

Texture and Sorption Characteristics of Bentonite-Based Sorbents

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ABSTRACT

Studies have been carried out by modifying local bentonites with acid and heat treatment, which activate the sorption properties, due to the cation exchange of the initial elements of montmorillonite minerals.

The physicochemical properties of natural bentonite and sorbents based on it have been studied. It was found that the modification of natural bentonite leads to a change in the chemical composition, structural and sorption properties. It is shown that modified sorbents based on natural bentonite are finely porous (nanostructured) objects with a predominance of pores with a size of 1.4-8.2 nm. The specific surface area of the sorbents depends on the modification method and the amount of the modifying component introduced.

The relevance of the development is determined by the growing interest in the creation of new environmentally friendly sorbents from natural aluminosilicates. However, the lack of effective granulation technologies is a deterrent to the widespread use of bentonites for water purification, since clay minerals are subject to the dispersion effect in aqueous media.

Methods and materials: to justify the possibility of using the developed technology for granulating bentonite, as well as the obtained samples of sorbents in the process of water treatment, the mineralogical composition of the granules was studied by X-ray phase analysis, the analysis of the specific surface, porosity (pore volume, pore distribution along the radius) by the method of sorption and capillary condensation of gases, their chemical and mechanical resistance were determined. After studying the physical properties, the sorption capacity of the samples was evaluated.

Results: a detailed analysis of the physical and chemical properties of the developed sorbents showed that the developed sorbents meet the requirements of national standard and are effective for use both as an independent filter material and as a component in water treatment systems.

The selective sorption properties of bentonite clay samples from the Navbakhor deposit in the Republic of Uzbekistan were studied. Mechanical enrichment of the selected bentonite clay samples was carried out, and the parameters of their selective sorption with respect to phenol were determined. A comparative analysis of the sorption properties of natural and enriched samples of bentonite clay showed that the main mineral is montmorillonite.

The purpose of this work is to study the properties of sorption materials based on bentonite clay.

KEYWORDS

bentonite, modifications, activation, sorption, activation energy.

INTRODUCTION

Promising sorbents for wastewater treatment from heavy metal ions are clay alumina silicate minerals, mainly bentonite clays (montmorillonite and beidellite), kaolinite, biotite, vermiculite, attapulgite (palygorskite). Natural clay minerals are inferior to zeolites in terms of adsorption characteristics. The adsorption activity of minerals is determined by the features of their crystal structure, as well as by their chemical and mineralogical composition. The specific surface area of clay minerals is $15-500 \text{ m}^2 / \text{g}$ [1]. Clay natural sorbents are highly dispersed polymer systems with a complex chemical composition. For clay minerals, as well as for zeolites, along with ion exchange, physical and molecular sorption is characteristic. Physical sorption is caused by the presence of some excess negative charge on the faces of crystals and surface hydroxide groups of an acidic and basic nature, capable of ionization. The presence of OH groups also causes a small ability to anionic exchange observed in layered minerals. In molecular sorption, the sorbed substances are located between the planes of the packages, destroying the original aquocomplexes, without changing the structure of the layers themselves. At the same time, the distance between the layers increases, since the clay mineral swells intra-laminar, which distinguishes it from zeolite, which is not capable of swelling [2]. In order to increase the sorption properties, the materials used for cleaning solutions containing heavy metal ions are subjected to various modifications [3-5]. The resulting changes in the properties due to the dissolution of a certain part of the oxides leads to an increase in the specific surface area and porosity of the sorbents. However, for these purposes, expensive reagents are used, and new waste water is formed, which must be treated. Modern technologies that use sorbents require qualitatively new sorption materials with increased capacity and high selectivity of action. Conventional methods of modifying sorbents by grafting chemical functional groups on their surface do not

achieve a drastic change in the properties of natural sorbents. With the usual modification of a natural sorbent, the porous structure of the material remains the same, but only the chemical nature of its surface changes by fixing the modifying agent on its active centers in quantities that usually do not exceed 1-5% of the mass of the sorbent. The search for other ways of modifying natural sorbents led to the creation of a new type of sorption-active materials, the so-called semi-synthetic sorbents [6-13]. The adsorption and other properties of natural sorbents and their optimal activation conditions are determined on the basis of a complex of physico-chemical and adsorption structural properties. The final stage of laboratory testing is to determine the suitability of sorbents for a particular technological process. Activation and modification of inorganic natural sorbents is carried out in order to change their properties in a targeted manner. There are a number of effective methods of chemical and physical modification of the surface and regulation of the porosity of sorbents [14-18].

Physical methods of sorbent activation are reduced to the processing of samples in mills, the effects of vacuum (vacuum drying), high pressure and temperature (hydrothermal treatment), ultrasonic vibrations, radiation, and high-frequency current [19-25]. Chemical methods of sorbent activation consist in the action of chemical reagents on samples [26-31]. Depending on the chemical reagent, acid, alkaline, salt and combined activation are distinguished. The reagents used are: 1) Mineral acids - sulfuric, hydrochloric, phosphoric. 2) Organic acids - oxalic, acetic. 3) Alkalis - caustic soda, caustic potassium, sodium carbonate, calcium oxide hydrate, etc. 4) easily water-soluble salts. In this case, as a rule, an exchange reaction occurs between the cations of the water soluble salt and the cations that are part of the absorbing complex of the natural sorbent, resulting in the formation of mono cations substituted forms of the sorbent. 5) Salts those are difficult to dissolve in water, such as calcium carbonate. In this case, after processing, the sample is calcined at a given temperature. 6) Organic substances - amines, amides. In this case, specific forms of organophilic sorbents are obtained. 7) Water-soluble polymer substances. Known effective methods of chemical and physical modification of the surface of natural mineral sorbents are carried out in order to regulate their adsorption and cation exchange capacity [32-36].

However, many of these methods often lead to contradictory and disparate results. Therefore, in this work, we not only experimentally tested the existing methods for modifying and activating the most promising inorganic natural mineral sorbents ("zeolite, bentonite, zeolite-containing siliceous formation") [37-42], but also proposed new technological techniques that can significantly improve the sorption characteristics of natural sorbents.

EXPERIMENTAL PART

Acid activation in the boiling mode was carried out by the following method. In a heat-resistant round-bottom flask with a reverse refrigerator, the initial natural sorbent and an acid solution are loaded (Fig. 1). In this case, the ratio of natural sorbent: acid solution varies from 1:1 to 1:10. To select the most optimal reagent, hydrochloric and sulfuric acids were tested. The activation time is set from 1 to 5 hours. Boiling (at 95-100°C) ensures good mixing of all components of the mixture. Immediately after activation with an acid solution, the sorbent is washed with water until a neutral reaction of the washing waters occurs. Next, the sorbent is dried first in a water bath to an air-dry state, and then in a drying cabinet at a temperature of 100-110°C for 2 hours.

