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Natural mineral raw materials as granular filtering materials in industrial and waste water treatment

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ABSTRACT

Analysis of currently existing methods of natural and waste water treatment from heavy metals has shown that one of the most promising is the sorption method using natural inorganic materials as sorbents: zeolite, diatomite, and vermiculite. The relevance of the topic is dictated by the need to develop an original technology of wastewater treatment and water treatment, which allows constant monitoring of the level of pollution of natural waters by industrial wastewater from metallurgical enterprises. The proposed methods of sorbent modification create a basis for studying the structure, porosity, and sorption capabilities of the mentioned natural materials. All three minerals: zeolite, diatomite, and vermiculite belong to highly porous, structured materials, promising for use as stable sorption systems in water treatment and water purification. The influence of physical and chemical characteristics of natural sorbents and the application of new promising environmentally safe materials and reagents for water treatment are considered. The sorption of copper ions on natural zeolite material before and after its modification by hydrothermal method was investigated. The activity of sorbents was estimated by the value of sorption capacity, i.e. the amount of heavy metal ions absorbed by a unit mass of sorbent based on zeolite and its modified form.

Keywords: wastewater, treatment, sorbents, zeolite, diatomite, vermiculite, clinoptilolite, waste, minerals.

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Introduction

Today, an urgent environmental task for mineral resource complex facilities is the development of highly efficient and economical technological and technical solutions for wastewater treatment.

Of particular concern is the constant increase in contamination of water supplies with heavy metals, which are toxic to living organisms even at extremely low concentrations. Removing or reducing the total concentration of heavy metals to less than 10 mg/l is a primary objective when treating any natural and wastewater. The relevance of the issues of efficiency of purification and preparation of drinking, industrial and waste water and their cost reduction explains the existence of many purification methods. among which the most commonly used is sorption because it allows you to increase the degree of purification to almost any required concentration. For the production of environmentally friendly sorbents, the most attractive are natural organic raw materials and production waste of plant origin, therefore sorbents based on them are most compliant with environmental requirements.

Such sorbents are also promising for the extraction of ionic impurities and contaminants from solutions, natural and waste waters, liquid radioactive waste, extraction and concentration of ions of heavy and radioactive metals (uranium, cesium, arsenic, chromium, manganese, strontium, zirconium, etc.)

A promising direction for intensifying deep cleaning processes is the use of natural mineral raw materials as effective sorbents. The main advantages of natural sorbents are environmental friendliness, a wide raw material base, high hydrophobicity and sorption capacity at a relatively low cost.

When treating industrial and waste water, the practical task is to select local natural materials as raw materials that optimally combine economic profitability and effective treatment. Our research suggests minerals zeolite, diatomite, vermiculite and their modified forms. Their structure and properties have been studied, because there is little in the literature The listed minerals were studied as sorbents for solving environmental problems.

Modern highly effective and safe sorbents for wastewater treatment include zeolites, which are minerals with ion-exchange properties. Generalized formula of zeolites:

$$K_{x/n}Al_xSi_yO_2(x+y)*ZH_2O$$

where K are cations of alkali and alkaline earth metals, ammonium, etc.; n is the charge of the cation.

More than 40 types of natural zeolites are known. The most common ones are :

1. Phillipsite $K_2(Ca_{0.5}Na)_4[Al_6Si_{10}O_{32}]*12H_2O$

2.Mordenite $(Na_{2,}Ca,K_{2)}4(Al_8Si_{40)}O96•28H_2O$

3.Chabazite $(Ca,Na_{2)\ 2}[Al_4Si_8O_{72]}*28H_2O$

4.Clinoptilolite $(K_2Na_2Ca)_3[Al_6Si_{30}O_{72]}*20H_2O$

Zeolite contains approximately 70% silicon oxide, the rest is oxide compounds of titanium, iron, manganese, magnesium and several other metals, including copper. The mineral has a special mature microporous structure with pore sizes from 3 to 10 A $^{0.}$ Molecular-sized pores, like a sponge, can absorb and firmly hold a wide variety of contaminants. These include heavy metals (lead, cadmium, zinc, strontium, chromium), radionuclides, nitrates and nitrites, ammonium salts, oils, petroleum products and a whole range of chemical and biological contaminants.

