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ACID MODIFICATION OF BENTONITES KALZHAT AND ORTA TENTEK AND STUDY THEIR PHYSICOCHEMICAL PROPERTIES

K. Aryp[1,2], S. Kabdrakhmanova[1], S. Nauryzova[1,2], E. Shaimardan[2], N. Kantay[2,3], A. Zh. Kerimkulova[1*], M. M. Beisebekov[1,2], A. D. Kukhareva[1,2], N. Berdibay[1]

[1] *Satbayev University, Almaty, Kazakhstan*
[2] *Scientific Center of Composite Materials, Almaty, Kazakhstan*
[3] *S. Amanzholov East Kazakhstan University, Ust-Kamenogorsk, Kazakhstan*

* *E-mail: kerimkulova07@mail.ru*

In this work was investigated the methods of acid modification of bentonite clays from the Kalzhat and Orta Tentek deposits located in the Almaty region of Republic Kazakhstan with sulfuric and nitric acid in various concentrations and determined the effective concentration. In this study, the effective concentrations of 20% H_2SO_4 and 10% HNO_3 were selected, because bentonite was modified with 10–98% H_2SO_4 and 10–65% HNO_3 acids during the study. Among these concentrations, according to the results of various studies, 20% H_2SO_4 and 10% HNO_3 acids were found to be effective, and the concentrations given in this work were considered effective. The obtained results were studied by the IR-spectroscopy FTIR, X-ray fluorescence, X-ray diffractometer and scanning electron microscope methods. Bentonite modification has a positive effect on opening the interlayer space of clay and improving its properties. After purification and modification, bentonite clay can be used in agriculture, manufacturing, medicine, and various fields such as drilling, construction and water purification.

Keywords: *bentonite, acid modification, montmorillonite, Orta Tentek, Kalzhat, clay.*

INTRODUCTION

Bentonite is a versatile clay mineral that is widely used in all industries due to its unique properties. In recent years, scientists have found many applications for bentonite clay due to its diverse physical and chemical properties. Bentonite belongs to the group of smectite minerals, characterized by the ability to swell in water. This unique property of smectite clays, such as bentonite, makes them very effective for various industrial and commercial purposes. Conventional natural bentonite clay is often unsuitable for use in its finished form and requires preliminary activation. As a result of such processing, the number of its active sites increases, porosity and surface area increase, and other useful properties are improved [1–2]. After activation, bentonite turns into a porous material with 2-4 times greater adsorption capacity and increased catalytic activity than the original natural clay. Typically, such activation is carried out using mineral acids. The structural parameters of the resulting activated bentonite (specific surface area, pore volume and pore diameter distribution) determine its adsorption properties and directly affect the selective absorption of substances and the adsorption rate [3]. Many researchers have attempted to modify bentonite to improve its adsorption properties.

This paper proposes a new, simple modification strategy combining acid activation and pillaring with iron hydroxide to improve the adsorption properties of bentonite [4]. Bentonite clay materials are heterogeneous hard clays and can be improved by acid treatment, which involves the removal of various pore-clogging ions by creating surface pores and cracks, resulting in an increase in surface area and pore volume [5–7]. In this work [8], a modification with H_2SO_4 (98%) was made, the clay/acid ratio (by weight) was 3.6. Sulfuric acid (0.1M, 1M, 2M and 3M) was used for modification [9]. As a result of the studies, it was found that 2M is effective. Modification of bentonite clay by microwave irradiation with sulfuric acid showed changes in the surface and acidity. In addition, HCl was used to activate the acid. In this work, 0.05–0.6 M hydrochloric acid solutions were used, as a result it was found that a concentration of up to 0.1 M is effective [10]. Acid modification of bentonite clays is the most necessary method of technology for obtaining a high specific surface of clays. It is noted that there are no universal methods of acid treatment to obtain the maximum specific surface, and its values obtained during testing of bentonite samples with different chemical compositions are given [11]. Different methods are used for acid modification of bentonite, but its efficiency depends on the temperature, duration of the process, acid concentration, chemical nature and chemical composition of bentonite. Therefore, it is important to find new effective methods for modifying bentonite, searching for ways to improve these properties by optimizing the parameters of existing technology [12].

In this paper, the acid modification of bentonite clays from the Orta Tentek and Kalzhat deposits located in the Almaty region of Republic Kazakhstan was studied, and the effective concentration was determined. These deposits are poorly studied natural sources of bentonite and smectite clays in Kazakhstan, their mineral and chemical composition is not fully described in foreign scientific literature. The scientific novelty of the study is that for the first time, clays of the Kalzhat and Orta Tentek deposits in Kazakhstan were comprehensively characterized, changes in their mineralogical and physicochemical properties after acid treatment were determined.

2. Materials and methods

2.1 Materials

Sulfuric and nitric acids (H_2SO_4 (95–98%) and HNO_3 (63–65%) "Sigma Tech", extra pure). Clays "Kalzhat" and "Orta Tentek" are mixtures of particles of different sizes with many additives of other minerals.

