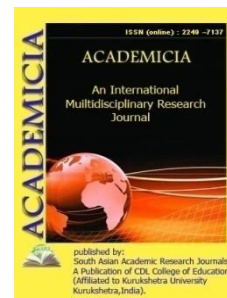




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## OPTIMIZATION OF THE ACID ACTIVATION PROCESS OF BENTONITE

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### ABSTRACT

*The fields of application of Bentonite clays are expanding due to imparting new properties to them as a result of various types of activation. One of the most effective treatments is acid treatment. Acid-activated montmorillonites are used as acid catalysts for various reactions. Acid activation with HCl solution of calcium and sodium montmorillonites of the Navbakhor deposit was carried out. It was shown that, as a result of acid treatment, the development of a porous structure occurs due to the removal of both interlayer cations ( $\text{Na}^+$ ,  $\text{Ca}^{2+}$ ) and cations of the octahedral layer ( $\text{Al}^{3+}$ ,  $\text{Fe}^{2+}$ ,  $\text{Mg}^{3+}$ ). The textural characteristics and strength properties of moulded composites based on acid-activated montmorillonites have been investigated. The purpose of this study is to identify the optimal conditions for acid activation and to study the textural characteristics of bentonite from the Navbakhor deposit.*

**KEYWORDS:** *Bentonite, Activation, Processing, Modification, Optimization, Texture Characteristics.*

### INTRODUCTION

Bentonite clays are good inexpensive sorbents for various substances, such as heavy metal ions [1], organic dyes [2]. Sorption occurs due to the presence of layered silicates (phyllosilicates,

smectites) in the composition of clays, such as 2 montmorillonite, palygorskite, illite. Acid activation of bentonite clay is a widespread method of obtaining porous sorbents for organic and inorganic substances [5, 6], acid catalysts [4, 7].

In addition, acid activation with preservation of the layered structure is necessary as an initial stage in the chemical modification of phyllosilicates [8]. It should be noted that activation must be cost-effective. Preference is given to, if possible, dilute solutions of acids, relatively low temperatures and activation times. It should also be taken into account that washing clay from excess acid is a rather laborious process, therefore, in most cases, it is advisable to calculate in advance the optimal acid concentration, taking into account the peculiarities of the chemical composition of natural clay, the presence of impurities. The study of the physicochemical parameters of clays of various deposits, activated by various acids, is devoted to a sufficient number of works [9–14].

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 bentonite clays will expand by imparting new properties to them as a result of various types of activation [7–11]. 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].

The first group consists of organic and dilute mineral acids, which extract only exchangeable cations from the lattice of montmorillonite into solution and do not affect aluminosilicate layers and the order of their packing. At the same time, the porous structure remains practically unchanged: the specific surface is 60–70 m<sup>2</sup>/g, the limiting specific volume of the sorption space is about 0.13 cm<sup>3</sup>/g. The second group - solutions of mineral acids of medium concentrations (2–4 N). Under the influence of these acids, exchangeable (Na<sup>+</sup>, Ca<sup>2+</sup>) and octahedral cations (Al<sup>3+</sup>, Fe<sup>3+</sup>, Mg<sup>2+</sup>) are extracted into the solution, once the aluminosilicate packets are ordered in the basal direction, but the structure of the aluminosilicate layers is not disturbed.

As a result, the pore space of montmorillonites develops significantly, S<sub>sp</sub> increases to 330 m<sup>2</sup>/g, V<sub>pore</sub> to 0.5 cm<sup>3</sup>/g. Concentrated mineral acids (more than 4 N) belong to the third group, which destroy the crystal structure of montmorillonite, wash out all cations, except for silicon, resulting in the formation of highly porous silicon oxide [14].

Acid-activated montmorillonites are widely used as acid catalysts for such reactions as cracking, isomerization, alkylation, acetylation, dimerization and polymerization of unsaturated hydrocarbons, hydrogenation and dehydrogenation of hydrocarbons, dehydration of alcohols, hydration of olefins, hydrotreating, formation of esters, etc. [7, 15–19].