The composition of zeolites can be described by the following empirical formula:

$$(Me+,Me^{2}+0.5)x...[AlxSiyO^{2}(x+y)]\ (H_2O)n, \quad (1)$$

where Me - metal cation, values x and y determine the number of atoms of silicon, aluminum, oxygen in the unit cell, x|y, depending on the type of zeolite, usually takes values from 1 to 5, n is the number of water molecules.

Thanks to the relatively high The Si / Al crystalline frame of clinoptilolite is heat-resistant (in air up to 700 ^{0} C), and is also resistant to aggressive substances and ionizing radiation (Fig. 1).

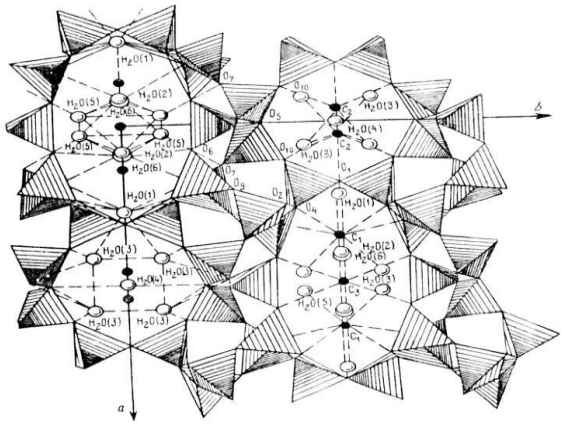

Figure 1 - Clinoptilolite frame fragment

In Kazakhstan, large deposits of zeolites are in Tayzhuzgen (Tarbagatai district of the East Kazakhstan region, approved reserves - 7 million tons, forecast - 215 million tons) and Chankanai (Kerbulak district of Almaty region, approved reserves - 5.5 million tons, forecast - 120 million tons). Zeolites from the Chankanai and Taizhuzgen deposits are medium-porous zeolites and can be used to extract pollutants from natural waters.

In Southern Kazakhstan, the Altyn- Emel (41 million tons), Karzhantau and Daubabinskoe zeolite deposits have been preliminarily assessed [1].

Modern wastewater treatment technologies include the development and implementation of technological processes that ensure:

- complex processing of mineral raw materials;
- reduction or complete elimination of environmental pollution from production waste;
-processing of production and consumption waste by obtaining commercial products;
-creation of closed systems providing recycling water supply [2].

Improvement of the main technological process, wastewater treatment methods, and disposal of resulting sediments make it possible to

create closed (drainless) water supply systems at enterprises. In this case, the main technological process and wastewater treatment are considered as a single integrated system.

Inorganic compounds of heavy metals belong to the group of highly hazardous pollutants with a wide range of toxic effects. Under natural conditions, along with surface waters, they also enter underground waters. One of the promising ways to solve this problem is the use of sorption technologies, which ensure the removal of pollutants of a wide nature to almost any residual concentration. An alternative raw material for removing toxicants from aquatic environments can be natural minerals: zeolite, diatomite, vermiculite and their modified forms. Modern enterprises are in dire need of a methodology for assessing the state of wastewater, solving problems of their treatment and prospects for the development of this area based on forecasting and management. This requires setting the problem of the need for theoretical and practical developments that ensure the implementation of organizational innovations designed to adapt the problems being solved in the field of wastewater treatment in various industries, including the metallurgical industry.

Experimental part

The need to increase the sorption capacity of filters in wastewater treatment plants leads to the search for new, cheaper and widespread materials characterized by sufficiently high mechanical strength, and chemical stability and at the same time possessing a more developed specific surface area and greater porosity. A significant indicator of the quality of filter materials is their resistance to acidic and alkaline environments. The use of natural zeolites in solving environmental problems of environmental objects is due to their adsorption properties, as well as thermal, chemical and radiation resistance. Zeolites are suitable for the sorption of amines and heavy metals from industrial wastewater. Data [3] show that long-term contact of the natural material, clinoptilolite, with a NaOH solution at 100-150 °C did not lead to any noticeable changes in the composition and structure of the zeolite, which confirms its physical and chemical stability. To test the resistance of this mineral to acids, the effect of a five-hour treatment of clinoptilolite with hydrochloric acid of various concentrations at 100-2 00^0C on the dissolution of heavy metal oxides: zinc, lead, cadmium, etc. was studied. It was established that clinoptilolite rocks meet the requirements for granular filter materials. Clinoptilolite, as mentioned earlier, is characterized by a noticeable cation exchange capacity and high selectivity to ions with a large radius: Cs^+, Rb^+, NH_4^+, Ba^{2+},Sr^{2+} [4].

