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Д.В.Сокольский атындағы «Жанармай,
катализ және электрохимия институты» АҚ

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ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
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АО «Институт топлива, катализа и
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PHYSICOCHEMICAL INVESTIGATIONS OF VERMICULITE – MICROPOROUS COMPONENT FOR HEAT-RESISTANT MATERIALS

Abstract. The results of investigations of Kulantau field (Turkestan region) vermiculite's physicochemical and physical-technical properties are presented. The vermiculite's chemical and mineralogical composition and its main crystalline phases, which are composed of ferrous magnesium and calcium aluminosilicates of complex composition, are determined by chemical, X-ray diffraction, spectral analysis methods. The complex thermal analysis method established the processes occurring during heating, which allow to judge the physicochemical and structural changes occurring in vermiculite during heat treatment. The electron microscopy method investigated the vermiculite particles' surface morphology. The main physical-technical properties of the expanded vermiculite are determined. It was established that the expanded vermiculite has low bulk density and low thermal conductivity, has high porosity, fire resistance, and refractoriness characteristics. Due to its unique properties, the expanded vermiculite can be effectively used as a microporous component for production of composite heat-resistant materials.

Key words: vermiculite, methods of physicochemical analysis, physical-technical properties, microporous component, composite heat-resistant materials.

Introduction. The Republic of Kazakhstan has huge reserves of natural and technogenic mineral raw materials, which under certain conditions of their processing can become a full-fledged resource for various industries [1-4].

The development of effective types of composite binding materials from natural and technogenic mineral raw materials is one of the most urgent scientific, technical, economic and environmental problems.

Vermiculite is a natural mineral from the group of hydromica, formed in natural conditions as a result of hydration and other secondary changes of various micas. Vermiculites, formed from ferrous magnesium micas – biotite and phlogopite, are of practical importance. The vermiculite's chemical composition corresponds to the approximate formula $(Mg, Fe^{2+}, Fe^{3+})_3 [(Si, Al)_4 O_{10}](OH)_2 \cdot 4H_2O$. However, vermiculite does not always exactly correspond to the general formula, since it contains various impurities [5].

In Kazakhstan, the most famous deposits of vermiculite are Altyntas, Karatas, Sholak-Kairaktin in Aktobe region; Barchinsk in North Kazakhstan region; Neozhidannoye in Karaganda region; Kulantau in Turkestan region [6].

A special feature of vermiculite is its ability to increase its volume by 10-20 times with a sharp roasting. This expansion phenomenon is due to the fact that during a sharp roasting, the molecular water between the vermiculite flakes quickly turns into steam, which moves the mica flakes always in the direction perpendicular to the mica cleavage. The vermiculite expanded during roasting during cooling retains increased volume with the thinnest air interlayers between the mica flakes, which gives the mineral many valuable properties, in particular fire resistance, refractoriness and low thermal conductivity [7, 8].

Introduction of the expanded vermiculite, as a refractory microporous component, which, being a damper, suppresses the resulting stresses under thermal effects, which contributes to increase in the heat resistance of heat-resistant concrete [9, 10].

In the works [11, 12], proceeding from the theoretical model applied to refractory materials, it was found that the vermiculite use contributes to decrease in the thermal conductivity of heat-resistant vermiculite concrete in comparison with other highly porous materials.

Methods. The *physicochemical investigations* of the vermiculite samples' microstructure were performed using JSM-6490LV scanning electron microscope (JEOL, Japan), with the help of which the elemental chemical composition was also determined by the energy dispersive method.

The *petrographic investigations* of the vermiculite samples were performed using MIN-8 optical polarization microscope by examining immersion preparations in the transmitted light.

The *chemical analysis* of the studied vermiculite samples was performed according to the method adopted for inorganic materials [13, 14].

The vermiculite samples' *microstructure was investigated* using JSM-6490LV scanning electron microscope (JEOL, Japan), which also determined the elemental chemical composition by the energy dispersive method.

The *X-ray diffraction investigation* was performed using Bruker AXS X-ray diffractometer (Germany) on CaK_{α} radiation at a voltage of 40kV and at a current of 40mA, at an angle coverage interval of 2θ 5-80º at a recording step of 0.1º and a recording speed of 2s/step.

The diffraction patterns were decoded automatically using EVA software package of Bruker AXS X-ray diffractometer.

