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Activation of Natural Bentonite and Study of Physico-Chemical and Texture Characteristics

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ABSTRACT: In the study, modification of bentonite of Navbahor fog of Navoi region of the Republic of Uzbekistan was carried out in hydrochloric, sulfuric and nitric acids at their boiling point. Modified samples were analyzed by IR-spectroscopic, X-ray phase, elemental and electron-microscopic analysis methods. A study of the morphology, specific surface area, structure and phase composition of the bentonite powder of the Navbahor deposit, subjected to mechanical treatment in a planetary mill, has been carried out. The following research methods were used: scanning electron microscopy (SEM), X-ray structural analysis, Brunauer-Emmett-Taylor (BET) method, laser diffraction, elemental analysis. It is shown that during the first 60 min of mechanical treatment in a planetary mill, powder particles are ground and the specific surface area increases to 33 m²/g. Further mechanical activation is due to the agglomeration of particles and a decrease in the specific surface area. X-ray structural analysis showed that the studied powder consists of four phases: hexagonal, monoclinic, orthorhombic, and tetragonal modifications. It is shown that prolonged mechanical action on the zeolite leads to an increase in the amount of the amorphous phase in the powder from 13% to 52%. 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 Nicolet IS-10 in the frequency range 400-5000 cm⁻¹. The phase composition was studied using an X-ray analyzer 2DPHASER "Bruker" (CuK α - radiation, 2 θ , 20-80 degrees). Electron microscopic images were taken under a Hitachi TM-3000 microscope. Elemental analysis was performed at Oxford Science. According to X-ray phase data, in the acidic treatment of Navbahor natural zeolite (boiling acids) its crystallinity level decreases (89%) and varies in the range of 70-80%. After acid treatment with each reagent, the IR spectra were calculated as the crystallinity of zeolite-based on the absorption lines, the value of which is well consistent with the data obtained by X-ray phase analysis. The purpose of this work is to study the effect of mechanical and acid treatment on the structure and properties of bentonite, to study their structure and physicochemical properties.

KEYWORDS: bentonite, mechanical treatment, specific surface area, crystal structure, phase composition, acid activation.

I. INTRODUCTION

Natural zeolites are inorganic sorbents. They constitute the largest group of aluminosilicates with framework structures [1]. Natural zeolites with several unique properties, such as the ability to ion exchange, high cation exchange capacity, micro and nanoporous structure, have long been widely used as highly efficient systems for the purification of paraffinic hydrocarbons, separation of mixtures of various gases and liquids, as well as used in the development of sorbents for technical and medical purposes [2-3]. In addition, the diversity of the composition, structural characteristics of natural minerals does not allow fundamental studies of the effect of structural and physicochemical parameters of materials on their sorption, catalytic and other practically significant properties [4]. One of the promising methods for changing the physicochemical properties of a material is mechanical processing, which is accompanied by



changes in morphology, specific surface area, structure and phase composition of materials, which can lead to a change in the operational properties of materials [5, 6].

Since prehistoric times, bentonite clays have been used to purify wine, oils, and water. They are non-toxic and have an extremely developed surface, which leads to a significant adsorption capacity, i.e., the ability to actively absorb various substances from solutions. A limiting factor in the widespread use of natural bentonite sorbents for the purification of drinking water and industrial effluents is the lack of effective granulation technologies since clay minerals are subject to the effect of peptization in aqueous media, i.e., dispersion [7]. As you know, bentonite clays (montmorillonite) are characterized by a layered crystal structure (3-layer) based on an Al-Fe-Mg-octahedral network enclosed between silicon-oxygen tetrahedra (TOT structure) with a silicate layer thickness of 0.94 nm [8-9]. One unit cell of montmorillonite is formed by 20 oxygen atoms and 4 hydroxyl groups, which is 8 silicate tetrahedra and four Al-containing octahedra [8-12]. The interlayer space contains exchangeable cations (Na^+ , K^+ or Ca^{2+}) and water molecules, which prevent the layers from sticking together. In montmorillonite, the charge is concentrated in octahedral layers and is distributed over all oxygen atoms in the structure. Usually, the lack of a positive charge is from 0.4 to 1.2 e per unit cell of Si_8O_{20} [13-14], which, when converted to a cation-exchange capacity, corresponds to 0.5-1.5 mg. eq/g. Typically, the exchange capacity of montmorillonites is about 0.7-1.0 mg. eq/g. The exchange complex of montmorillonite is mainly represented by Na^+ and Ca^{2+} ions located in the space between the silicate layers. Another source of exchange centres is weakly acidic hydroxyl groups (Si-OH) and basic (Al-OH) groups on the side faces and edges, which, depending on pH, participate in ion exchange. For example, the presence of OH^- groups determines the ability of layered minerals to anion exchange [15-18]. Along with ion exchange, bentonite clays are characterized by physical and molecular sorption. Physical sorption is due to the presence of an excess negative charge on the crystal faces and surface hydroxyl groups of an acidic and basic nature, capable of ionization. During molecular sorption, the adsorbed 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 centres as exchangeable cations, hydroxyl groups, as well as the activation of bentonite clays can significantly expand the practical application of layered silicates for the purification of aqueous media by modifying their surface [18-21].

According to the obtained X-ray phase analysis data, the studied samples of granules annealed at different temperatures have a similar mineralogical composition. Heat treatment of the samples practically does not affect the mineralogical composition and structure of the granular material, and with an increase in the firing temperature, there is a slight increase in the intensity of the peaks, which indicates crystallization and dehydration of the phases.

II. EXPERIMENTAL PART

The bentonite of the Navbakhor deposit, the Republic of Uzbekistan was investigated. Mechanical activation of zeolites was carried out in a Retsch PM 400 planetary ball mill at a speed of 350 rpm. The acting load in the planetary mill was 26.8 g; ceramic balls with a diameter of 10, 15, 20, 30 mm and a mass of 430 g were used as grinding bodies. The processing time varied from 1 to 600 min [22].

The morphology of the powders was studied by scanning electron microscopy (SEM) on a Zeiss Supra 55VP scanning electron microscope [23].

Particle size analysis and measurement were performed using an LA-950 HORIBA laser diffraction analyzer. The specific surface area was determined by the Brunauer-Emmett-Taylor (BET) method on a TriStar 3000 instrument [24]. The error in measuring the specific surface area was no more than 3%. The study of the structure of the zeolite was carried out on a Rigaku Mini Flex II diffractometer in $\text{CuK}\alpha$ radiation in the range of 2θ angles from 3 to 90° with a step of 0.4° and an exposure time of 1 s. The analysis of the phase composition of zeolites under heating conditions from 20 to 1050°C was carried out on a D8-Advance Bruker diffractometer in $\text{CuK}\alpha$ radiation in the 2θ angle range from 5 to 85° . The X-ray diffraction patterns were decoded using the ASTM file, PDF-2 [25].

Based on the results of X-ray diffraction analysis, the sizes of coherent scattering regions (CSR) and micro-distortions of the crystal lattice were determined by the Hall-Williamson and Selyakov-Scherrer methods [26].