Due to their unique properties: - developed internal surface, zeolites have high absorption characteristics for impurities found in the water being purified.

The advantage of sorption wastewater treatment is the ability to optimize the parameters of sorption columns, which is carried out through the use of natural sorbents, their modification and the combination of two or more granular sorbents in one adsorption column.

Table 1 shows the capacity values of clinopthiolite for cations of mono and divalent metals (Me $^{+,}$ Me $^{2+),}$ expressed both in milligram equivalents per gram of zeolite in the cationic form of the corresponding ion (E), and in milligram equivalents per gram of Na – forms of zeolite (E $^{*)}$. Capacity values in milligram equivalents per gram of zeolite in the cationic form of the corresponding ion (E) are given to show its dependence on the atomic mass of the cation, while the analysis of the results obtained was carried out using capacity values expressed in milligram equivalents per gram Na – zeolite forms, which were calculated using the formula:

$$E^* = E\, d_{kat} / d_{Na,}$$

where d_{kat} / d_{Na} are the specific gravities of the zeolite in the corresponding cationic and Na – forms.

To increase the adsorption capacity of natural zeolites about various pollutants, it is recommended to pre-grind them to particles of about 150 microns in size, due to which the specific surface area of the sorbent increases. This makes it possible to remove up to 90-95% of polluting ions from industrial wastewater.

Based on structural features, chemical composition, and physicochemical properties, three large groups of natural sorbents can be distinguished: dispersed silicas, layered and layered-tape silicates, frame silicates, and zeolites [5]. Dispersed silicas are of sedimentary origin. They are 60-95% composed of amorphous SiO_2 – silicon dioxide.

Table 1- Exchange capacity of high-silica zeolite

Cation metals	Clinoptilolite		
	E, mg eq, / g	d_{kat}/d_{Na}	E^*, mg eq, / g
Li^+	1.65	0.98	1.62
Na^+	1.95	1.00	1.95
Ag^+	1.73	1.12	1.94
K^+	1.93	1.02	1.97
NH_4^+	1.95	0.99	1.93
Rb^+	1.77	1.09	1.93
Cs^+	1.68	1.15	1.93
Ca^{2+}	1.26	0.99	1.25
Sr^{2+}	1.32	1.03	1.36
Ba^{2+}	1.42	1.06	1.51
Zn^{2+}	1.09	1.01	1.10
Cu^{2+}	1.09	1.01	1.10
Cd^{2+}	1.25	1.06	1.33
Pb^{2+}	1.45	1.11	1.61

One of the most promising filter materials in treatment facilities of metallurgical plants is the highly porous minerals zeolite, diatomite, and vermiculite, used to purify wastewater from iron, ammonium ions, heavy metals, radionuclides, organic compounds and various trace elements [6].

The sorption properties of clinoptilolite rocks, and zeolites from the Chankanai and Taizhuzgen deposits with a clinoptilolite content of at least 60-70%, were studied. The studies assessed the sorption exchange capacity (SOE, mg/g, mg-ion/g) in static mode (ion concentration 7.0±0.1 mg-ion/l, ratio of solid and liquid phases 1:10, particle size 0,8÷1.2 mm, sorption time 24 hours). Zeolite samples have significant sorption capacity not only for particularly toxic ions ($Cd^{2+,}$ $Pb^{2+)}$ but also for other heavy metals ($Cu^{2+,}$ $Zn^{2+)}$ [[7], [8], [9]].

The studies were carried out under static conditions with aqueous solutions of metal salts at a concentration of 0.1-0.5 g/l, T = 20 ^{0} C. Ion content heavy metals in powder samples and depending on time defending model solutions determined X-ray fluorescent method according to a certified methodology. The activity of sorbents was assessed by the sorption capacity, i.e., the quantity of heavy metal ions, absorbed unit mass of sorbent based on zeolite and its modified form [[10], [11], [12], [13], [14]].

Analysis of the sorption capacity showed that the sorption capacity of zeolite can be significantly increased due to its modification (Fig. 2).