The *thermogravimetric investigations* were performed using TGA/DSC1 Mettler Toledo thermal-analytical system (Switzerland) – a device for combined thermographic analysis and differential scanning calorimetry. The device is designed to measure thermodynamic characteristics (heat and temperature of phase transitions and physicochemical reactions), as well as recording changes in the mass of solid and powdered materials in the temperature range from 25 to 1600ºC.

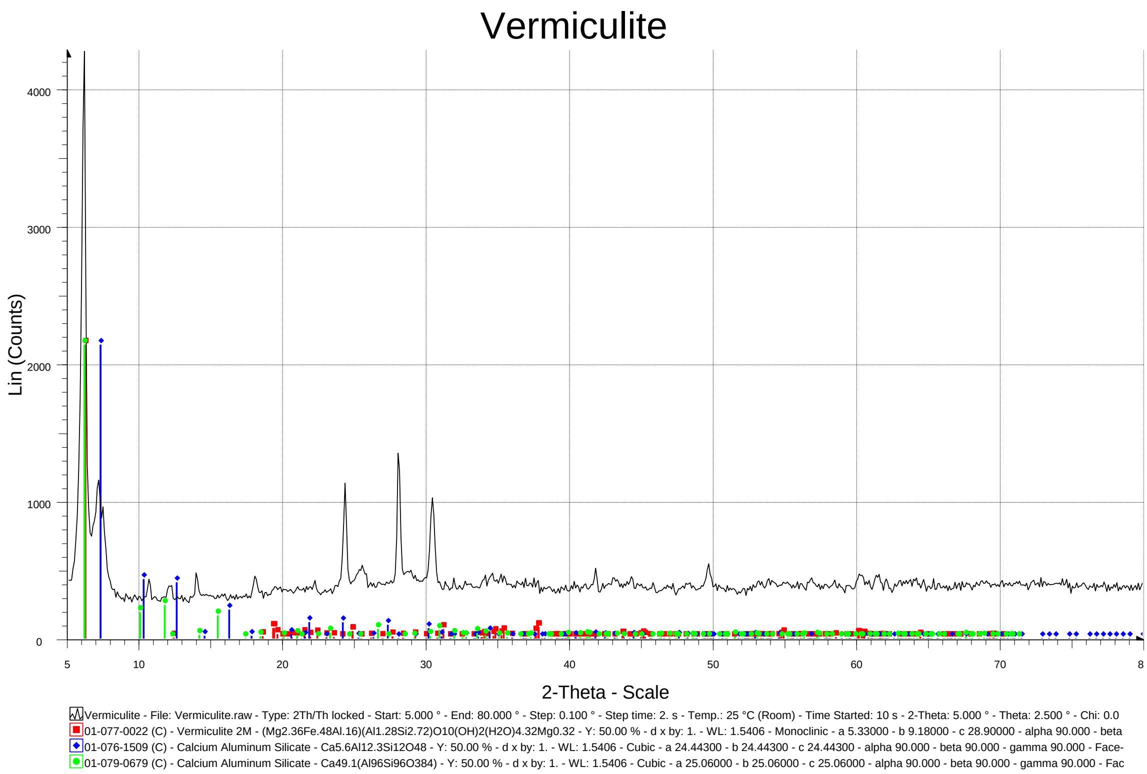

Figure 1 – The diffractogram of the vermiculite sample

Results. The Kulantau field (Turkestan region) vermiculite samples were used for physicochemical investigations.

The chemical composition of the vermiculite is shown in Table 1.

Table 1 – Chemical content of the vermiculite

Content of oxides, mass percent									
SiO_2	Al_2O_3	CaO	MgO	Fe_2O_3	FeO	Na_2O	K_2O	MnO	H_2O
32.5	11.4	2.6	16.6	11.5	1.7	5.5	5.6	8.1	4.6

The diffractogram of the vermiculite sample is shown in Figure 1.

As a result of the X-ray investigation of the vermiculite sample, the following main crystalline phases were identified:

- vermiculite $(Mg_{2.36}Fe_{0.48}Al_{0.16})(Al_{1.28}Si_{2.72})O_{10}(OH)_2(H_2O)_{4.32}Mg0_{0.32}$;
- calcium alumosilicates $Ca_{5.6}Al_{2.3}Si_{12}O_{48}$; $Ca_{49.1}(Al_{96}Si_{96}O_{384})$.