2.2 Methods

2.2.1 Acid modification of clays

Bentonite clays from the Kalzhat and Orta Tentek deposits (Almaty region, Republic of Kazakhstan) were used as the main raw material in the research work. The process of treating Kalzhat and Orta Tentek clays with various concentrations of mineral acids (H_2SO_4, HNO_3) for chemical modification was studied in the course of the research. Before acid treatment, bentonite clay samples were ground in a ball mill (X, X) to a size of 0.08 mm. To modify bentonite, 10 g of bentonite was weighed and 10 ml of acid solution was added to it, continuously stirred for 30–60 min, and then the filter was washed with distilled water to pH = 6.0–8.0 using a "blue ribbon" of filter paper. Then the bentonite with filter paper was placed in a drying cabinet (X, X) at a temperature of 150 °C and dried for 3–5 hours. Before determining the chemical composition, the samples of acid-modified bentonite were dried to a constant weight.

The obtained results were determined using the XRF spectrometer KPB-1M. Structural and morphological characteristics of clay before and after modification were studied using a SEM JEM 1400 (JEOL, Japan). The chemical structure was investigated by FTIR FT-801. X-ray phase analysis was performed on X'PertPRO XRD.

2.2.2 Qualitative indicators of bentonites

The sorption of methylene blue (adsorption of Meth. blue) of primary and acid-treated clays was determined using the methods specified in state standard 28177-89.

2.2.3 XRF spectrometer

The elemental composition of the initial and modified clays was determined using a (KPB-1 M, Russia) X-ray fluorescence spectrometer.

2.2.4 IR analysis

IR analysis of clays was carried out on an FT-801 IR spectrometer (Simex, Russia) using a standard method with a resolution of 1 cm^{-1} in the range of 450–4700 cm^{-1} and at a temperature of 25 °C using additional equipment for measuring the coefficients of attenuated total reflection (ATR) and specular-dynamic-scattered reflection (SDR).

2.2.5 XRD analysis

The crystalline structures of the clay were studied by X-ray diffraction on an X'Pert Pro diffractometer (Malvern Panalytical Empyrean, the Netherlands), monochromatic copper radiation (CuKa) with a scanning step of 0.05, K-Alpha1 [Å] 1.54187. The measurement angle was 5–70°, the voltage in the X-ray tube was 45 kV, the current was 30 mA, the measurement time was 0.5 s at each step. For measurements in the reflection mode, an aluminum rectangular universal sample holder (PW 1172/01) was used.

2.2.6 SEM analysis

The clay surface morphology was studied using a JEM 1400 (JEOL, Japan). The measurements were carried out using a secondary electron detector at an accelerating voltage of 15 kV in high vacuum mode.

2.2.7 BET analysis

The specific surface area of bentonite was determined using a BSD-660 analyzer (China), which is capable of measuring surface areas starting from 0.0005 m^2/g, pore diameters in the range of 0.35–500 nm, and pore volumes from 0.0001 cm^3/g.

3. Results and discussion

3.1 Qualitative indicators of bentonites

The sorption capacity for methylene blue in the initial Kalzhat clay was 81%, and in Orta Tentek – 87%. In Kalzhat clay, after treatment with 20% H_2SO_4, an increase in the sorption capacity to 85% was observed, and after treatment with 10% HNO_2 – up to 88%. In Orta Tentek clay, after treatment with 20% H_2SO_4, an increase in the sorption capacity to 94% was observed, and after treatment with 10% HNO_2 – up to 89%. This acid modification is associated with the opening of the silicate layer, cleaned of large residues and water-soluble minerals in bentonite [13].

3.2 Initial and modified chemical composition of clays

The chemical composition of the initial and modified Kalzhat and Orta Tentek clays was determined using XRF analysis (Table 2).

Table 2 shows the chemical composition of the Kalzhat and Orta Tentek clays. The SiO_2 content increases due to the acid leaching of aluminum and other exchange cations (Ca^+, Mg^+, Fe^{3+}). The proportion of Al_2O_3, Fe_2O_3, CaO, MgO and other oxides decreases. As a result, the chemical composition of bentonite is associated with silicon, which ensures acid resistance and high adsorption capacity. It can be seen from the table that the main components of the clays are SiO_2, Al_2O_3 and Fe_2O_3, but Cr_2O_3, CuO, ZnO, As_2O_3, Ag_2O, PbO, SnO_2, Sb_2O_3 are found in very small quantities. Modifications with sulfuric and nitric acids lead to changes in the elemental composition. In particular, it can be seen that the amount of silicon oxide increases in the two clays after washing with two different acids. When Kalzhat clay is modified with sulfuric and nitric acids, the content of oxides of phosphorus, sulfur, calcium, titanium, chromium, manganese, copper and zinc decreases. It is evident that some composition increases relatively when modified with nitric acid. After washing with sulfuric and nitric acid, a decrease in the amount of iron, phosphorus, calcium, vanadium, chromium, manganese, nickel, copper and zinc oxides is observed in the Orta Tentek clay. At the same time, after modification with 10% nitric acid, the concentration of aluminum, magnesium, potassium, titanium and silver increased, and the amount of other elements decreased.

