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PHYSICOCHEMICAL AND TEXTURE CHARACTERISTICS OF NATURAL BENTONITE

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ABSTRACT:

The study was conducted on the modification of bentonite of Navbahor district of Navoi region of the Republic of Uzbekistan in chloride, sulfuric and nitric acids and their boiling point. Modified samples were analysed by IR-spectroscopic, X-ray phase, elemental and electron-microscopic analysis methods. Morphology, specific surface structures and spatial composition of bentonite powder found in Navbahor were studied. Mechanical processing was carried out in a planetary mill.

The aim of the study is to investigate the mechanical effects and the effects of acidic processing on the structure and composition of bentonite, its structure and physicochemical properties.

The following methods of inspection were used: scanning electron microscopy (SEM), X-ray structural analysis, Brunauera - Emmeta - Taylor (BET), method laser beam diffraction and element analysis.

It was shown that in the first 60 minutes of mechanical processing, a further grinding of the powder was carried out in a planetary mill, with an increase in the specific surface area of 33m^2/g. Subsequent mechanical activation resulted in particle agglomeration and shrinkage of the specific surface area.

Keywords: crystallinity level of zeolite, bentonite, the structure of bentonite, natural zeolites.

INTRODUCTION:

Natural zeolites are inorganic sorbents. They form a large group of carcass structural aluminosilicates [1]. Natural zeolites have a number of unique properties, including ion exchange capacity, high cation exchange capacity, micro and nanoporous structure, long and widespread use, are used as a high-efficiency system for cleaning paraffinic hydrocarbons, separation of various gases and liquids, and technical and medical sorbents. used as a reference for processing [2-3].

In addition, the diversity of the composition of natural minerals, structural characteristics do not allow to conduct of fundamental studies of the effect of the structure and physicochemical size of materials on their sorption, catalytic and other practical bonds. One of the most promising ways to change the physicochemical properties of materials is mechanical processing, in which the relative surface structures of the morphology and spatial composition of materials change together, leading to changes in the serviceability of materials [5,6].

Bentonite clays have been used for historical purposes to purify wine, oil and water.

Since they are non-toxic and have a highly developed surface, this significantly determines the volume of adsorption i.e. demonstrates the ability to actively absorb in various solutions. Absorption factors of natural bentonite sorbents are the lack of technologies for the formation of effective granules for the treatment of drinking water, industrial effluents because clay minerals are effectively peptized in an aqueous medium, ie dispersed [7].

It is known that for bentonite clays (montmorillonite) the layered crystalline structure (3-layered) is characteristic, based on Al-Fe-Mg-octahedral networks, the thickness of tetroedric silicon oxide (structure TOT) silicate layer is between 0.94 nm [8-9].

One elemental cell of montmorillonite consists of 20 oxygen atoms and 4 hydroxyl groups, which consist of 8 silicate tetroeders and 4 aluminium octahedrons [8-12].

In the space between the layers (Na^+, K^+ or Ca^{2+}) there are exchangeable cations and water molecules, which prevent the layers from sticking together. In montmorillonite, the charge is concentrated in the octahedral layer and distributed in all oxygen atoms in the structure. Typically, the lack of positive charges occurs in one of the elemental cells of Si_8O_{20}, ranging from 0.4 to 1.2 e [13-14], when we convert it to a cation exchange capacity of 0.5–1.5 mg-eq. / G. corresponds to. The exchange complex of montmorillonite was presented based on Na^+ and Ca^{2+} ions located between the silicate layers. Another source of exchange centres is based on weakly acidic hydroxyl groups (Si-OH) and lateral margins and ribs (A1 – ON), which are involved in ion exchange depending on pH. For example, the presence of ON in the group indicates the ability of layered minerals to exchange anions [15-18].

In addition to ion exchange, physical and molecular sorption is characteristic of bentonite sludges. Physical sorption corresponds to the basic characteristic of the edges of crystals and the surfaces of the acid hydroxide group, and ionization, the ability to form an excess negative charge. In molecular sorption, the substance being sorbed is located in interplanetary packages. It does not change the structure of the layers themselves, breaking down the primary active complexes. Thus, the presence of active centres expands to such an extent that the activation of hydroxyl group exchange cations and thus bentonite sludge is practically used to clean aqueous media by modifying the surface of layered silicates [18-21].

