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Research on the production of sorbent based on bentonite clay for wastewater treatment from chemical industries

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ABSTRACT
In our country, the problem of water purification remains urgent, with the growth of external factors contributing to this, which can include an increase in the number of industrial enterprises, the development of agriculture, urban growth and others. To solve the problem of water purification it is economically advantageous to create new sorbents, with the available resources in our country.For the processing of of bentonite clay under experimental conditions, instrumental test methods were chosen using a scanning electron microscope (SEM) of the Jeol JSM-6490l V brand, a multi-parameter portable cyberscanner (PCD 650 Eutech) and a Q-1500 Derivatograph. In this article, we studied the bentonite of the Darbaza deposit. For physico-chemical analysis of clay from the Darbaza deposit, a specially selected sample was crushed, and sieved and its chemical composition was determined. *Results.* Based on the results of instrumental studies, the elemental and mineralogical composition of bentonite clay from the Darbazinsky deposit was determined using a scanning electron microscope JSM-6490l V (Jeol, Japan) using the energy-dispersive method. The resulting sorbent based on bentonite clay has a high sorption capacity and is recommended for use in the treatment of wastewater from chemical industries. The resulting sorbent based on bentonite clay from the Darbaza deposit makes it possible to purify wastewater from phosphate production from phosphate and other ions up to 60%. The developed sorbent based on bentonite clay has environmental and economic efficiency and connections with the use of local natural resources. Thus, it should be noted that for adsorption treatment of wastewater from chemical industries with a high degree, it is possible to use effective sorbents based on bentonine clays from the Darbaza deposit. It should also be noted that the use of bentonite clays for water purification by the sorption process is an effective and affordable alternative to adsorbents that show high adsorption capacity for various compounds.
Keywords: bentonite, Darbaza deposit, water treatment, montmorillonite, adsorption, heavy metals.

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Introduction

The problem of wastewater treatment, since the second half of the 20th century, is relevant for all countries of the world. From the colloid-chemical point of view, wastewater is a heterogeneous mixture of dissolved, colloidal and suspended-in-water impurities of organic and inorganic nature. One of the main pollutants of natural waters is heavy metal ions coming with the wastewater from galvanic shops, mining, ferrous and non-ferrous metallurgy, and machine-building plants.

There are quite a lot of purification methods, but the simplest and most effective methods of water purification are adsorption methods [[1], [2], [3], [4]]. The advantages of these methods are high efficiency, possibility to treat wastewater containing several substances, as well as recovery of these substances. The efficiency of adsorption purification reaches 80-95 % and depends on the chemical nature of the adsorbent, the size of the adsorption surface and its availability, the chemical structure of the substance and the chemical form of its presence in the medium. Kazakhstan is famous for its rich deposits of various minerals, as well as natural clay - bentonite, the properties of which will be investigated in this article. The diverse application of bentonite is conditioned by its physical and chemical abilities. The greatest use of bentonite clay is in medicine, drilling, metallurgy, foundry, construction, agriculture, environmental programs, adsorbents, fillers, etc [[5], [6], [7], [8], [9]].

The world bentonite reserve is 5.5 billion tons, 45% of which is accounted for by China, and 15% by the United States, the remaining reserve is accounted for by Russia, Turkey, Greece, India, etc. Kazakhstan has large reserves of bentonite clay, only in South Kazakhstan there are more than 6 deposits of bentonite, Keles, Darbaza, Lenger, Andreevs, Dzerzhin, Ildersay, etc., in East Kazakhstan there are known such deposits as Tagans, Dinosaur, Manraksk, etc. The total reserve of bentonite clay is more than 150 million tons [10].

Since the problem of water purification from various pollutants remains urgent, and our country is rich in the presence of many deposits of bentonite clay, which in composition is not inferior to Wyoming bentonite (USA), it is economically advantageous to use it for water purification. Of the many known methods of purification, sorption methods of purification are economically advantageous and effective, as the efficiency of sorption can reach 80-90% depending on the nature of the adsorbent used [11].