Table 1. Qualitative indicators of clay samples

Methylene blue adsorption capacity %	Kalzhat clay			Orta Tentek clay		
	Initial	H_2SO_4 20% modified	HNO_3 10% modified	Initial	H_2SO_4 20% modified	HNO_3 10% modified
	81	85	88	87	94	89

Table 2. Chemical composition of clay samples

Composition %	Kalzhat clay			Orta Tentek clay		
	initial	H_2SO_4 20% modified	HNO_3 10% modified	initial	H_2SO_4 20% modified	HNO_3 10% modified
SiO_2	67,6	70,1	69,6	66,2	72,1	69,5
Al_2O_3	18,1	18,2	18,1	18,1	17,2	18,5
Fe_2O_3	7,43	6,50	7,59	8,0	6,27	7,74
MgO	1,86	1,74	2,08	1,53	1,25	1,63
P_2O_5	0,132	0,0847	0,0813	0,156	0,0760	0,0905
SO_3	1,65	0,672	0,348	2,09	0,936	—
K_2O	0,550	0,564	0,605	0,555	0,541	0,596
CaO	1,46	0,943	0,468	2,09	0,611	0,383
TiO_2	0,940	0,895	0,903	0,952	0,842	0,994
V_2O_5	0,0321	0,0170	0,0195	0,0346	0,0156	0,0194
Cr_2O_3	0,0088	0,0071	0,0077	0,0081	0,0063	0,0078
MnO	0,0435	0,0265	0,0322	0,0536	0,0245	0,0206
Co_2O_3	0,0212	0,0169	0,0218	0,0235	0,0159	0,032
NiO	0,116	0,0063	0,0076	0,0120	0,0061	0,0074
CuO	0,0085	0,0039	0,0050	0,0084	0,0043	0,0051
ZnO	0,0061	0,0039	0,0048	0,0064	0,0035	0,0050
As_2O_3	0,0014	0,0007	0,0009	0,0023	0,0005	0,0007
Ag_2O	0,0007	0,0009	0,0012	0,0009	0,0012	0,0013
BaO	0,0207	0,0189	0,0245	0,0369	0,0231	0,0207
PbO	0,0031	0,0024	0,0026	0,0033	0,0023	0,0027
SnO_2	0,0080	0,0076	0,0097	0,0099	0,0083	0,0092
Cl	0.0084	0,0465	0,0481	0,0107	0,0032	0,0095
Sb_2O_3	0,0011	0,0012	0,0014	0,0014	0,0012	—

3.3 Results of IR analysis of clays

The FTIR spectrum of initial and acid-modified Kalzhat and Orta Tentek clays in the wave number range of 500–4000 cm^{-1} is shown in Figures 1–2. The increase in the peak intensity at 3734 cm^{-1} indicates a decrease in the number of exchangeable cations during acid modification. These results are consistent with the XRF data, which revealed a significant decrease in the Ca content in acid modified clay compared to the initial clay due to partial dissolution of calcium ions during acid modification (see Table 1). An intense peak at 1040 cm^{-1}, a decrease in the intensity of the bands can be observed at 820 and 730 cm^{-1}, which is associated with a change in the silicon environment caused by acid exposure. Weak absorption peaks indicate that these clays belong to the montmorillonite group [14–15]. Similar results were obtained in previous studies [16–18].

The FTIR spectrum of initial and modified Orta Tentek clay is shown in Figure 2.

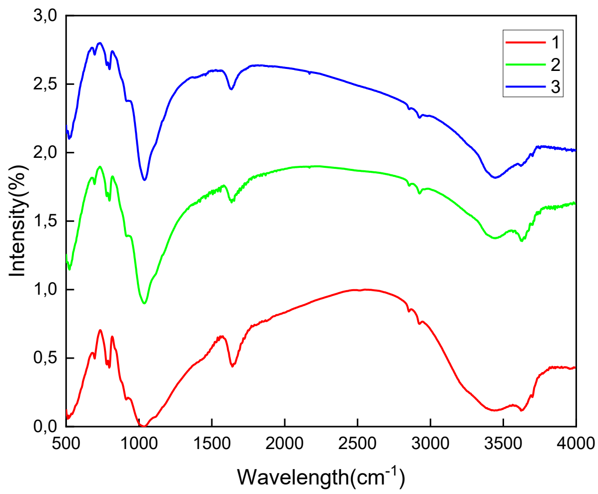

Figure 1. IR spectrum of clay Kalzhat. 1 – initial Kzh, 2 – H_2SO_4 20% modified, 3 – HNO_3 10% modified

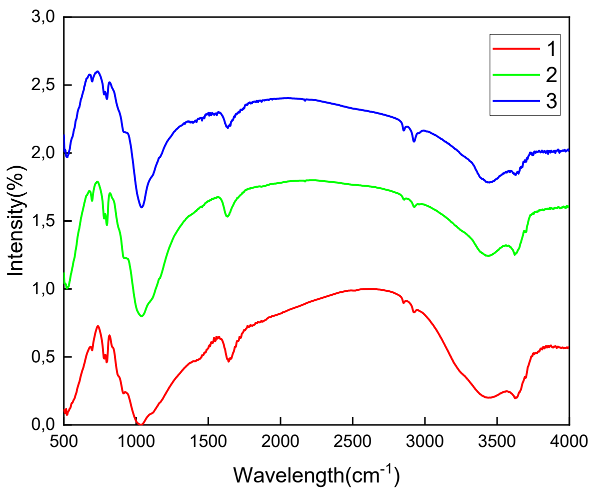

Figure 2. IR spectrum of Orta Tentek clay. 1 – initial Orta Tentek, 2 – H_2SO_4 modified 20%, 3 – HNO_3 modified 10%