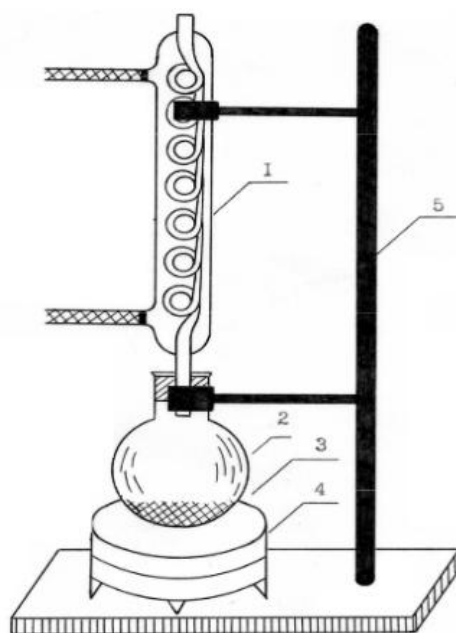


Fig.1. Installation of activation of natural mineral sorbents: 1-glass return cooler; 2 –round bottom heat-resistant bulb; 3-sorbent; 4-electric plate; 5-tripod

To determine the sorption capacity of the obtained high-silica zeolites, model solutions of phenol with a concentration of 200 mg/l were prepared. The VD-1-125 dividing funnel was used for testing the sorbent. Funnel diameter 40 mm, height 160 mm, loading weight 60 g.

The flow direction of the solution during filtration was set from the bottom up. The filtration process of the model solution is carried out by means of circulation in order to compensate for the small volume of loading. The time spent on filtering took 1 hour. As a reference sorbent, activated carbon of the BAU-A brand (state standard 6217-74) was taken as having the highest sorption capacity in relation to phenol. The phenol sorption was determined on a spectrofluorometer (RF-6000 Shimadzu) and on a gas chromatography "Kristallux-4000M".

The static adsorption capacity of the adsorbent (A , mg / g) is determined by the formula

$$A = (C_{\text{н}} - C_{\text{к}}) * V/m$$

Where S_i and S_c are the initial and final concentrations of petroleum products in the solution, mg/l; V is the volume of the solution, l; m is the weight of the sorbent, g.

Studies of the created sorbing materials on the sorption capacity for petroleum products, in particular, for diesel fuel, were also carried out. To prepare the model solution, 100 mg of diesel fuel was taken. This volume of diesel fuel was mixed with 20 ml of acetone.

All components were well mixed and filled with 1 liter of distilled water in a conical flask. Next, the resulting solution was distributed in conical flasks with a volume of 250 ml. The sorbent and BAU-A coal produced by us were filled into the conical flasks. Static tests were carried out on a magnetic stirrer for 30 minutes with the loaded sorbents. Then, the sample was filtered through a paper filter and sent for preparation for sorption capacity tests on a GCMS–QP2010 Ultra chromat-mass spectrometer. The preparation was as follows. To extract the samples, a solution was made from the following components: magnesium sulfate with a mass of 18 g was taken, hexane with a volume of 20 ml was taken by the dispenser, and 200 ml of water sample was taken by the dispenser after static tests. All components were thoroughly mixed for 30 minutes in a 250 ml conical flask on a magnetic stirrer. After this time, a sample was taken from the near-surface layer by a dispenser using a transition and a glass tube (since the density of the extractant hexane is less than the density of water), then it was placed in a vial and sent for studies on the sorption capacity on a GCMS-QP2010 Ultra chromat-mass spectrometer, in comparison with a sample after cleaning with a conventional sorbent activated carbon of the BAU-A brand.

RESULTS AND DISCUSSION

Analysis of texture properties of natural sorbents, the chemical and mineral composition of natural sorbents, as well as structural features and texture characteristics are fundamental in the study and creation of sorbents for adsorption processes.

The method of chemical modification is one of the most promising methods for controlling the porous structure of sorbents.

The method of low-temperature nitrogen adsorption is used to characterize the texture parameters of the sorbents (the surface of the dissolved substance, the

distribution of porous volumes and pore sizes). According to experimental data, in a wide range of pressures – from 0 to 1 P/Ps - adsorption isotherms are formed, which are measured. Macro genesis is determined by mercury porosimetry. When studying the porous structure of natural sorbents, the IUPAC classification is used.

As is known, the adsorption properties of natural sorbents are determined by both the porous structure and the chemical nature of the surface. The presence of active centers on the surface of the sorbents leads to the fact that the adsorption in them makes a significant contribution to the total amount of adsorption. The heterogeneity of the porous structure of natural sorbents, the nature and number of active centers, undoubtedly affect the shape of the adsorption isotherms.

The adsorption isotherms shown in Figure 2 confirm the heterogeneous porous structure of bentonite: bentonite is characterized by a micro-and mesogamous structure. As a result, bentonite differs in its probable mechanism and type of adsorption.

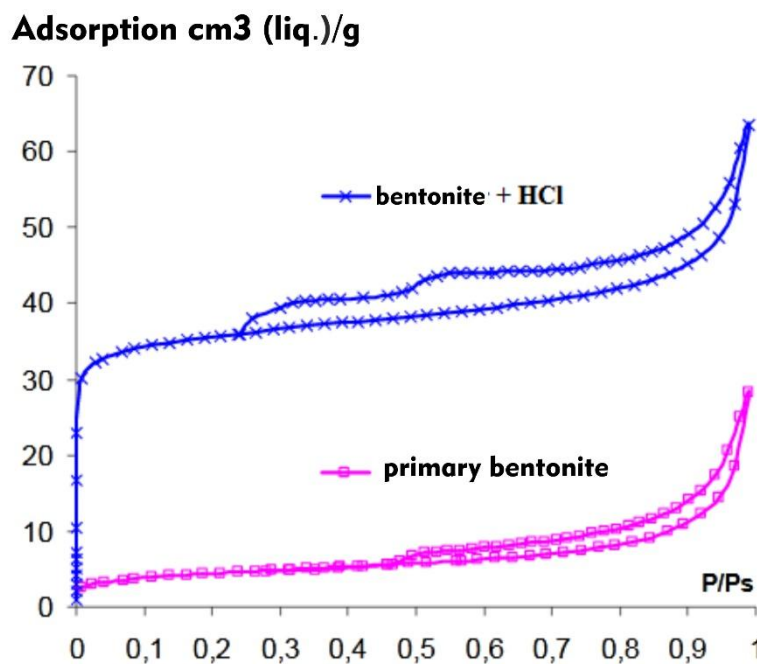


Fig.2. Isotherms of nitrogen adsorption-desorption at 77 K on bentonite

Bentonite adsorption isotherms treated with a natural and acidic solution have the simplest appearance. According to the IUPAC classification, these isotopes belong to the first type. They are reversible and fry towards partial pressures. The adsorption isotherms at $P/P_s \rightarrow 0$ reach their maximum values. The adsorption-desorption isotherms in bentonite correspond to type IV isotherms with complex hysteresis loops that reflect the presence of mesotherms of different sizes and shapes.