Figure 2 shows the derivatogram of the vermiculite sample.

As a result of the complex thermal investigation of the vermiculite sample, the following thermal effects were determined.

When analyzing the vermiculite's TG, DTG and DSC curves, it was found that when the vermiculite is heated, complex thermal transformations were observed: the 1st endo-effect is removal of moisture at a temperature of 90.7ºC with 6% mass loss; the 2nd endo-effect at 176.85°C is removal of moisture with 7.5% mass loss. At a temperature of 653.4°C there is a prolonged release of water (the 3rd effect) with a mass loss of up to 10%, another 0.5% of water is lost at 917°C (the 4th effect). The first two endo-effects are due to the loss of zeolitic water from large and small pores, the 3rd and the 4th effects are due to the loss of water present in the vermiculite in the form of structural OH groups, the removal of which leads to the zeolite structure destruction. The total water mass loss is 11.1%.

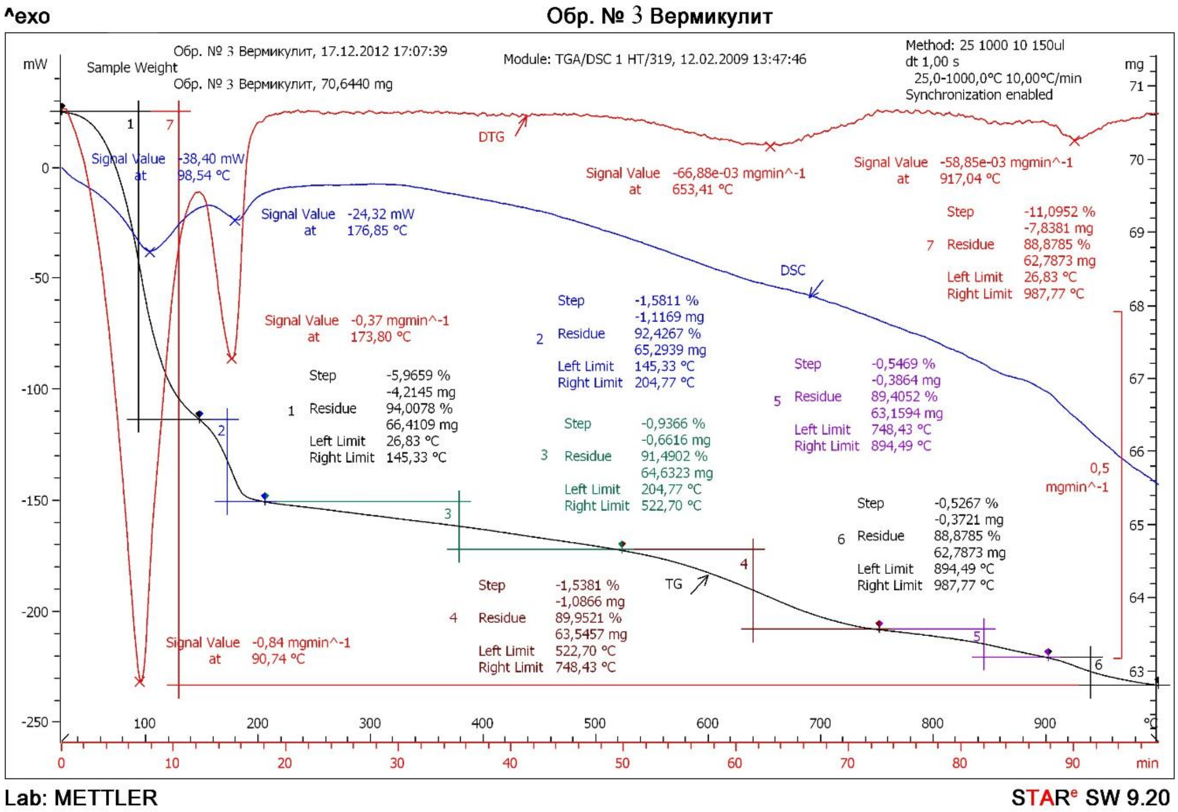

Figure 2 – The derivatogram of the vermiculite

The vermiculite's complex thermal analysis results allow to judge the physicochemical processes occurring in it when heated and associated with various states of water in the vermiculite.

The observed intense endothermic effect in the temperature range of 80-100°C is associated with the adsorption water loss.