3.4 XRD analysis

The results of X-ray structural analysis of the original and modified Kalzhat clay are presented in Figure 3. According to the results, the main rock-forming mineral of the initial Kalzhat clay is quartz (57%), its chemical formula corresponds to SiO_2. The crystal lattice of the initial clay Kalzhat has a hexagonal structure. Modified with 20% H_2SO_4, was found to be that the main constituent mineral of modified Kalzhat clay is quartz 62%, its composition corresponds to the following formula: SiO_2. The structure of the crystal lattice of the clay mineral is hexagonal. However, the main rock-forming mineral of Kalzhat clay, modified with 10% HNO_3, is montmorillonite, the chemical formula of which is $Si_{7.80}\,Al_{1.72}\,Cs_{0.16}\,Fe_{0.20}\,Mg_{0.28}\,O_{20.00}$. The crystal lattice of the modified Kalzhat clay is cubic.

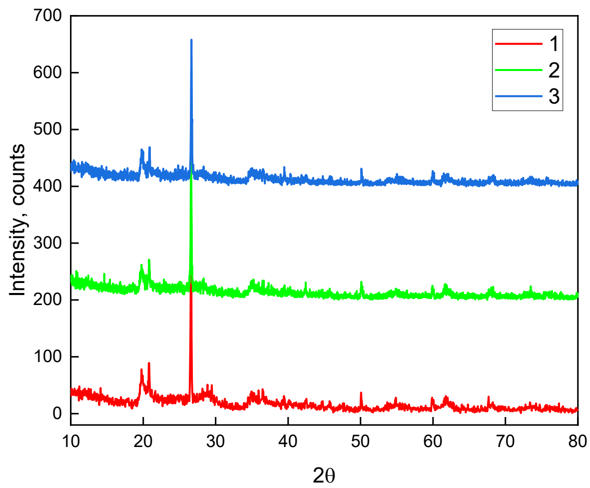

Figure 3. XRD spectrum of Kalzhat clay. 1 – initial Kzh, 2 – (20%) H_2SO_4 modified Kzh, 3 – (10%) HNO_3 modified Kzh

The results of XRD structural analysis of the initail and modified Orta Tentek clay are presented in Figure 4. It was established that the main rock-forming mineral of Orta Tentek clay is quartz SiO_2 – 66%. The crystal lattice of the primary clay is hexagonal. Modified with 20% H_2SO_4, the main mineral constituting the Kalzhat clay is montmorillonite 60%. The crystal lattice of the clay mineral is monoclinic. The main rock-forming mineral of the Orta Tentek clay, modified with 10% HNO_3, is quartz 59%. Its composition corresponds to the following formula: SiO_2. The structure of the crystal lattice of the clay mineral is hexagonal.

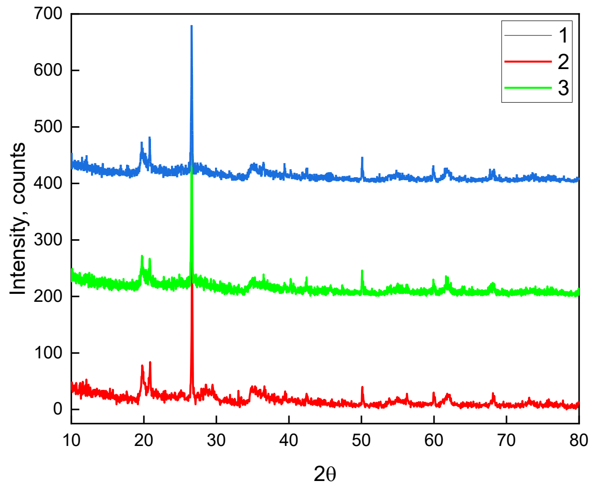

Figure 4. XRD spectral spectrum of the Orta Tentek clay. 1 – initial O.T., 2 – (20%) H_2SO_4 modified, 3 – (10%) HNO_3 modified

3.5 Results of morphological analysis

As a result of acid treatment, a significant change in the morphology of clays is observed, in particular, the exchange of interlayer cations, which leads to an increase in the pore volume and surface area. The crystal lattice is partially destroyed, new micro- and mesopores are formed. The structural stability of bentonite increases, its granules are crushed and evenly distributed.

It is evident from Figure 5B that the sizes of the initial Kalzhat clay layers are in the range of 21.2–245.7 µm. The surface of the clay layers is uniform, and the clay layers modified with 20% H_2SO_4 decrease to the range of 15.6–115.3 µm, which is clearly seen in Figure 5E. It can be seen the formation of pores on the clay surface and the opening of the interlayer space. Also, after modification with 10% HNO_3, the sizes of the layers decrease to the range of 8.5–124.7 µm. For comparison, an increase in the specific area of bentonite is observed in bentonite clays modified with nitric and sulfuric acid.

