

## Physicochemical Analysis of Bentonite Used in The Preparation of Catalysts for Methanol and Dimethyl Ether Synthesis

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### Abstract

*The article presents a method of purification of obtaining bentonite clay for use in catalysis. It was found that the cleaning process includes the stages of centrifugation, drying and mechanical activation. Clay cleaning frees it from impurities and increases the proportion of the useful component - the sorption-active mineral montmorillonite. The porosity of the BET method was determined (measurement of specific surface area and porosity). The accuracy and standardization of the method for determining the adsorption activity have been carried out. An exponential dependence of the adsorption activity*

on the weight of the sample, (1.0 g), sorption time (35 min) and the equilibrium concentration of the dye solution (39 ml 0.15%) were established. The physicochemical and textural properties of betonies clay for the use of catalysis have been studied. It was found that bentonite clay for use in catalysis is a light gray powder, odorless, practically insoluble in water and organic solvents, with a particle size of less than 0.3 mm.

Keywords: Betonies clay, specific surface area, porosity, adsorption activity.

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## 1. Introduction

Increasing the efficiency of use and development of the mineral resource base of betonies clays in Uzbekistan is an urgent problem. Betonies clays are good inexpensive sorbents for various substances, such as heavy metal ions [1], organic dyes [2]. The study of the physicochemical parameters of clays of various deposits, activated by various acids, is devoted to a sufficient number of works [3-9]. Much attention in the literature is paid to natural framework aluminosilicates, especially zeolites. These materials have a negatively charged three-dimensional aluminosilicate framework. In the gaps of the framework, there are hydrated positive ions of alkali metals, which compensate for the charge of the framework, and water molecules. When zeolites are heated, water is released from them, and adsorption cavities are formed. The areas of application of betonies clays will expand by imparting new properties to them as a result of various types of activation [8–11]. One of the most effective types of exposure is acid treatment [5, 10–12]. According to the nature and strength of the effect on the crystal structure of montmorillonites, acids can be divided into three groups [13-18].

All over the world, active research in the field of directed synthesis of new highly effective functional nanostructured materials for various purposes, such as for use as catalysts, sorbents, dosage forms carriers, membranes, fillers of composites, ceramics, etc., is continuing. Catalytic and adsorption processes are virtually impossible without the use of materials with a developed nanoporous structure. Oxide materials containing silicon and aluminum, both natural and synthetic, are widely used and are of considerable

scientific interest, since it is possible to regulate their porous structure and composition during synthesis and subsequent modification to give the necessary functional properties [6-8]. The modification of aluminosilicates by the introduction of metals of variable valence allows their use in redox catalysis, including for solving environmental problems. Catalytic oxidation of organic substances is an effective method for neutralization of gas emissions from source water.

Adsorption and other properties of natural sorbents and their optimal activation conditions are determined on the basis of a complex of physicochemical and adsorption-structural properties. The final stage of laboratory tests is to establish the suitability of sorbents for a specific technological process. The activation and modification of inorganic natural sorbents is carried out with the aim of a directed change in their properties. There are a number of effective methods for chemical and physical modification of the surface and regulation of the porosity of sorbents [14-18].

Pillarization is a particular case of intercalation, in which inorganic compounds are introduced into the interlayer space. Regardless of the specific features, the modification process can be reduced to three main stages: 1) hydrolysis of metal ions with formation of polynuclear hydroxocomplexes (PNHC)-pilling solution, 2) ion exchange, and 3) heat treatment of the modified material. Pillarization processes of layered aluminosilicates. Montmorillonite swells in aqueous solutions, and the distance between the aluminosilicate layers increases. The aluminosilicate is placed in a pilling solution containing polynuclear metal hydroxocations, the ionic exchange of interlayer clay cations into larger polynuclear hydroxocomplexes takes place.

Polyhydroxocomplexes are usually obtained by hydrolysis of cations with NaOH solution. Almost all transition metal ions with a charge greater than or equal to two can polymerize as a result of hydrolysis and form polynuclear hydroxocompounds, which can be achieved by selecting certain conditions for this process, first of all the rate of pH change during hydrolysis, the degree of hydrolysis and temperature.

Physical methods of sorbent activation are reduced to sample processing in mills, vacuum drying, high pressure and temperature, ultrasonic vibrations, radiation and high frequency current [19-20].

## 2. Experimental

On the basis of existing methods of clay purification, a technological method of purification of mineral raw materials of bentonite clay, applicable in laboratory conditions, has been developed. The technique includes four stages: centrifugation, drying and mechanical processing. The efficiency of mechanical activation was monitored by observing the main parameters confirming the change in the structure of the clay: the size and shape of particles in electron micrographs, and the indicators of adsorption activity.

Centrifugation was carried out for 5 minutes at a centrifuge operating mode of 3000 rpm. Drying of clay was carried out in a dry heat oven at a temperature of 130 °C for 190 minutes. The result is a clay with a particle size of 1-30 μm. The specified drying standards are established in a practical way as the most effective. Mechanical processing was carried out in a ball mill, followed by control of the shape, particle size and adsorption activity. It has been experimentally established that the optimal mechanical activation time is

90 minutes. The working hypothesis of mechanical activation for 90 minutes is based on the fact that with an increase in the processing time, an increase in the specific surface occurs, as well as a change in the shape of the solid and the accumulation of defects on its surface.

Carnabotin kaolin, a natural soil obtained from the bentonite deposits of Cattacorgon and Navbahor, was used as an object of study. The solution containing  $Al_{13}$  polycation was prepared by hydrolysis of aluminum chloride.  $AlCl_3 \cdot 6H_2O$  was added dropwise to the solution at room temperature until the  $NaOH[OH^-]/[Al^{3+}] = 2.4$  ratio was 4.3-4.7. The solution was then heated at 600 °C for 24 hours, resulting in the formation of an  $Al_{13}$  polyhydroxocomplex. According to the formulation of the  $Al_{30}$  polycation solution,  $Al_{13}$  was obtained from a solution quenched at 115 °C for 5 hours by hydrothermal treatment. The melts of large Al/Fe polyhydroxy complexes were hydrothermally treated in a high pressure reactor (at 135 °C for 20 hours) in a mixture of  $Al_2(OH)_5Cl$  and  $FeCl_3 \cdot 6H_2O$  solutions at Al:Fe molar ratio =15. Relative to  $Al^{3+}$  (x) solution concentration  $x=2,5,3,7,4,3,5,1M$  (precipitation is formed at  $x>6M$ ).

20 g of bentonite was added to 1 L of distilled water and the upper layer of the suspension was centrifuged after 24 hours. The separated particles of the MM (montmorillonite) fraction were dried at 600 °C to an average size of 2 μm. Intercalation of MM  $Al_{13}$ ,  $Al_{30}$  and the Al/Fe polyhydroxy complex was performed by adding the intercalating solution (3 mol  $Al^{3+}/g$  MM) to the 1% aqueous suspension by ion exchange and using a strong magnesium stirrer at 800 °C for 2 hours. After 12 hours at room temperature, the suspension was washed of  $Cl^-$  ions (Fig. 1).

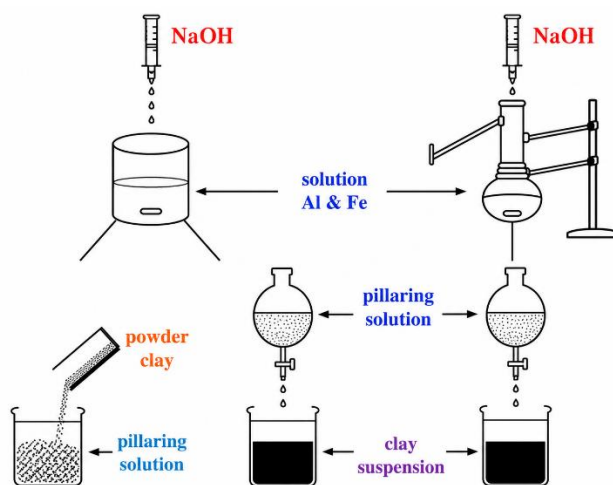


Figure 1. Bentonite implementation scheme

**Methods for testing the adsorption properties.**

Adsorption experiments were carried out as follows: 0.1 g of adsorbent was placed in a cylindrical beaker 50 mm in diameter, 20 ml of paint mixture was added and stirred from 15 to 1440 minutes at a constant speed of 400 rpm at 20 °C. After the experiment was completed, the adsorbent was separated from the mixture by centrifugation at 8000 rpm for 4 min. Dye concentration in the mixture was studied using a spectrophotometer UV-IS U-2001 (Hitachi) at the wavelength of maximum absorption ( $\lambda_{\max}=514\text{nm}$  for red acid 17,  $\lambda_{\max}=663\text{nm}$  for metallic blue dye). To study the effect of pH on the adsorption process (in the range of 2.0-13.0), HCl (0.1 M) or NaOH (0.1 M) was added to the mixture and measured using a pH meter. To obtain an adsorption kinetic curve, samples were taken from the experimental cups after 15, 60, 120, 180, 300, and 1440 min. Analysis of the residual concentration of the dye on the adsorption isotherm obtained an increase in the dye content in the aqueous solution from 10 to 100 mg/L. The amount of dye in the sample adsorbed during (qt) is determined by the following equation.

$$q_t = \frac{V(C_0 - C_t)}{m} \quad (1)$$

Here  $C_0$  and  $C_t$  (mg/ml) are the initial and  $t$  (min) values of dye concentration,  $V$  is the volume of mixture (ml),  $m$  is the mass of dry adsorbent.