Natural clay minerals are widely used as sorbents. They have good physical and chemical characteristics as well as high absorption capacity. Adsorption properties of bentonites can be regulated both by methods of physical action and by chemical activation.

From a practical point of view, bentonite occupies a special place among clay minerals. One of the areas of bentonite use is wastewater treatment from chemical contaminants, where it is used as a sorbent.

Experimental part

The initial sample of clay with sorbing properties was selected in the South Kazakhstan region, Saryagash district, Darbaza deposit.

The following methods of physical and chemical analysis were used to study the structure and structure of clay: scanning electron microscope, FTIR spectrometer and X-ray phase analysis. The chemical composition of bentonite was determined using a JSM6490LV scanning electron microscope. The IR absorption spectra were taken on a ShimadzuIRPrestige-21 FT-IR spectrometer with a Miracle total internal reflection (TIR) attachment by PikeTechnologies.X-ray phase analysis was carried out on a Bruker D8ENDEAVOR diffractometer (Germany).

Differential-thermal analysis of the initial sample of bentonite clay of Darbazin deposit, selected on the floodplain of the Keles River was performed on a Q-1500 derivatograph in the laboratory "Sapa" of M. Auezov SKU. For physico-chemical analysis of clay from Darbaza deposit, a specially selected sample was crushed, and sieved and its chemical composition was determined. Figure 1 shows bentonite samples in the original and pulverized form.

Bentonite clay is a complex rock, the base of which is montmorillonite, which has the formula $Si_8Al_4O_{20}(OH)_4 \times nH_2O$, where silicon can be replaced by various cations (aluminum, iron, zinc, magnesium, calcium, sodium, potassium, etc.). Montmorillonite has a three-layer crystalline structure, the outer layers are silicon-oxygen tetrahedrons, with silicon in the middle and associated hydroxyl groups or oxygen at equal distances from it. The middle layer is represented as octahedrons with oxygen atoms or hydroxyl groups in the tops and aluminum, iron or magnesium atoms between them [[12], [13]].

Figure 1 - Bentonite samples in initial and crushed form of Darbaza deposit

In order to study the composition of bentonite clay, the result of a scanning electron microscope (SEM) was obtained (Figure 2), and the elemental composition of the initial clay sample of the Darbaza deposit was determined (Table 1).

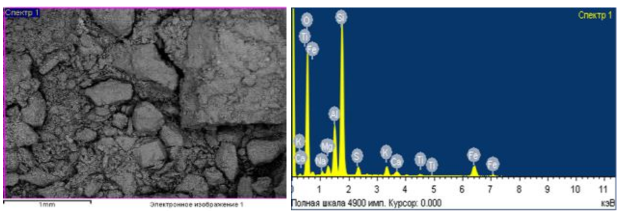


Figure 2 – Microstructure and elemental analysis of bentonite clay of Darbaza deposit

Table 1 - Elemental composition of bentonite clay of Darbaza deposit

Elemental composition of clay Weight %							
Na_2O	MgO	Al_2O_3	SiO_2	K_2O	CaO	TiO_2	Fe_2O_3
1.48	2.61	14.87	51.4	2.66	1.29	0.80	9.98

The elemental composition of the sample shows that the bentonite of the Darbaza deposit contains large amounts of silicon, aluminum and iron compounds, which explains the adsorption capacity of the studied object, it should also be noted the content of associated metal compounds in the form of oxides, hydroxides, carbonate and sulfate salts, which reduce the adsorption capacity of the object. In practice, to improve adsorption properties bentonite is subjected to activation, which in turn is divided into chemical and acidic [14].

To determine the mineral composition of the clay of the Darbaza deposit, IR spectroscopy and X-ray phase analysis were performed (Figure 3,4).

