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Metallurgy



Purification of wastewater from heavy metal ions using nanostructured adsorbents

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ANNOTATION

The activities of industrial enterprises in ferrous and non-ferrous metallurgy and other industries lead to environmental pollution with wastewater containing harmful substances that, even in small quantities, have a rather serious negative impact on human health and the state of the biosphere. There are a large number of natural sorbents used to solve water treatment problems. Among inorganic sorption materials, zeolites are widely used in practice. These natural materials have thermal and radiation stability and high selectivity. The purpose of this article is to study the sorption capacity of zeolites modified with nanostructured rare metals in several ways, with different options for activating the matrix to improve sorption properties with respect to ions of heavy and non-ferrous metals. Based on the experiments conducted, it was proven that zeolites modified with vanadium and titanium nanocompounds are highly effective in removing heavy metal ions from wastewater. The resulting composition on a zeolite matrix creates a highly dispersed solid phase of nanoparticles in the form of a sol-gel. Such systems have an excess of energy, which leads to increased reactivity and adsorbing properties. It is obvious that the activation of zeolites makes it possible to obtain a wider range of active centers of different nature. This determines the varied use of zeolites in the technological system for treating wastewater from heavy and non-ferrous metal ions, which will make it possible to achieve MPC standards.

Key words: zeolite, sorption technologies, heavy metals, wastewater, adsorbent.

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Introduction

Heavy metals, which have a toxic effect on aquatic organisms in even relatively low concentrations, are extremely dangerous in natural waters. The harm of heavy metals to a living organism is due to their ability to bioaccumulate and concentrate, which leads to disruption of the functioning of organ systems. The difficulty of removing heavy metal ions (HTI) from the body is due to the fact that they form strong bonds with proteins and other components of cellular structures [1].

In this regard, wastewater treatment from industrial enterprises must be carried out until heavy metals are almost completely removed. But using only traditional methods this is difficult to achieve.

One of the common methods for treating wastewater from ITM is sorption and ion exchange methods, hence the need to obtain cheaper sorbents with improved physicochemical properties increases [2]. Despite the variety of adsorbents used, many of them do not satisfy the full range of requirements for materials of this type, and therefore the search and development of new sorption materials is ongoing. The most efficient and

cost-effective treatment of water from heavy metal ions is when using materials such as aluminosilicates of various types. It is known that the high sorption properties of natural aluminosilicates, their low cost, and their large reserves in nature serve as the basis for their choice as adsorbents for metal impurities from wastewater from various industries. To expand the scope of natural zeolites and give them new additional properties (anion-exchange, adsorption, magnetic, bactericidal, etc.), zeolites are modified with both organic and inorganic modifiers.

Recently, there has been an increased interest in the use of complex zeolite-containing reagents in the treatment of wastewater of various origins [3]. It should also be noted here that an important approach to the creation of effective and environmentally safe sorbents is the modification of the structure of zeolites due to the formation of new functional groups (sorption-active centers) that strongly bind heavy metal ions. Adsorbents on a zeolite carrier, activated by rare and rare earth elements, deserve special attention, since many of them show selectivity to heavy metals. Therefore, developments devoted to the study of the sorption activity of zeolites modified with rare metals with different types of activation with respect to non-ferrous metal ions are relevant.

In English and Russian language publications, much attention is paid to the toxic effects of heavy metals on living organisms and methods of its neutralization. Works [[4, [5], [6]] provide data on the sources of heavy metals and characteristics of toxic effects.

The solubility of individual heavy metals in aqueous solutions and the extent of their removal using alkaline agents and sodium sulfides are strictly regulated by the United States Environmental Protection Agency.

Among modern methods that provide effective wastewater treatment from various pollutants, including heavy metal ions, a special role belongs to physicochemical technologies [[7], [8], [9], [10]]. One of the environmentally safe methods of surface water purification is the use of the adsorption mechanism, which is most often implemented as one of the stages of the complex technological process of water purification since other methods can lead to secondary pollution of the natural environment [9].