Modification of bentonite of Navbahor district of Navoi region of the Republic of Uzbekistan [27-32] 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 a neutral reaction, dried at room temperature, and analyzed 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 NicoletetS-10 in the frequency

range 400-5000 cm⁻¹. The phase composition was studied using an X-ray analyzer 2DPHASER "Bruker" (CuKα - radiation, 2nd, 20-80 degrees). Electron microscopic images were taken under a Hitachi TM-3000 microscope. Elemental analysis was performed at Oxford Science. The modulus of silicate of zeolite, i.e. the mole ratio of SiO₂/Al₂O₃, 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_{SiO₂}- silicon oxide mass fraction,%;

C_{Al₂O₃}- mass fraction of aluminum oxide,%;

M_{Al₂O₃}, M_{SiO₂} are molecular masses of aluminium and silicon oxides, respectively.

The degree of crystallinity was determined from the absorption lines of the IR spectra. The specific surface area and porosity of the sorption materials were determined by the method of low-temperature adsorption of nitrogen on a SORBI-MS device (ZAO META, Novosibirsk) by the BET method; the chemical composition of the surface of the sorbents was determined by a scanning electron microscope LEO1450 (Germany) with an energy-dispersive X-ray microanalyzer with a Si- (Li) semiconductor, an EDXINCA Energy detector from Oxford Instruments; the phase composition was determined on an EmpyreanSeries2 X-ray diffractometer (Panalytical, Holland); thermal studies were carried out on a NETZSCHSTA409 PC/PG thermal analyzer (Germany).

Sorption studies were carried out using a thermostatic system and a pH meter of the EXPERT-001 series. The concentration of elements in the solution was determined by atomic absorption spectrometry (AAS) on a contraAA700 instrument (Analytik Jena, Germany) using the appropriate State standard samples.

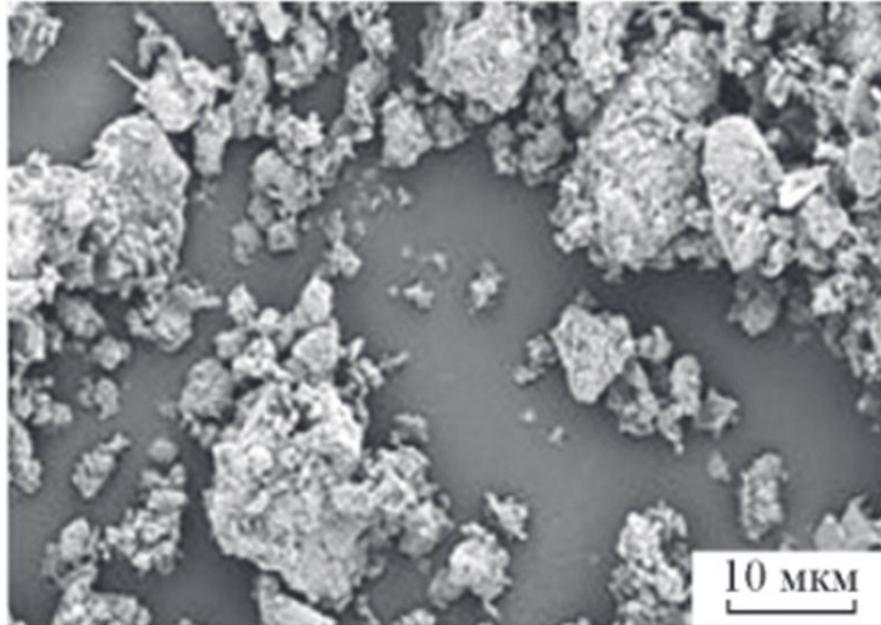
III. RESULTS AND DISCUSSION

In fig. 1 shows the elemental analysis, SEM image and diffractogram of Navbakhor bentonite. The results of the elemental analysis showed that the zeolite powder in the initial state consists mainly of Al, O, Si, Ca, Mg, Fe. The powder is represented by particles of irregular shape, tightly adjacent to each other, of different sizes, with an average size of 27 microns. The particle size distribution is unimodal. Identification of X-ray diffraction patterns of Navbakhor bentonite showed that the initial powder contains seven phases with different mineral content: smectite (smectite 15A), quartz (quartz), cristobalite-low, clinoptilolite (clinoptilolite), illite (illite 2M1), orthoclase (orthoclase), calcite (calcite) with a value of 20, 8, 15, 14, 15, 9, 6%, respectively. In this case, the content of the amorphous phase in the original zeolite was 13%. With an increase in the time of mechanical treatment, there is a change in the average particle size of the Navbakhor bentonite powder (Fig. 2). The initial average particle size of the zeolite particles was 27 μm, after treatment for 20 minutes, 5.5 μm, and after 600 minutes of treatment, 28 μm. Moreover, most of the particles lost their original shape during mechanical activation and began to have a spherical shape (Table 1). Table 1, it is shown that during the first 60 minutes of mechanical treatment in a planetary mill, grinding occurs, and, consequently, a decrease in the average particle size, while the particles lose their original shape and are converted into particles with a shape close to spherical.

Table 1. Specific surface area and average particle size of zeolites after mechanical activation in planetary mills

A. Machining time, min	B. Specific surface, m ² /g	C. Particle size calculated from specific surface area, nm	D. Particle size, dispersion using a laser analyser, μm
E. 0	F. 19±0.1	G. 137	H. 27
I. 10	J. 23±0.4	K. 108	L. 10
M. 60	N. 33±0.7	O. 75	P. 21
Q. 180	R. 25±0.4	S. 100	T. 27
U. 600	V. 20±0.2	W. 125	X. 28

Further mechanical activation is due to agglomeration, when each micro-particle consists of many nanoparticles, the structure of which provides a large specific surface area [12].



<i>Ement</i>	<i>Mass. %</i>	<i>At. %</i>
OK	48.5	65.5
MgK	0.3	0.2
AlK	3.7	2.9
SiK	46.3	33.9
CaK	0.5	0.3
FeK	0.6	0.2

a)