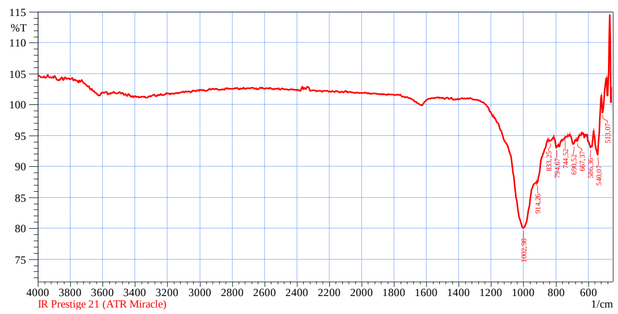


Figure 3 - IR spectrum of bentonite from Darbaza deposit

Decoding of spectra shows that in bentonite of Darbaza deposit small peaks in the region of 1600-1700cm^{-1} and 3600-3700cm^{-1} are observed, which are characteristic for Na-O and O-H groups. The presence of peaks in the region of 950-1050 cm^{-1} confirms the presence of the main group Si-O, which is one of the main constituents of bentonite. Peaks in the region of 740-800 cm^{-1} which are characteristic of Al-O are observed.

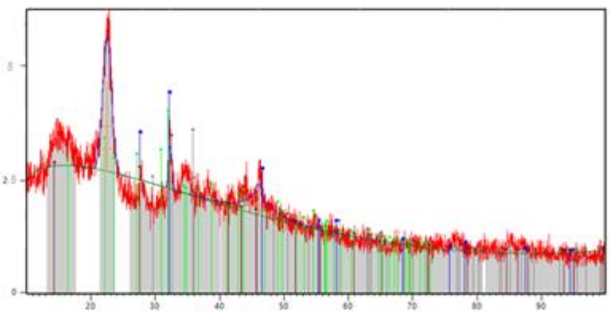


Figure 4 - XRF of Darbaza bentonite

Analysis of the data obtained by XRF showed that the bentonite clay sample from Darbaza deposit has three intense and several non-intense diffraction maxima: - d equal to 5.02; 3.342; 1.55 are characteristic of the phases of muscovite, which has the formula $K_2O{\cdot}3Al_2O_3{\cdot}6SiO_2{\cdot}2H_2O$;

- d equal to 3.21; 2.77; 2.227; 1.995; 1.560; 1.49; 1.292 are characteristic phases of potassium feldspar, which has the formula $Na_2O{\cdot}Al_2O_3{\cdot}6SiO_2$;

- d equal to 4.47; 3.42; 3.06; 2.60 are characteristic of phases of montmorillonite, which has the formula $Si_8Al_4O_{20}(OH)_4 \times nH_2O$;

- d equal to 3.85; 2.291; characteristic of kaolinite phases, $Al_2O_3{\cdot}2SiO_2{*}2H_2O$.

The presence of minerals, bentonite muscovite explains the sorption properties of the studied bentonite sample. Graphical dependences of DTG and DTA curves on temperature are shown in Figure 5.

Figure 5 - DTA of Darbaza bentonite

Analysis of the obtained graphical data showed that the studied sample is characterized by two pronounced and one insignificant endo-effects and four exo-effects. Endoeffect at T=180°C indicates the removal of surface moisture and at 550 °C partial desulfatization of iron-containing impurities. The exoeffect at 870°C is characteristic of decarbonization of magnesium and calcium carbonates. Insignificant exo-effects at 280-450°C are characteristic of burnout of organic impurity components. Exoeffects at 525-530 °C indicate the burnout of sulfur impurities of bentonite clay.

Activation [15] of bentonite clay is carried out as follows: A certain mass of bentonite clay is suspended in a water-sprite solution (ratio 1:1.5 cm^3, temperature 80-100 ^{0}C) for 60 minutes and at the same time a modifier solution is prepared by dissolving octadecylamine in aqueous-alcohol a solution containing an equimolar amount of hydrochloric acid (up to pH = 7.0) at the same temperature. A solution of octadecylammonium chloride salt is added in portions to the bentonite clay suspension with continuous stirring throughout the entire period of the activation process. After dosing of the modifier solution is completed, the suspension is stirred for an additional 30 minutes and cooled to room temperature.