A distinctive feature of sorption is the ability to extract substances from multicomponent mixtures, high efficiency even at low concentrations of pollutants, as well as the quality of the sorption material used.

There are a large number of natural sorbents used to solve water treatment problems, such as quartz sand, activated carbon, chitosan, jute, cellulose, sawdust, bentonite clay, etc. The most common sorbent in domestic and foreign practice for deep purification of drinking water is activated carbons: granular and powdered [11]. But the use of such traditional sorbents in wastewater treatment, in the presence of specific contaminants in the source water, does not provide a guaranteed water quality that meets sanitary rules and regulations.

Among inorganic sorption materials, zeolites are widely used in practice. These natural materials have thermal and radiation stability, high selectivity, fairly good sorption properties, and the possibility of obtaining them in granular form make them very attractive for use in the water sector [12]. Zeolites can absorb not only ions of heavy metals, organic pollutants, oil products, pesticides, radioactive elements, but also pathogenic microorganisms, bacteria and viruses. The ability to extract heavy metals, surfactants, organic substances and other toxicants from water, including biologically stable ones that cannot be removed by other methods, as well as the absence of secondary contaminants, make zeolites especially valuable when used in systems industrial wastewater treatment [13].

An analysis of modern literature [[6], [7], [8], [9], [10], [11], [12], [13]] showed a significant increase in the rate of publications devoted to the study of numerous modifications based on zeolites with the introduction of metal nanoparticles and the scope of the corresponding sorption and catalytic systems. The high surface energy of nanosystems makes them especially attractive for use in many catalytic industries since this property makes it possible to carry out technological processes under milder conditions than traditionally accepted ones. Nanomaterials based on oxides of rare and rare earth metals have unique properties that make them promising for wide use in chemistry and technology.

A promising direction for water purification from impurities of priority environmental pollutants is the use of modified synthetic or natural zeolites, which have a high sorption capacity with respect to many organic and inorganic substances [6].

In order to improve the adsorption properties of natural zeolites, they are modified or activated by heat treatment without destroying the crystal structure [3]. This leads to the fact that their porous open microstructure is capable of regulating the structure, for example, modifying with nanosized particles of rare and rare earth metals, which

determines their unique sorption properties, increases their selectivity, and reduces the sorption time [4].

In order to use natural zeolites for the purification of wastewater from industrial and mining enterprises from salts of heavy metals, the authors of [13] studied the adsorption characteristics of natural zeolites with an exchange capacity of 95–140 meq/100 g during the exchange of sodium cations or ammonium ions for ions lead, cadmium, copper and zinc from aqueous solutions at an adsorption time of 10 - 100 minutes, and the degree of extraction of heavy metal ions from the solution is 90 - 98% and decreases in the indicated series of heavy metals: $Pb > Cd > Zn > Cu > Ni$.

The paper [14] proposes a method for wastewater treatment from heavy metal cations, based on the use of slag formed during the complex processing of steel, which has a complex chemical composition and is characterized by a highly developed surface and the presence of a large number of microcracks, pores, and active centers, which determine the sorption properties. slag. With an increase in the amount of added slag, the purification efficiency increases, which is explained by an increase in pH and an increase in the number of active centers. During the purification process, the efficiency of purification from Fe^{2+} and Fe^{3+} ions was up to 99.5%, from Zn^{2+} ions - up to 97.5% with the addition of 0.7 g/l of slag and the initial concentration of model solutions of 30 mg/l. Shungite rocks of Karelia, which exhibit sorption activity in the process of wastewater treatment from heavy metals and oil products, are also material for solving environmental problems [15].

One of the possible ways to increase the efficiency of wastewater treatment technologies from heavy metals is the use of new nanodispersed sorbents that act as sorbents-coprecipitators of toxic components. Their basis is natural highly dispersed aluminosilicates, zeolites, the surface of which is modified by substances of inorganic and organic nature [[16], [17], [18], [19], [20], [21], [22]].