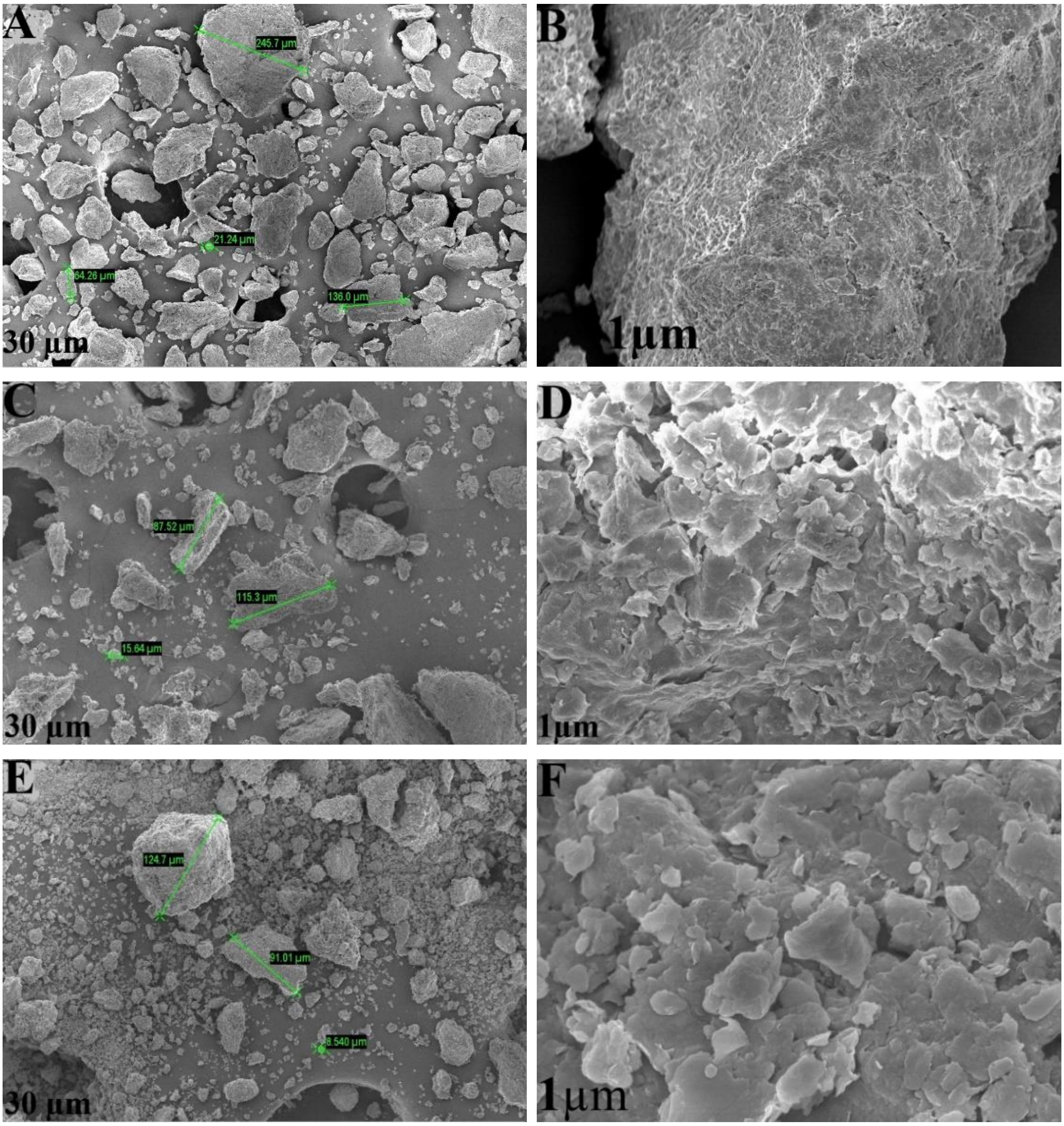

Figure 5. SEM images of Kalzhat clay. A, B – initial Kzh.; C, D – modified (20%) H_2SO_4; E, F – modified (10%) HNO_3

The morphology of the initial Orta Tentek clay is not visible on the clay surface in the 100 µm range (Figure 6A). Figure 6D clearly shows that the surface of the clay modified with 20% H_2SO_4 is smooth and pores have formed, and Figure 6G also clearly shows that pores have formed on the surface of the clay modified with 10% HNO_3. It can be seen that the particle size of the initial clay is a maximum of 177.2 µm, while in the clay modified with 20% H_2SO_4 it decreases to 86.3 µm, and in the clay modified with 10% HNO_3 it decreases to 52.9 µm. Modification with sulfuric and nitric acids helps to improve the properties of the clay by expanding the interpacket space of mineral particles. In addition, the adsorption properties are improved, which allows for the effective sorption of heavy metals, organic molecules and dyes.