In addition, recently all over the world, there has been an increase in interest in the use of natural minerals and composites based on them as carriers for catalysts and desiccants, which can compete with traditional SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, zeolites both at the expense of a lower price and due to higher operational characteristics of the material.

The proposed classifications of porous bodies are based mainly on experimental data on the size of pores or their fractional composition. They do not reflect the origin and structure of porous bodies [20-21]. M.M. Dubinin is known to divide the pores as follows (by the effective radius):

- - macropores with  $r_{eff} > 100$  nm;
- - mesopores with  $1.5 < r_{eff} < 100$  nm;
- micropores with  $r_{eff} < 1.5$  nm.

Of particular interest is the characteristic of the microporous structure of adsorbents in the case of an activated nature of adsorption [22-25]. It should be recalled that according to the classification of M.M. Dubinin, the effective radius of micropores is in the range of  $r \leq 1.6 \dots 1.8$  nm. In a number of works, formations with  $r < 0.6 \dots 0.7$  nm are attributed to micropores [25-26]. Larger micropores, the so-called. super micropores are represented by the transition region between the micro-and mesopores proper [24].

### EXPERIMENTAL PART

The enriched fraction of Navbakhor bentonite was used for the work, which was obtained as follows: a suspension of 30 g of native clay in 300 ml of distilled water was prepared with thorough stirring; the particles of all the minerals that make up the clay were divided into fractions. The resulting suspension was left for a day. Then, the enriched clay fraction was separated by centrifugation at 8000 rpm for 5 min. The resulting fraction was dried in air for 12 hours, then at a temperature of 65 °C for 12 hours. Activation was carried out with hydrochloric acid of concentration 0.5 M and 2.0 M at a temperature of 88 °C for two hours. After activation, the clay suspension was not slowly cooled by pouring it into 200 ml of distilled water. Then the clay was washed several times with distilled water, centrifuged and dried for 12 hours at room temperature, then for 12 hours at 65 °C, ground in a mortar and stored in polyethylene containers. Mechanical activation of bentonites in the process of grinding makes it possible to achieve an increase in the total activity of particles by reducing their size, increasing the total surface, achieving a more compact form of particles, and profound transformations in the crystal structure of the material. To study the activation parameters of clays, an AGO-2 planetary-centrifugal mill-activator with varying activation time from 10 to 180 seconds was used in order to achieve the maximum degree of grinding of bentonite clays with their maximum activity while maintaining the characteristics of the crystal-chemical structure and the absence of aggregation.

Mechanical activation (MA) in the AGO-2 planetary-centrifugal mill was carried out at a ratio of the mass of clay and grinding bodies 1: 2 (for bentonite clays –30–180 seconds), at a constant speed  $v_{ob} = 1500$  rpm. During the activation of clays, their quality was assessed by geometric parameters and technological properties. The technological properties of the clays were evaluated according to various indicators according to GOST. The physicochemical properties of clays as binders are largely determined by the peculiarities of their interaction with water; therefore, the influence of the activation time on the swelling, colloidal and water absorption of clays was investigated. The textural characteristics of the synthesized samples were calculated on the basis of nitrogen adsorption and desorption isotherms at 77 K, obtained on a Nova 1200 e Quantachrome volumetric apparatus. The specific surface area ( $S_{sp}$ ) of the samples was calculated by the BET method, the volume and surface of micropores ( $V_{mi}$ ,  $S_{mi}$ ) - by the t-method according to the adsorption curve, the average mesopore diameter ( $D_{me}$ ) - by the