Thus, the information obtained from published sources is fragmentary and does not contain sufficient information about the possibilities and advantages of using modified aluminosilicates for water purification from heavy metal ions, which necessitated the present research.

Experimental part

The purpose of this work is to study the sorption capacity of zeolites modified with nanostructured rare metals in several ways, with different variants of matrix activation to improve the sorption properties with respect to ions of heavy and nonferrous metals. The results obtained will be used for further development of a hybrid technology for the treatment of process and wastewater.

In this regard, the task of our research is to create a rational method for treating industrial wastewater, which would ensure the implementation of sorption technology, taking into account the economic and environmental requirements for it. The effectiveness of the developed adsorbents in the process of removing heavy metals was assessed using model water.

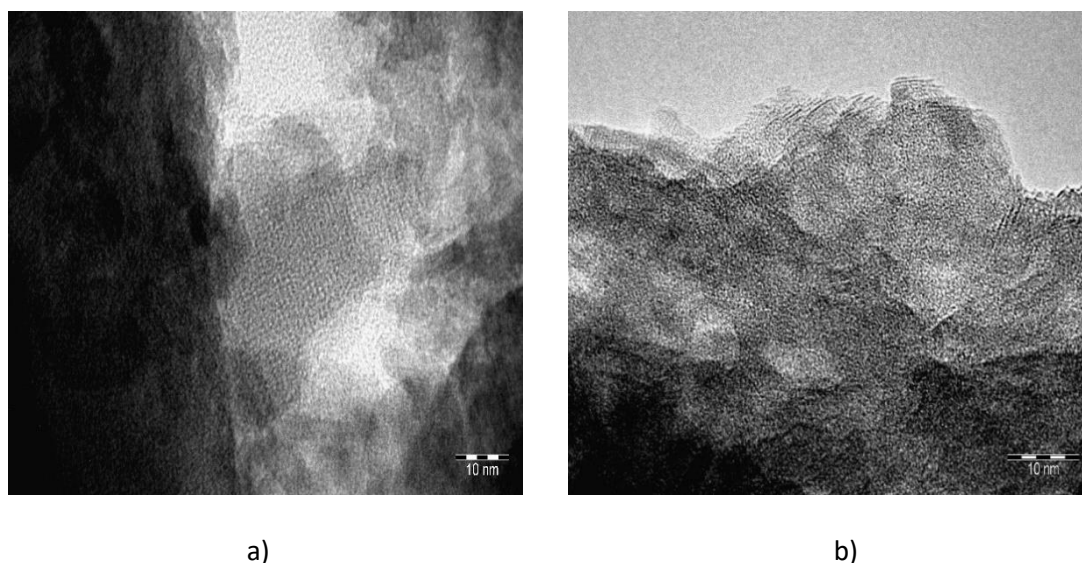


Figure 1 - Scanning electron microscopy of the original zeolite (a) and activated (b)

As adsorbents with high metal consumption, we used aluminosilicate (clinoptilolite), zeolites of the KN-30 brand in statics and dynamics, and modified with a mixture of vanadium and titanium nanopowder. Figure 1 shows SEM images of the original zeolite (A) and activated (B) by nanosized particles of a mixture of rare metals (V + Ti).

According to electron microscopy data, finely dispersed structures with $d \approx 2.0 - 4.0$ nm predominate on the surface of the unmodified ZSM- Al_2O_3 zeolite. The straight channels of the ZSM-5 zeolite intersect with the zigzag channels, forming a three-dimensional structure of narrow channels with sizes of 5.1–5.6 Å. Such a structure causes, on the one hand, the selectivity of the zeolite, and, on the other hand, diffusion restrictions, which lead to low exchange activity.