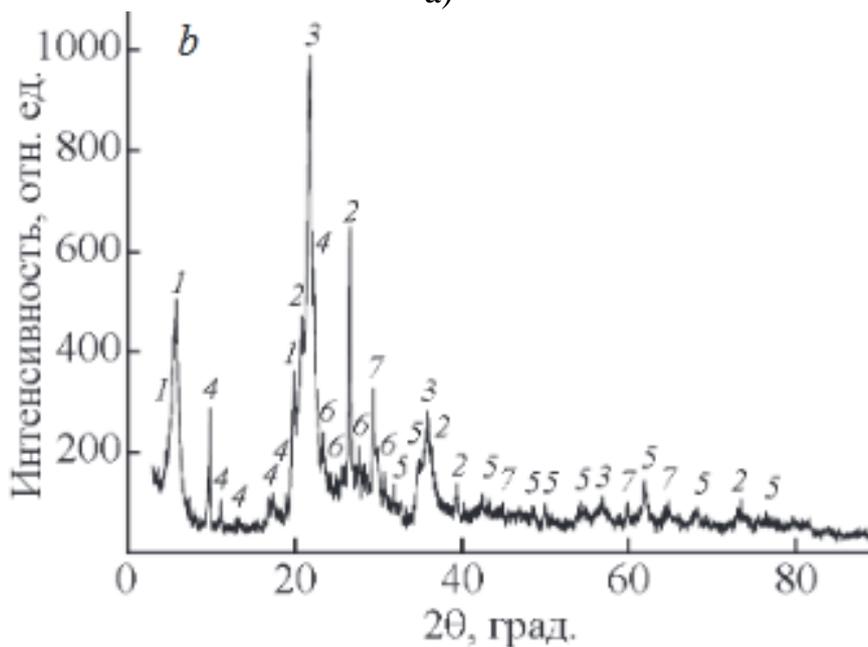


Fig. 1. Initial zeolite: a-SEM image and elemental analysis, b-diffractogram: 1-smectite, 2-quartz, 3-cristobalite, 4-clinoptilolite, 5-illite, 6-orthoclase, 7-calcite.

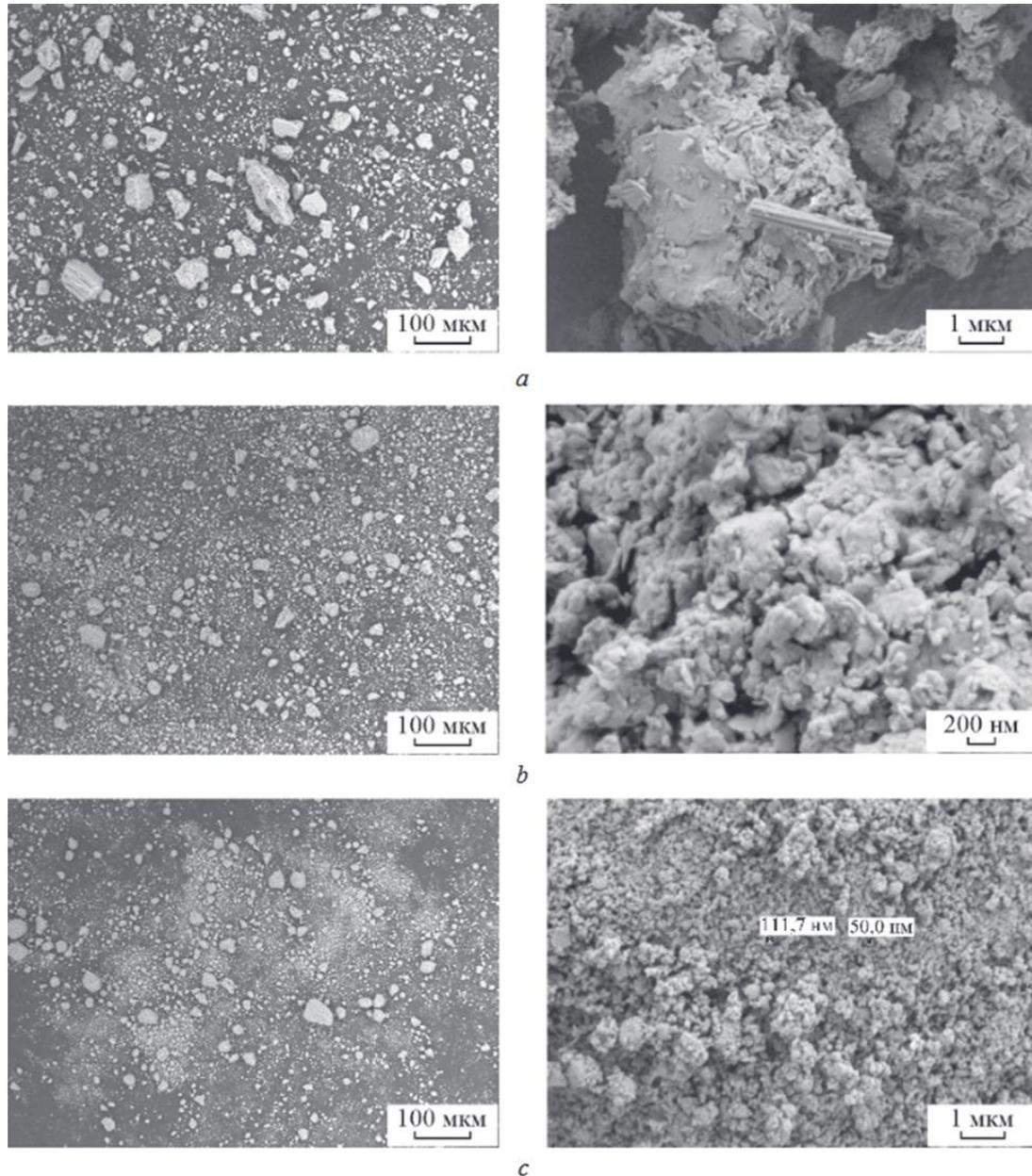


Fig. 2. SEM images: a) - the original zeolite, b) - after mechanical treatment 20 min, c) - 60 min.

Quantitative phase analysis of Navbakhor bentonite during mechanical processing up to 600 min showed radical changes in the mineralogical composition. The content of minerals smectite, clinoptilolite, illite, calcite, cristobalite reached a minimum value and amounted to 0.2, 1, 2.5, 1, 14%, respectively, and the content of quartz increased to 17%, orthoclase - up to 12%. It is shown that prolonged mechanical action on the zeolite leads to an increase in the amount of the amorphous phase in the powder from 13 to 52%. The specific surface area of the investigated powder was determined using the CSR dimensions. In the initial state, the specific surface of the powder was 32 m²/g. Within 60 minutes, the specific surface area increases, reaching a maximum of 36 m²/g. After 600 min, the specific surface area of Navbakhor bentonite was 20 m²/g.

Table 2 shows the results of determining the specific surface area by BET and the specific surface area calculated from the CSR. Thus, the changes in the specific surface area according to the two methods are of the same nature, and the phase composition determines the specific surface area of Navbakhor bentonite. The results of determining the porosity

of bentonite clay and sorbents based on it are presented in table 1. From the data in the table, it can be seen that the initial bentonite powder (sample No. 1) had the highest specific surface area. An increase in the firing temperature leads to a significant decrease in the surface area, as well as an increase in the percentage of macropores with a radius of 10-52 nm.

Table 2. The porous structure of bentonite clay samples after high-temperature firing

Sample	Specific surface area <i>S</i> , m ² /g	Total pore volume <i>V</i> pore, cm ³ /g	Pore distribution by radius, %			
			1,5-2,0 nm	2,0-4,0 nm	4,0-8,0 nm	10,0-52,0 nm
No. 1 Source 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

To determine the chemical and mechanical resistance, samples of granules weighing 100 g were placed in glass flasks with a capacity of 250 cm³ and 150 cm³ of distilled water were poured into them. The flasks with the studied sorbent, filled with water and closed with stoppers were shaken continuously for 24 hours on an AVU-6 device at a speed of 120 shakes per minute. After drying, the sorbent was dispersed on sieves of 0.5 and 0.25 mm. The granules that passed the 0.5 mm sieve and the granules remaining on the 0.25 mm sieve characterize grindability, and those that passed the 0.25 sieve abrasion. The data obtained are shown in the table. 2. It has been established that an increase in the temperature of heat treatment has a positive effect on the mechanical strength of granules, which is associated with the evaporation of all crystallization water and structural water.