The second endo-effect is caused by the release of water (interpacket), bound to exchangeable water ions, which is contained between the mica flakes and is strongly adsorbed on their cleavage planes. In this case, there is a significant samples' mass loss.

The stretched third endo-effect is characteristic of all micaceous materials, including vermiculite, and is associated with the removal of hydroxyl groups.

The results of the surface morphology's electron microscopic investigations and the vermiculite sample's energy dispersive analysis are shown in Figure 3.

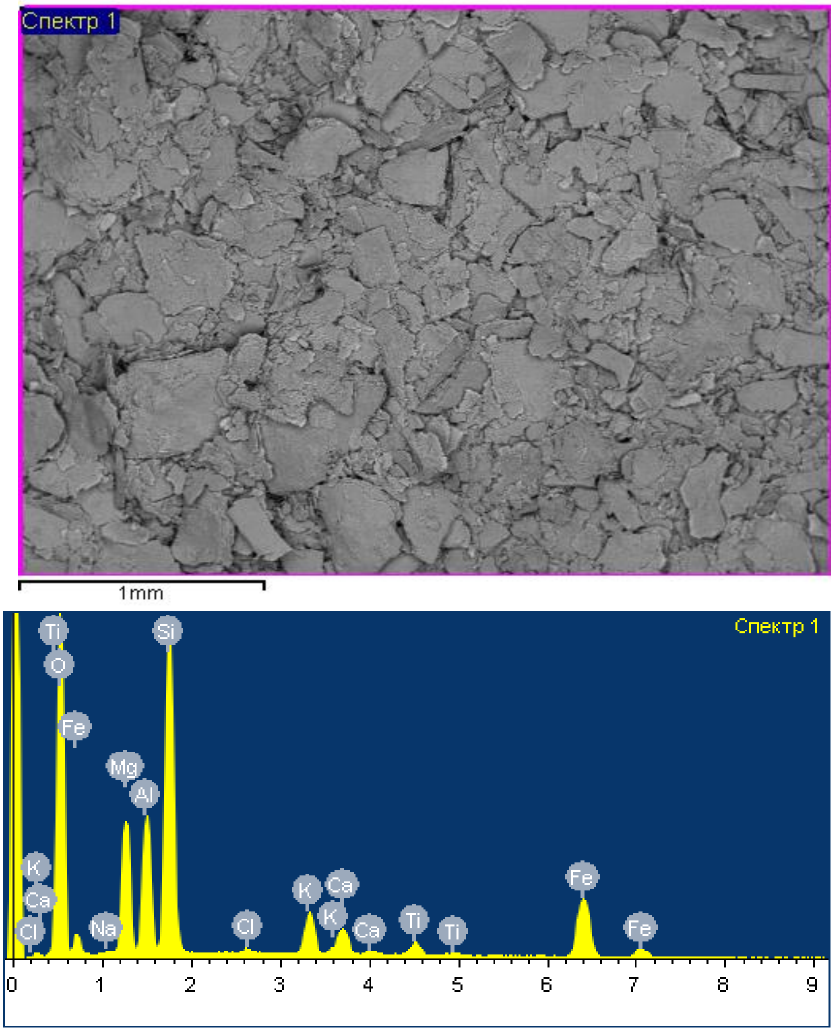

Figure 3 – The surface morphology and the elemental composition when scanning the vermiculite sample

The investigations of the elemental chemical composition (Table 2) showed that the vermiculite composition has a significant content of such elements as Si, Mg, Al, Ca, Fe, K, which characterizes its mineralogical composition.

Table 2 – The elemental chemical composition of the vermiculite

Content of elements, mass percent									
O	Na	Mg	Al	Si	Cl	K	Ca	Ti	Fe
45.89	0.11	8.63	7.82	17.42	0.27	3.04	2.12	1.42	13.28

Table 3 shows the main physical-technical properties of the expanded vermiculite.

Table 3 – The main physical-technical properties of the expanded vermiculite

Main physical-technical properties	Indicators
Loss on ignition, mass percent	1.2 – 11.8
Bulk density, fraction 0.6-5 mm, kg/m^3	100-150 kg/m^3 (Mark 150)
Porosity, including intergranular, %	84 – 95
Operating temperature, ºC	+1350

The expanded vermiculite has a number of valuable properties [15], among them the most important ones include the following:

- low density – 60-200 kg/m^3;
- low thermal conductivity coefficient – 0.048-0.06 W/m · degree (°C);
- high fire resistance, melting point > 1300°C;
- low coefficient of thermal expansion – 0.000014;

Currently, the actual scientific and technical challenge is to create new efficient types of heat-resistant materials, in particular heat-resistant concretes, the production of which saves deficit and expensive raw materials, reduces fuel and energy consumption and reduces labor costs [16].