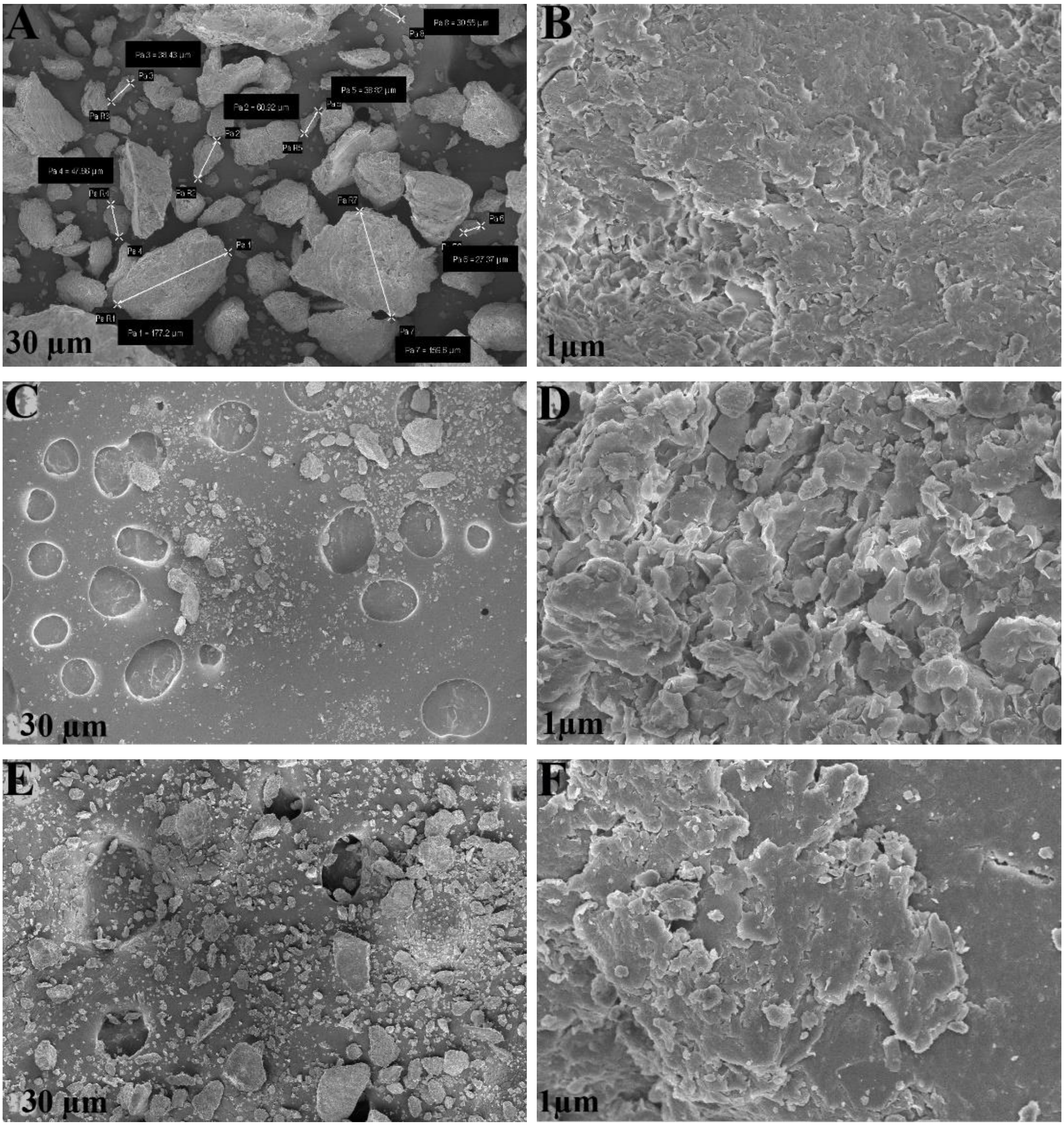

Figure 6. SEM images of Orta Tentek clay. A, B – initial O.T.; C, D – modified H_2SO_4 20%; E, F – modified HNO_3 10%

3.6 BET analysis

The adsorption/desorption isotherms and average pore sizes of the initial Kalzhat and Orta Tentek clays, as well as clays modified with sulfuric and hydrochloric acids, are presented in Tables 3–4, as well as in Figures 7 and 8. The main indicators characterizing the adsorption properties of bentonites are their specific surface area, volume and pore size. The results of the study showed that the isotherms of all samples belong to type IV according to the Brunauer classification. [19].

According to the comparative values of the textural indicators of bentonite samples, presented in Table 3, the initial Kalzhat clay had a specific surface area of 73.98 m^2/g, pore volume of 0.04 m^3/g, pore size of 3.29 nm. In clay modified with 20% H_2SO_4, the surface area increased by 1.1 times, the pore volume by 2.25 times, and the pore size by 1.6 times. At the same time, after modification with 10% HNO_3, the surface area was 78.16 m^2/g, the volume was 0.05 m^3/g, and the size was 4.89 nm. This may be due to the rearrangement of nanocrystals or the replacement of large cations with small ones [20].

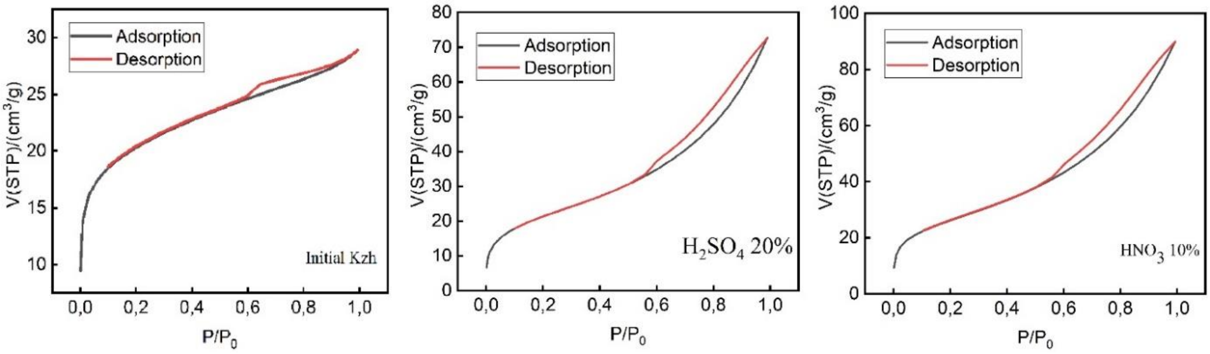

Figure 7. Initial Kalzhat; H_2SO_4 20% modified; HNO_3 10% modified; N_2 adsorption/desorption isotherms