However, under the influence of a higher temperature (over 700 °C) in the granules, a significant decrease in the values of the specific surface area is observed (Table 3), thereby reducing the sorption capacity, which makes the practical use of the obtained granules less expedient. It is also not advisable to carry out firing at a temperature of less than 450 °C, since the samples do not have sufficient strength due to the incomplete yield of crystallization water. Thus, the experimentally established optimal firing temperature is in the range of 450–550 °C.

Table 3. Indicators of mechanical strength of bentonite granules

Sample	Abrasion, % (norm ≤ 0.5)	Grindability, % (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 firing t = 850 °C	0,0	1,00

For an objective analysis of the distribution of clay particles by size and fixing changes in the structure of clay, photographs were selected (Fig. 2-3) taken at such magnification of the microscope, which made it possible to easily determine the minimum and maximum particle sizes using the scale bar, as well as consider changes in appearance particles of the test sample. The thermogram of this bentonite is shown in Fig. 3.

The first endothermic effect is observed at a temperature of 373–453K, associated with the release of free and inter-packet water. The second endothermic effect is less pronounced than the first and is observed in the temperature range 933–1053K and is caused by the loss of constitutional water, as well as the dissociation of magnesium and calcium carbonates, which are present in this bentonite as a modifier.

The decarbonization of carbonates is a topochemical process. In general, the endothermic decarbonization reaction is described by the equation: A_m → B_m + C_r, where A_m – CaCO₃, MgCO₃; B_m – CaO, MgO и C_r – CO₂.

A special feature 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 that of the original phase.

As a result, the volume of the new solid phase formed is much less than the initial one. So, instead of 1 cm³ CaCO₃ (MgCO₃) 0.455 cm³ CaO and 0.397 cm³ MgO are formed. The temperature of decomposition of calcium and magnesium carbonates is influenced by the size of the crystals of the original bentonite

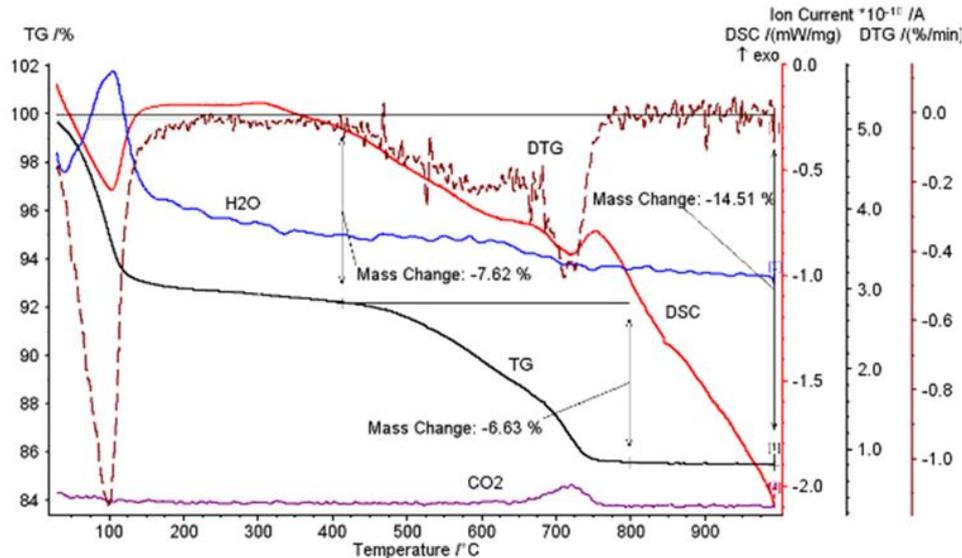


Fig. 3. Thermogram of Navbakhor bentonite

. A decrease in the decomposition temperatures with an increase in the degree of dispersion is associated with the well-known phenomenon of vapour pressure with a decrease in the particle radius. On the thermogram, the largest amount of released CO₂ is observed at a temperature of 993–1013 K, and a further increase in temperature does not lead to a significant change in mass. In general, the mass of the sample in the considered temperature range decreased by 14.51%, where 7.62–8% is water and released CO₂. A further increase in temperature leads to the destruction of bentonite. The specific surface area of bentonite is very high and is equal to 65.30 m²/g. Micrographs of a bentonite particle are shown in Fig. 4.

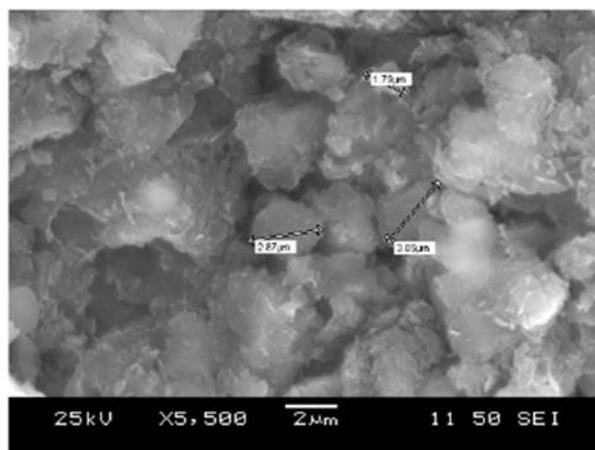


Fig.4. Dimensions of Navbahor bentonite particles

Microchemical analysis of separate plots with high frequency (Fig. 5) is presented in Table 1. Thermogram Navbahorskogo bentonite after mechanical processing is presented on ris. 4. The first bright endothermic effect occurs in the range of 343–423K, where the release of free and chasting mezhpaketnoy water and the reduction of the mass of the image is 4.86%. The second endothermic effect occurs at a temperature of 733–773K and is associated with the separation of constitutional moisture and the release of SO₂, associated with the decomposition of organic compound bentonite.