Acid activation of bentonite and silicon-retaining bentonite is observed at comparable surface surfaces, pore volumes and diameters, increased porosity, and

decreased actual density. The most serious changes in the structure of acid activation occur in bentonite, in which the specific surface area increases from 15 to 108 m² / g, the size of microcapsules increases from 0.006 to 0.054 cm³/g, and the total volume of porosity increases from 0.045 to 0.103 cm³ / g, which affects the redistribution of microcapsules.

The activation of bentonite in both acidic and alkaline solutions leads to a decrease in the specific surface area, but at the same time to an increase in the total volume and diameter of the pores. This is due to a redistribution of pores in size, a decrease in micropores and an increase in mesopores. It was found that during acid activation, the reaction of ion exchange between N⁺ and the exchangeable metal cations in the sorbents occurs, as well as the release of Al³⁺ and Fe³⁺ from the primary structure of minerals, which is confirmed by a decrease in alkali. Oxides of alkaline earth metals in natural sorbents after acid activation.

Thus, by changing the type of activating agent, it is possible to form a porous structure in which micro -, meso- and macropores change in a wide range for the subsequent selective sorption of various molecules. Bentonite is characterized by micro- and mesoporous properties. This makes it possible to predict the adsorption properties of substances by varying the diameter of the molecules in a wide range.

Changing the surface state and relative proportions of the mineral phases in the cinemas, which are analyzed as a result of their activity with acid and alkali solutions, undoubtedly, affects their sorbitional properties - electronic microscopic studies, X-ray structural analysis, IR spectroscopy and nuclear magnetic resonance results testify to this.

The effect of activators such as HCl and NaOH solutions on the surface state, the specificity of morphology (microgeometry of the resulting sorbent particles), and the microstructure of bentonite were determined using a SEM-100U scanning electron microscope. The results obtained are shown in Figures 3-5.

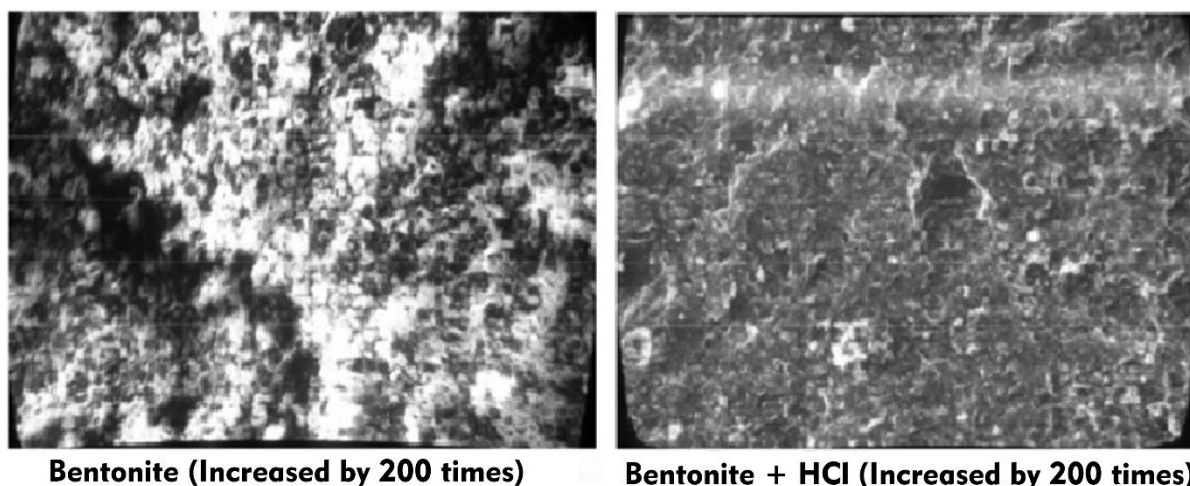


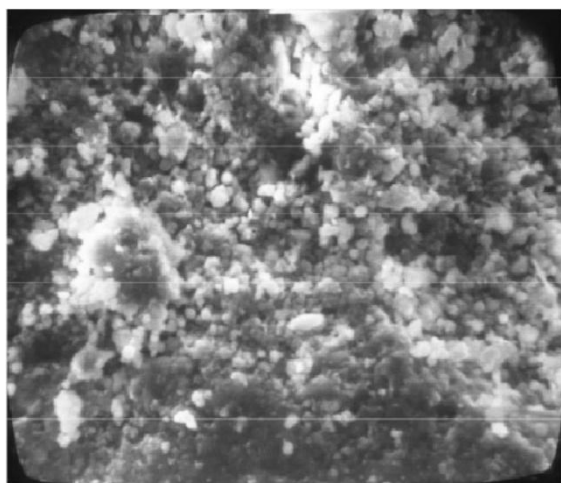
Figure 3. Effect of HCl solution on bentonite microstructure

Bentonite: spherical-globular microstructure with fragments of new derivatives - individual crystals of globular cristobalite (clinoptilolite). The size of globules is from 0.5 to 3 microns, elliptic-smectite ones - up to 20 microns.

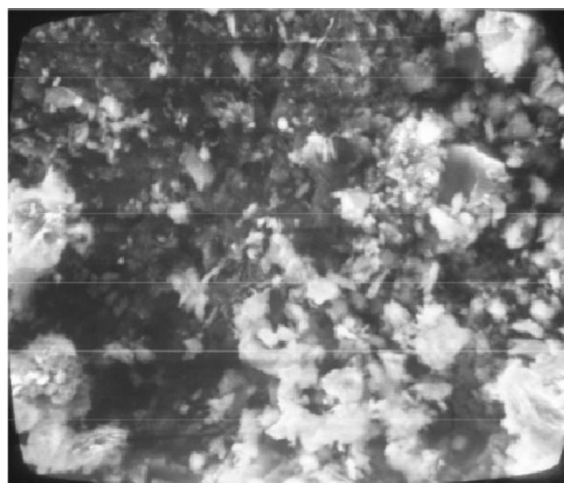
The first sample of bentonite has a crystalline granular microstructure, pagular, globular. Uniformly unevenly distributed sediment (sediment) is visible on the surface of the sample. The size of individual particles varies from 2 to 20 microns. The black area is formed by geometric depths in a wide range of measurements - from a point ($\sim 1 \mu\text{m}$) to an elongation ($> 100 \mu\text{m}$).

