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Satbayev University

Х А Б А Р Л А Р Ы

ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН
Satbayev University

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Қазақстан Республикасы Ұлттық ғылым академиясы «ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы» ғылыми журналының Web of Science-тің жаңаланған нұсқасы Emerging Sources Citation Index-те индекстелуге қабылданғанын хабарлайды. Бұл индекстелу барысында Clarivate Analytics компаниясы журналды одан әрі the Science Citation Index Expanded, the Social Sciences Citation Index және the Arts & Humanities Citation Index-ке қабылдау мәселесін қарастыруда. Webof Science зерттеушілер, авторлар, баспашылар мен мекемелерге контент тереңдігі мен сапасын ұсынады. ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы Emerging Sources Citation Index-ке енуі біздің қоғамдастық үшін ең өзекті және беделді геология және техникалық ғылымдар бойынша контентке адалдығымызды білдіреді.

НАН РК сообщает, что научный журнал «Известия НАН РК. Серия геологии и технических наук» был принят для индексирования в Emerging Sources Citation Index, обновленной версии Web of Science. Содержание в этом индексировании находится в стадии рассмотрения компанией Clarivate Analytics для дальнейшего принятия журнала в the Science Citation Index Expanded, the Social Sciences Citation Index и the Arts & Humanities Citation Index. Web of Science предлагает качество и глубину контента для исследователей, авторов, издателей и учреждений. Включение Известия НАН РК. Серия геологии и технических наук в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по геологии и техническим наукам для нашего сообщества.

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MECHANICAL ACTIVATION OF NAVBAHORSK BENTONITE AND ITS TEXTURAL AND ADSORPTION CHARACTERISTICS

Abstract. The article presents a method of purification of obtaining betonies 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. 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.

Key words: betonies clay, specific surface area, porosity, adsorption activity.

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 [13-15].

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 [14-16].

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 [17].

Materials and methods. On the basis of existing methods of clay purification [18-21], a technological method of purification of mineral raw materials of betonies clay, applicable in laboratory conditions, has been developed. The technique includes four stages: centrifugation, drying and mechanical processing [22-25].

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 130^0C for 190 minutes. The result is a clay with a particle size of 1-30 mm. 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.

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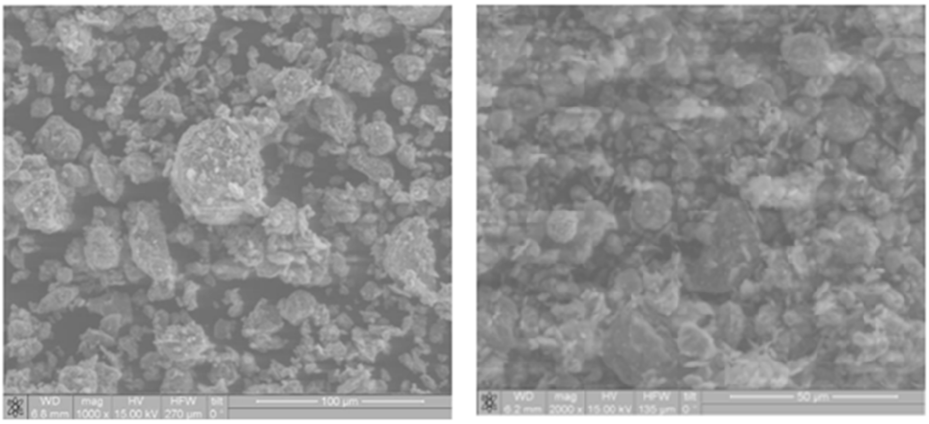

Fig 1. Micrograph of the initial clay sample (a) and after 30 minutes of mechanical activation (b)