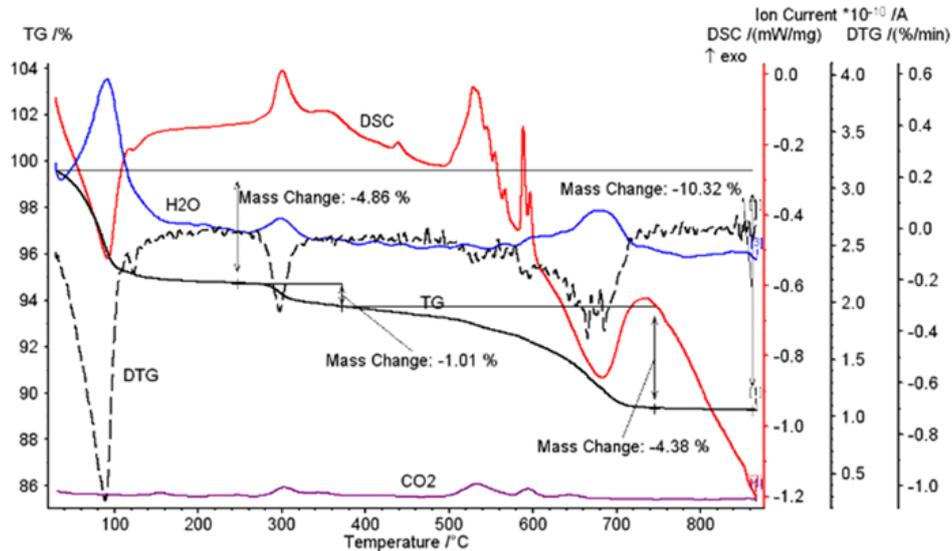


Figure 5. Thermogram of Navbahor bentonite after mechanical treatment

The third endothermic effect on the heating curve after mechanical treatment of Navbakhor bentonite is observed in the temperature range 853–873K and is caused by the destruction of the crystal lattice of the mineral, decomposition of carbonates that 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 studied interval, the weight reduction is 10.3%, which is significantly less than that of Greek bentonite (14.51%).

During heating, two exothermic effects are observed. The first, in the range 553–953K, is associated with the decomposition of the organic component of bentonite. The second exo-effect is at a temperature of 853–873K, which is associated with the decomposition of montmorillonite. The fourth exothermic effect is observed in the temperature range 1003–1053K, where the components are sintered with the formation of mullite. In the process of heating, CO₂ is released, especially at a temperature of 833–893K, which is caused by the decomposition of calcium and magnesium carbonates, which are part of bentonite. In fig. 5 shows the differential curve of the size distribution of mesopores of natural bentonite. It can be seen from the figure that there is a rather narrow pore size distribution.

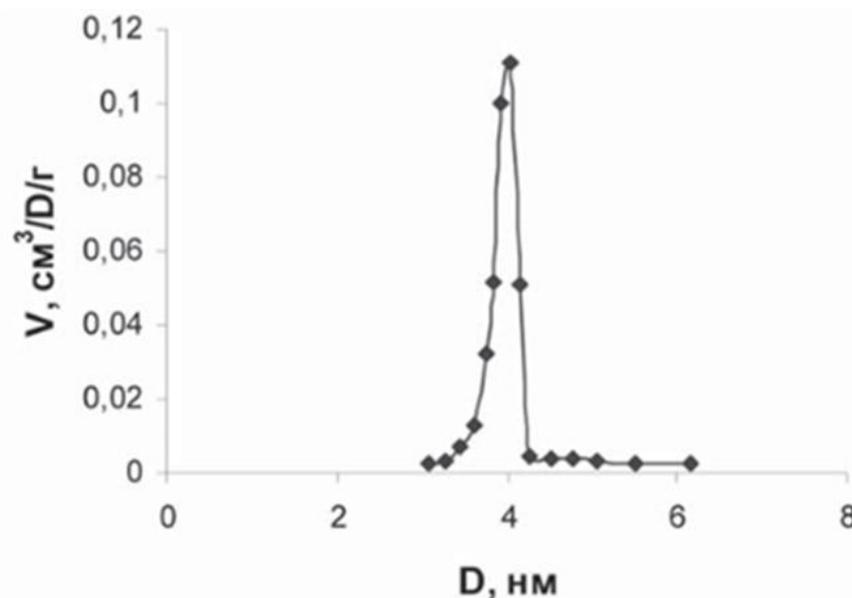
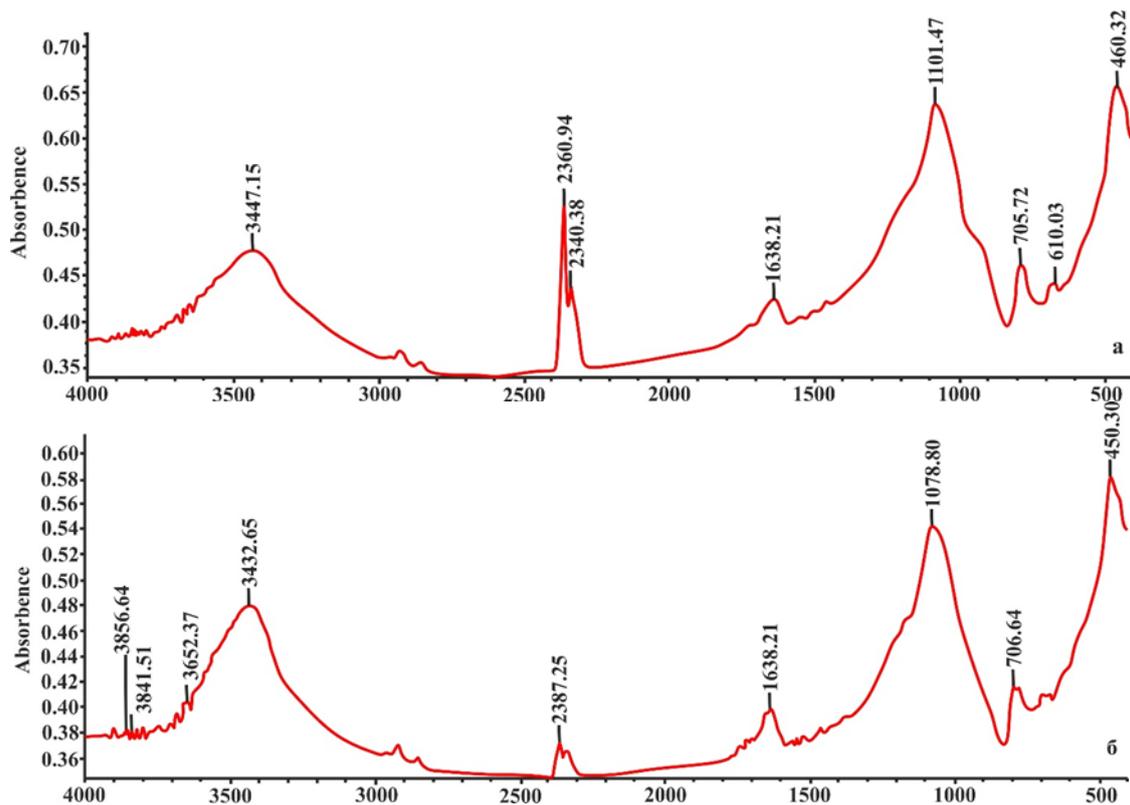


Fig. 6. The differential size distribution curve of natural bentonite mesopores

IV. EXPERIMENTAL RESULTS

The processing efficiency of the zeolites studied increases with increasing acid concentration. In acid decantation, the value of the silicate modulus Si/Al in zeolite increases with increasing acid concentration from 1 to 5 N. their IR spectra were analyzed to explain the specificity of the structural changes that occur when Navbahor natural zeolites are treated with boiling acids. 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 oxygen 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.