In addition to zeolites, diatomites have been studied as sorbents. In Kazakhstan, the largest deposits of diatomite in the world are located in the Aktobe region. In total, the forecast resource of Kazakhstan's diatomite reserves is three billion tons. For comparison: in the United States, diatomite reserves are estimated at 500 million tons. It is a sedimentary rock, usually loose or weakly cemented. Diatomite is a good absorbent material with low heat and sound conductivity; in addition, it is refractory, acid-resistant, and non-flammable. This material is unique in that in one case it can be used as a hygroscopic material, a very strong absorbent that absorbs moisture, and in another case - as a superhydrophobic material that repels liquid. Diatomite itself is a natural nanomaterial containing up to 80% silicon dioxide. Diatomite is promising as:

- filtration material for water treatment of industrial wastewater;
- Universal sorbent for eliminating emergency spills of oil products and aggressive environments;
- filtration material for purification of food and technical liquids**.**

One of the promising materials for water treatment is also vermiculite. Vermiculite is a mineral from the group of hydromicas that have a layered structure.

Important properties of expanded vermiculite are chemical inertness, heat resistance, strength, safety, high adsorption capacity, and ion exchangeability. It is thanks to these properties that vermiculite serves as an inorganic matrix for creating a sorbent. During the expansion of vermiculite, water crystallized between mica flakes evaporates, which leads to the formation of macro- and micropores in the vermiculite particle and an increase in the specific surface area by approximately 5 times. Expanded vermiculite itself

is hydrophilic. To turn vermiculite into an effective sorbent, it must be chemically modified. In this case, macropores become hydrophobic, while micropores remain hydrophilic. Pores of different sizes and wettability characteristics give modified expanded vermiculite unique sorption and ion exchange characteristics [[15], [16], [17], [18], [19]].

Expanded vermiculite is used:

- for removing petroleum products, organic and toxic liquids from the surface of water bodies and soils;
- for localization and removal of oil in case of accidents of underwater oil pipelines and during offshore oil production;
- for wastewater treatment from oil and other contaminants.

The above-listed macroporous granular sorbents with a high exchange capacity have good kinetic characteristics for the sorption of heavy metal ions.

Results and discussion

Composition and content are given in Table 2 of model solutions of heavy metal salts.

Sorbents based on mineral raw materials and their modified forms can be used for selective or group extraction and concentration of metal ions. The kinetic laws of the cleaning process were studied to establish the optimal cleaning regime. The proposed methods for modifying sorbents create the basis for studying the structure, porosity, and sorption capabilities of these natural materials [[20], [21], [22]]. Zeolite has a significant and similar sorption capacity not only for particularly toxic ions (Hg^{2+}, Cd^{2+}, Pb^{2+}), but also for other heavy metals (Cu^{2+}, Co^{2+}, Ni^{2+}, Zn^{2+}, Ba^{2+}, Sr^{2+}), present in natural and waste waters in different quantities**.** A special role is played by the significant sorption