Pre-metric composition of clay ore. Preparation of the soil for pre-metric analysis We place it in a porcelain cup and divide it into grains up to 1 mm in size. The granular (granular) content is prepared by pipetting according to GOST 21216.2-81. This method is based on the fact that particles of different sizes fall into water at different rates. The suspension is washed only once, and after a certain time a sample from a certain depth is taken in a pipette for examination.

To study the grain composition of the soil it is placed in a wooden sieve and crushed with a porcelain probe until it reaches a size of 1 mm. Properties of the crushed particles by pipette method GOST 21216.2-81. This was defined in the method. Sodium pyrophosphorus with a concentration of 40 g/l was used to separate the grains.

The obtained results show that the composition of fission fraction (size less than 1 micron) of natural samples taken from Kattakurgan and Navbakhor deposits (samples 2 and 3) refers to coarse-grained soil as per GOST 9169-75. It should be noted that the sample taken from.

Navbakhor mine (sample 3) contains a large proportion of sand, so it belongs to the class of sandy soils. Among investigated objects only the soil taken from Karnabat pit (sample 1) is the raw material belonging to the group of fine separating plastic soils.

Comparison of obtained results shows that difference between soils of natural fraction and artificially enriched soils is very small, which indicates absence of influence of enrichment process. For example: in Karnabat and Kattakurgan soils change of natural and enriched soils is 4-6%, while change of Navbakhor soil is only 5%.

In order to clearly observe adsorption property in soil samples before enrichment these samples should be mechanically crushed to small pieces with thieves, then washed day and then soil fraction should be selected and samples should be taken not deeper than 10 cm.

Very small influence of enriched soils on studied components in chemical composition of studied soils is also unnoticeable. Navbakhor and Kattakurgan soils contain high amount of dye oxide. It should be said that the increase of alkali metal oxides in the samples shown above is the result of the presence of feldspar, hydromica and other compounds in the soil.

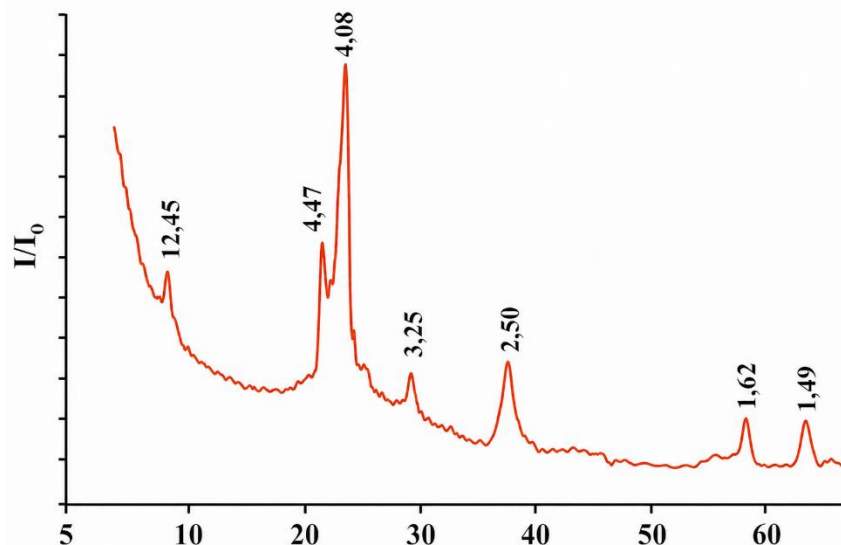
The results of the analysis and comparison show that all the studied soil samples are raw materials of semi-mineral composition, the soil composition of which is very complex and consists of a mixture of hydrosilicate in the form of illite and kaolinite.

These reflexes contain montmorillonite (d-14.73-14.56; 6.43-6.48; 2.54-2.60 Å), illite (d- 4.47; 3.38; 3.31-3.24; 2.98 Å), and kaolinite (d-7.15-7.20; 3.58-3.57; 2.56Å). Due to the addition of quartz material in the coarse-dispersed (coarse-dispersed) part (sand-foam fraction) (d-2.46; 3.36-3.34; 2.29; 2.24-2.25 Å), in orthoclase-like feldspar (d-3.80; 3.20-3.18; 2.92; 2.53 Å), in Navbakhor soil (d-5.03; 3.50; 3.20; 2.34-2.35 Å).

The amount of minerals in the studied samples was determined by the intensity of the spectra in X-ray reflexes. The results obtained are presented. All studied samples consist of montmorillonite-caolinite-hydrosilicate soils. The samples studied show that the enriched soil of Karnabat Ota (4 samples) contained more earthy minerals. The amount of montmorillonite in Kattakurgan and Navbahor soil samples differed from natural and enriched conditions by a factor of 2. Koallinite and hydromica differ by a factor of 1.5. The Cavert reflex is clearly visible on the X-ray diffraction.

Figure 2 shows a diffractogram of a Fe-soil-k sample obtained by adding PGK iron to natural soil. According to RFA, the structure of the modified soil

layer changes when it is heated to 500 °C. Fe-soil-k changes into a layered structure, which is called "house of cards" in the literature. Reflections corresponding to d001 disappear on the diffractogram and fuzzy bands of



**Figure 2. Diffractogram of Fe-soil-to-500, the distance between the planes is given in Å.**

The results show that the modified specific surface area, average diameter, and ore volume depend on sampling, polyhydroxylation composition, and method of application. The increased surface area of modified samples compared to natural soil. The average diameter of Al-soil-1, Fe-soil-0 samples increases less and decreases in Al-soil-2, Fe-soil-k, Fe-Al-soil-o, Fe-Al-soil-k samples, the natural total volume changes less in relation to the soil.

Recently, much attention has been paid to the conservation and treatment of industrial wastewater from various organic contaminants. Sorption and catalytic methods are widely used for these purposes. Water treatment with sorbents is very relevant. For this purpose it is important to buy cheap sorbents. Obtaining inexpensive sorbents from naturally dispersed materials, including montmorillonite soil, increases economic efficiency. Montmorillonite soil dramatically reduces the dye content of contaminated water, reduces water treatment costs, and does not re-pollute water when used. Natural soil is cation-exchange, so sorbent technology is important. Anion-type dissociators are good at cleaning harmful organic compounds in dirty water. In addition to the sorption method of treating polluted water, a catalytic method is also used to purify it from dyes, which uses environmentally safe oxidants such as ozone, oxygen and hydrogen peroxide. The Fenton and Raff system of Fe<sup>2+</sup>

low intensity appear, indicating the formation of irregular silicate layers.

and Fe<sup>3+</sup> ions is used as a catalyst. However, homogeneous catalysts and heterogeneous oxidation catalysts are used to remove other heavy metal ions from water in a certain pH range. The resulting modified soil was used to remove dyes and surfactants. Also, an aqueous solution of azocrystalline and hydrogen peroxide thiocyanate was used in the reaction of our samples for purification .

Study of adsorption properties. The magnitude of the adsorption capacity is related to the fact that natural soil has more cation-exchange centers than anion-exchange ones. The structure of natural soil expands the dyes, affecting not only the outer surface of the sorbents, but also the space between the layers, providing a half-layer adsorption of cationic dyes.

Compared to anionic dyes, cationic dyes have a much lower adsorption capacity. Adsorption of anions with positive charges occurs at the lateral damage boundary of the MM, which does not cover a very large area of the soil surface. Tests show that when soil is heated to 400 °C and 500 °C, the natural adsorption capacity of soil is lower than that of soil dried at 250 °C. Adsorption capacity in soils heated to 500 °C and 400 °C does not differ much.

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When there is a strong attraction between the hydroxyl group and the surface, such as during adsorption on polar adsorbents, the alkyl chain is compressed, similar to silicate oxides. When the concentration approaches the concentration per unit volume, the tendency for aggregation in the chain of adsorption molecules begins. This leads to a vertical orientation of the molecules, which leads to a sharp increase in adsorption. The second sharp rise of the isotherm corresponds to the coverage of the surfactant molecules in the vertical direction prior to surface saturation at low concentrations. The second rise at high concentrations indicates formation of a layer or formation of a micellar aggregate on the sorbent surface. At low values of Na-dodecyl sulfate the adsorption capacity changes very little. The concentration of Fe and Al is higher than normal values compared to natural soil. Adsorbent capacity is mainly characterized by the total surface area of the sorbents and the number of major groups.

Fe - soil extraction technology goes soil preparation natural soil taken from the mine, the fraction size < 0,1 microns includes gravel. Pre-washed natural soil is placed in a mixer, water is added in a ratio of 1:10 = T:W (W:T) and left to swell at room temperature for 24 hours. The ground suspension is then placed in a mixer and treated with 22 kHz ultrasound for 3 minutes.

A mixture of basic iron (III) chlorides is prepared and placed in the next mixer, 1 drop of 1.0 M FeCl<sub>3</sub>·6H<sub>2</sub>O NaOH solution is added to it and the desired concentration is obtained. The resulting ash is left to age at room temperature for 24 hours. The hydrolyzed iron salt should have a pH of 1.55-1.95. Then add the ash while stirring into the next mixer with the soil slurry. For each 1 kg of soil, 10 liters of ash are applied.