To separate unbound activated bentonite clay, it is centrifuged repeatedly, washed with a hot aqueous-alcohol solution, and then with distilled water [16]. The completeness of washing is assessed by the residual content of chlorine ions by adding a few drops of a 5% silver nitrate solution to the mother solution. The resulting product is dried at a temperature of 80^0C in a vacuum to constant weight. The cation exchange capacity of bentonoite clay is 90 mEq/100 g with an average silicate platelet diameter of ~75 nm. To determine the functional groups of the resulting activated bentonite clay, spectral analysis was carried out with an IR Fourier spectrometer (Zhimadzu IR Prestige-21). The result of IR spectroscopy after the activation process is shown in Figure 6.

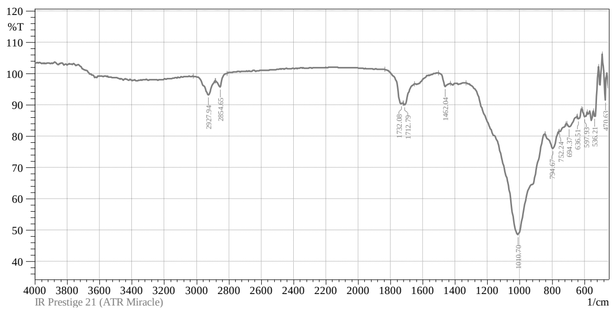


Figure 6 – IR absorption spectra of Darbaza bentonite clay after activation

According to the results of the IR spectra it was revealed that Darbaza activated bentonite clay appears absorption bands in the region of 2924 and 2854cm^{-1} related to stretching vibrations of the methylene group and also corresponds to the asymmetric bending vibration of methyl groups in the region of 1458cm^{-1}. The absorption bands at 1712-1735 cm^{-1} are characteristic of stretching vibrations of acrylate groups. Pendulum vibrations of methylene groups in the region of 798 cm^{-1} are also observed. There is a slight increase in the absorption band with a peak at 1010 cm^{-1} (1100-837 cm^{-1}) corresponding to the stretching vibrations of the Si-O-Si group.

Table 2 – Physico-chemical and mechanical properties of sorbents based on bentonite clay

Name	Density, g/cm^3	Specific surface area, cm^2/g
Bentonite clay from the Darbaza deposit	2.07	1380
	Average granule, mm	Strength, MPa
	10-13	4.4
Activated bentonite clay from the Darbaza deposit	Density, g/cm^3	Specific surface area, cm^2/g
	2.30	1445
	Average granule, mm	Strength, MPa
	10-13	5.25

From Table 2 it follows that the physico-chemical and physico-mechanical parameters of acid-activated bentonite clay are higher than those of a conventional bentonite clay sample and can increase the mechanical strength of sorbents to 5.25 MPa, which facilitates repeated use for wastewater treatment.

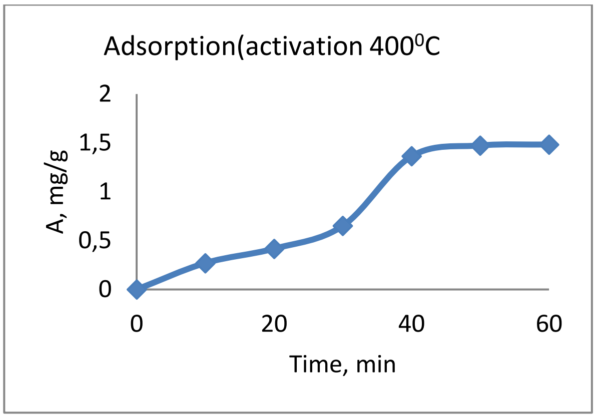
Determination of the physicochemical and mechanical parameters of the sorbent based on bentonite clay before and after activation was carried out by GOST 28177-89 [[17], [18]].

The discussion of the results

The obtained results of elemental analysis in Table-1 confirm that the bentonite clay of the Darbaza deposit belongs to the alkaline earth group. Since its complex consists of calcium and magnesium cations. Bentonite has a porous structure, due to porosity - high swelling, which determines their optimal sorption characteristics. The mechanism of sorption of bentonite clays is that sorbates are introduced between planes, while the structure the layers themselves remain unchanged. It should be noted that scientists [19] have concluded that the sorption process on bentonite clay is predominantly a physical process, includes ion exchange and is controlled by diffusion in the solution film.