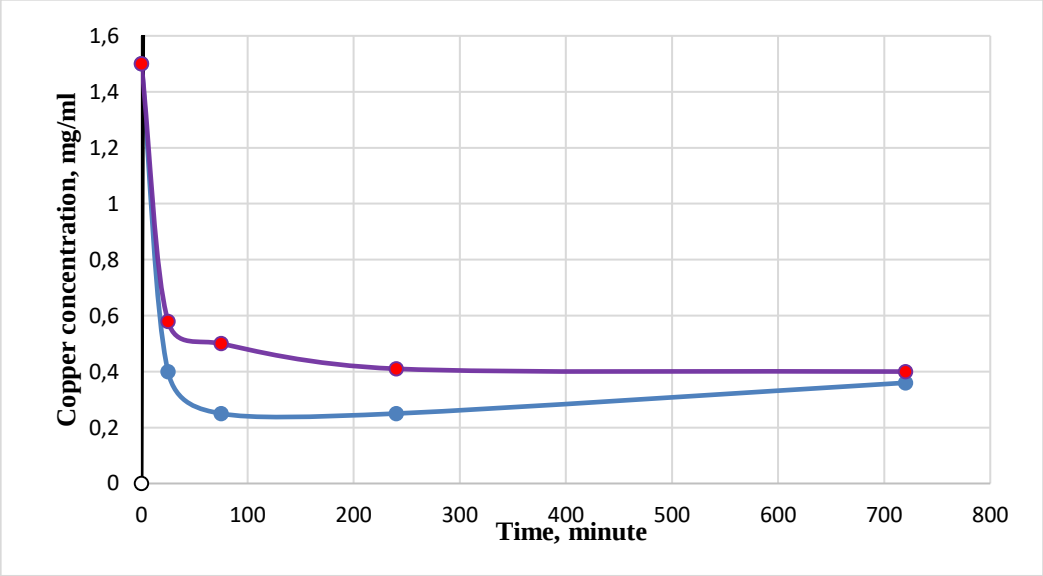

Figure 2 - Kinetics of copper ion sorption by natural sorbents
(1 - copper concentration on zeolite sorbent; 2 - on modified zeolite sorbent)

Table 2 - Composition and content of the model solution

Cation	NH_4^+	Pb^{2+}	Ba^{2+}	Sr^{2+}	Cd^{2+}	Cu^{2+}	Zn^{2+}	Co^{2+}	Ni^{2+}
Concentration (in mg/l) of metal in wastewater	40.0	2.95	3.68	1.65	2.15	2.20	2.5	2.5	1.2

capacity of the studied clinoptilolite samples for lead ion, which makes it possible to use them for wastewater treatment and soil detoxification in the adjacent territories of metallurgical enterprises**.**

Diatomite, untreated thermally, with high adsorption rates and low permeability values, can be used as an adsorbent for fine wastewater treatment in a stationary mode. The advantage of using diatomite for fine purification of wastewater from pollutants is the possibility of restoring the adsorption properties of diatomite after calcination at 300-400 ^{0} C. Diatomite used for fine purification of wastewater from heavy metal cations can also be regenerated or recycled. Calcined diatomite, which has a relatively high permeability with a fairly low adsorption value, can also be used as a filter material for rough water purification [[23], [24], [25], [26]]. It is possible to use diatomite in the form of granules of fractions 0.5-1.0, 0.8-2.0; 1.0-4.0, 2.5-5.0 mm.

Conclusions

In the EU countries and the USA, lists of priority environmental pollutants have been compiled, which for various matrices (water, soil, air, etc.) contain approximately 100-150 of the most dangerous pollutants that are constantly found in environmental objects.

In Kazakhstan, there is not yet a scientifically based (from the point of view of ecology, toxicology, hygiene and eco-analytics) list of priority pollutants for water, air and soil, which makes it difficult to periodically systematically monitor their content in various natural environments.

Therefore, it is necessary to compile a scientifically based list of priority pollutants in the natural waters of Kazakhstan.

One of the most promising filter materials are zeolite, clinoptilolite, and diatomite vermiculite, which can be used in wastewater treatment from iron, ammonium ions, heavy metals, radionuclides, organic compounds and various trace elements. The sorption properties of clinoptilolite rocks from the Chankanai and Taizhuzgen deposits with a clinoptilolite content of at least 60–70% have been studied.

Based on a detailed analysis of foreign experience in wastewater treatment technologies, research has been carried out on the possibilities of using effective adsorption methods along with traditional reagent treatment methods.

The experience of American and German scientists were studied in detail: modern methods of purification of natural and waste waters and the capabilities of new generation equipment in identifying toxic substances in natural and waste waters. The development of new and improvement of existing treatment methods, and their implementation at enterprises in Kazakhstan will allow solving the following practical problems: fundamentally new approaches to organizing energy- and resource-saving highly efficient technologies for wastewater treatment and water treatment will be developed.

Zeolite, diatomite, and vermiculite rocks for the extraction and concentration of metals in their modified forms through preliminary thermochemical treatment.

Research on the extraction of heavy metals on granular filters with zeolite, diatomite, and vermiculite loading, with a mineral content of 90-95%, fractions of 0.5-1.0 mm at a temperature of 300 ^{0} C - 400 ^{0} C, has established the almost complete extraction of large Pb cations $^{2+,}$ Fe $^{2+,}$ Ni $^{2+,}$ Mn $^{2+,}$ Zn $^{2+.}$

Zeolite exhibits sorption capacity not only for particularly toxic ions: Hg $^{2+,}$ Cd $^{2+,}$ Pb $^{2+,}$ but also for heavy metal ions Cu $^{2+,}$ Co $^{2+,}$ Ni $^{2+,}$ Zn $^{2+,}$ Ba $^{2+,}$ Sr $^{2+,}$ present in natural and waste waters in different quantities.