As can be seen from the electron microscopic image (Fig. 1, B), formations with $d \approx 3.0-5.0$ nm, consisting of V_2O_5 and TiO_2 , prevail on the surface of the activated zeolite. There are structures, the size of which varies within 5.0-7.0 nm, formed by $\text{V}_x\text{Ti}_{1-x}\text{O}_2$. There is also an insertion of vanadium and titanium atoms into the structure of the zeolite and Al_2O_3 with the formation of $\text{V}_4\text{Ti}_2\text{Si}_3\text{O}_{10}$ and $\text{VO}_x\text{Ti}_x\text{AlO}_3$.

Nanoparticles of many substances exhibit properties that make it possible to use them as catalysts and adsorbents, and the question of the size at which the catalytic and sorption features of the nanostate of the studied materials begin to appear becomes important. In addition, it was found that at the nanolevel, the properties of the elements used for modification, for example, nanovanadium, nanocarbon, nanotitanium, etc., change significantly [[5], [19]]. This is apparently due not only to an increase in the active surface of the adsorbent, consisting of nanoparticles, but also to the fact that

a significant proportion of the atoms that form its surface are in the so-called low-coordinated state, in which they exhibit maximum activity, both catalytic and sorption [20].

Results and Discussion

The zeolite sorbent used in the work was activated with nanostructured titanium-vanadium powder prepared from titanium dioxide and ammonium metavanadate. A promising raw material for obtaining a Ti-V-O hybrid composition is middlings and wastes of titanium-magnesium production, as the most accessible and cheap.

During the research, conditions were worked out that combined optimal adsorbent concentrations, mixing mode, and reagent supply rate, ensuring the precipitation and removal of heavy metals from the solution. The work studied sorption properties, and assessed the static exchange capacity (COE, mg/g) under standard conditions (initial concentration of heavy metal ions, ratio of solid to liquid phases 1:10, particle size, static sorption time). Determination of the concentrations of various ions from aqueous solutions was carried out using certified methods in accredited laboratories.

Figure 2 shows the technological scheme of wastewater treatment from ITM under static conditions using the obtained activated zeolites. The process of sorption from wastewater can be carried out under static and dynamic conditions. In the static mode, the liquid particle does not move relative to the sorbent particle; moves along with the latter in a sorption reactor with a stirrer.

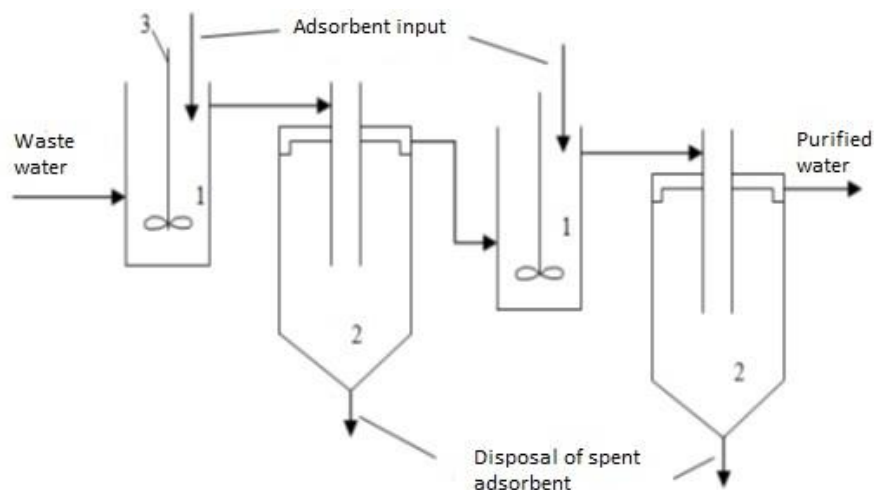


Figure 2 - Scheme of the adsorption plant in static conditions: 1 - sorption reactor; 2 - sump; 3 – stirrer

Table 1 - shows the ZOE values (mg/g) for heavy metal ions when using zeolites.