The resulting sorbent based on bentonite clay has a high sorption capacity and is recommended for use in the treatment of wastewater from chemical industries.

The sorption capacity and the dependence of the process on the time and temperature of the resulting sorbent are shown in Figure 7.



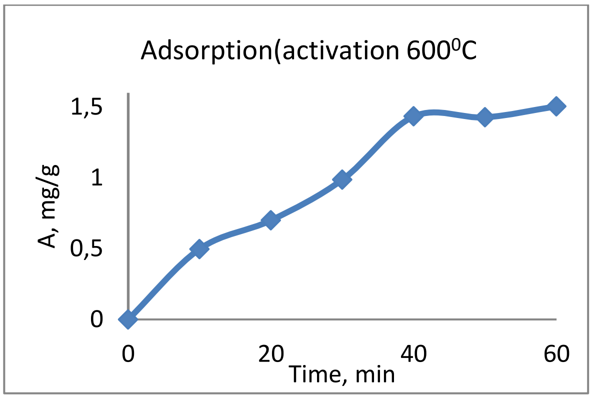


Figure 7 - Dependence of adsorption on process time.

From Figure 7 it follows that the adsorption capacity of the resulting sorbent increases by 10-15% after activation. The studies were carried out on a laboratory installation with software control of a liquid flow rate of 3.5-4.0l/min, in the temperature range of 20-40^{0}C. In laboratory studies, we used wastewater from the mineral fertilizer plant (MFP) of LLP «Kazphosphate», characterized by the presence of the following chemical compounds, %: P_2O_5-2,0-2,1, SO_3-0,6-0,8, CaO-2,8-3,1, K_2O-0,6-0,8, MgO -0,8-1,1, $A1_2O_3$-0,8-0,9, Fe_2O_3-0,3-0,4.

After using the sorbent, the chemical composition of the wastewater has the following composition, %: P_2O_5-1,1,0-1,2, SO_3-0,3-0,33 CaO-1,5-1,7, K_2O-0,2-0,2,5 MgO -0,3-0,4, $A1_2O_3$-0,3-0,4, Fe_2O_3-0,1-0,15.

The composition of wastewater before and after the sorption process was determined according to GOST 26449.1 [20].

Based on the results of the experimental studies, it can be concluded that the resulting sorbent based on bentonite clay from the Darbaza deposit makes it possible to purify wastewater from phosphate production from phosphate and other ions up to 60%.

Conclusions

Based on the results of instrumental studies, the elemental and mineralogical composition of bentonite clay from the Darbazinsky deposit was determined using a scanning electron microscope JSM-6490I V (Jeol, Japan) using the energy-dispersive method. Differential thermal analysis was used on a bentonite clay sample to monitor changes in the raw material that occur as a result of heat treatment. Based on X-ray phase analysis, the phase composition of bentonite clay was determined. IR spectroscopic analysis was also carried out to determine the structural features of the starting raw materials and the finished product.

The developed sorbent based on bentonite clay has environmental and economic efficiency and connections with the use of local natural resources. Thus, it should be noted that for the adsorption treatment of wastewater from chemical industries, it is possible to use effective sorbents based on bentonine clays from the Darbaza deposit to a high degree. It should also be noted that the use of bentonite clays for water purification by the sorption process is an effective and affordable alternative to adsorbents that show high adsorption capacity for various compounds.