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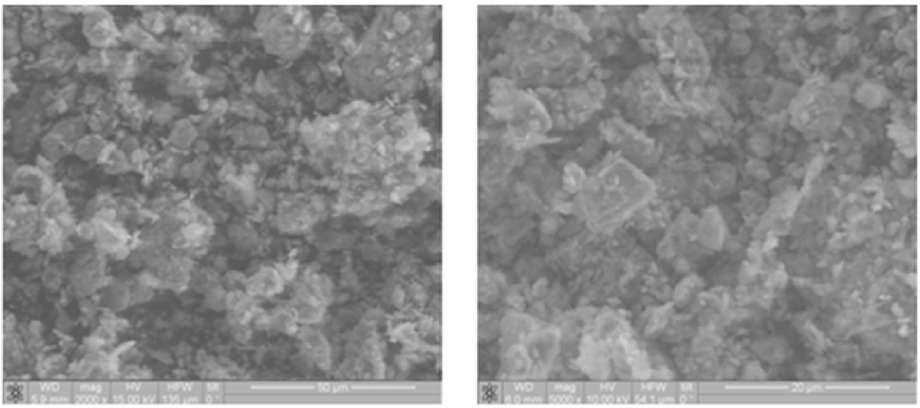

Fig 2. Micrograph of a clay sample after 40 minutes (a) and after 80 minutes (b) mechanical activation

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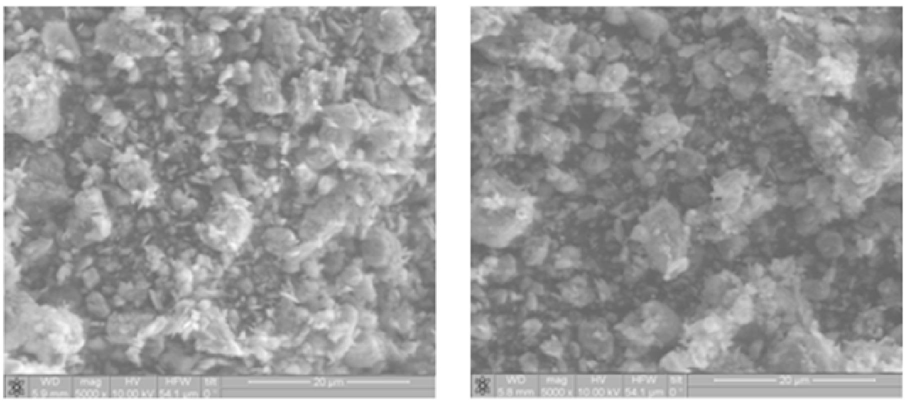

Fig 3. Micrograph of a clay sample after 90 minutes (a) and after 100 minutes (b) mechanical activation

Results. Figure 1 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. 2). After 80 minutes of mechanical activation (Fig. 2), 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 increases. Sticking of small plate elements is observed. After 90 minutes of mechanical activation (Fig. 3), 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. 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	-	-	-

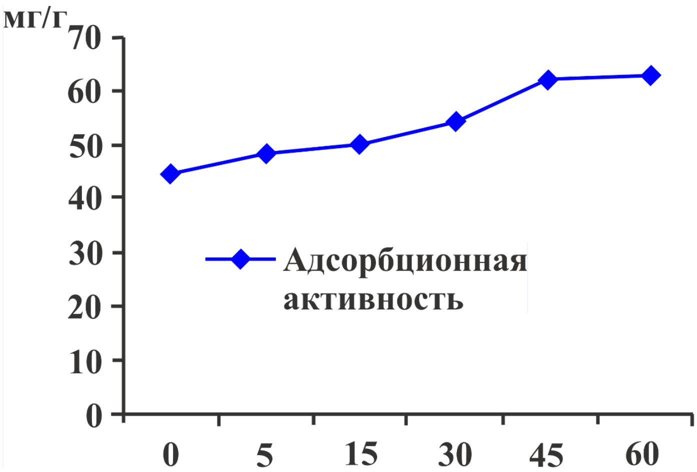

Fig. 4. Dynamics of changes in adsorption activity depending on the time of mechanical activation.

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 4.

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.

Discussion. 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 Navbakhor betonies was determined, presented in Table 2.