Two-stage technology of purification of polluted water. The two-stage adsorption-catalytic technological scheme for purification of polluted water from organic dyes and surfactants is based on experiments with sorbents and

catalysts obtained from natural soil and iron polyhydroxocomplex, mixed aluminum polyhydroxocomplex [79].

The high efficiency of this technology has been proven in many experiments, for example, the results of dyeing wool products and purification of contaminated water went well in the scientific laboratory. Tests of mixed dyeing models were carried out at the training and experimental complex. The results of oxidation of organic dyes and surfactants in this process prove that the catalysts obtained in the future have great potential.

### 3. Results and Discussion

Clay purification frees it from impurities and increases the proportion of the useful component - the sorption-active mineral montmorillonite. The linearity of the VET method (measurement of specific surface area and porosity) was determined. Accuracy, reproducibility and standardization of the method for determining adsorption activity were evaluated. Exponential dependence of adsorption activity on the sample weight (0.8 g), sorption time (20 min) and the equilibrium concentration of dye solution (35 ml 0.15 %) was found. Physico-chemical, technological properties of bentonite clay for chemical applications were studied. It has been found that bentonite clay for chemical industry application is a light gray powder, odorless, practically insoluble in water and organic solvents, with particle size less than 0.1 mm, the pH of 2% suspension is 7.1-8.7. According to the technological characteristics it is a fine powder of medium gravity with medium flowability index. In terms of adsorption characteristics - it is a combined meso-macro-microporous adsorbent with a predominance of mesopores, its specific surface area is 53.5 m<sup>2</sup> / g, pore volume - 0.065 cm<sup>3</sup> / g, the average pore size - 4.8 nm, adsorption activity by methylene blue - 62.0 bentonite/g. The effectiveness of mechanical processing was controlled by observing the main parameters that confirm the change in the structure of clay: the size and shape of the particles on electron microphotographs, the indicators of adsorption activity. Weaning is a method of separation of solids based on different rates of their fall in a liquid medium. Disturbed sedimentation was carried out in a reactor with a stirrer: one part of the mineral raw material bentonite clay and 10 parts of water were loaded into the reactor and stirred for an hour, then it was left to stand for 24 hours, then stirred for another 10 minutes and left to stand for an hour. After sedimentation, the middle colloidal layer of clay suspension was decanted from the liquid phase and selected by siphoning.

Weakening was carried out three times. Control of release of clay from sandy impurities was made by rubbing it between two glasses. The criterion for the effectiveness of distilling is the absence of sand particles in the clay. Centrifugation was carried out for 5 minutes at a centrifuge operating mode of 3 thousand revolutions per minute. Drying of clay was carried out in a drying oven at 120 ° C for 180 minutes. As a result, clay with a particle size of 1-20 mm was obtained. The specified drying rates were established by practical way as the most effective.

Mechanical processing was carried out in a ball mill with subsequent control of the shape, particle size and adsorption activity. It was found experimentally that the optimal time of mechanical processing is 80 minutes. The working hypothesis of mechanical processing for 80 minutes is based on the fact that with increasing processing time there is an increase in the specific surface area, as well as changes in the shape of the solid and the accumulation of defects on its surface. The final stage includes packing, packaging and shipment of finished products to the warehouse. Packing was carried out by 25 kg in paper bags with polyethylene inserts according to State Standard 19360-74. Each package had a label with the name of the product, name of the manufacturer, net weight, series number, date of manufacture, expiration date.

Figure 3 shows the initial sample with an average size of 10-30 microns; the amount of the specified fraction is 54%, the amount of the fraction with a particle size of 25-35 μm. After 30 minutes of mechanical activation, the number of particles with an average size of 9-13 microns increases to 53%, the number of particles with a size of 15-30 microns decreases to 30%.

After 40 minutes of mechanical activation, a further decrease in clay particles occurs, elements with an uneven surface of 10-30 μm appear, an increase in the amount of this fraction from 34% to 84% is observed (Fig. 4). After 80 minutes of mechanical activation (Fig. 4), particles with a size of 1-4 microns appear, particles with a size of 33-53 microns disappear. The number of particles with a size of 8-10 microns and 13-30 microns is 49% and 34%, respectively. The number of elements with an uneven surface increase. Sticking of small plate elements is observed. After 90 minutes of mechanical activation (Fig. 5), the content of the fine fraction (3-5 μm) increases from 39% to 84%, the amount of the 8-10 μm fraction decreases from 49% to 33%, and fractions larger than 30 μm disappear. In this case, the maximum number of particles with uneven edges and defects on the surface is observed.

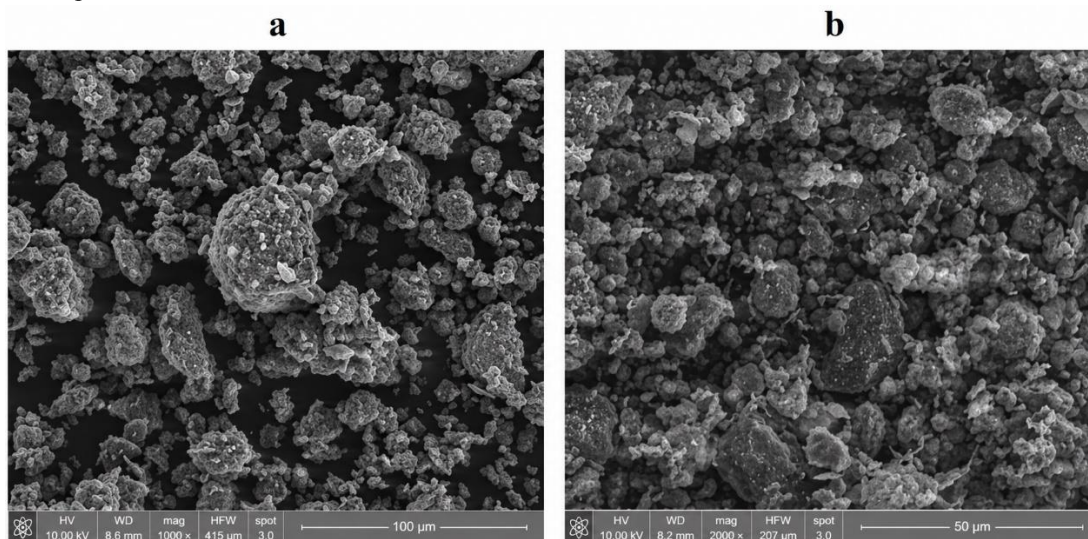


Figure 3. Micrograph of the initial clay sample (a) and after 30 minutes of mechanical activation (b)

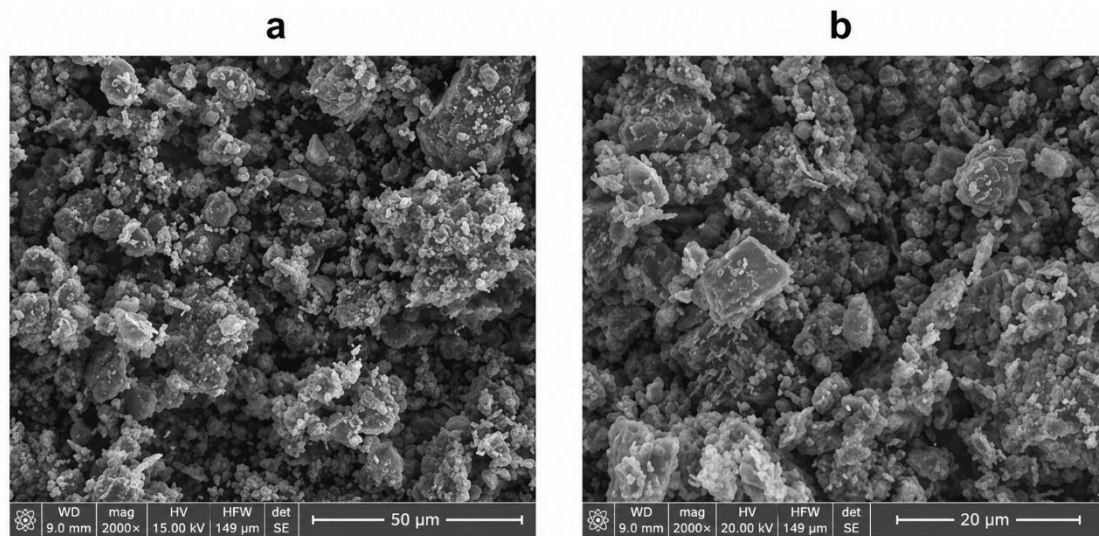


Figure 4. Micrograph of a clay sample after 40 minutes (a) and after 80 minutes (b) mechanical activation