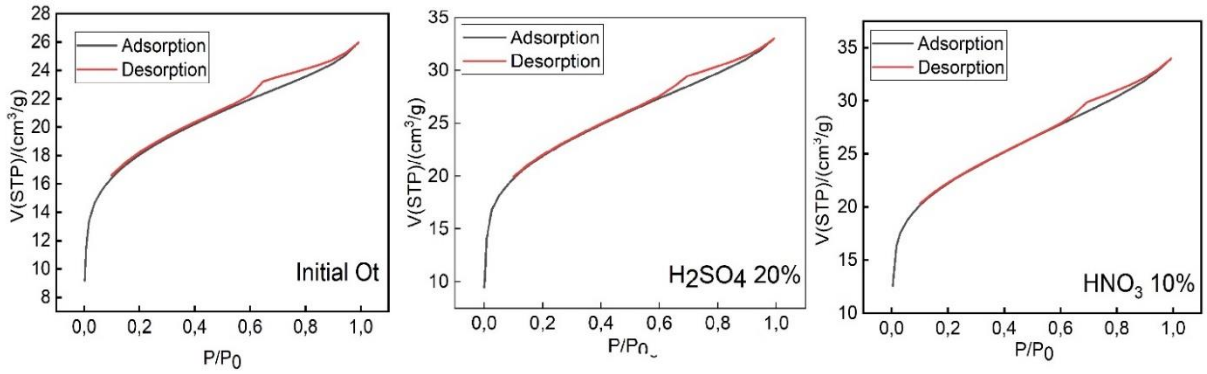

Figure 8. Initial Orta Tentek; H_2SO_4 20% modified; HNO_3 10% modified; N_2 adsorption/desorption isotherms

Table 3. Structural parameter values of the initial Kalzhat and clays modified with H_2SO_4 20%, HNO_3 10%

Parameters	Initial Kalzhat	H_2SO_4 20%	HNO_3 10%
Surface area, m^2/g	73,98	77,73	78,16
Pore volume, m^3/g	0,04	0,09	0,05
Pore size, nm	3,29	5,42	4,89

Table 4 shows the adsorption/desorption isotherms and average pore sizes of the initial Orta Tentek clay and clays modified with sulfuric and hydrochloric acids. The specific surface area of the initial Orta Tentek clay was 65.62 m^2/g, the pore volume was 0.04 m^3/g, and the pore size was 3.24 nm. For the clay modified with 20% H_2SO_2, the surface area increased by 1 time and was 68.14 m^2/g. The pore volume was 0.05 m^3/g, and the pore size was 3.33 nm. Moreover, after modification with 10% HNO_3, the surface area was 7.03 m^2/g, the volume was 0.05 m^3/g, and the size was 3.15 nm. The results showed that bentonite has a high specific surface area and a large pore volume, which determines its high adsorption capacity and confirms its exceptional suitability for use as an adsorbent.

Table 4. Structural parameter values of the initial Orta Tentek and H_2SO_4 20%, HNO_3 10% modified clays

Parameters	Initial Orta Tentek	H_2SO_4 20%	HNO_3 10%
Surface area, m^2/g	65,62	68,14	70,03
Pore volume, m^3/g	0,04	0,05	0,05
Pore size, nm	3,24	3,33	3,15

4. Conclusion

The acid modification of clays from two deposits in the Republic of Kazakhstan – Kalzhat and Orta Tentek – was studied by their structure and composition. It was established that silicon and iron are the main components of Kalzhat and Orta Tentek clays. Treatment with sulfuric and nitric acids of various concentrations leads to a change in the elemental composition of clays. Compared to the initial clays, a decrease in the amount of phosphorus, manganese, potassium, sodium and calcium is observed. A relative increase in iron content was found when modified with 10% nitric acid. In addition, modification with two acids completely destroyed the amount of vanadium, bromine and chromium. The results of X-ray structural analysis showed that the clay samples contained montmorillonite, quartz, mixed-layer clay mineral and smectite. The results of IR analysis showed that the main rock-forming minerals of these two clays are montmorillonite and quartz. The morphology of the Tentek clays of Kalzhat and Orta is characterized by a clear layered structure. With acid modification, it is clearly seen that the interlayer folds open and mesopores are formed. Acid modification increases the adsorption and catalytic efficiency of bentonite by increasing its surface area, porosity and acid sites, as its elemental composition is enriched with silicon, and its morphology acquires a fine-porous structure. Under the influence of acid, interlayer cations (Ca^{2+}, Mg^{2+}, Fe^{3+}) are released, new vacancies and pores are formed. This increases the adsorption properties of clay. Under the influence of modification, the surface area increases by 2–4 times, a pore structure develops, as a result of which it effectively absorbs heavy metals, dyes, and organic compounds. High sorption properties open up wide opportunities for the production of bentonite as effective sorbents for removing organic and inorganic pollutants from water, for the preparation of drilling fluids in the oil industry, and as a carrier of drugs in pharmaceuticals.

