ БИОФОРТИФИКАЦИИ ЗЕРНА РИСА

Аннотация. Разработан способ получения хелатных полимерсодержащих микроудобрений на основе техногенных отходов и установлены оптимальные условия их получения с использованием водорастворимых полиэлектролитов. Элементный состав коттрельной пыли и бурого угля отхода был изучен растровым электронным микроскопом ISM-6490-LV (JEOL, Япония).

Изучен механизм образования хелатных полимерсодержащих микроудобрений методом элементного анализа и электронной микроскопии. При этом установлено, что в основном в микроудобрениях происходит структурообразование в виде аморфной структуры с небольшим включением металлов. Также изучена характеристика микроэлементов и их физиологическое значение до и после биофортификации при выращивании зерна риса. При этом установлено, что полученное микроудобрение, содержит наряду с гуматами калия и натрия также Ва, Fe, Mn, Ti, Mg. Эти микроэлементы участвуют в формировании урожая и определяют его качественные и количественные составляющие, т.е богаты микроэлементами, активизирующими действие ферментов, гармонов и витаминов.

Полученные микроудобрения характеризуются высоким содержанием гуминовых веществ, которые участвуют в структурообразовании почвенного слоя находящегося вокруг биофортифицированных семян, накоплении питательных элементов и микроэлементов в доступной для растений форме, способствуют регулированию геохимических потоков металлов в водных и почвенных экосистемах. В конечном итоге все это обеспечивает живой организм минеральными веществами и витаминами, что влияет на защитные функции живого организма, в значительной степени активизирует его иммунные свойства.

Ключевые слова: хелатные полимерсодержащие микроудобрения, техногенные отходы, котрельная пыль, гуминовая кислота, этерифицированные производные гидролизованного полиакрилонитрила.

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