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Химия өндірісінің ағынды суларын тазалау үшін бентонит сазы негізінде сорбент алу әдісін зерттеу

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ТҮЙІНДЕМЕ
Біздің елімізде суды тазарту мәселесі өзекті болып қала береді. Бұған өнеркәсіптік кәсіпорындар санының көбеюі, ауыл шаруашылығының дамуы, қалалардың өсуі және т.б. сыртқы факторлардың өсуі ықпал етеді. Суды тазарту мәселесін шешу үшін біздің елімізде бар ресурстардан жаңа сорбенттер алу экономикалық тұрғыдан тиімді болып табылады. Эксперименттік жағдайларда бентонит сазын өңдеу үшін Jeol JSM-6490l V сканерлеуші электронды микроскоп (SEM), көп параметрлі портативті кибер сканер (PCD 650 Eutech) және Q-1500 дериватографы арқылы аспаптық сынақ әдістері таңдалды. Зерттеулердің нәтижелері бойынша энергия-дисперсиялық әдіспен JSM-6490l V (Джеол, Жапония) сканерлеуші электронды микроскоптың көмегімен Дарбаза кен орнындағы бентонит сазының элементтік және минералогиялық құрамы анықталды. Алынған бентонит балшық негізіндегі сорбент жоғары сорбциялық қабілетке ие және химия өнеркәсібінің ағынды суларын тазартуда қолдануға ұсынылады. Дарбаза кен орнынан алынған бентонит сазының негізінде алынған сорбент фосфат өндірісінің ағынды суларын фосфаттан және басқа иондардан 60%-ға дейін тазартуға мүмкіндік береді. Бентонит сазы негізінде жасалған сорбент жергілікті табиғи ресурстарды пайдаланылғандықтан экологиялық және экономикалық тиімді болып келеді. Сонымен, химия өнеркәсібінің ағынды суларын адсорбциялық тазарту үшін Дарбаза кен орнының бентонитті саз негізіндегі тиімді сорбенттерді жоғары дәрежеде қолдануға болатындығы анықталды. Сондай-ақ, сорбциялық процесс арқылы суды тазарту үшін бентонит саздарын пайдалану әртүрлі қосылыстар үшін жоғары адсорбциялық қабілетін көрсететін адсорбенттерге тиімді және қолжетімді балама болып табылатынын атап өткен жөн.

Түйін сөздер: бентонит, Дарбаза кен орны, суды тазарту, монтмориллонит, адсорбция, ауыр металдар.

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Исследование получения сорбента на основе бентонитовой глины для очистки сточных вод химических производств

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АННОТАЦИЯ
В нашей стране проблема очистки воды остается актуальной, причем этому способствует рост внешних факторов, к которым можно отнести увеличение количества промышленных предприятий, развитие сельского хозяйства, рост городов и другие. Для решения проблемы очистки воды экономически выгодно создание новых сорбентов, из имеющихся в нашей стране ресурсов. Для обработки бентонитовой глины в экспериментальных условиях были выбраны инструментальные методы испытаний с использованием сканирующего электронного микроскопа (СЭМ) марки Jeol JSM-6490l V, многопараметрического портативного киберсканера (PCD 650 Eutech) и дериватографа Q-1500. По результатам инструментальных исследований определен элементный и минералогический состав бентонитовой глины из Дарбазинского месторождения с использованием сканирующего растрового электронного микроскопа JSM-6490l V (Jeol, Япония) энерго-дисперсионным методом. Полученный сорбент на основе бентонитовой глины имеет высокую сорбционную способность и рекомендуется применять при очистке сточных вод химических производств. Полученный сорбент на основе бентонитовой глины из Дарбазинского месторождения позволяет очистить сточные воды фосфатного производства от фосфатных и других ионов до 60%. Разработанный сорбент на основе бентонитовой глины имеет экологическую и

экономическую эффективность, связи с использованием местных природных ресурсов. Таким образом, следует отметить, что для адсорбционной очистки сточных вод химических производств с высокой степенью возможно использование эффективных сорбентов на основе бентониновых глин из Дарбазинского месторождения. Также следует отметить, что использование бентонитовых глин для очистки воды процессом сорбции является эффективной и доступной альтернативой адсорбентов, которые показывают высокую адсорбционную емкость по отношению к различным соединениям.

Ключевые слова: бентонит, месторождение Дарбаза, очистка воды, монтмориллонит, адсорбция, тяжелые металлы.

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