Zeolites	Cations			
	Cu ²⁺	Ni ²⁺	Zn ²⁺	Pb ²⁺
Aluminosilicate	3.97	1.75	2.28	2.50
(clinoptilolite)	2.53	1.53	1.81	2.20
Zeolite KN-30	2.04	1.47	1.78	1.88
(processing in static)	1.83	1.17	1.53	1.61

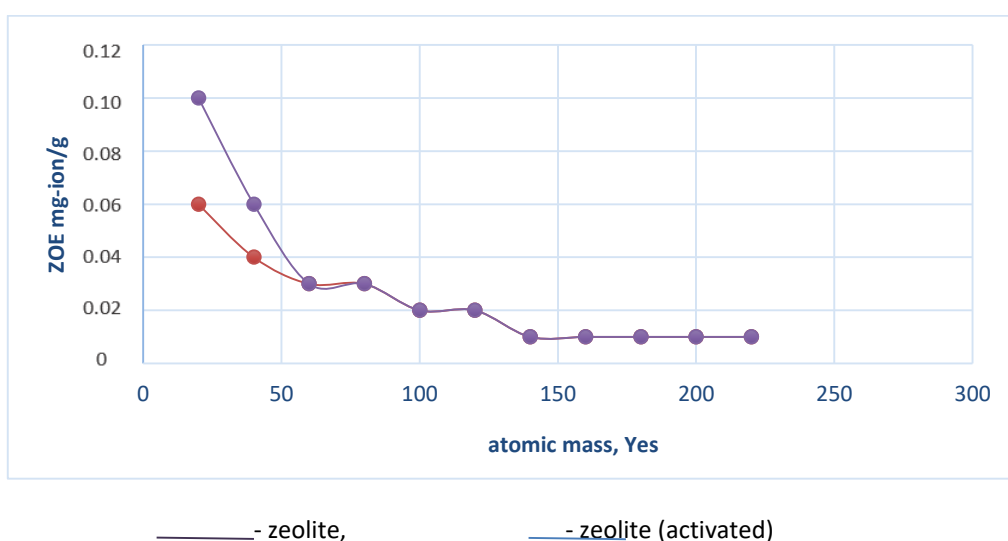


Figure 3 - Dependence of ZOE values (mg-ion/g) for heavy metal ions on atomic mass

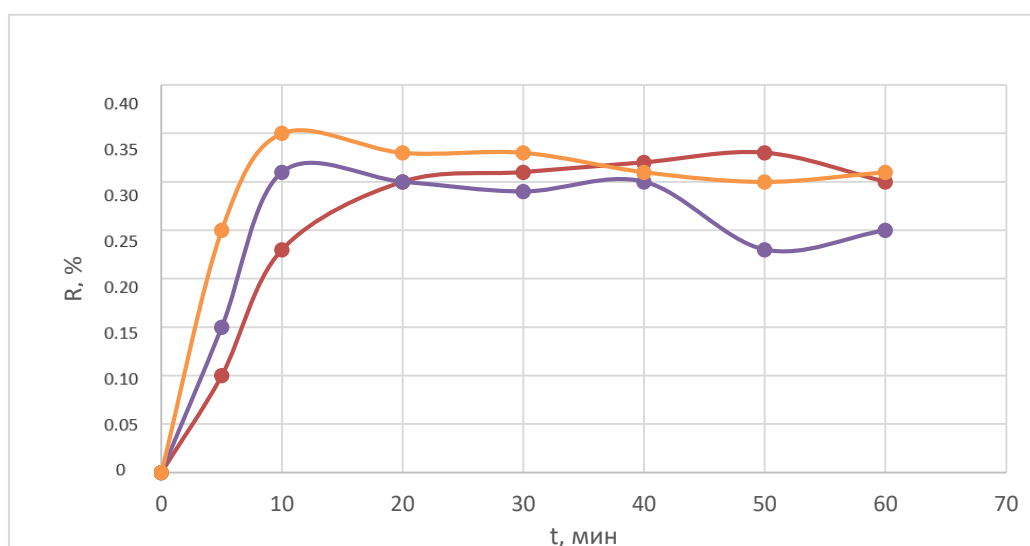


Figure 4- Determination of the sorption capacity of the zeolite from the mass of the introduced element:
 1- Cu(II), 2- Ni(II) 3- Zn(II)