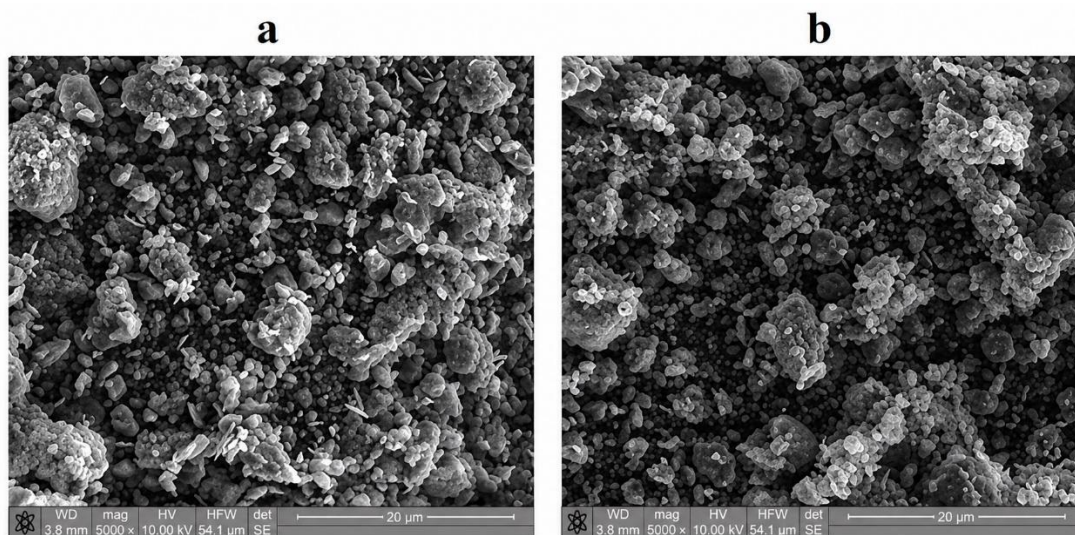


Figure 5. Micrograph of a clay sample after 90 minutes (a) and after 100 minutes (b) mechanical activation

The aggregation of particles increases. After 100 minutes of mechanical activation, the fractional picture, in comparison with the previous one (after 90 minutes of mechanical activation), changes little. The particles are aggregated and enlarged. Based on the above, the calculation of the particle size distribution was carried out depending on the duration of mechanical activation, presented in Table 1. As follows from the table, the sizes of clay particles decrease from 50 µm (after 30 minutes

of mechanical activation) to 3-5 µm (after 100 minutes of mechanical activation) activation). After 40 minutes, the elements stick together. After 90 minutes, the maximum number of lamellar elements and particles with ragged edges and defects on the surface is recorded. Clay processing for more than 90 minutes is impractical due to increased energy consumption. Thus, the most favorable is the 90-minute mode of mechanical activation.

Table 1. Changes in the particle size of the studied clay sample depending on the duration of its processing

Processing time, minutes	Particle size, µm					
	3-5	5-10	10-20	10-30	30-50	More than 50
	Fraction content, %					

Without processing	-	19	51	34	8.5	1.5
5	-	49	33	15	5	-
15	-	39	80	9	4	-
30	39	49	31	3	-	-
90	83	31	8	-	-	-
80	85	30	5	-	-	-

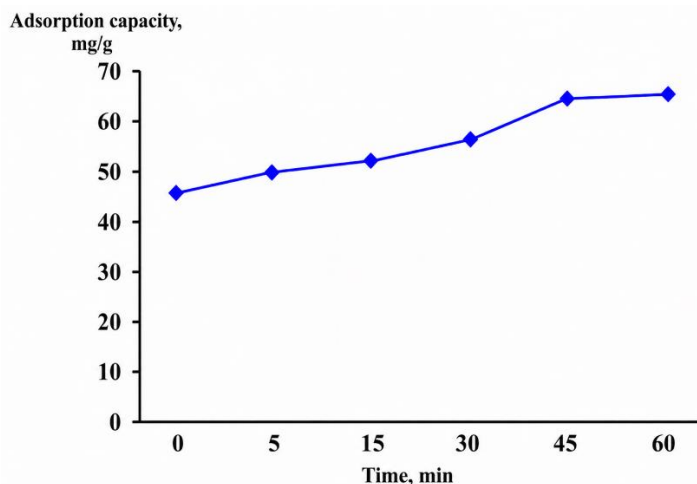


Figure 6. Dynamics of changes in adsorption activity depending on the time of mechanical activation

Control over the process of mechanical activation of bentonite in terms of "adsorption activity" was carried out using methylene blue dye. The results of the analysis of the adsorption activity of clay, obtained depending on the time of its mechanical activation, are shown in Figure 6.

As follows from the diagram, the maximum adsorption activity observed in samples that have been machined for 90 and 100 minutes. In our opinion, the most optimal mechanical activation time is 90 minutes. Clay processing for more than 90 minutes is impractical due to increased energy consumption.

Sorption of methylene blue occurs due to the isomorphic substitution of atoms with the lowest valence in the tetrahedral layers of the crystal lattice structure of clay minerals. The number of active adsorbed centers depends on the structural features of the crystal lattice of clay

minerals. The connection of methylene blue with the active adsorbed center occurs through its amino group or the central nitrogen atom. These processes are accompanied by a change in the color of the reagent used. The color of the clay after cleaning according to our method changes: the shade characteristic of the mineral montmorillonite becomes more pronounced. This allows us to assume that the proportion of the sorption-active mineral montmorillonite in the clay composition increases after its purification and enrichment. The study of the spatial structure showed that morphologically betonies consist of symmetric equated particles with an average particle size of 3-5 microns, which characterizes it as a mineral raw material with a high specific surface. Based on the energy dispersive spectrum, the chemical composition of Navahos betonies was determined, presented in Table 2.

Table 2. Chemical composition of betonies clays of the Navahos deposit

Name	The content of oxides, (the masses %)											
	SiO <sub>2</sub>	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>	SO <sub>3</sub>	FeO	p.p.p.

Alkaline bentonite clay	59,91	0,35	13,89	5,10	1,94	0,49	1,53	1,95	0,43	0,95	----	18,91
Carbonate - playworker clay	48,99	----	9,83	----	3,94	10,09	----	1,80	1,99	----	3,41	34,33
Alkaline earth clay	58,33	0,81	13,58	8,50	3,98	0,89	0,99	3,30	0,93	0,49	----	14,08

According to the standards of the International Union of Theoretical and Applied Chemistry, the study of the specific surface area and porosity is mandatory when studying sorbents, since it allows you to identify a clay sample with the most optimal adsorption characteristics. Adsorption is determined by the presence of pores in the test sample. Pores with a diameter less than 0.4 nm are called sub micropores, 0.4-3 nm in size - micro pores, 3-50 nm - micro pores, with a diameter of more than 50 nm - macro pores. Micropores act as channels for the penetration of substances into the sorbent. Mesopores are much smaller than macropores; their radius of curvature is from 3 to 50 nm, which is much larger than the dimensions of the adsorbed molecules. Filling the volume of these pores is already possible by the method

of capillary condensation. At pressures below the corresponding capillary condensation, adsorption occurs on the mesopore surface. Micro pores are filled with volumetric filling. Table 3 shows the texture and adsorption characteristics of betonies clay.

From table 3 it follows that betonies are a fine crystalline powder of medium weight with satisfactory flow characteristics. The use of betonies powder in catalysis can be difficult. According to its adsorption characteristics, betonies is a combined micro-macro-micro porous adsorbent with a predominance of mesopores. Since the specific surface area is an average characteristic of the size of internal pores, its high indicator is due to the average pore size, which in betonies clay is 4.9 nm.

**Table 3. Textural and adsorption characteristics of bentonite clay**

#	Characteristics	index
1	Main faction	Less than 0.1 mm - 95%
2	Maximum bulk density, g/cm <sup>3</sup>	0.935
3	Adsorption activity, bentonite, g	88.0±0,3
4	Cation exchange capacity, bentonite, eq	31.4
5	Specific surface area according to the single-point BET method, sm <sup>2</sup> /g	58.5
6	Specific surface area according to the BET five-point method, sm <sup>2</sup> /g	58.5
7	Pore volume at pressure P/P <sub>0</sub> = 0.99, cm <sup>3</sup> /g	0.089
8	Average pore size, nm	4.9

In order to standardize the method for determining the adsorption activity, the optimal conditions were selected according to the following indicators: the weight of the sample of the sorbent; sorption time; the volume or

equilibrium concentration of the dye solution. Nine studies were conducted for each indicator. The results are shown in Table 4.