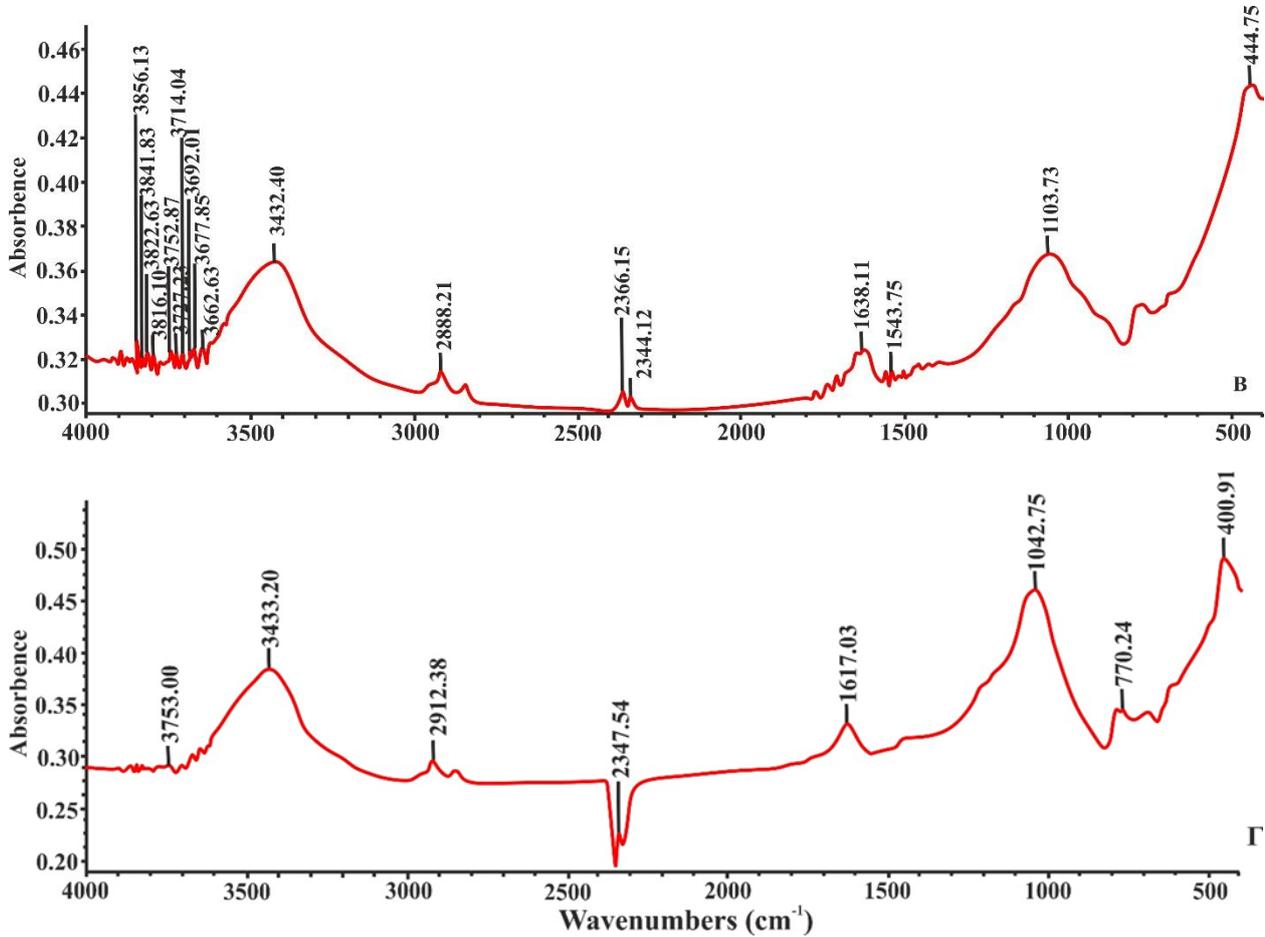


Fig. 7. IR spectra of the original natural zeolite sample treated with boiling chloride (a), nitric (b) and sulfuric (c) acids at a concentration of 4 N

In the field of vibrational frequencies of the aluminosilicate carcass, the IR spectra of the original zeolite and its samples treated with boiling acids were compared.

As the acid concentration increases, the lines weaken in the range of 550–660 and 796 cm^{-1} , which is typical for the oscillations of the outer bonds of tetrahedrons.

These lines, as noted above, are inextricably linked with the tetrahedrons of the zeolite crystal lattice.

At the same time, the intensity of the lines at 460 and 1062 cm^{-1} , which is typical for the internal tetrahedral oscillations of the bonds, is almost unchanged during acid treatment.

The complete breakdown of the Navbahor natural zeolite crystal lattice occurs at acids only at 5 N, as evidenced by the loss of lines in the range 460–463, 550–660, 796, 1078–1081 cm^{-1} .

X-ray phase analysis confirms the data obtained by IR spectroscopic method.

Figure 7 shows zeolite diffractograms treated with Navbahor early natural zeolite and boiling zeolites.

X-ray phase analysis data showed that despite some changes in chemical composition, the crystal lattice of zeolite remains unchanged after acid treatment.

Diffractograms of zeolite treated with natural and boiling acids are similar in appearance to bentonite diffractograms in the initial state.

In Fig. 7, when comparing the spectra with the initial natural bentonite diffractogram, neither the shifts of the interference maxima nor the distortions of their shapes were detected.

The differences noted in the diffractograms are only related to changes in the intensity of some basic (main) return lines. In all cases (at different concentrations of acids) at $d = 13.56\text{\AA}$ the intensity of the strongest base return of bentonite remained unchanged.