A sample of bentonite activated with a 7% HCl solution is more homogeneous, both large globular fragments of 100-150 microns in size and small rock particles (up to 5 microns) are clearly visible. Much less rare (light) separations are observed, the size of which does not exceed 50 microns. Less common are black pits of various visible shapes. Their size ranges from 20 to 100 microns.

The NaOH solution has a greater effect on the state of the bentonite surface. Figure 4 clearly shows a twisted-fibrous texture (structure) with many unevenly distributed spherical shapes and a particle size of 2-3 microns.



**Electron microscopic image
of bentonite (magnification)**



**Electron microscopic image of
bentonite + NaOH (magnification)**

When analyzing micrographs taken with a scanning electron microscope, it becomes clear that the surface of Navbakhor bentonite is formed by both small (< 5 microns, Fig. 5a) and large particles (up to 30 microns, Fig. 5b), and on such a surface there are many chips characteristic of montmorillonite.

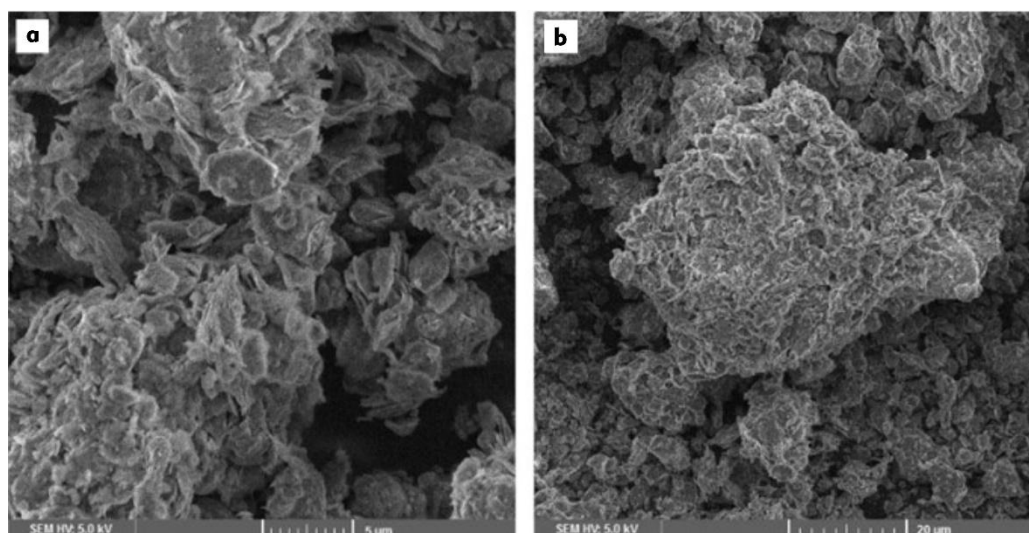


Fig.5. Micrographs of the surface of natural MM-containing Navbakhor bentonite

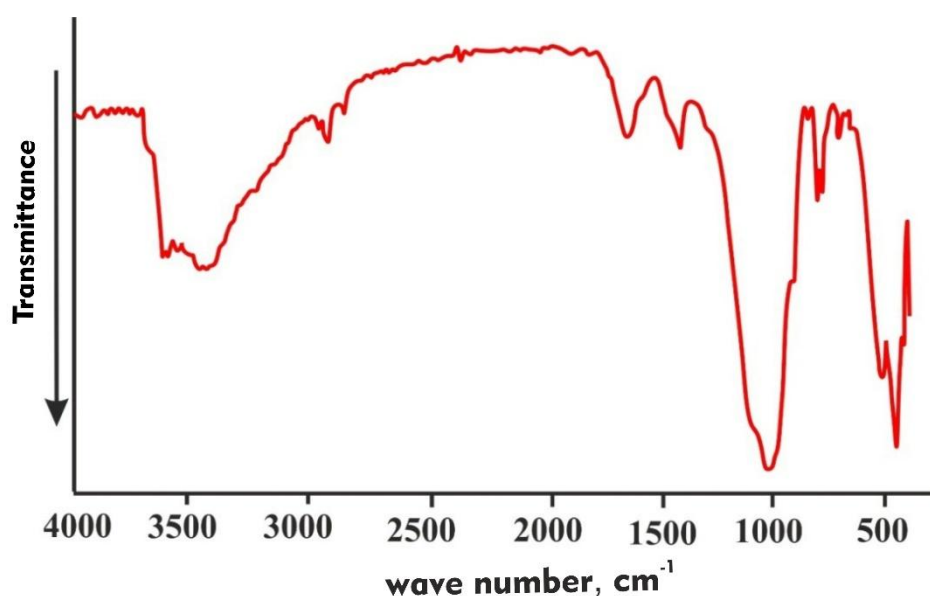


Fig. 6. IR spectrum for a sample of natural MM-containing Navbakhor bentonite

The analysis of the IR spectra of MM-containing clay on the Avatar 360 FT-IR ESP instrument (Fig. 6) allows us to associate the peak at 3437 cm^{-1} with the presence of Mg^{2+} and Fe^{2+} cations in the structure of the inorganic material, and to attribute the low-frequency band in the region of 2920 cm^{-1} to the valence vibrations of OH groups. The peak of 870 cm^{-1} , in turn, corresponds to the plane deformation vibrations of hydroxyl groups in montmorillonite. In the region of $860\text{--}798\text{ cm}^{-1}$, valence Al-O vibrations of condensed AlO_4 tetrahedra are observed. In the wavenumber range of $1680\text{--}1536\text{ cm}^{-1}$, the polymer form of water was found (in montmorillonite), and in the region of $1060\text{--}920$ (peak at 1020 cm^{-1}), deformation vibrations of hydroxyl groups bound to the aluminum atom (Al-OH) were found. The peak at 3620 cm^{-1} corresponds to the in-phase valence vibrations

of the internal OH groups or 2Al-OH ligaments of montmorillonite, whereas the peak at 3690 cm^{-1} is characteristic of paired surface hydroxyl groups. In the 540 cm^{-1} region, mixed Si-O and Al-O(H) strain oscillations are observed, and at $920\text{--}910\text{ cm}^{-1}$, a small flat peak is observed for groups of layered montmorillonite in contact with a silicon atom.