The granules of the studied samples have a similar mineralogical composition when heated at different temperatures, according to the X-ray phase analysis obtained. Thermal processing of samples practically does not affect the mineralogical composition of the granular structure of the material.

EXPERIMENTAL PART:

Bentonite from Navbahor district of the Republic of Uzbekistan was inspected. The mechanical activation of zeolites was carried out at a planetary spherical mill Retsch RM 400 at a speed of 350 rpm. The load acting on the planetary mill is 26.8g. ceramic balls with a diameter of 10, 15, 20, 30 mm and a weight of 430 g were used as the decomposing body. Processing time 1 to 600 min. change in the interval [22].

The morphology of the powder was studied by Zeiss Supra 55VP in a scanning electron microscope using a scanning electron microscope method [23].

Laser diffraction analyzer LA-950 HORIBA was used in the analysis and measurement of particle size. The reference surface was determined by the Brunauera-Emmeta-Taylor (BET) method on a TriStar 3000 instrument [24].

The error in measuring the reference surface was less than 3%. Examination of zeolite structures was performed using a CuK_α -irradiator on a diffractometer Rigaku Mini Flex II at a time exposure of 1 s with a 0.4° step at an angle of 3 to 90° from the 2nd. Spatial analysis of zeolite was carried out at a diffractometer D8-Advance Bruker heated from 20 to 1050 °C using a CuK_α -irradiator at an angle of 5 to 85° at the 2nd angle. We performed the radiograph reading using ASTM, PDF-2 cardboard [25]. According to the results of X-ray diffraction analysis, coherent scattering (OKR) and micro-displacement of the crystal lattice were detected by the Holla-Williamson and Selyakova-Sherrera methods [26].

Modification of bentonite of Navbahor district of Navoi region of the Republic of Uzbekistan was carried out in hydrochloric, sulfuric and nitric acids at their boiling point. In tubes containing 1 g of local zeolite, 10 ml of boiling sulfuric, nitric or hydrochloric acid was poured under constant stirring. After 24 h, the zeolite was washed with distilled water to the neutral reaction of the washed water, dried at room temperature, and analysed by IR-spectroscopic, X-ray phase, elemental, and electron-microscopic analysis methods. Acid treatment at different concentrations of acids: 1; 2; 2.5; 3; 4; 5 N was carried out equally. IR spectroscopic studies were performed on an IR spectrometer NicoleteIS-10 in the frequency range 400-5000 cm^{-1}. The phase composition was studied using an X-ray analyser 2DPHASER "Bruker" (CuK_α - radiation, 2nd, 20-80 degrees). Electron microscopic images were taken under a Hitachi TM-3000 microscope. Elemental analysis was performed at Oxford Science.

The silicate modulus of zeolite, i.e. the mole ratio of SiO_2/Al_2O_3, was calculated as follows: $M = (SiO_2)/(Al_2O_3) = (C_{SiO_2} \cdot M_{Al_2O_3})/(C_{Al_2O_3} \cdot M_{SiO_2})$, where C_{SiO_2}- silicon oxide mass fraction,%; C_{SiO_2}- mass fraction of aluminium oxide,%; $M_{Al_2O_3}$, M_{SiO_2} are molecular masses of aluminium and silicon oxides, respectively.

EXPERIMENTAL RESULTS AND DISCUSSION:

Figure 1 shows the element analysis for SEM images and diffractograms Navbahor bentonites. The results of the elemental analysis show that the zeolite powder in the initial state consisted mainly of elements Al, O, Si, Ca, Mg, Fe. The particles that make up the powder have indeterminate shapes, are densely adjacent to each other, and come in a variety of sizes, averaging 27 μm. X-ray identification of Navbahor bentonite showed that the initial powder contained minerals of different compositions in seven phases: smectite (smectite 15A), quartz (quartz), cristobalite (cristobalite-low), clinoptite-illite (clinoptilolite), orthoclase (orthoclase), calcite (calcite) with values of 20, 8, 15, 14, 15, 9, 6% respectively. The amorphous phase in the composition is 13% in the original zeolite. As the mechanical processing time increases, the average size of Navbahor bentonite powder particles changes (Figure 2). The initial average size of the zeolite particle was 27 μm, 5.5 μm after 20 minutes of processing, and 28 μm after 600 minutes of processing. In addition, many particles lost their original shape and took on a spherical shape during mechanical activation (Table 1). In Table 1, the first 60 minutes were crushed by mechanical processing in a planetary mill, which means that the average size of the particles decreases, during which time the particles lose their initial shapes and become spherical shapes.