Barrett-Joyner- Halenda method according to the desorption curve, the average micropore diameter was determined by the equation:  $D_{mi} = 2V_{mi} / S_{ud}$ . Before measuring the adsorption isotherms, the samples were degassed at 300 °C and a residual pressure of 10<sup>-3</sup> mm Hg. Art. within 4 hours. The content of zirconium and zinc in the modified bentonite was determined by laser mass spectrometry using an EMAL-2 device. The chemical composition of the samples was determined by the X-ray fluorescence method using VRA-30 (Carl Zeiss, Germany) and Optima X (MRU, Germany) instruments. To determine the average particle size, sieve analysis was performed using a set of sieves (µm): 0.071, 0.100, 0.160, 0.200, 0.250, 0.315. Fractionation was carried out on an Analysette 3 Spartan Fritsch vibrating scatter (Germany) for 15 min. The masses of the fractions retained on the sieves were determined on a technical balance Gosmetr VLTE- 1100 (Russia). The average grain size on the sieve and the average particle size= in the crushed material were calculated according to the method described in [22]. X-ray diffraction patterns of substances were taken on an X-ray diffractometer Bruker D8 ADVANCE (Germany) in CuK α -radiation; The 6 obtained X-ray diffraction patterns were identified using the VA program with a PDF-2 powder data bank. To quantitatively determine the elemental composition of the samples, we used the energy dispersive X-ray fluorescence method using a Shimadzu EDX 800 HS spectrometer (Japan). The analysis was carried out without taking into account light elements. The concentration of the determined elements was calculated by the method of fundamental parameters using the software of the spectrometer. The relative determination error did not exceed ± 2%. IR absorption spectra of phosphorus-containing samples were recorded in the range 400–4000 cm<sup>-1</sup> in vaseline oil using a Shimadzu FTIR Prestige-21 Fourier spectrometer (Japan) at room temperature. The specific surface area of the samples was determined by the method of low-temperature nitrogen adsorption using a Sorbtometer-M device (Russia).

## RESULTS AND DISCUSSION

To determine the optimal conditions for acid treatment, the method of planning extreme experiments was used [11]. As a design, a full factorial experiment was chosen, in which all possible combinations of factors are implemented at all levels selected for the study. The following factors were chosen: C – acid concentration, T - activation temperature and t - time of the clay activation process. As a response function, we used the specific surface area and the average pore radius of the clay. If the specific surface area is used as a response function, then the following regression equation can be obtained:

Equation (1) satisfies the Fisher criterion and therefore is adequate, at least in the range of variation of the parameters. Table 2 shows the values of the factors in a full factorial experiment for clays I-IV of domestic deposits. The results on the reproducibility of some characteristics of the porosity of clay I of the Navbakhor deposit treated at CHCl = 10 N, T = 80 °C, t = 2 h (plan centre conditions) are given in Table 3. In the figure, and for clay I, the response surface is shown for two values of the time t (1 h - the lower surface, 3 hours – 7 the upper surface). The figure shows that the response surface is complex. Thus, at low acid concentrations, the specific surface area increases with decreasing process temperature, while at high acid concentrations, on the contrary, surface growth is observed with increasing temperature. As for the time of acid treatment, an increase in its duration significantly affects the size of the surface.

**TABLE1. VALUES OF FACTORS IN A FULL FACTORIAL EXPERIMENT FOR CLAYS I-IV OF VARIOUS DEPOSITS**

Experiment number	Values of factors for different clay samples								
	Concentration HCl,N			T,°C			t,ч		
	IиII	III	IV	IиII	III	IV	IиII	III	IV
1	6	3	4	70	40	90	1	2	6
2	10	7	7	70	40	90	1	2	6
3	6	3	3*	90	80	90	1	2	6
4	10	7	4**	90	80	90	1	2	6
5	6	3	4***	70	40	90	3	4	6
6	10	7		70	40	-	3	4	-
7	6	3		90	80	-	3	4	-
8	10	7		90	80	-	3	4	-

\*Used nitric acid HNO<sub>3</sub>;

\*\*clay treated in hydrochloric acid was subjected to ion exchange with Al<sup>3+</sup>;

\*\*\*hydrochloric acid after experiment No.1.Reused.