STUDY OF SORPTION CAPACITY

Below are the results of the phenol sorption capacity obtained on the gas chromatograph "Kristallux-4000M" (Figure 7-8).

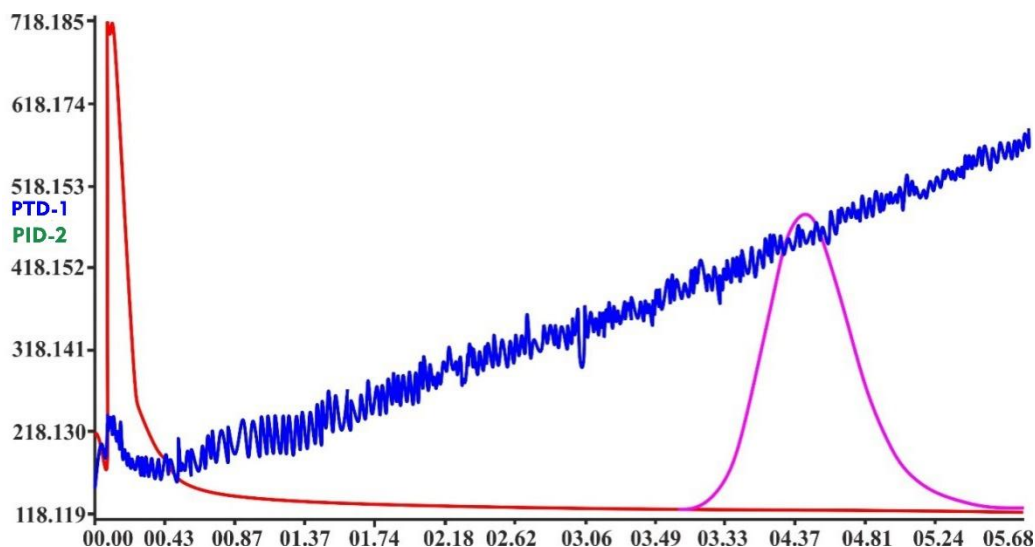


Figure 7. Chromatogram of the sorption capacity for phenol

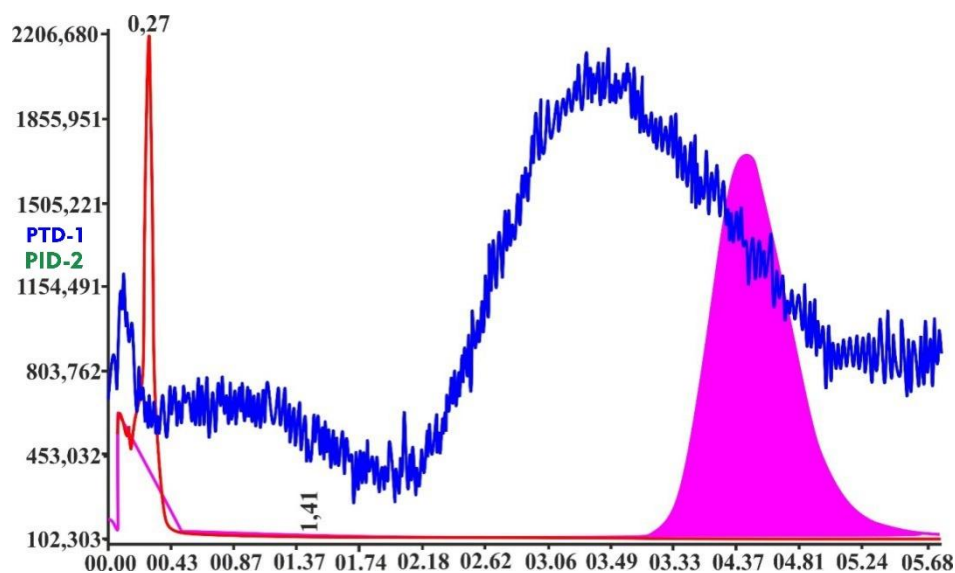


Figure 8. Chromatogram of the sorption capacity for phenol

High-silica zeolites obtained from Navbahor bentonite were also tested for phenol sorption (Table 1).

Table-1
Test results of high-silica zeolites

Type of sorbent	Loading weight, g	Initial concentration of phenol, mg/l	Residual concentration of phenol, mg/l
HSZN ₁	1	200	64
HSZN ₂	1	200	70,3
HSZN ₃	1	200	59
HSZN ₄	1	200	67

As can be seen from the presented data, the best result of the sorption of phenol from water has HSZN₃.

The efficiency of the resulting sorption in relation to petroleum products is slightly higher than the efficiency of activated carbon of the BAU-A brand (Table 2).

Table-2
Comparison of the actions of N₃ and N₁ of sorbents and BAU-A

	HSZN ₃	HSZ N ₁	BAU-A
Initial concentration of diesel fuel *	96,8mg / l	96,8mg / l	96,8 mg / l
Final concentration of diesel fuel *	53,6 mg / l	54,9 mg / l	76,7

From all of the above, it can be seen that the best result of sorption capacity in relation to phenols has HSZN₁.

And it showed itself best of all in the sorption capacity in relation to petroleum products, in particular, to diesel fuel, HSZN₃.

To determine the sorption capacity of these high-silica zeolites, experiments were carried out on a dye-methylene blue. To conduct these experiments, model solutions of methylene blue were prepared in distilled water. To do this, distilled water was poured into a conical flask with a volume of 1 liter and methylene blue powder was filled in. Everything was well mixed. Next, the UV spectrophotometer was heated for a time of 20 minutes. During this time, a transparent sample cell with a wall size of 10 mm was prepared.

First, the spectrum of the model solution was taken, and then the methylene blue solution was poured into conical flasks in a volume of 200 ml. Sorption complexes HSZN₁ and N₃, weighing 0.4 g each, were filled in. The flasks with the solution and the sorbent in it were mounted on magnetic stirrers for static testing for 30 minutes. After that, samples were taken into cuvettes and studies were conducted on the sorption capacity of these sorbents on a UV spectrophotometer.

The optical density and wavelength of the radiation passing through the cuvette were taken from the obtained spectra. Using these values, as well as the values of the extinction coefficients, it is possible to calculate the values of the concentrations of methylene blue in the initial solution and after sorption using the following formula: $A = \varepsilon \cdot l \cdot C$

$$A = \varepsilon \cdot l \cdot C$$

Where, **A** is the optical density, ε is the extinction coefficient, **l** is the length of the cell wall, and **C** is the concentration of the substance.