The specific surface area and the average size of zeolites after mechanical activation in a planetary mill.

Mechanical processing time, min.	Specific surface, m^2/g	Particle size calculated from the specific surface, nm	Particle size obtained from laser analysis, mkm
0	19±0.1	137	27
10	23±0.4	108	10
60	33±0.7	75	21
180	25±0.4	100	27
600	20±0.2	125	28

Subsequent mechanical activation due to agglomeration, each micro-particle is composed of many nanoparticles, the structure of which forms a large specific surface [12].

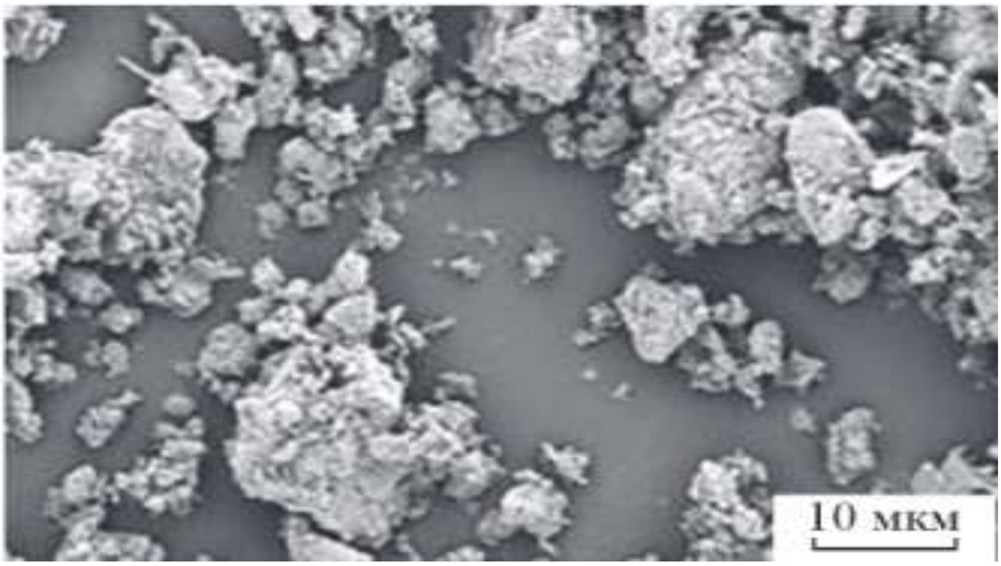


Figure 1. SEM image of early zeolite

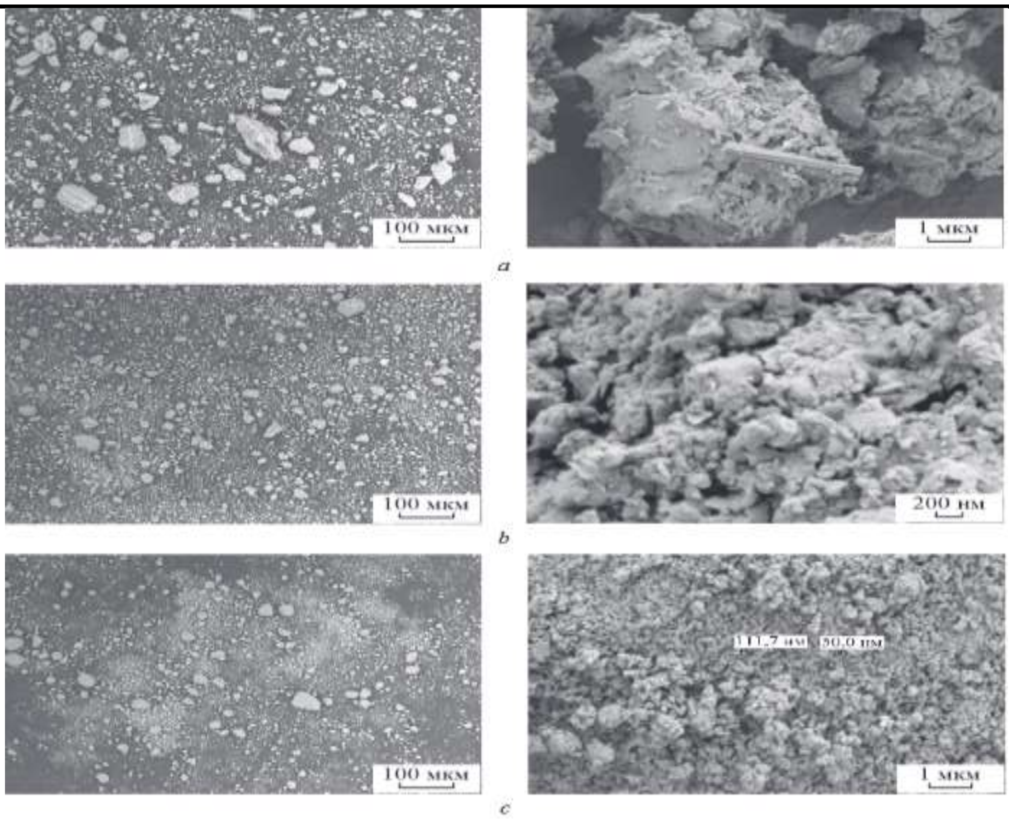


Figure 2. SEM -image: a-initial zeolite, b- 20 min. after mechanical processing, c) 60 min. after mechanical processing.

Quantitative spatial analysis of Navbahor bentonite showed that there were radical changes in the mineralogical composition when the mechanical processing process reached 600 min. The mineral content of smectite clinoptilolite, illite, calcite, cristobalite reached a minimum value and was 0.2, 1, 2.5, 1, 14%, respectively, the content of quartz increased by 17% and that of orthoclase by 12%. The long-term mechanical effect on the stressed zeolite resulted in an increase in amorphous phases in the powder from 13 to 52%. We determined