**Table 4. The results of the quantitative determination of the adsorption activity of betonies clay according to the following parameters: weight of the sorbent sample, sorption time, volume or equilibrium concentration of the dye solution**

Sample #	1	3	3	4	5	8	9	9	9
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m*, g	0,4	0,5	0,8	0,9	0,9	0,9	1,0	1,1	1,3
t, min	30								
V, ml	35								
A, betonies	81,9	81,9	81,9	83,0	83,0	59,9	59,3	58,0	53,1
Sample #	10	11	13	13	14	15	18	19	19
m*, g	0,9±0,003								
t, min	3	5	10	15	30	35	30	35	40
V, ml	35								
A, betonies	5,9	13,3	39,9	49,1	83,0	83,1	83,0	83,3	83,3
Sample #	19	30	31	33	33	34	35	38	39
m*, g	0,9								
t, min	30								
V, ml	15	30	35	30	35	40	90	50	55
A, betonies	31,3	35	44,9	55,3	83,0	83,1	83,1	83,3	83,1

As shown by the tabular data, an increase in the weight of a sample of betonies clay at the same sorption time and a constant volume of the dye solution leads to a decrease in the fixation of the adsorption activity of betonies. Increasing the sorption time or volume of methylene blue dye does not change the adsorption activity reading. A decrease in the sorption time or the volume of methylene blue dye leads to incomplete adsorption and a decrease in the adsorption activity index. Thus, according to the results of the conducted studies, an exponential dependence of the adsorption activity on the weight of the sample, the sorption time and the equilibrium

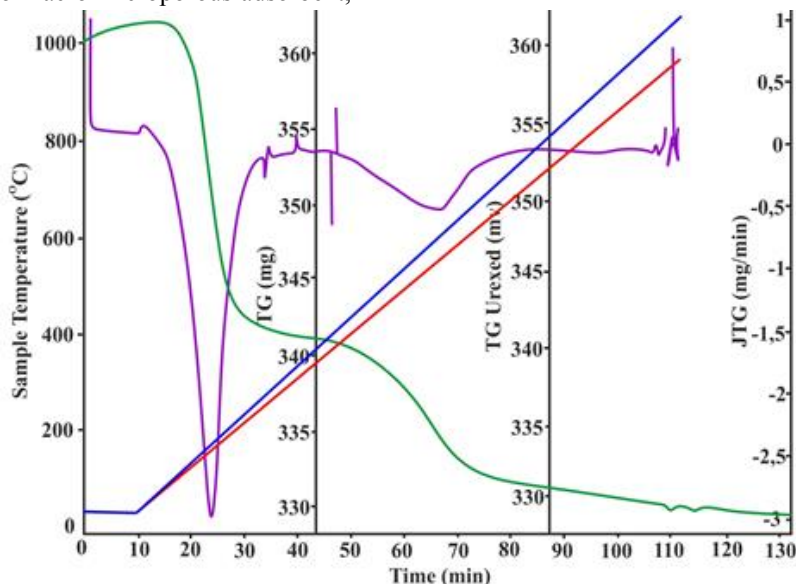
concentration of the dye solution was established, which confirmed the advisability of choosing the correspondence of the sample (0.9 g), the sorption time (30 min) and the equilibrium concentration of the dye solution (35 ml 0.15%). Betonies was standardized according to the following parameters: description, pH of the aqueous suspension, weight loss on drying, adsorption activity, cat ion exchange capacity, heavy metals (arsenic), specific surface area, volume and average pore size. The obtained indicators are presented in table 5.

**Table 5. Indicators of the quality of betonies clay**

#	Characteristics	Bentonite clay
1	Description	Light gray powder, odorless, practically insoluble in water and organic solvents
2	pH of a suspension (5 in 100) in water	9.1-9.9
3	Weight loss on drying,%	88.0±0,3
4	Adsorption activity, bentonite, g	83.0±0,3
5	Cation exchange capacity, bentonite, eq	19.4
6	Arsenic	Absent
7	The ratio of the elements Si <sup>4+</sup> and Al <sup>3+</sup>	3:1
8	Specific surface area according to the BET five-point method, m <sup>2</sup> /g	54,5±3,0
9	Pore volume at pressure P/P <sub>0</sub> = 0.99, cm <sup>3</sup> /g	0.085±0.005
10	Average pore size, nm	4.9

Thus, bentonite is a light gray powder, odorless, practically insoluble in water and organic solvents, the pH of the suspension (5 in 100) is 9.1-9.9. The weakly alkaline nature of the suspension is explained by the presence of alkaline earth and alkali metals in the clay composition. According to the adsorption characteristics, this is a combined meso-macro-microporous adsorbent,

the specific surface of which is  $54.5 \text{ m}^2/\text{g}$ , the pore volume is  $0.085 \text{ cm}^3/\text{g}$ , the average pore size is  $4.9 \text{ nm}$ , the adsorption activity for methylene blue is  $83.0 \text{ mg/g}$ . According to its technological characteristics, it is a finely dispersed medium-weight powder with an average flow ability index.



**Figure 7. Derivatogram of bentonite from the Navbakhor field**

Analysis of keratographic curves of bentonites lines (Fig. 7) shows images of TG - thermal logarithmic gravimetric line, where a decrease in the initial mass of bentonites is noted for a certain time, and DTG - differential thermal gravimetry shows a line of temperature parameters change.

The primary decomposition of bentonites began at  $90 \text{ }^\circ\text{C}$  and ended at  $300 \text{ }^\circ\text{C}$ . The loss was due to absorbed structural water and other volatile impurities.

At the second stage of decomposition at a temperature of  $480 \text{ }^\circ\text{C}$ , the remaining mass of 339 bentonite is reduced to 335 bentonites, or 3.9% of the original mass. At this stage, the decrease in mass occurs due to the evaporation of carbonates and other volatile impurities that can decompose at given temperatures. This took about 19 minutes. In total, the decomposition of bentonite took about 59 minutes, and the weight loss was 14% in total.

After that, the decomposition process stopped, and stable metal oxides may have remained on the solid support (bentonites). Thus, when the keratographic lines of coal and bentonites were combined, they, in principle, turned out to be the same. In the process of keratographic analysis of coal and bentonites, the optimal parameters of decarburization were established:  $800 \text{ }^\circ\text{C}$  for 40 min and activation at  $950 \text{ }^\circ\text{C}$  for 90 min. The main parameters for

further checking the quality of the obtained granules are the establishment of the sorption capacity, strength and the ability to regenerate for further repeated use.

One of the main properties of bentonites, which is decisive for its use in industry, is the ability to swell, that is, the ability to absorb water. In almost all areas of application, bentonites are used in ground form.

Enriched kaolin mainly consists of kaolinite with admixture of hydromica up to 5-20 %. Concentrated kaolin according to the average chemical composition is close to kaolinite, formula of which is  $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$  (39.5 % + 46.5 % + 14.0 %). The yield of marketable kaolin at enrichment is not lower than 40 %. The percentage of white color of samples after drying at  $110 \text{ }^\circ\text{C}$  (whiteness) for groups I and II - 84-87%; for groups III and IV - 84-74%. Fire resistance - from  $1730$  to  $1780 \text{ }^\circ\text{C}$ ; mechanical strength - from 0 to  $200 \text{ kg/cm}^2$ , depending on quantity of swelling mineral montmorillonite in kaolin's; sintering ability - not lower 2 %; air shrinkage - 2,9-7 %; fire shrinkage - 11,9-16,4 %; threshold of structure formation - from 10,078 to  $1,21 \text{ g/cm}^3$ . According to the above characteristics, enriched kaolin meets the requirements and is suitable for use.

Plastic properties of clay were evaluated by plasticity and its binding capacity. Volga sand was used as a sinking material in determining the binding capacity. Plasticity

was determined by the method (Table 6). According to clay plasticity classification, clay belongs to moderately plastic clays.

**Table 6. Results of determining the plasticity of masses of mixtures of clay and Volga sand**

Mass composition, %		Average yield strength, %	Medium rolling limit, %	Number of plasticity
Clay	Sand			
100	-	46,8	34,2	12,6
60	40	35,0	23,2	11,8
40	60	23,2	14,2	9,0
20	80	13,9	10,7	3,0

Binding ability of clay was determined by the change in plasticity of the mass with different contents of sinking agents (Volga sand) and the limiting amount of Volga sand, the addition of which forms a dough with a number of plasticity of not less than 7. According to the binding capacity (with 60 % of Volga sand bound clay, the

number of plasticity of the mass is 9.0) clay belongs to the plastic clays (the amount of Volga sand bound clay, 20-60%).

Sintering temperature of clay was set by firing samples at 600-1400 °C with subsequent determination of properties (Table 7).

**Table 7. Properties of samples fired at different temperatures**

Firing temperature, °C	Sample properties			
	Linear shrinkage, %	Water absorption, %	Porosity, %	Apparent density, g/cm <sup>3</sup>
600	3,3	30,2	43,5	1,44
800	4,5	30,7	43,8	1,43
1000	6,8	17,7	30,1	1,80
1100	11,9	17,0	30,5	1,80
1200	14,2	7,6	16,8	2,23
1250	18,1	2,1	5,5	2,61
1300	20,7	1,1	2,7	2,65
1400	22,2	1,5	3,6	2,39

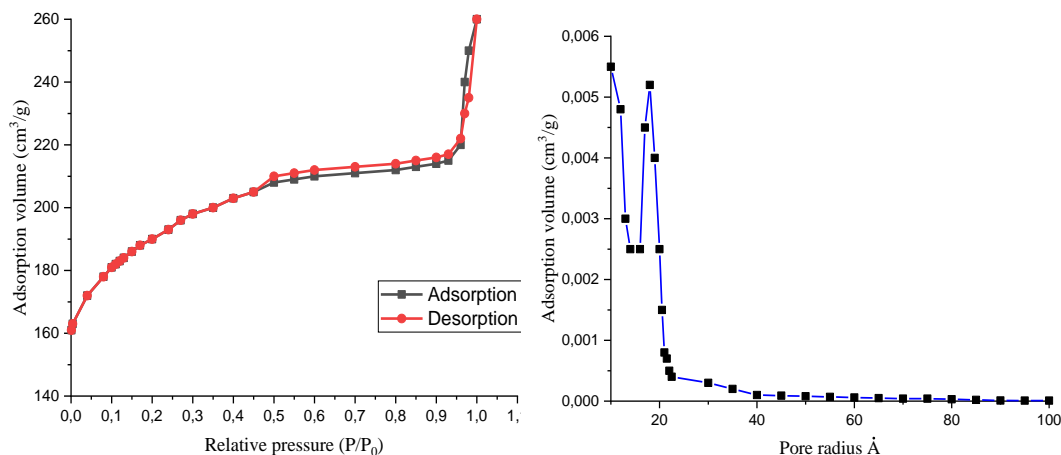
Other clays studied contain coloring oxides, so their use in the masses may cause a yellowish-grayish hue of the tiles, which would require the use of highly muted glazes. According to the data obtained, modification of bentonite with aluminum and iron (III) hydroxocations leads to an increase in the ultimate adsorption capacity of bichromate ions. The optimum number of modifying components is 5 [Me<sup>n+</sup>] mmol/g of bentonite. A further increase in the number of modifying components leads to a relative decrease in the value of adsorption capacity.