**TABLE2.SOME CHARACTERISTICS OF THE POROSITY OF CLAYI**

Sample no.	S <sub>уд</sub> ,m <sup>2</sup> /g	V <sub>ер.пор</sub> ,cm <sup>3</sup> /g	D <sub>ер.пор</sub> ,Å	S <sub>пор</sub> ,m <sup>2</sup> /g	V <sub>micropores</sub> cm <sup>3</sup> /g
0	75	0,205	146	56	0,00596
1	93	0,231	140	66	0,0058
2	92	0,216	142	60	0,0082
3	103	0,221	129,7	68,3	0,0084
4	103	0,226	125	72,3	0,0067
5	124	0,23	111	83	0,0076
6	96	0,23	133	68,9	0,0059
7	115	0,237	113	83,8	0,006
8	105	0,228	114,7	79	0,006
9	99	0,21	126,8	66	0,0084
10	103	0,217	126,4	68,6	0,0084
11	104	0,227	125	72	0,0067

\*0-original clay sample before acid treatment;1-8- samples processed under the conditions given in the table.2;

9, 10 and 11 are samples (reproducibility study), treated under the conditions of the centre of the plan (10 NHCl, 80° C, 2h).

Only at a low acid concentration, and with an increase in concentration, the influence of the time factor decreases. The extrapolation of the response surface beyond the variation intervals, most likely, does not make much physical sense, . since the resulting surface is not essentially linear and a different compositional design should be used to find the extremum, namely, the second order. However, the second-order design for three factors assumes 27 experiments and does not guarantee that the extremum will be found, since with an increase in the number of experiments,

the likelihood that the resulting regression equation will be adequate decreases. This is one of the reasons why it is legitimate to stop at linear designs of a full factorial experiment.

Additional experiments in the range of values of factors corresponding to the maximum value of the surface ( $\text{CHCl} = 2 \text{ N}$ ,  $T = 40 \text{ }^\circ\text{C}$ ,  $t = 4 \text{ h}$ ) actually made it possible to obtain a surface of less than  $100 \text{ m}^2/\text{g}$ . Thus, it can be assumed that the extremum is in the range of concentration values from 4 to 6 N, temperature from 70 to 90  $^\circ\text{C}$ , and time from 4 to 10 hours. For clay II in the table. 4 shows the values of specific surfaces and average pore diameter, as well as several other porosity characteristics. From table. 4 it follows that the toughening of acid treatment, i.e. an increase in acid concentration, an increase in the temperature and duration of the processing, and, entails an increase in the volume of pores, their average diameter and leads to a decrease in the contribution to the total surface of micropores and pores of large diameter. In this case, the average pore diameter naturally decreases. Results of a planned experiment on acidizing clays of the Navbakhor deposit. X-ray phase analysis of bentonite clays of the Navbakhor deposit (clay III) showed that the mineralogical composition of clays is distinguished by the content of a large amount of montmorillonite.

Therefore, the limiting values of the factors in the full factorial experiment were slightly changed. The changes consisted in softening the acidizing conditions, i. E. in decreasing acid concentration and processing temperature (Table 2). For clay III, the main characteristics of porosity are given in the table. 4. For verification, an extreme experiment was set up at a point with the following values of the factors:  $\text{CHCl} = 10 \text{ N}$ ,  $T = 90 \text{ }^\circ\text{C}$ ,  $t = 10 \text{ h}$ , As a result, the 9 characteristics of the porous structure of clay were obtained, shown in Table 4. Comparison of the data of sample 9 with the data of samples 4,7,8 for clay III shows that the toughening of the acid treatment conditions does not at all lead to an increase in the specific surface area, but, on the contrary, leads to its decrease (up to  $105 \text{ m}^2/\text{g}$ ) in comparison with milder conditions. processing. Based on the totality of the data obtained, the optimal conditions for acid treatment for bentonite clay IV (Navbakhor deposit) were selected, shown in Table 2. For clay IV, in addition to variations in acid concentration, the effect of acid type was also investigated. Nitric acid was used instead of hydrochloric acid, and it was also investigated how the use of spent acid affects porosity. Chemical analysis of the spent acid showed that as a result of acid treatment with nitric acid, iron and aluminium ions are washed out of the clay. The results of studying the porous structure of clay IV are presented in Table 5.