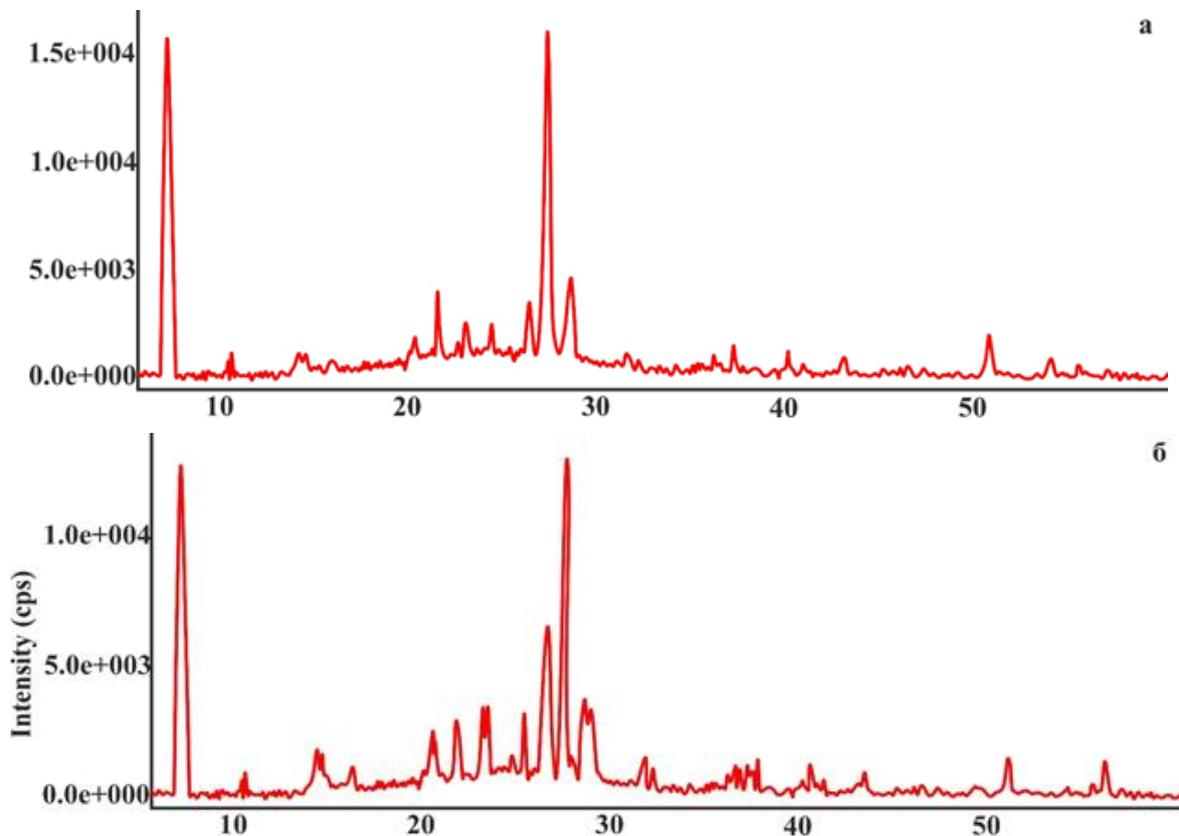
X-ray phase analysis of samples of pre-acid and post-acid zeolite (at acid concentrations up to 5 N) does not lead to a change in the scattering angles, but only to the intensity of the characteristic lines, where changes in the intensity of different characteristic lines do not occur uniformly. This suggests that the bentonite grid will be more defective, along with the possible partial disruption of the crystal structure. The detected changes in the line intensities in the diffractograms of the acid-treated bentonite are, in our view, the result of melting. The application of X-ray phase analysis also made it possible to determine whether an increase in the thermal stability limit of Navbahor bentonite occurs as a result of acid treatment. However, slight changes in the crystal lattice structure can be seen in the IR spectrum of a sample treated with 1 N acid, although the radiograph does not detect this. This indicates that the IR spectroscopy method is more sensitive to structural changes of zeolite compared to radiography.

The subsequent increase in acid concentration leads to a significant deterioration of the crystal lattice and the formation of aluminosilicate - anorthite, which is reflected in the diffractogram, ie the processing of natural zeolite with 5 N boiling acid leads to a violation of the IR spectrum and mineral structure according to diffractogram (respectively 6- Figure 7).

In the IR spectra of zeolites treated with 5 N acids, streaks are lost in the 400–500 cm^{-1} region, which is probably due to the rupture of Si (Al) -O bonds and the partial loss of aluminium from alumina-silica tetrahedra.

This is also evidenced by the radiograph of the sample under consideration, which does not have the sharp lines typical of early zeolite. As can be seen in Figure 6, the zeolite structure of the natural specimen is preserved, indicating that the zeolite structure is not degraded after Navbahor bentonite has been treated with boiling acids up to a certain concentration (5 N). X-ray phase data showed that in the acid treatment of Navbahor natural zeolite (boiling acids) its crystallinity level decreases (89%) and varies in the range of 70-80%.

After acid treatment with each reagent, the IR spectra were calculated as the crystallinity of zeolite-based on the absorption lines, the value of which is well consistent with the data obtained by X-ray phase analysis.



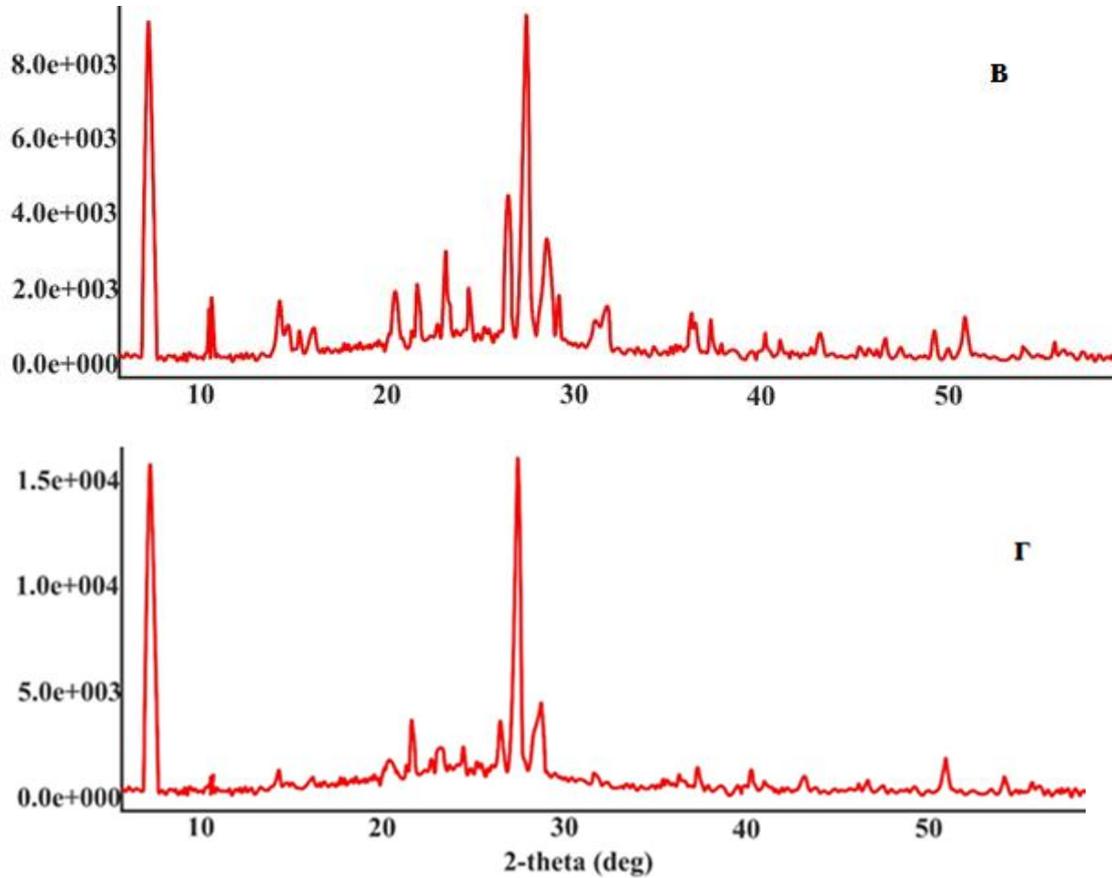


Fig. 8. Diffractograms of the original natural zeolite sample treated with boiling chloride (a), nitric (b) and sulfuric (c) acids at a concentration of 4 N.