High-temperature combustion leads to a decrease in the adsorption-active surface of the sorbents.

It should also be noted that the value of the maximum adsorption capacity for Al-modified sorbents increases in proportion to the relative surface area of the sorbents. The increase in the adsorption activity and specific surface area of Fe-modified sorbents also testifies to the efficiency of the technology of surface modification of bentonite-based sorbents with iron (III) polyhydroxycationic compounds.

In the present work a sorbent on the basis of bentonite clay of Navbakhor deposit was synthesized for the first

time. SEM and BET showed that as a result of modification the textural and sorption characteristics of bentonite clay were significantly improved. The effect of the amount of modifier, initial salt concentration, and reagent contact time on ion adsorption was established. Comparison of surface and microwave size was calculated by BET method for mesoporous sorbents and Dubinin method for mesoporous sorbents. Adsorption isotherms are typical of type IV according to the de Bour classification and characterize the adsorption of mesoporous materials. In the isotherm, monomolecular adsorption in the shells and  $P/P_0 = 0-$



**Figure 8. Adsorption-desorption isotherms and pore size distribution differential curves related to radius of synthesized sorbents.**

Activation was carried out by optimizing a multi-factorial experiment according to a complex plan 2<sup>4</sup>. Obtained as an optimization criterion, after activation, the wet zeolite residue ( $y_1$ , r/100g), the equilibrium capacity for nitrogen ( $y_2$ , cm<sup>3</sup>/g), the equilibrium capacity for oxygen ( $y_3$ , cm<sup>3</sup>/g) and the separation coefficient of the nitrogen-oxygen mixture ( $y_4 = y_2/y_3$ ). The optimization factors selected were: temperature,  $X_1$  (400-500 °C), heating rate,  $X_2$  (2-5 °/min), the amount of CO<sub>2</sub> in the washing nitrogen gas,  $X_3$  (0-1 volume %), the duration of the process,  $X_4$  (0.5-1 hour) and the cationic composition of zeolite  $X_5$ . Process Research Parameters (temperature, heating rate and others) are determined experimentally.

For absorption into the CaCl<sub>2</sub>·MnCl<sub>2</sub>/HQZ system, the following model of the composition of the mixtures was prepared. Studies have shown that CaCl<sub>2</sub>·MgCl<sub>2</sub>/HQZ is 14.3% (mass fraction) of sulfur in terms of hydrogen sulfide. The dynamic sulfur capacity at 1000 pmm is 11.3%.

0.05, as well as adsorption in microwave microwaves are observed. At  $P/P_0 = 0.05$ , mono- and multimolecular adsorption in the pores is observed. Area  $P/P_0 = 0.05-0.4$  is the polymolecular adsorption that is used to compare surface surfaces ( $S_{sol}$ ) in the BET equation. Adsorption isotherms characterize capillary condensation in the intermediate shells  $P/P_0 = 0.4-1.0$ .

The paper presents the results of the influence of thermal activation factors on zeolites with different cationic composition. Silicon enriched zeolite obtained from Navbakhar bentonite was used as sorbent (Fig. 8).

According to the experiment, we can conclude that in the apparatus with a flow of 4000 m<sup>3</sup>/day of gas, this sorbent can be used 1.5 weeks continuously. To confirm CaCl<sub>2</sub>·MnCl<sub>2</sub>/HQZ, the possibilities were used. The experiment was conducted at a research industrial device in the Bukhara oil refinery filled with an adsorber with a diameter of 1.2 m, a height of 8 m, and a mass of 2 tons. In the industrial apparatus of the mass fraction in the gas studied, 3.5% H<sub>2</sub>S after the start of the test, after 24 hours, the concentration of hydrogen sulfide released from the adsorber was 180 ppm. Sample results showed that CaCl<sub>2</sub> · MnCl<sub>2</sub> /HQZ. The dynamic capacity for hydrogen sulfide mass fraction was 2.76%. The full dynamic capacity of the adsorbent for H<sub>2</sub>S is less than 20-25%.

Local raw materials: kaoline and bentonite were activated using physical and chemical methods. Textural characteristics and surface morphology of prepared high silicaon zeolites. The Branauer-Emmet-Teller (BET) method was used to determine the specific surface; the Barret – Jouner –Halenda (BJH) method was used to determine the pore volume and size. It was proved that

$P/P_0 = 0-0.05$  in the mesopore interval monomolecular adsorption,  $P/P_0 = 0.05$  in the mesopores mono- and multimolecular adsorption, when  $P/P_0 = 0.05-0.4$  in the observation zone polymolecular adsorption. The activation process of the HQZ obtained from kaolin Nurabad was optimized by a multi-factor  $2^4$  experiment. The following factors were selected as optimization factors: temperature,  $X_1$  (400-500 °C), heating rate,  $X_2$  (2-5 °/min), the amount of  $CO_2$  in the nitrogen washing gas,  $X_3$  (0-1 volume %), process duration  $X_4$  (0.5-1 hour), and the cationic composition of zeolite  $X_5$ .

The obtained regression equations in the studied range allow us to determine the assessment of the criteria for any combination of the studied factors, analyze the intensity of the indicators of each factor, process and determine the optimal conditions for the process.

Bentonite clays (montmorillonite) are characterized by a layered crystalline structure (3-layer) based on Al-Fe-Mg-octahedral grid enclosed between silica-oxygen tetrahedrons (TOT structure) with silicate layer thickness of 0.94 nm. One unit cell of montmorillonite is formed by 20 oxygen atoms and 4 hydroxyl groups, which makes 8 silicate tetrahedrons and four Al-bearing octahedrons. The interlayer space contains exchangeable cations ( $Na^+$ ,  $K^+$  or  $Ca^{2+}$ ) and water molecules that prevent the layers from sticking together. In montmorillonite the charge is concentrated in the octahedral layers and distributed over all oxygen atoms in the structure. Usually the lack of positive charge is from 0.4 to 1.2 e per one unit cell of  $Si_8O_{20}$ , which when translated into cation-exchange capacity corresponds to 0.5-1.5 mg-eq./g. Typically, the exchange capacity of montmorillonites is approximately 0.7-1.0 mg-eq/g. The exchange capacity of montmorillonite is mainly represented by  $Na^+$  and  $Ca^{2+}$  ions located in the space between the silicate layers.

Another source of exchange center are weakly acidic hydroxyl groups (Si-OH) and basic (Al-ON) on lateral faces and edges, which participate in ionic exchange depending on the pH. For example, the presence of OH-groups causes layered minerals to be capable of anion exchange. In addition to ionic exchange, bentonite clays are characterized by physical and molecular sorption. Physical sorption is caused by the presence of excessive negative charge on the crystal faces and surface hydroxide groups of acid and basic nature capable of ionization. During the molecular sorption the sorbed substances are located between the planes of the packages, destroying the original aqua complexes without changing the structure of the layers themselves. Thus, the presence of such active centers as

exchangeable cations, hydroxyl groups, and activation of bentonite clays makes it possible to considerably expand the practical application of layered silicates for cleaning aqueous media by modifying their surface.

According to the obtained data of X-ray phase analysis, the investigated samples of pellets annealed at different temperatures have similar mineralogical composition. Heat treatment of samples practically does not affect the mineralogical composition and structure of granulated material, and with increasing firing temperature the intensity of peaks slightly increases, indicating crystallization and dehydration of phases.

To determine the chemical and mechanical stability, 100 g samples of pellets were placed in glass flasks with a capacity of 250 cm<sup>3</sup> and 150 cm<sup>3</sup> of distilled water were poured into them. Flasks with the sorbent under study, poured with water and sealed with plugs, were shaken in continuous mode for 24 hours on the device AVU-6 with a speed of 120 shakes per minute. After drying the sorbent was dispersed on 0.5 and 0.25 mm sieves. The pellets that passed the sieve 0.5 mm and remained on the sieve 0.25 mm characterize the grindability, and the pellets that passed the sieve 0.25 mm-grindability. The positive effect of increasing the temperature of heat treatment on the mechanical strength of granules has been established, which is associated with the evaporation of all crystallization water and structural water. However, under the influence of higher temperature (over 700 °C) a significant decrease of specific surface values is observed in granules, thus the sorption capacity decreases, which makes the practical application of the obtained granules less feasible. It is also not expedient to carry out firing at temperatures less than 450 °C, as the samples do not have sufficient durability because of the incomplete yield of crystallization water. Thus, the experimentally established optimum firing temperature is in the range of 450-550 °C. Thermogram of this bentonite is shown in Figure 9.