**TABLE-3. SOME CHARACTERISTICS OF THE POROSITY OF CLAYS II AND III**

Sample no.	$S_{ud}, \text{m}^2/\text{g}$ (byBET)		$V_{er.pore}, \text{cm}^3/\text{g}$		$D_{ep.nop}, \text{Å}$		$S_{por}, \text{m}^2/\text{g}$		$V_{micropores}, \text{cm}^3/\text{g}$	
	II	III	II	III	II	III	II	III	II	III
0	70	61	0,177	0,096	151	125	46,7	31	0,006	0,087
1	96	78	0,172	0,101	115,7	102	59,6	39	0,008 9	0,011
2	98	76	0,166	0,095	113	103	58,7	37	0,009	0,012
3	112	109	0,179	0,119	102	78	70,5	61	0,008 8	0,01
4	119	124	0,192	0,153	94	67	81,7	91	0,007	0,003

5	112	66	0,19	0,093	106	111	72	34	0,008 7	0,0096
6	111	68	0,199	0,096	107,6	120	74	32	0,007 7	0,0094
7	136	136	0,224	0,182	92,5	68,7	97	106	0,006	0,004
8	138	146	0,246	0,208	86,3	68	114	122	0,005 7	0,003
9	-	105	-	0,226	-	99	-	92	-	0,0048

\*0- original clay sample before acid treatment; 1-8-samples processed under the conditions given in the table.2; 9-sample (reproducibility test) treated under conditions: 10NHCl, 90°C, 10h.

**TABLE-4. POROUS CHARACTERISTICS OF CLAY IV**

Sample no.	$S_{ud}, m^2/g$ (byBET)	$V_{er.pore}, cm^3/g$	$D_{ep.por}, \text{Å}$	$S_{por}, m^2/g$	$V_{micropores}, cm^3/g$
0	124	0,164	99	66	0,0146
1	300	0,511	75	272	0,0068
2	266	0,574	90	254	0,004
3	285	0,480	77	268	0,0052
4	290	0,510	76	270	0,0060
5	290	0,512	76	271	0,0061

\*0- original clay sample IV, 1-5-samples processed under the conditions given in Table 2.

In this case, the use of nitric acid (sample 3) and there use of hydrochloric acid (sample 5), as well as treatment with hydrochloric acid followed by ion exchange with  $Al^{+3}$  (sample 4), to deeply purify the solution from impurities by removing the ion-exchange products reactions from the reaction sphere resulted in practically the same properties of the porous structure as for sample 1. A significant decrease in the specific surface area (sample 2) should be noted with an increase in the acid concentration from 4 to 7 N.

**TABLE-5. OPTIMUM CONDITIONS FOR ACIDIZING CLAYS I AND IV**

Clay number	$S_{ud}, m^2/g$ (byBET)	$C_{HCl}, N$	$T^\circ C$	t, h
I	105	4-6	70-90	4-10
II	137	10	90	3
III	266	10	90	10
IV	308	4	90	6

## CONCLUSION

Thus, the dependence of the specific surface area of clay on the parameters of acidizing is non-linear. The optimal results of acid treatment are shown in the table. 5. Analysis of the tabular data shows that for all clay samples, the optimal porosity values are achieved at  $CHCl = 6-10 N$ ,  $T = 90^\circ C$ ,  $t = 6-10 h$ . The use of waste hydrochloric and nitric acids instead of hydrochloric acid for the treatment of clays practically does not affect their porous characteristics.

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