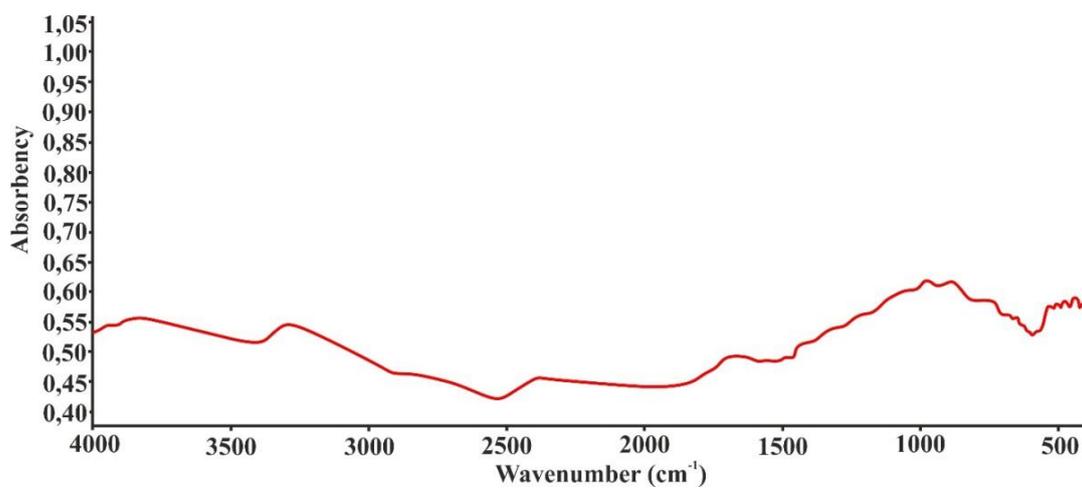


Fig. 9. IR spectrum of natural zeolite treated with boiling hydrochloric acid at a concentration of 5 N

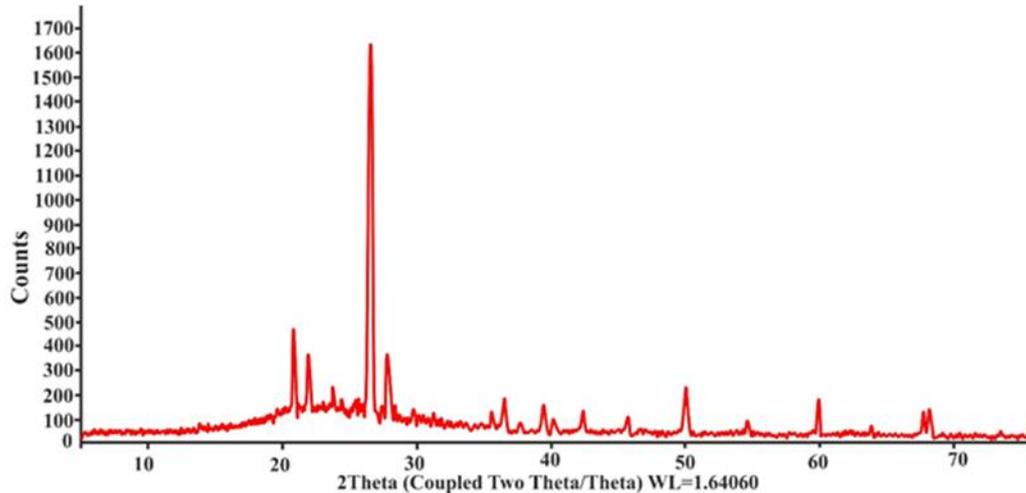


Fig. 10. Diffractogram of natural zeolite treated with boiling hydrochloric acid at a concentration of 5 N.

Figure 10 shows samples treated with boiling hydrochloric (a), nitric (b), and sulfuric (c) acids, and micrographs of the original natural zeolite sample.

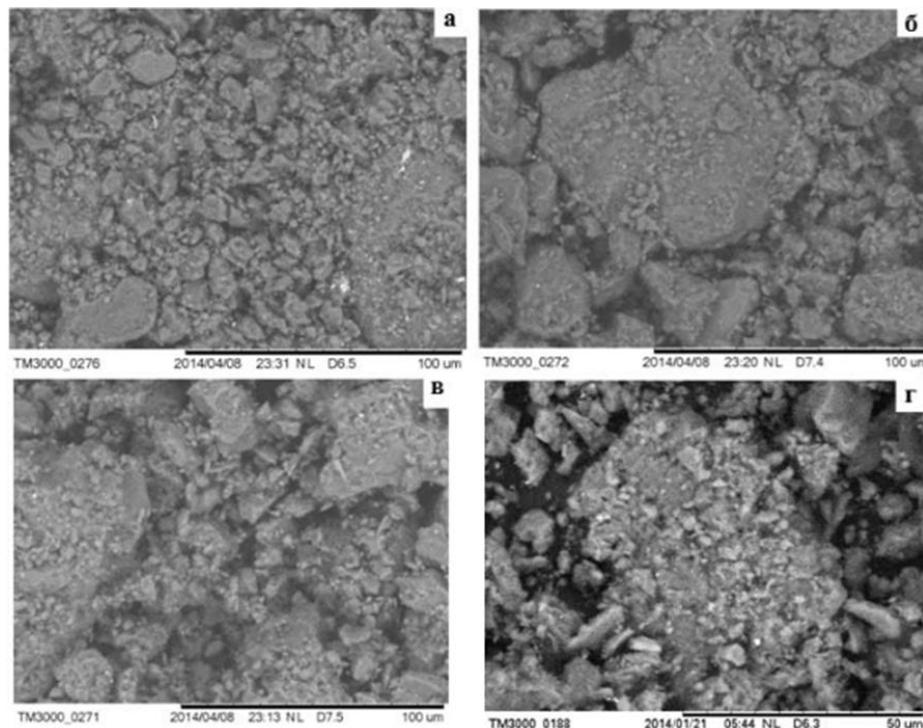


Fig. 11. Samples treated with boiling chloride (a), nitric (b) and sulfuric (c) acids and micrographs of the original natural zeolite sample

V.CONCLUSION

It was found that mechanical treatment of the bentonite powder of the Navbakhor deposit in a planetary mill during the first 60 min leads to a decrease in the average particle size from 27 to 5.5 μm and an increase in the specific surface area of the powder (according to the BET method) from 19 ± 0.1 to 33 ± 0.7 m^2/g . The further mechanical treatment causes agglomeration of particles to 28 μm and a decrease in the specific surface area to 20 ± 0.2 m^2/g . Mechanical



treatment of natural zeolite leads to a decrease in CSR and an increase in micro distortions of the crystal lattice for the phases of clinoptilolite and smectite.

With an increase in the time of mechanical treatment, the amount of X-ray amorphous phase of zeolites increases from 13 to 52%. Thus, it is known from the results of our research that despite some changes in the 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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