The first endothermic effect is observed at 373-453K due to the release of free and interstitial water. The second endothermic effect is weaker than the first one and is observed in the temperature range 933-1053K and is caused by loss of constitutive water as well as dissociation of magnesium and calcium carbonates, which are present in this bentonite as a modifier. Decarbonization of carbonates is a topochemical process. In general terms, the endothermic decarbonization reaction is described by the equation:  $A_m \rightarrow B_m + C_r$ ,

where  $A_m$  is  $CaCO_3$ ,  $MgCO_3$ ;  $B_m$ - $CaO$ ,  $MgO$ , and  $C_r$ - $CO_2$ .

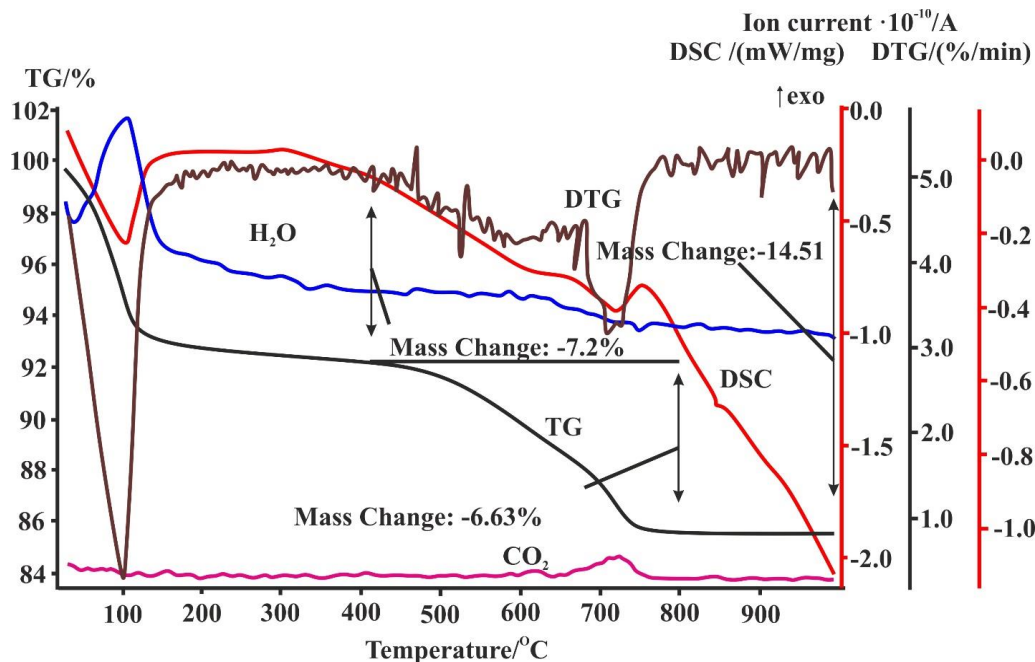


Figure 9. Thermogram of bentonite

The peculiarity is the formation of two new ones from the solid phase: solid and gaseous. The density of the newly formed solid phase is much higher than the initial phase. As a consequence, the volume of the newly formed solid phase is much less than the initial one. Thus, instead of  $1\text{ cm}^3\text{ CaCO}_3(MgCO_3)$   $0.455\text{ cm}^3\text{ CaO}$  and  $0.397\text{ cm}^3\text{ MgO}$  are formed. The decomposition temperature of calcium and magnesium carbonates is influenced by the size of the initial bentonite crystals. Decrease in decomposition temperature with increasing dispersion degree is related to the well-known phenomenon of vapor elasticity with decreasing particle radius. In the thermogram, the greatest amount of released  $CO_2$  is observed at temperatures of  $993\text{--}1013\text{ K}$  and further increase in temperature no longer leads to a significant change in mass. On the whole, the mass of the sample in the temperature range under consideration decreased by  $14.51\%$  where  $7.62\text{--}8\%$  is water and evolved  $CO_2$ . Further increase in temperature leads to bentonite destruction. The specific surface of bentonite is very high and is equal to  $65.30\text{ m}^2/\text{g}$ .

Thermogram of Navbahor bentonite after mechanical treatment is shown in Fig. 10. The first pronounced endothermic effect is observed in the interval  $343\text{--}423\text{ K}$ , where the removal of free and partially interstitial water and reduction of the sample mass by  $4.86\%$  occur. The

second endothermic effect occurs at  $733\text{--}773\text{ K}$  and is associated with the removal of constitutive moisture and release of  $CO_2$  associated with the decomposition of the organic component of bentonite.

The third endothermic effect on the heating curve after mechanical treatment of Navbakhor bentonite is observed in the temperature range  $853\text{--}873\text{ K}$  and is caused by destruction of the crystal lattice of the mineral, decomposition of carbonates which are in bentonite. Subsequent heating is accompanied by the destruction of the crystal lattice of bentonite and its transformation into an amorphous substance. During heating in the investigated interval mass decrease is  $10.3\%$ , which is much less than for Greek bentonite ( $14.51\%$ ). During heating, two exothermic effects are observed. The first one in the interval of  $553\text{--}953\text{ K}$  is connected with the decomposition of the organic component of bentonite. The second exothermic effect at  $853\text{--}873\text{ K}$ , which is associated with the decomposition of montmorillonite. The fourth exothermic effect is observed in the temperature range of  $1003\text{--}1053\text{ K}$ , where sintering of the components with the formation of mullite occurs. In the process of heating  $CO_2$  is emitted, especially at  $833\text{--}893\text{ K}$ , which is caused by the decomposition of calcium and magnesium carbonates that are part of bentonite.

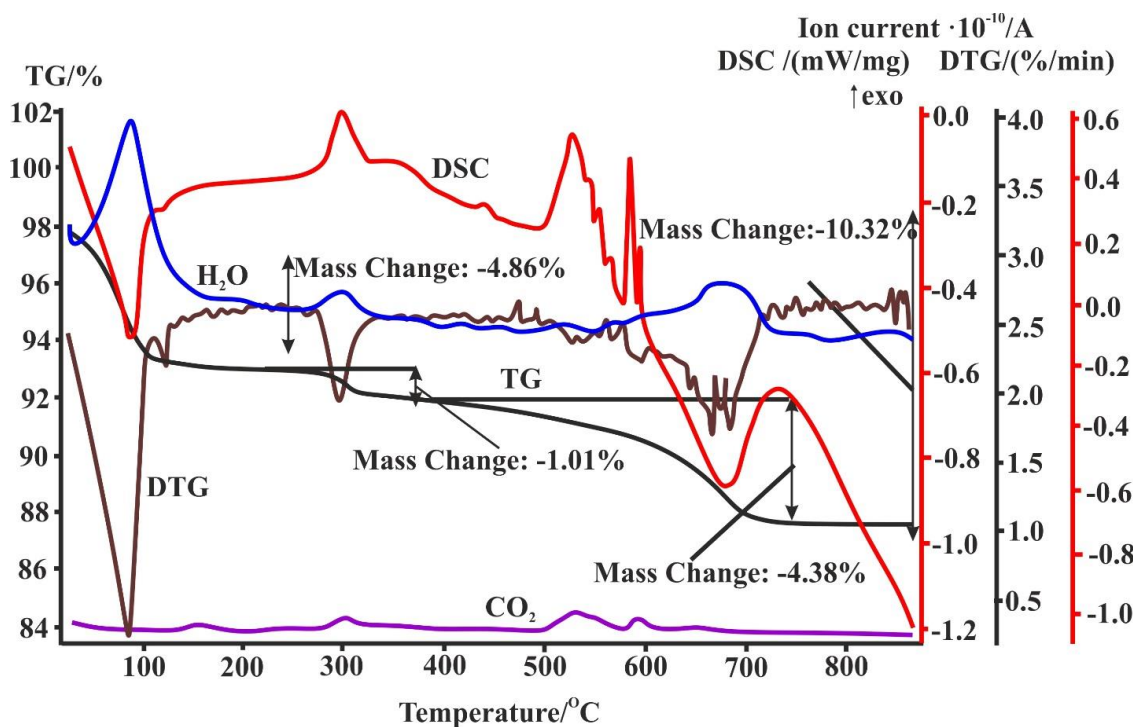


Figure 10. Thermogram of Navbahor bentonite after machining

Figure 11 shows a differential curve of mesopore size distribution of natural bentonite. From the figure you can see that there is a fairly narrow pore size distribution.