The three most characteristic wavelengths were chosen: 246, 291, and 665.

Table 3
Optical density values

	Initial solution	№1	№3
λ, nm	A	A	A
246	0,351	0,164	0,190
291	0,560	0,255	0,358
665	0,987	0,392	0,578

Table 4
Values of extinction coefficients for each wavelength

λ, nm	ε
246	10070
291	23900
665	32000

Formal kinetics considers the process without taking into account the mechanism and describes the result of all reactions as a whole. The task of formal kinetics is to construct equations for calculating the rate constant of the same reaction, that is, equations that allow us to calculate the reaction rate as a function of concentration at any given time. To do this, you need to determine the order of the reaction for each of the reagents, build a differential equation for the reaction rate and integrate it.

Methods for determining the reaction order can be divided into two groups - integral and differential. All methods based on the use of integral forms of kinetic equations of procedures belong to the group of integral methods for determining the reaction rate. There are several ways to do this. One of them is the selection method, which involves applying equations corresponding to the first, second, and third reaction modes. This method has algebraic and graphical solutions. The graphical method consists in constructing an experimental dependence $C = f(t)$ in a certain coordinate system, in which the presence of linearity confirms the correctness of the choice of order. According to this method, the kinetic curve at each of the temperatures corresponds to the first order of the reaction, which can be seen in Figures 9-11.

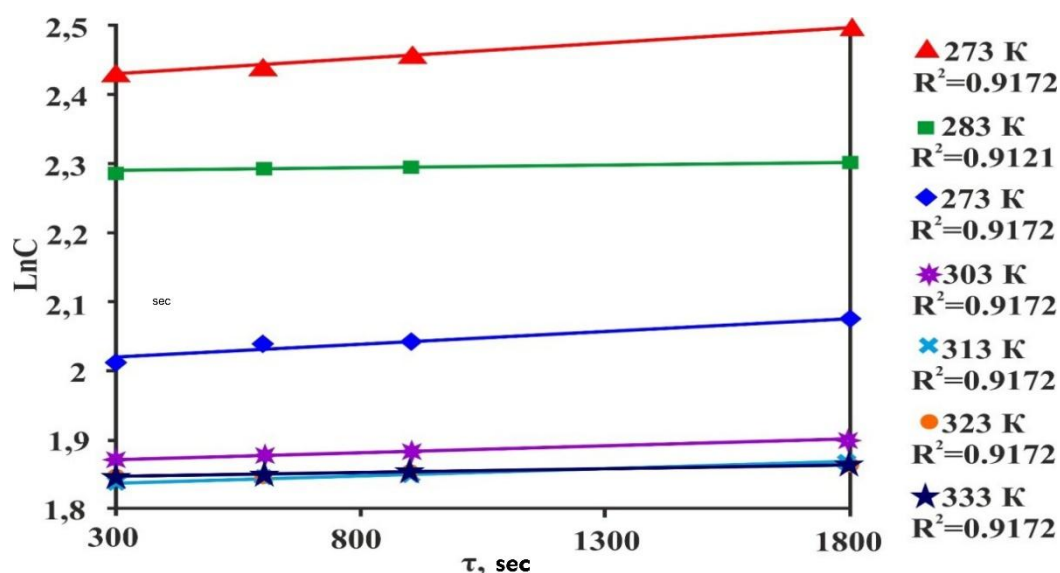


Fig.9. Dependence of the logarithm of concentration on time for first-order reactions during adsorption

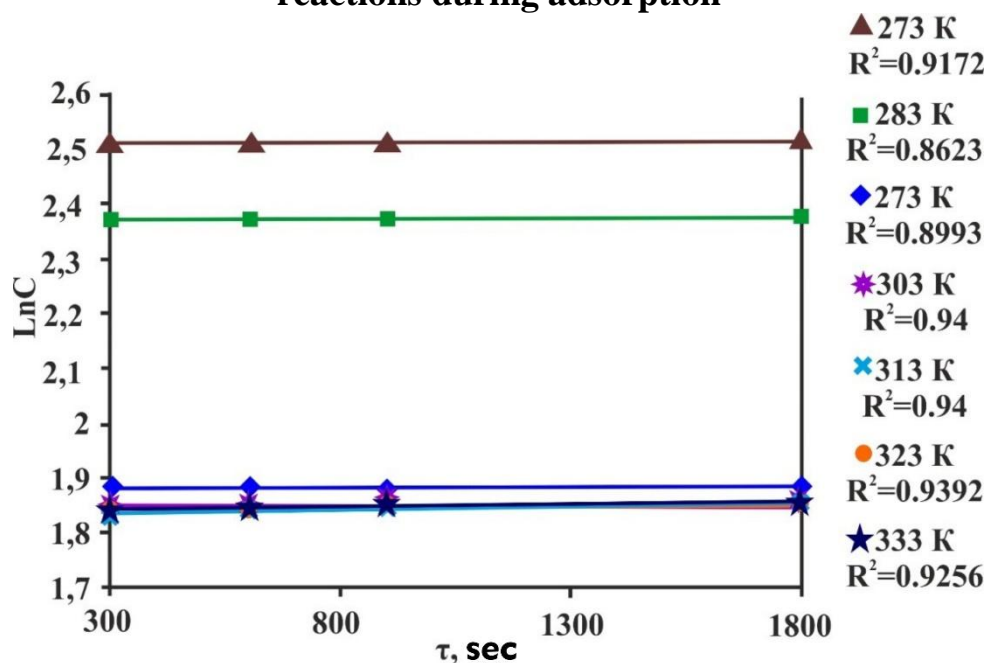


Figure 10. Time dependence of the logarithm of concentration for first-order reactions during adsorption

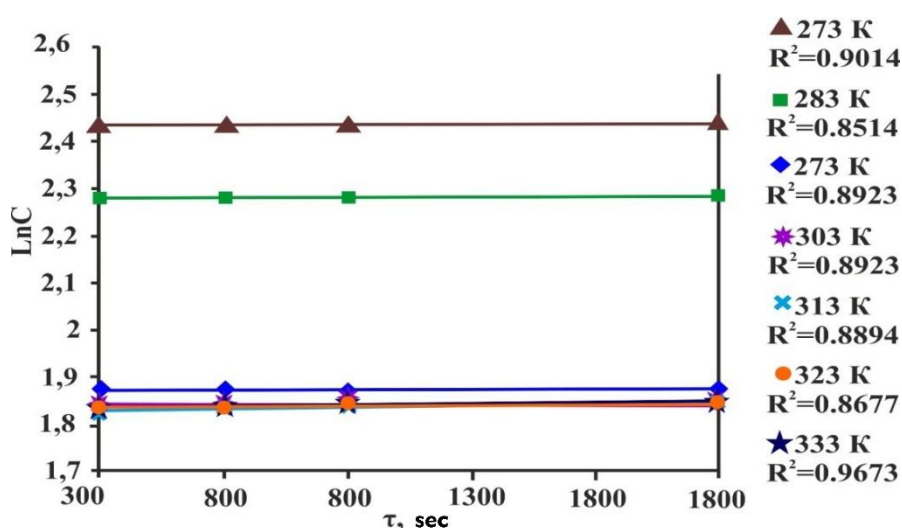


Figure 11. Dependence of the logarithm of concentration on time for first-order reactions during adsorption