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БЕНТОНИТТЕРДІ, ҚАЛЖАТТЫ ЖӘНЕ ОРТА ТЕНТЕКТІ ҚЫШҚЫЛДЫ ТҮРЛЕНДІРУ ЖӘНЕ ОЛАРДЫҢ ФИЗИКАЛЫҚ ХИМИЯЛЫҚ ҚАСИЕТТЕРІН ЗЕРТТЕУ

Қ. Арып[1,2], С. Қабдрахманова[1], С. Наурызова[1,2], Е. Шаймардан[2], Н. Қантай[2,3], А. Ж. Керімқұлова[1*], М. М. Бейсебеков[1,2], А. Д. Кухарева[1,2], Н. Бердібай[1]

[1] «Қ.И. Сәтбаев атындағы Қазақ ұлттық техникалық зерттеу университеті» КеАҚ, Алматы, Қазақстан
[2] Композиттік материалдар ғылыми орталығы, Алматы, Қазақстан
[3] С.Аманжолов атындағы Шығыс Қазақстан университеті, Өскемен, Қазақстан

* *Байланыс үшін E-mail: kerimkulova07@mail.ru*

Бұл жұмыста Қазақстан Республикасының Алматы облысында орналасқан Қалжат және Орта Тентек кен орындарының бентонит саздарын әртүрлі концентрациядағы күкірт және азот қышқылымен қышқылдық модификациялау әдістері зерттеліп, тиімді концентрациясы анықталды. Бұл зерттеуде 20% H_2SO_4 және 10% HNO_3 тиімді концентрациясы таңдалды, өйткені зерттеу барысында бентонит 10–98% H_2SO_4 және 10–65% HNO_3 қышқылдарымен модификацияланды. Осы концентрациялардың ішінде әртүрлі зерттеулердің нәтижелері бойынша 20% H_2SO_4 және 10% HNO_3 қышқылдары тиімді деп танылып, осы жұмыста берілген концентрациялар тиімді деп танылды. Алынған нәтижелер ИҚ-спектроскопиялық FTIR, рентгендік флуоресценция, рентгендік дифрактометр және сканерлеуші электронды микроскоп әдістерімен зерттелді. Бентонит модификациясы саздың қабат аралық кеңістігін ашуға және оның қасиеттерін жақсартуға оң әсер етеді. Тазарту және модификациялаудан кейін бентонит сазын ауыл шаруашылығында, өндірісте, медицинада және бұрғылау, құрылыс және суды тазарту сияқты әртүрлі салаларда қолдануға болады.

Түйін сөздер: *бентонит, қышқылдық модификация, монтмориллонит, Орта Тентек, Қалжат, саз.*

КИСЛОТНАЯ МОДИФИКАЦИЯ БЕНТОНИТОВ КАЛЖАТ И ОРТА ТЕНТЕК И ИЗУЧЕНИЕ ИХ ФИЗИКО-ХИМИЧЕСКИХ СВОЙСТВ

К. Арып[1,2], С. Кабдрахманова[1], С. Наурызова[1,2], Е. Шаймардан[2], Н. Кантай[2,3], А. Ж. Керимкулова[1*], М. М. Бейсебеков[1,2], А. Д. Кухарева[1,2], Н. Бердибай[1]

[1] НАО «Казахский национальный исследовательский технический университет им. К.И. Сатпаева», Алматы, Казахстан
[2] Научный центр композиционных материалов, Алматы, Казахстан
[3] Восточно-Казахстанский университет им. С. Аманжолова, Усть-Каменогорск, Казахстан

* *E-mail для контактов: kerimkulova07@mail.ru*

В данной работе исследованы способы кислотной модификации бентонитовых глин месторождений Калжат и Орта Тентек, расположенных в Алматинской области Республики Казахстан, серной и азотной кислотой различных концентраций и определены эффективные концентрации. В данном исследовании были выбраны эффективные концентрации 20% H_2SO_4 и 10% HNO_3, так как в ходе исследования бентонит модифицировали кислотами 10–98% H2SO₄ и 10–65% HNO_3. Среди этих концентраций по результатам различных исследований эффективными оказались кислоты 20% H2SO₄ и 10% HNO_3, эффективными же считаются концентрации, приведенные в данной работе. Полученные результаты изучены методами ИК-спектроскопии FTIR, рентгеновской флуоресценции, рентгеновской дифрактометрии и сканирующей электронной микроскопии. Модификация бентонита положительно влияет на раскрытие межслоевого пространства глины и улучшение ее свойств. После очистки и модификации бентонитовая глина может использоваться в сельском хозяйстве, производстве, медицине, а также в различных областях, таких как бурение, строительство и очистка воды.

Ключевые слова: *бентонит, кислотная модификация, монтмориллонит, Орта Тентек, Калжат, глина.*