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

Name	The content of oxides, (the masses %)											
	SiO_3	TiO_3	Al_3O_3	Fe_3O_3	MgO	CaO	Na_3O	K_3O	P_3O_5	SO_3	FeO	ppp
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 - palygorskite 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 submicropores, 0.4-3 nm in size - micro pores, 3-50 nm - micro pores, with a diameter of more than 50 nm - macro pores. Macropores act as channels for the penetration of substances into the sorbent. Mesopores are much smaller than macrospores; 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.

Table 3. Textural and adsorption characteristics of bentonite clay

№	Name	index
1	Main faction	Less than 0.1 mm - 95%
2	Maximum bulk density, g/cm^3	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^2/g	58.5
6	Specific surface area according to the BET five-point method, sm^2/g	58.5
7	Pore volume at pressure $P/P_0 = 0.99$, cm^3/g	0.089
8	Average pore size, nm	4.9

From table 3 it follows that betonies is 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 combined micro-macro-micro porous adsorbents 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.

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 no.	1	3	3	4	5	8	9	9	9
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 no.	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 no.	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.

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 m^3/g, the pore volume is 0.085 cm^3/g, the average pore size is 4.9 nm, the adsorption activity for methylene blue is 83.0 betonies, g. According to its technological characteristics, it is a finely dispersed medium-weight powder with an average flow ability index.

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^{4+} and Al^{3+}	3:1
8	Specific surface area according to the BET five-point method, m^2/g	54,5±3,0
9	Pore volume at pressure P/P_0 = 0.99, cm^3/g	0.085±0.005
10	Average pore size, nm	4.9

Analysis of derivatographic curves of betonies lines (Fig. 5) shows images of TG - thermal logarithmic gravimetric line, where a decrease in the initial mass of betonies is noted for a certain time, and DTG - differential thermal gravimetry shows a line of temperature parameters change.

The primary decomposition of betonies began at 90^0C and ended at 300^0C. The loss was due to absorbed structural water and other volatile impurities.

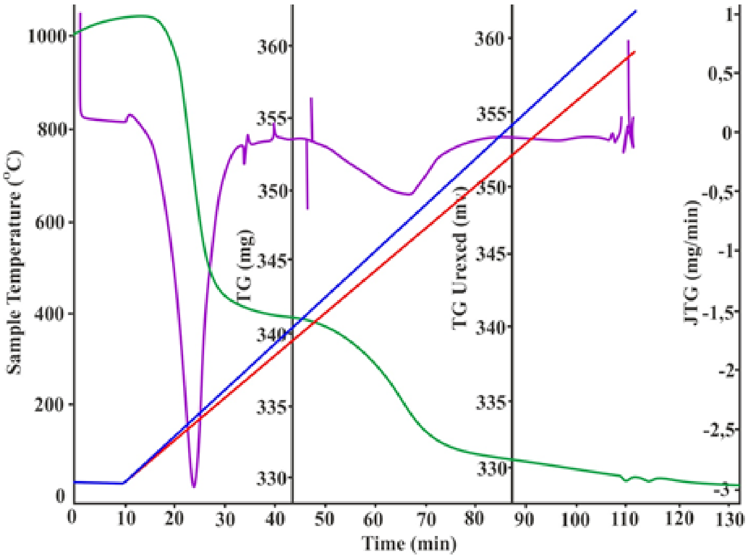

Figure 5. Derivatogram of bentonite from the Navbakhor field

At the second stage of decomposition at a temperature of 480^0C, the remaining mass of 339 bentonite is reduced to 335 bentonite, 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.

Conclusion. After that, the decomposition process stopped, and stable metal oxides may have remained on the solid support (betonies). Thus, when the derivatographic lines of coal and betonies were combined, they, in principle, turned out to be the same. In the process of derivatographic analysis of coal and betonies, the optimal parameters of decarburization were established: 800^0C for 40 min and activation at 950^0C 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 betonies, 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, betonies is used in ground form.

The chemical composition of the considered betonies has been determined. It has been established that the particles of the montmorillonite mineral have sizes of 1.0-0.3 μm and more, with a thickness of 0.001-0.03 μm. 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 betonies clays of the deposits under consideration were established.