The table shows that the studied zeolite samples have a significant and close sorption capacity for heavy metal ions (Cu^{2+} , Ni^{2+} , Zn^{2+}), and a particularly toxic Pb^{2+} ion, which is currently very likely to be found in natural waters. At the same time, the activated zeolite sample is more selective to the Ni^{2+} and Zn^{2+} ions, and the zeolite (in dynamics) is more selective to the Ni^{2+} ion, although they also have an increased selectivity to the Cu^{2+} and Pb^{2+} ions.

It was shown [11] that the removal of lead from natural waters using natural zeolites is very effective both on pure clinoptilolite rocks and in combination with clinoptilolite with other minerals. In the conducted studies, a sample of the zeolite activated with nanocompounds shows an increased selectivity in relation to the lead ion.

Hence, it follows that the sorption capacity of activated zeolite is much higher than that of synthetic non-activated ones. For example, the dynamic sorption capacity before the breakthrough of 0.01 mg/l was at least 0.022 mg/l, and before the breakthrough of 0.1 mg/l - at least 0.034 mg/l. It should be noted that the dynamic curves of lead ion sorption on natural zeolites differ from the curves on activated samples in a flatter form, which requires the use of a sorption load height of at least 2–2.5 m to obtain effective purification. Figure 3 shows the dependences of ZOE mg-ion/g on the atomic mass of these metals.

The dependences obtained for various zeolite samples practically converge in the region of high values of atomic masses. This makes it possible to determine with high accuracy by extrapolation the limiting value of ZOE for metals, which is very difficult to determine experimentally for high concentrations due to the high contamination of wastewater with multiple impurities.

The sorption capacity of the zeolite was determined experimentally in the process of studying Cu^{2+} , Ni^{2+} , and Zn^{2+} ions.

For the studied "sorbent-metal" system, a series of solutions were prepared with the same content of the sorbent (0.1 g) and different increasing content - 60, 100, 140, and 180 $\mu\text{g/ml}$ of metal cations. The optimum acidity of the medium was created and mixed on magnetic stirrers for the optimum time and at the optimum temperature, which were previously selected for these systems. The amount of the adsorbed element was determined in each experiment, as described above, and a graph was plotted in the coordinates: "the degree of sorption (R, %) - the mass of the introduced element" (Fig. 4).

A specific property of zeolites is the rate of sorption of metal ions, which depends on the structure and properties of the zeolite matrix, the nature of the ion, and the degree of dispersion of the sorbent. When the temperature rises to 60°C, the sorption time is slightly reduced, by an average of 10 minutes.

Studies of the properties and structure of the substrate of various sorption materials and modified zeolite activated with a mixture of titanium and vanadium nanopowder applied powder showed that an increase in adsorption on the sorbent occurs on a highly developed substrate structure due to the formation of adsorption layers on the surface and in the pores activated by the powder. The activity of the modified zeolite depends on the rate of formation and subsequent transformation of surface intermediates, which is determined by the nature of the interaction, that is, the nature of emerging and breaking bonds, in some cases also by the spatial configuration.

Table 2 - Results of chemical analysis of wastewater treatment from heavy metals in the presence of various sorption materials

Applied sorption materials	Metal content after treatment, mg/l				Degree of removal, %			
	Cu^{2+}	Zn^{2+}	Ni^{2+}	Pb^{2+}	Cu^{2+}	Zn^{2+}	Ni^{2+}	Pb^{2+}
Clinoptilolite	0.06	0.16	0.08	0.13	89.2	74.2	86.7	59.4
Zeolite KN-30 (processing in static)	0.05	0.12	0.042	0.11	91.1	80.6	93.0	65.6
Zeolite processing in dynamics	0.05	0.09	0.024	0.08	91.2	85.5	96.0	75.4
Zeolite activated	0.02	0.02	0.012	0.04	96.4	96.8	98.0	87.5