These kinetic lines are constructed with a high degree of correlation (within the error of the experimental data), and they all relate to heterogeneous reactions of the first order. The area of formation is limited to a time interval from 300 to 1800 seconds, since then the process remains unnoticed or the desorption of oil from the pores of the sorption materials begins or does not occur. Thus, the logarithm of the concentration for a first-order reaction is linear in time, and the rate constant is numerically equal to the tangent of the angle of inclination of the straight line to the time axis:

$$k_1 = - \operatorname{tg} \alpha$$

The temperature dependence of the reaction rate is expressed by the Arrhenius equation and the Van't-Hoff rule. The Arrhenius equation has the following form:

$$k = A e^{\frac{E_a}{RT}}$$

where k is the rate constant of the chemical reaction;

A is the old exponent;

e is the base of the natural logarithm;

R is molar gas constant, J / mol • K;

T is temperature, K; activation

E_a – en is ergy, J / mol

The integral form of the equation looks like this:

$$\ln k = \ln A - \frac{E_a}{RT},$$

There is A does not depend on the temperature in all cases in the temperature range under study. The inverse temperature dependence of the initial value of the logarithm of the chemical reaction rate constant is found by the tangent of the slope angle:

$$E_a = -R \cdot t g \alpha$$

Knowing the activation energy and the velocity constant at a certain temperature T_1 , we can calculate the value of the velocity constant at any T_2 in accordance with the Arrhenius equation. Table 3.5 shows the calculated values of the activation energy by integrating the Arrhenius equation in the range from T_1 to T_2 , which allows us to calculate E_a for the velocity constants at two temperatures:

$$\lg \frac{k_{(T_2)}}{k_{(T_1)}} = \frac{E_a}{2,3R} \left(\frac{1}{T_1} - \frac{1}{T_2} \right)$$

Table 5
The value of the activation energy of oil sorption of high-silicon zeolites (HSZ № 3) obtained from Navbakhor bentonite at various temperatures.

Activation energy (E_a , Kj/mol)	speed constants (k , c^{-1})	Temperature range (ΔT , K)
HZS №3		
11,16	$1,78 \cdot 10^{-6}$	273-283
12,09	$2,12 \cdot 10^{-6}$	283-293
15,94	$2,53 \cdot 10^{-6}$	293-303
13,53	$3,13 \cdot 10^{-6}$	303-313

To explain the physical meaning of the activation energy, Arrhenius suggested that not all particles, but only those in the activated state, are able to react. In the activated state, the molecules are energetically excited and unstable, and in the normal state, they are stable. The fraction of particles in the excited state is relatively small, while the number of particles in the normal state is almost equal to the total number of particles. Since the activation process is endothermic, the equilibrium constant of this process increases with increasing temperature. As the concentration of particles in the active state increases, the reaction rate increases.

The activation energy can be defined as the thermal effect of the activation process, in other words, it is the energy that individual molecules must accumulate in order to be able to react. It is necessary that the affected molecules overcome the energy barrier caused by the repulsion of the electron shells. It is known that if the activation energy is 15-20 kJ / mol, then the slowest step is to move the reagent to the surface of the solid or remove the resulting product from the surface of the solid, if the activation energy is in the range of 50 to 200 kJ / mol - a chemical reaction on the surface of the body itself. As can be seen from Table 3.5, the sorption process depends on the temperature in the target region and can occur in both kinetic and diffusion fields.

In addition, the activation energy of diffusion in the sorption process usually ranges from 8.5-12.6 kJ / mol (diffusion in a thin film) to 12.6–42.0 kJ / mol (diffusion in a grain). The given values of the activation energy indicate a mixed diffusion-kinetic mode of sorption.

Alkaline activation was performed on bentonites with a zeolite-containing siliceous formation.

The most valuable properties in practical terms, these sorbents acquire only after chemical modification. The cation exchange capacity of bentonite in acidic neutral and alkaline media is different; it is associated with the reactivity of silanol groups $\text{Si} - \text{OH}$, their amount of surface, which depends on the degree of hydration of silica.

In addition, it should be taken into account that natural bentonites contain impurities, which also have a certain effect on the value of the exchange capacity. The choice of optimal activation conditions is shown on the bentonite. It is experimentally proved that for a zeolite-containing siliceous formation (bentonite), the activation conditions will be similar

To determine the activation conditions, mathematical methods of experimental planning and statistical processing of experimental data (specific surface area, total pore volume, true density, and mechanical strength) were used. The main purpose of the mathematical experiment is to develop a mathematical model that adequately describes the activation process and allows for its control in the final result.

It is with the help of such a model that you can effectively manage the activation and modification processes, quickly changing their parameters in accordance with the requests of the received characteristics. The planning of the experiment begins with the selection of the center of the plan, i.e. the point corresponding to the initial value of all the factors used in the experiment, the values of which are then set for a series of planned experiments. The center of the plan is usually chosen based on a priori knowledge of the process. If they are not present, the center of the study area is usually taken as the center of the plan.

The following factors were chosen for the activation of bentonite: the concentration of NaOH from 1 to 10% (X_1) and the activation time from 0.5 to 5 h (X_2) at a constant Phases ratio:G, equal to 1:2; the range of variation in the concentration of NaOH, equal to 3.2; the activation time-1.6 (Table 6).

Table 6
Levels of factor variation

Factors	Codedesignations	Lowerlevel $X_1=-1$	Upperlevel $X_1=+1$	Mainlevel $X_1=0$	Variationinterval
SolutionconcentrationNaOH H%	X_1	2,3	8,7	5,5	3,2
Time activations, h	X_2	1,15	4,35	2,75	1,6

Usually, the experiment plan is set in the form of a planning matrix (Table 7).