the specific surface area of the powder under test using OKR measurements. The specific surface area of the powder was 32 m^2/g in the initial case. 60 min. overtime, the specific surface area increased, reaching a maximum value of 36 m^2/g. After 600 min, the specific surface area of Navbahor bentonite was 20 m^2/g. The second table shows the results of the surface reference surface found by the BET method and the results of the reference surface calculated from OKR. Thus, the change in the specific surface area of the two different methods determines the specific surface areas of Navbahor bentonite with spatial content to the same properties. The results of determining the porosity of bentonite sludges and sorbents based on them are presented in Table 1.

Table 1. Porosity structure of bentonite clay samples after high-temperature firing.

Examples	Relative surface S, m²/g	The total volume of porosity $V_{por.}$, sm³/g	Radial distribution of porosity, %			
			1,5-2,0 nm	2,0-4,0 nm	4,0-8,0 nm	10,0-52,0 nm
No. 1 primary bentonite powder	65,3	0,064	9	21	21	49
No. 2. Firing t = 450 °C	57,2	0,096	6	16	23	55
No. 3. Firing t = 550 °C	46,8	0,098	6	17	23	54
No. 4. Firing t = 650 °C	45,4	0,100	5	13	25	57
No. 5. Firing t = 750 °C	36,9	0,098	4	14	24	58
No. 6. Firing t = 850 °C	26,4	0,036	5	15	24	56