Table 2 shows the results of processing a model solution containing heavy and non-ferrous metals with the sorption materials used. Initial concentration of metals in the model solution, mg/l: Cu^{2+} -0.56; Zn^{2+} - 0.62; Ni^{2+} - 0.60; Pb^{2+} - 0.32

An analysis of the results obtained (Table 2) allows us to draw the following conclusions: the best sorption capacity under static conditions with respect to heavy metal ions has a zeolite modified with vanadium and titanium, after activation, the degree of extraction for the studied ITMs ranged from 87.5 to 98, 0%. The chemical modification of the zeolite with the use of nanostructured vanadium and titanium compounds significantly increased the sorption capacity with respect to $\text{Cu}(\text{II})$, $\text{Ni}(\text{II})$, and $\text{Zn}(\text{II})$ ions; good sorption properties are also observed for Pb^{2+} , but the recovery is lower. It can be seen that the sorption capacity of zeolites activated with rare metals is much higher than that of activated carbons [5]. It is known [13] that after water treatment with technical aluminum sulfate and two-stage mechanical filtration, the zinc content in it increases significantly. Cleaning with activated zeolite allows, as can be seen from the table, not only post-treatment for zinc but also to reduce the odor, color, and ammonium ion content. To remove the ammonium ion, the sorption method using natural zeolites, including modified ones, is also the most effective, which was shown in [[20], [22]].

Based on the experiments conducted, it was proven that zeolites modified with vanadium and titanium nanocompounds are highly effective in the process of removing heavy metal ions from wastewater. The degree of metal removal was (%): Cu^{2+} - 96.4; Zn^{2+} - 96.8; Ni^{2+} - 98.0; Pb^{2+} - 87.5. Particular consideration should be given to the removal of lead ions, which are widespread in industrial wastewater, using the developed nanosorbent. As has been shown [6], the removal of lead from natural waters using natural zeolites is very effective both on pure clinoptilolite rocks and in a combination of clinoptilolite with other minerals but does not ensure water quality that meets sanitary standards [11]. The significant sorption capacity of the studied samples for the lead ion Pb^{2+} makes it possible to use them for the purification of stormwater and detoxification of soils in adjacent areas of large highways. As a result of studies of the

sorption properties of nanostructured adsorbents based on a zeolite matrix, their ability to extract heavy metal ions from water, as well as to remove ammonia nitrogen, has been proven.

Conclusion

The resulting composition on a zeolite matrix creates a highly dispersed solid phase of nanoparticles in the form of a sol-gel. Such systems have an excess of energy, which leads to increased reactivity and adsorbing properties. It is obvious that the activation of zeolites makes it possible to obtain a wider range of active centers of different natures. This determines the varied use of zeolites in the technological system for treating wastewater from heavy and non-ferrous metal ions, which will make it possible to achieve MPC standards.

Modification of zeolite-containing rocks with nanostructured compounds of rare metals changes their physical and chemical characteristics, their molecular structure is ordered and acid and adsorption properties are improved.

Thus, the study of the processes of sorption of heavy non-ferrous metals by natural materials, such as zeolites, is of practical interest to the water sector of industry. Of particular priority is the production of relatively cheap sorption materials based on industrial waste, since this waste is reused. Industrial wastes used as adsorbents do not have the main disadvantage of most used adsorbents - high cost. Taking into account the high cost of individual compounds of vanadium and titanium, samples of nanostructured adsorbents were obtained from technogenic vanadium-titanium-containing solutions of the titanium-magnesium plant (Ust-Kamenogorsk), which showed high efficiency. This advantage will reduce the amount of production waste, which will have a positive impact on capital costs. The use of waste and by-products of the metallurgical complex to activate matrix structures is promising and economically beneficial for purifying aqueous solutions of various compositions from ITM, ranging from wastewater from industrial enterprises to natural waters and food systems.