Table7
Primary Experiment planning matrix

ExperimentPlan	experiment№	X_1	X_2	$X_1 \cdot X_2$	X_1^2	X_2^2
Fullfactorialexperiment	1	2,3	1,15	2,645	5,25	1,322
	2	8,7	1,15	10,00	75,69	1,3225
	3	2,3	4,35	10,00	5,29	18,92
	4	8,7	4,35	37,84	75,69	18,92
Experimentsinstarpoints	5	10	2,75	27,5	100	7,56
	6	1	2,75	2,75	1	7,56
	7	5,5	5,0	27,5	30,25	25
	8	5,5	0,5	2,75	30,25	2,5
Experiments in the center of the plan	9	5,5	2,75	15,12	30,25	7,56
	10	5,5	2,75	15,12	30,25	7,56
	11	5,5	2,75	15,12	30,25	7,56
	12	5,5	2,75	15,12	30,25	7,56

The first column of the matrix represents the numbering of the experiments. The following columns of the matrix contain dimensionless symbols corresponding to the upper and lower levels of variation of factors and their interactions. When constructing the planning matrix, the first row in the columns corresponding to the factors considered in the experiment is filled with a dimensionless symbol corresponding to the lower level of the values of the experimental factors, i.e., the symbol (-). The continuation of filling in the column corresponding to the first factor in order is carried out by successive alternation of opposite signs (without dimensional values of the levels of variation of the factor).

All subsequent columns that correspond to other factors numbered in order are filled in with a sign change frequency that is half that of the previous column. The experiment planning matrix constructed in accordance with this rule is shown in Table 8.

Table 8
Adsorption-structural properties of bentonite activated by NaOH, according to the experimental planning matrix (se-experimental data sd-calculated data)

ExperimentPlan	Conditionsofthe experiment		Specific surface area according to BET, m ² /g		Total pore volume, cm ³ / g		Truedensity g / cm ³		Mechanicalst rength, Kg / cm ²	
	NaOH %	Time h	y ^o	y ^p	y ^o	y ^p	y ^o	y ^p	y ^o	y ^p
Fullfactori alexperime nt	2,3	1,15	133	135	0,400	0,413	2,29	2,29	140	131
	8,7	1,15	126	126	0,433	0,439	2,27	2,27	110	109
	2,3	4,35	132	135	0,429	0,431	2,30	2,30	145	123
	8,7	4,35	130	131	0,432	0,426	2,29	2,28	150	136
Experi-ments in star points	10	2,75	128	128	0,430	0,431	2,28	2,29	100	106
	1	2,75	140	137	0,425	0,416	2,32	2,31	95	113
	5,5	5,0	136	133	0,428	0,432	2,28	2,28	125	146

	5,5	0,5	130	129	0,441	0,429	2,27	2,27	130	133
Experiments in the center of the plan	5,5	2,75	125	125	0,444	0,444	2,27	2,27	169	169
	5,5	2,75	124	125	0,446	0,444	2,27	2,27	165	169
	5,5	2,75	126	125	0,443	0,444	2,26	2,27	171	169
	5,5	2,75	125	125	0,442	0,444	2,265	2,27	170	169
Bentonite source	-	-	154	-	0,422	-	2,359	-	79	-

Each row of the planning matrix defines the experience conditions, and each column defines the values of the controlled and controlled parameters of the activation process, i.e., the values of the factors corresponding to the experience condition. In the last columns, enter the values of the response function obtained experimentally in each number of the experiment conducted in accordance with the conditions specified in the rows of the experiment planning matrix.

The values of the factors in each experiment, if the experiment planning matrix is used, differ from their initial values by the amount of the variation interval.

According to the selected levels of variation of factors, the conditions for activation are entered into the experimental planning matrix, which is used to conduct experiments to determine the specific surface area of the total pore volume, density, and mechanical strength of activated bentonite (Table 6). This table also shows the data obtained during the processing of the planning matrix.

Based on the results of processing the experimental planning matrix, a system of regression equations is obtained that adequately describes the process of activation of bentonite with alkali.

The regression equations have the following form:

Y_1 – specific surface by BET;;

$$Y_1 = 125 - 3,2X_1 + 1,4X_2 + 1,25X_1X_2 + 3,6X_1^2 + 3,1X_2^2$$

Y_2 – total pore volume;

$$Y_2 = 0,443 + 0,005X_1 + 0,001X_2 - 0,007X_1X_2 - 0,010X_1^2 - 0,006X_2^2$$

Y_3 – true density;

$$Y_3 = 2,266 + 0,010X_1 + 0,004X_2 - 0,001X_1X_2 - 0,016X_1^2 - 0,003X_2^2$$

Y_3 – $P/P_s = 0,11$ static moisture capacity;

$$Y_4 = 1,724 + 0,033X_1 - 0,007X_2 - 0,025X_1X_2 - 0,085X_1^2 - 0,020X_2^2$$

Y_4 – $P/P_s = 0,47$ static moisture capacity;

$$Y_5 = 5,48 + 0,023X_1 + 0,01X_2 - 0,001X_1X_2 - 0,010X_1^2 - 0,020X_2^2$$

Y_5 – $P/P_s = 0,98$ static moisture capacity;

$$Y_6 = 23,64 + 0,15X_1 - 0,81X_2 - 0,15X_1X_2 - 3,03X_1^2 - 1,48X_2^2$$

Y_7 – mechanical strength;

$$Y_7 = 168,8 + 2,2X_1 + 4,7X_2 - 8,75X_1X_2 - 29,7X_1^2 - 14,7X_2^2$$

Using the system of regression equations, it is possible to optimally set the modes of conducting experiments, which significantly reduces time and material costs. Based on the tests performed using the experimental planning matrix, the

optimal conditions for the activation of bentonite were selected: the NaOH concentration of 5.5%; the activation time of 2.75 h; the phase ratio: $W=1:2$.

CONCLUSION

The physical and chemical properties of natural bentonite and sorbents based on it are studied. It is established that the modification of natural bentonite leads to a change in its chemical composition, structural and sorption properties. It was found that the modified sorbents based on natural bentonite are fine-pored (nanostructure) objects and with a predominance of pores with a size of 1.4-8.2 nm. The value of the specific surface area of the sorbents depends on the amount of the modifying component introduced.

It is established that the most effective method of activation of bentonite is the activation of 7% hydrochloric acid solution for 2 hours in the boiling mode. The T: W phase ratio is 1:2. The optimal ways to activate bentonite are activation of 5.5% NaOH for 2.75 h at a phase ratio of 1:2 to obtain a sorption-filter material for water purification and acid activation (HCl 20%, activation time 2 h) for air drying. The optimal method of activation of bentonite is acidic: the concentration of hydrochloric acid is 7%, the activation time is 20 minutes.

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