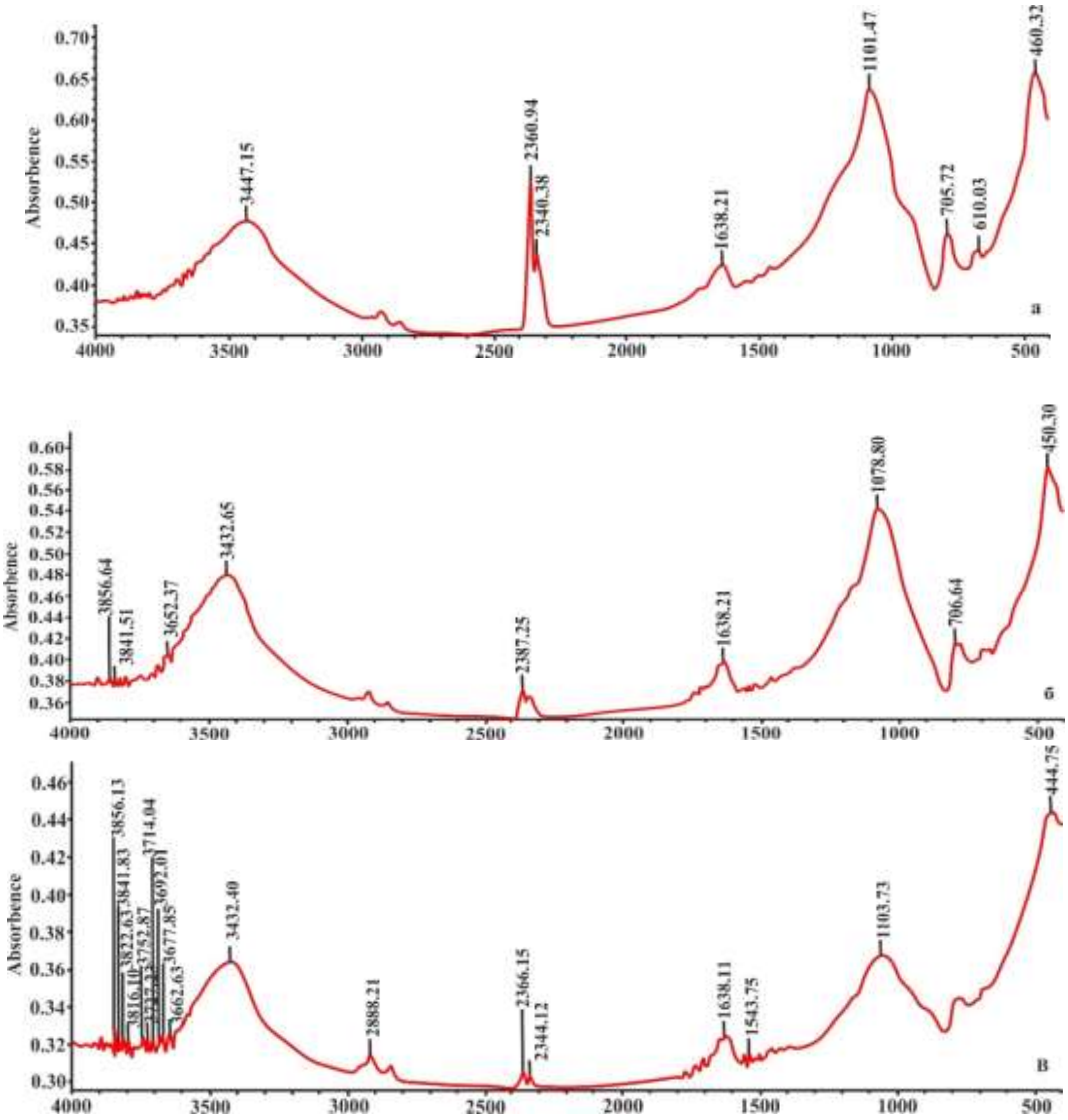
From the data given in Table 1, it can be seen that the largest specific surface area belongs to the initial bentonite powder (sample 1). The increase in the firing temperature led to an increase in the surface roughness level, in addition to an increase in the percentage of macropores with a radius of 10–52 nm. To determine the chemical and mechanical resistance of the samples, we placed granules with a mass of 100 g in glass tubes with a capacity of 250 cm³ and poured 150 cm³ of distilled water into them. The test sorbent tube was filled with water and sealed with foam, and the AVU-6 was shaken continuously at 120 rpm for 24 h. After drying the sorbent was passed through sieves of 0.5-0.25 mm. The granules that pass through the 0.5 mm sieve and remain in the 0.25 mm sieve characterize the fracture. 0.25 sieve friction. The quantities obtained are given in Table 2.

Table 2. Indicators of mechanical strength of bentonite granules.