Thus, natural mineral raw materials (zeolites, diatomites, vermiculites, and their modified forms), the reserves of which are sufficient in Kazakhstan, can be used as a granular load for wastewater treatment and water treatment. All three minerals are highly porous, structured materials that are promising for use as stable sorption systems in water purification and water treatment.

Conflict of interest. On behalf of all the authors, the correspondent author declares that there is no conflict of interest.

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Өндірістік ағынды суларды тазартуда түйіршікті сүзгі материалдары ретіндегі табиғи минералды шикізат

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ТҮЙІНДЕМЕ

Қазіргі уақытта табиғи және ағынды суларды ауыр металдардан тазартудың қолданыстағы әдістерін талдау сорбенттер ретінде табиғи бейорганикалық материалдарды: цеолит, диатомит, вермикулит қолданатын сорбциялық әдіс перспективалы болып табылатындығын көрсетті. Тақырыптың өзектілігі табиғи сулардың металлургиялық кәсіпорындардың өндірістік сарқынды суларымен ластану деңгейіне тұрақты мониторинг жүргізуге мүмкіндік беретін ағынды суларды тазарту мен су дайындаудың өзіндік технологиясын әзірлеу қажеттілігінен туындады. Сорбенттерді өзгертудің ұсынылған әдістері аталған табиғи материалдардың құрылымын, кеуектілігін, сорбциялық мүмкіндіктерін зерттеуге негіз болады. Аталған үш минерал: цеолит, диатомит, вермикулит жоғары кеуекті құрылымдық материалдарға жатады, оларды суды тазарту мен суды дайындауда тұрақты сорбциялық жүйелер ретінде пайдалану перспективті болып табылады. Бұл жұмыста табиғи сорбенттердің физика-химиялық сипаттамаларының әсері және су дайындау үшін жаңа перспективалы экологиялық қауіпсіз материалдар мен реагенттерді қолдану қарастырылады. Гидротермиялық әдіспен модификациялауға дейінгі және кейінгі табиғи цеолит материалында мыс иондарының сорбциясы зерттелді. Сорбенттердің белсенділігі сорбциялық сыйымдылықтың мөлшері бойынша, яғни цеолит пен оның модификацияланған түрлеріндегі сорбент массасының бірлігімен сіңірілген ауыр металл иондарының мөлшерімен бағаланды.

Түйін сөздер: ағынды сулар, сорбенттер, цеолит, диатомит, вермикулит, клиноптилолит, қалдықтар, минералды шикізат.

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Природное минеральное сырьё в качестве зернистых фильтрующих материалов при очистке природных сточных вод

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АННОТАЦИЯ

Анализ существующих в настоящее время методов очистки природных и сточных вод от тяжелых металлов показал, что одним из перспективных является сорбционный метод с использованием в качестве сорбентов природных неорганических материалов: цеолита, диатомита, вермикулита. Актуальность темы продиктованы необходимостью разработки оригинальной технологии очистки сточных вод и водоподготовки, позволяющей вести постоянный мониторинг уровня загрязнений природных вод производственными сточными водами металлургических предприятий. Предложенные способы модификации сорбентов создают основу для изучения структуры, пористости, сорбционных возможностей названных природных материалов. Все три минерала: цеолит, диатомит, вермикулит относятся к высокопористым, структурированным материалам, перспективным для использования в качестве устойчивых сорбционных систем в водоочистке и водоподготовке. Рассмотрены влияние физико-химических характеристик природных сорбентов, применение новых перспективных экологически безопасных материалов и реагентов для водоподготовки. Исследована сорбции ионов меди на природном цеолитном материале до и после его модификации гидротермальным методом. Активность сорбентов оценивали по величине сорбционной емкости, т.е. количеству ионов тяжелых металлов, поглощенных единицей массы сорбента на основе цеолита и его модифицированной формы.

Ключевые слова: сточные воды, очистка, сорбенты, цеолит, диатомит, вермикулит, клиноптилолит, отходы, минеральное сырье.

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