Heat-resistant materials for thermal units operating at high temperatures should have high strength, low thermal conductivity, high heat resistance, not high cost, and a number of other performance indicators.

Analysis of published works showed that the use of mineral industrial wastes for production of composite binders can be no less effective than the use of specially manufactured technical products [17].

The choice of the expanded vermiculite as a microporous component of heat-resistant materials is due to its high thermal insulation properties and fire resistance, due to which the expanded vermiculite is used in fire-retardant materials.

Conclusion. As a result of the physicochemical investigations, it was established that according to the mineralogical composition, the vermiculite is represented mainly by magnesian aluminosilicates of complex composition.

The expanded vermiculite has such valuable properties as fire resistance, high melting point, low thermal conductivity, low bulk density.

Due to its unique properties, the expanded vermiculite can be effectively used as a microporous component for production of composite heat-resistant materials.

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ЫСТЫҚҚА ТӨЗІМДІ МАТЕРИАЛДАРҒА АРНАЛҒАН МИКРОКЕУЕКТІ КОМПОНЕНТІ – ВЕРМИКУЛИТТІ ФИЗИКА-ХИМИЯЛЫҚ ЗЕРТТЕУ

Аннотация. Түркістан облысы Құлантау кен орнының вермикулитінің физика-химиялық және физика-техникалық қасиеттерін зерттеу нәтижелері келтірілген. Химиялық, рентгенография, спектральды талдау әдістерімен вермикулиттің химиялық және минералогиялық құрамы, және оның негізгі кристалды фазалары күрделі құрамды магнийтемір және кальций алюмосиликаттары анықталады. Кешенді термиялық талдау әдісімен термиялық өңдеу кезінде вермикулиттегі физика-химиялық және құрылымдық өзгерістерді бағалауға мүмкіндік беретін процестері анықтады. Электронды микроскопия әдісімен вермикулиттің бөлшектер бетінің морфологиясы зерттелді. Ісінген вермикулиттың негізгі физика-техникалық қасиеттері анықталды. Ісінген вермикулит төмен тығыздығы мен төмен жылу өткізгіштікке ие, жоғары кеуекті, өртке қарсы және қиын балқу сипаттамалары бар екендігі анықталды. Бірегей қасиеттері арқасында ісінген вермикулит ыстыққа төзімді композитті материалдарды өндіру үшін микрокеуекті компонент ретінде тиімді пайдалануға болады.

Түйінді сөздер: вермикулит, физико-химиялық талдау әдістері, физика-техникалық қасиеттері, микрокеуекті компонент, ыстыққа төзімді композициялық материалдар.

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ФИЗИКО-ХИМИЧЕСКИЕ ИССЛЕДОВАНИЯ ВЕРМИКУЛИТА – МИКРОПОРИСТОГО КОМПОНЕНТА ДЛЯ ЖАРОСТОЙКИХ МАТЕРИАЛОВ

Аннотация. Приведены результаты исследований физико-химических и физико-технических свойств вермикулита Кулантауского месторождения Туркестанской области. Методами химического, рентгенографического, спектрального анализа определены химико-минералогический состав вермикулита и составляющие его основные кристаллические фазы, представленные магнийжелезистыми и кальциевыми алюмосиликатами сложного состава. Методом комплексного термического анализа установлены процессы, протекающие при нагревании, которые позволяют судить о физико-химических и структурных изменениях, происходящих в вермикулите при термической обработке. Методом электронной микроскопии исследована морфология поверхности частиц вермикулита. Определены основные физико-технические свойства вспученного вермикулита. Установлено, что вспученный вермикулит обладает низкой насыпной плотностью и низкой теплопроводностью, имеет высокие характеристики по пористости, огнестойкости, тугоплавкости. Вспученный вермикулит, благодаря своим уникальным свойствам, может эффективно применяться в качестве микропористого компонента для производства композиционных жаростойких материалов.

Ключевые слова: вермикулит, методы физико-химического анализа, физико-технические свойства, микропористый компонент, композиционные жаростойкие материалы.

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