Examples	friction.,% (norm ≤ 0,5)	fragmentation,% (norm ≤ 0.4)
No. 2. Firing t = 450 °C.	0,45	2,70
No. 3. Firing t = 550 °C.	0,25	2,20
No. 4. Firing t = 650 °C.	0,25	2,00
No. 5. Firing t = 750 °C.	0,10	1,20
No. 6. t firing = 850 °C.	0,0	1,00

It has been shown that an increase in the thermosetting temperature has a positive effect on the mechanical strength of the granules, which is associated with the evaporation of all crystalline water and structural water. However, exposure to higher temperatures (above 700 °C) resulted in a significant decrease in the specific surface area of the granules (Table 1). Accordingly, the sorption capacity is reduced, which indicates that it is not advisable to apply the obtained granules in practice. It is also not advisable to burn at temperatures below 450 °C. Because the crystallization waters come out poorly, the samples will not have sufficient strength. Thus, experimentally, the optimal firing temperature is in the range of 450-550 °C. To explain the specificity of the structural changes that occur during the treatment of Navbahor natural zeolite with boiling acids, their IR spectra were analyzed. The IR spectra of Navbahor natural zeolite treated with boiling acids are shown in Figure 6. Comparison of the IR spectra of natural zeolites with those of acid-treated specimens showed that the aluminium-silicon oxygenated carcass of zeolite had all the characteristic absorption lines in the area of 400–1200 cm^{-1}. As can be seen from Figure 6, the most intensive absorption lines were observed in all cases in the range of 1000–1100 cm^{-1}, which corresponds to the Si-O-Si bond oscillation. The absorption line at 796 cm-1 is related to the valence oscillations of the Al-O bond, and at 460-463 cm^{-1} - by the deformation oscillations of AlO_4. Absorption lines in the range of 3100-3700 cm^{-1} are associated with the presence of zeolite water, and therefore with lines of deformation vibrations of water molecules in 1637-1640 cm^{-1}.

The absence of absorption lines at 960 cm^{-1} indicates high crystallinity and the absence of an amorphous phase mixture in all zeolites. The absence of absorption lines in the area of 3720-3740 cm^{-1} corresponding to amorphous SiO_2 also indicates high crystallinity and phase purity of all samples.



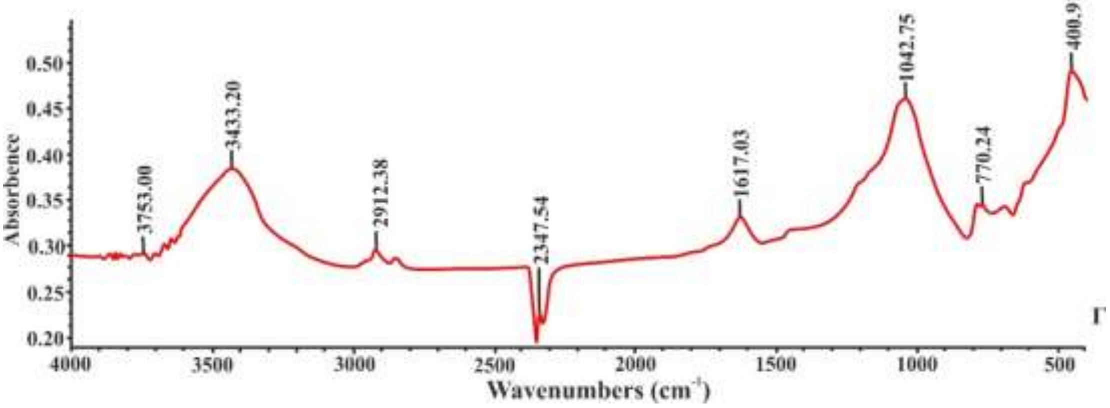


Figure 6. IR spectra of the original natural zeolite sample treated with boiling chloride (a), nitric (б) and sulfuric (в) acids at a concentration of 4 N.

CONCLUSION:

It was noted that mechanical processing of bentonite powder found in Navbahor for 60 min in a planetary mill reduced the average particle size from 27 to 5.5 μm and increased the specific surface area of the powder to 19 ± 0.1 to 33 ± 0.7 m^2/g (BET method).). Subsequent mechanical processing agglomerated the particles to 28 μm and reduced the specific surface area to 20 ± 0.2 m^2/g. Mechanical processing of natural zeolite has led to a decrease in OKR and an increase in microglia of crystal lattice for clinoptilolite and smectite phases. Increasing the mechanical processing time resulted in an increase in the amount of X-ray amorphous phase of zeolite from 13 to 52%. Thus, it is known from the results of our research that despite some changes in chemical composition, the crystal lattice of Navbahor-bentonite natural zeolite remains unchanged, i.e. it is resistant to aggressive environments (up to certain concentrations - 5 N).

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