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НАВБАХОРЛЫҚ БЕНТОНИТТІҢ МЕХАНИКАЛЫҚ АКТИВТЕНУІ ЖӘНЕ ОНЫҢ ҚҰРЫЛЫМЫ МЕН АДСОРБЦИЯЛЫҚ СИПАТТАМАЛАРЫ

Аннотация. Мақалада катализде қолдану үшін бентонит сазын алудың тазарту әдісі келтірілген. Тазарту үдерісі центрифугалау, кептіру және механикалық активтендіру кезеңдерін қамтитындығы анықталды. Сазды тазарту оны қоспалардан тазартады және пайдалы компоненттің - сорбциялық-белсенді минерал монтмориллонит үлесін арттырады. Зерттеу нәтижесінде BET әдісінің кеуектілігі (меншікті беттік ауданы мен кеуектілігін өлшеу) анықталды. Адсорбциялық белсенділікті анықтау әдісінің дәлдігі мен стандартталуы жүзеге асырылды. Адсорбциялық белсенділіктің үлгінің салмағына

(1,0 г), сорбция уақытына (35 мин) және бояғыш ерітіндісінің тепе-теңдік концентрациясына (39 мл 0,15%) экспоненциалды тәуелділігі анықталды. Бентонит сазының катализді қолдану үшін физика-химиялық және текстуралық қасиеттері зерттелді. Катализде қолдануға арналған бентонит балшығының ақшыл сұр, иісі жоқ ұнтақ екені, іс жүзінде суда және органикалық еріткіштерде ерімейтіні, бөлшектерінің мөлшері 0,3 мм-ден аспайтындығы анықталды.

Сорбентті активтендірудің физикалық әдістері диірмендерде үлгіні өңдеуге, вакуумдық кептіруге, жоғары қысым мен температураға, ультрадыбыстық тербелістерге, сәулеленуге және жоғары жиіліктегі токқа дейін азаяды. «Адсорбциялық белсенділік» тұрғысынан бентониттің механикалық активтену процесін бақылау метилен көк бояуын қолдану арқылы жүзеге асырылды. Механикалық активтену уақытына байланысты алынған саздың адсорбциялық белсенділігін талдау нәтижелері көрсетілді.

Түйінді сөздер: бентонит балшықтары, меншікті бетінің ауданы, кеуектілігі, адсорбциялық белсенділігі.

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МЕХАНИЧЕСКАЯ АКТИВАЦИЯ НАВБАХОРСКОГО БЕНТОНИТА И ЕГО ТЕКСТУРНЫЕ И АДСОРБЦИОННЫЕ ХАРАКТЕРИСТИКИ

Аннотация. В статье представлена методика очистки получения бентонитовой глины для применения в катализе. Установлено, что процесс очистки включает стадии центрифугирования, сушки и механической активации. Очистка глины освобождает ее от примесей и повышает долю полезного компонента – сорбционно-активного минерала монтмориллонита. Определена пористость метода BET (измерение удельной площади поверхности и пористости). Проведена оценка точности и стандартизация метода определения адсорбционной активности. Установлена экспоненциальная зависимость адсорбционной активности от массы навески, (1,0 г), времени сорбции (35 мин) и равновесной концентрации раствора красителя (39 мл 0,15%). Изучены физико-химические и текстурные свойства бентонитовой глины для применения катализа. Установлено, что бентонитовая глина для применения в катализе представляет собой порошок светло-серого цвета, без запаха, практически не растворимый в воде и в органических растворителях, с размером частиц менее 0,3 мм. Целью работы является исследование текстурных и адсорбционных характеристик бентонитовой глины. Контроль над процессом механической активации бентонит по показателю «адсорбционная активность» проводили с использованием красителя метиленового синего. Результаты анализа адсорбционной активности глины, полученные в зависимости от времени ее механической активации.

Ключевые слова: бентонитовая глина, удельная площадь поверхности, пористость, адсорбционная активность.

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