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

Наноқұрылымды адсорбенттерді қолдану арқылы ағынды суларды ауыр металл иондарынан тазарту

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ТҮЙІНДЕМЕ

Қара және түсті металлургия және басқа да салалардағы өнеркәсіптік кәсіпорындардың қызметі қоршаған ортаның зиянды заттары бар ағынды сулармен ластануына әкеліп соғады, олар аз мөлшерде болса да адам денсаулығына және биосфера жағдайына айтарлықтай теріс әсер етеді. Суды тазарту мәселелерін шешу үшін қолданылатын көптеген табиғи сорбенттер бар. Бейорганикалық сорбциялық материалдардың ішінде тәжірибеде цеолиттер кеңінен қолданылады. Бұл табиғи материалдар термиялық және радиациялық тұрақтылыққа және жоғары селективтілікке ие. Бұл мақаланың мақсаты ауыр және түсті металдардың иондарына қатысты сорбциялық қасиеттерін жақсарту үшін матрицаны белсендірудің әртүрлі нұсқалары бар наноқұрылымды сирек металдармен модификацияланған цеолиттердің сорбциялық қабілетін бірнеше тәсілдермен зерттеу болып табылады. Жүргізілген тәжірибелер негізінде ванадий және титан наноқосылыстарымен модификацияланған цеолиттердің ағынды сулардан ауыр металл иондарын кетіруде тиімділігі жоғары екендігі дәлелденді. Цеолит матрицасында алынған композиция золь-гель түріндегі нанобөлшектердің жоғары дисперсті қатты фазасын жасайды. Мұндай жүйелерде энергияның артық мөлшері болады, бұл реакциялық және адсорбциялық қасиеттердің жоғарылауына әкеледі. Цеолиттерді белсендіру табиғаты әртүрлі белсенді орталықтардың кең спектрін алуға мүмкіндік беретіні анық. Бұл ауыр және түсті металл иондарынан ағынды суларды тазартудың технологиялық жүйесінде цеолиттердің әртүрлі қолданылуын анықтайды, бұл ШРК (шекті рұқсат етілген концентрация) стандарттарына қол жеткізуге мүмкіндік береді.

Түйін сөздер: цеолит, сорбциялық технологиялар, ауыр металдар, ағынды сулар, адсорбент.

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Очистка сточных вод от ионов тяжелых металлов с применением наноструктурных адсорбентов

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АННОТАЦИЯ

Деятельность промышленных предприятий чёрной и цветной металлургии и других отраслей приводит к загрязнению окружающей среды сточными водами, содержащими в своём составе вредные вещества, оказывающие и в небольших количествах довольно серьёзное негативное воздействие на здоровье человека и состояние биосферы. Существует большое количество природных сорбентов, используемых для решения задач водоочистки. Среди неорганических сорбционных материалов на практике широко используются

цеолиты. Эти природные материалы обладают термической и радиационной стабильностью, высокой селективностью. Цель данной статьи — изучение сорбционной способности цеолитов, модифицированных наноструктурированными редкими металлами несколькими способами, при разных вариантах активации матрицы для улучшения сорбционных свойств по отношению к ионам тяжелых и цветных металлов. На основании проведенных экспериментов было доказано, что цеолиты, модифицированные наносоединениями ванадия и титана, обладают высокой эффективностью в процессах удаления ионов тяжелых металлов из сточных вод. Полученная композиция на цеолитной матрице создает высокодисперсную твердую фазу из наночастиц в форме золя-геля. Такие системы имеют переизбыток энергии, что и приводит к повышенной реакционной способности и адсорбирующим свойствам. Очевидно, что активирование цеолитов позволяет получить более широкий набор активных центров различной природы. Это обуславливает разнообразное использование цеолитов в технологической системе очистки сточных вод от ионов тяжелых и цветных металлов, ввиду чего станет возможным достижение нормативов ПДК.

Ключевые слова: цеолит, сорбционные технологии, тяжелые металлы, сточные воды, адсорбент.

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