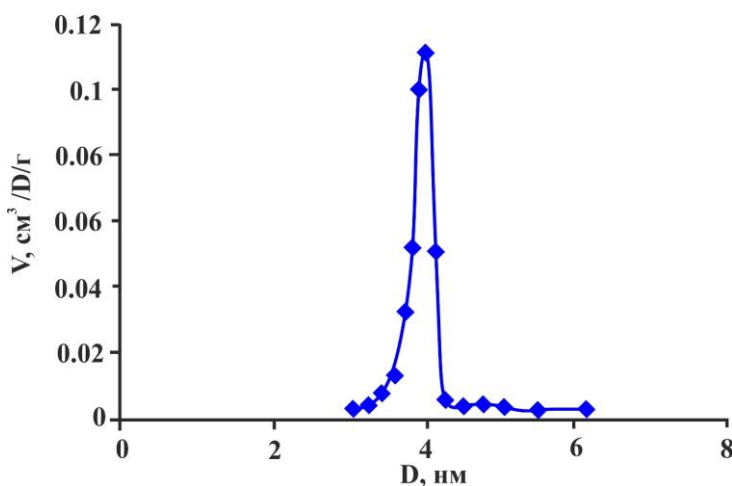


Figure 11. Differential curve of natural bentonite mesopores size distribution

It is found that the mechanical treatment of Navbahor deposit bentonite powder in a planetary mill during the first 60 min leads to a decrease of the average particle size from 27 to 5.5  $\mu\text{m}$  and an increase of the specific surface area of the powder (according to BET method) from  $19 \pm 0.1$  to  $33 \pm 0.7 \text{ m}^2/\text{g}$ . Further mechanical treatment causes agglomeration of particles to 28  $\mu\text{m}$  and reduction of the specific surface area to  $20 \pm 0.2 \text{ m}^2/\text{g}$ . Mechanical treatment of natural zeolite results in

reduction of ROC and growth of micro distortions of the crystal lattice for clinoptilolite and smectite phases. With increasing time of mechanical treatment the amount of X-ray amorphous phase of zeolites increases from 13 to 52 %.

Mechanical activation of bentonites in the milling process makes it possible to increase the overall activity of particles by reducing their size, increasing the overall surface, achieving a more compact particle shape, deep transformations in the crystal structure of the material. A

planetary centrifugal mill-activator AGO-2 with activation time varying from 10 to 180 seconds was used to study the parameters of clay activation in order to achieve the maximum degree of bentonite clay grinding at maximum activity with preservation of the features of their crystallochemical structure and absence of aggregation.

Since the modification of samples in the presence of ammonium hydroxide did not lead to an increase in the volume of micropores, it seems that zirconium and zinc are located in the interlayer space of bentonite in the form of mono-nuclear ions, rather than as polyhydroxo complexes. In addition, the growth of the total pore volume may be due to partial destruction of lamellar particles during thermal treatment of modified bentonite, with additional formation of mesopores and inter-partial voids between disordered layer fragments due to removal of adsorbed ammonium ions and water.

Acid activation of bentonite clay by 0.5 M hydrochloric acid solution leads to removal of calcium and sodium ions by 68.9 and 68.5 % respectively, washout of magnesium ions occurs by 39.2 %. Activation of bentonite clay with 2.0 M hydrochloric acid solution results in decreasing calcium ions concentration by 91.8 % and sodium ions by 92.5%, while magnesium ions are removed by 49%. Thus, for almost complete removal of exchangeable  $\text{Ca}^{2+}$  and  $\text{Na}^{+}$  cations from the interlayer space of the studied bentonite clay, activation with 2.0 M hydrochloric acid for 2 hours at 88 °C is recommended. As a result of activation, the layered structure of clay minerals is preserved, which is very important for further modification of the surface of the obtained samples.

The obtained oligomeric metal hydroxycations are converted by subsequent heating (the modified bentonite was calcined at 400 °C, while the  $\text{Al}_2\text{O}_3$  content increased from 14.4 to 18%) into metal oxides, which hold the montmorillonite layers separately from each other, acting like supports in the interlayer space.

The activity of modified bentonite clays depends on the ratio of aluminum and silicon atoms in them. X-ray patterns showed that modified bentonite clays are a certain chemical compound of montmorillonite mineral type rather than a simple mechanical mixture of oxides. Transition of montmorillonite into H/ $\text{NH}_3$ -form allows increasing the interplanar distance of the sample only slightly (up to 1.0 nm) in comparison with the original bentonite clay. Greater interplanar spacing (1.4 nm) was obtained on  $\text{Al}^{3+}$  modified bentonites as compared to the original and converted to the H/ $\text{NH}_3$ -form. According to X-ray diffraction data for the OH-form of bentonite,

structures resistant to swelling and to temperature exposure were formed in the interlayer space of montmorillonite with the predominance of interplanar distances of 1.8 nm. The specific surface of the original and modified bentonite clays was determined by the method of low-temperature nitrogen adsorption. Based on the obtained physico-chemical characteristics it was found that the modification of bentonite with salts of ammonium, aluminum and aluminum hydroxycation allows to increase the interplanar distance and specific surface of the catalyst.

Only at small concentrations of acid, and with increasing concentration the influence of the time factor decreases. Extrapolation of the response surface beyond the intervals of variation, most likely, does not make much physical sense, since the obtained surface is not essentially linear and for finding the extremum another composite plan should be applied, namely, of the second order. However, the second-order plan for three factors assumes 27 experiments and does not give a guarantee of finding an extremum, because as the number of experiments increases, the probability that the obtained regression equation will be adequate decreases. This is one of the reasons why it is legitimate to stop at linear plans of full factor experiment.

Additional experiments in the region of factor values corresponding to the maximum surface value ( $C_{\text{HCl}} = 2\text{N}$ ,  $T = 40\text{ }^\circ\text{C}$ ,  $t = 4\text{ h}$ ) actually yielded a surface of less than 100  $\text{m}^2/\text{g}$ . Thus, it can be assumed that the extremum is in the region of concentration values from 4 to 6 N, temperature from 70 to 90 °C, and time from 4 to 10 h.

#### 4. Conclusion

The aim of the work is to study the textural and adsorption characteristics of bentonite clay. The chemical composition of the considered bentonites has been determined. It has been established that the particles of the montmorillonite mineral have sizes of 1.0–0.3  $\mu\text{m}$  and more, with a thickness of 0.001–0.03  $\mu\text{m}$ . The shape of the particles is poorly expressed, rarely hexagonal, and tends to form aggregates of particles. The data of thermo gravimetric studies were obtained and the temperature ranges of structural transformations and destruction of bentonite clays of the deposits under consideration were established.

Cleaning clay frees it from impurities and increases the proportion of a useful component – the sorption-active mineral montmorillonite. The porosity of the VET method (measurement of specific surface area and porosity) was determined. An assessment of the accuracy

and standardization of the method for determining adsorption activity was carried out. The exponential dependence of the adsorption activity on the weight of the sample (1.0 g), sorption time (35 min) and the equilibrium concentration of the dye solution (39 ml 0.15%) was established. The physicochemical and textural properties of bentonite clay for the application of catalysis have been studied. It has been established that bentonite clay for use in catalysis is a light gray powder, odorless, practically insoluble in water and organic solvents, with a particle size of less than 0.3 mm.

Bentonite clays are good inexpensive sorbents for various substances, such as heavy metal ions, organic dyes. Enough works have been devoted to the study of the physico-chemical parameters of clays of various deposits activated by various acids. Much attention in the literature is paid to natural frame aluminosilicates, especially zeolites. These materials have a negatively charged three-dimensional aluminosilicate frame. In the gaps of the frame there are hydrated positive ions of alkali metals, compensating for the charge of the frame, and water molecules. When zeolites are heated, water is released from them, and adsorption cavities are formed. The fields of application of bentonite clays will expand by giving them new properties as a result of various types of activation. One of the most effective types of exposure is acid treatment. According to the nature and strength of the effect on the crystal structure of montmorillonites, acids can be divided into three groups.

Physico-chemical properties of natural bentonite and sorbents modified by aluminum and iron (III) polyhydroxylation by "sol-gel" method were studied. It has been shown that modification of natural bentonites leads to changes in their chemical composition, structure and adsorption properties.

Modified sorbents based on bentonite are granular (nanostructured) objects with a large number of pores sized 1.5-4.0 nm. The value of the specific surface of the sorbents depends on the amount of the modifying component - aluminum or iron (III) polyhydroxylation introduced. Adsorption capacity of the studied sorbents by bichromate ions has been studied. According to the form of isotherms it is determined that they have the form of Langmuir isotherms. It is shown that Al-modified sorbents have a higher adsorption capacity for dichromate ions than Fe-modified sorbents and natural bentonite.

Montmorillonite is the main component of bentonite clays-natural aluminosilicates. Due to the specific structure of the crystal lattice framework and developed

interfacial surface, montmorillonite clays have excellent adsorption and ion-exchange properties. The fields of application of bentonite clays expand due to giving them new properties as a result of various types of activation. One of the most effective types of exposure is treatment with acids. Acid-activated montmorillonites are used as acid catalysts for various reactions. Acid activation with HCl solution of calcium and sodium montmorillonites of the Navbahor deposit was carried out. It is shown that acid treatment results in porous structure development due to removal of both interlayer cations ( $\text{Na}^+$ ,  $\text{Ca}^{2+}$ ) and octahedral layer cations ( $\text{Al}^{3+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Mg}^{2+}$ ). The textural characteristics and strength properties of molded composites based on acid-activated montmorillonites have been investigated. The character of changes in the specific surface area, pore volume, pore volume distribution by the strength size of the samples when varying the montmorillonite content in the composite has been analyzed. It was found that the composite materials containing 80 wt. % bentonite have a highly developed monodisperse porous structure in the pore size range of 1.8 nm and a specific surface area of 290-330  $\text{m}^2/\text{g}$ . The strength of composites reaches 100-115  $\text{kg}/\text{cm}^2$ , which is sufficient for industrial carriers.

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