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PHYSICOCHEMICAL CHARACTERISTICS AND CARBON DIOXIDE SORPTION PROPERTIES OF NATURAL ZEOLITES

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ABSTRACT

The physicochemical characteristics of natural zeolites from the Tayzhuzgen and Shankanay deposits have been studied by BET, SEM, XRD, and FT-IR spectroscopy. Sorption properties of natural zeolites for carbon dioxide were studied in the flow mode in the temperature range of 50-300 °C. Among the studied samples, the most active in the process of capturing CO_2 was the natural zeolite of the Tayzhuzgen deposit, at a temperature of 300 °C, the adsorption capacity for carbon dioxide was 27.4%. The high CO_2 adsorption capacity of natural zeolite from the Tayzhuzgen deposit is due to its specific surface area and high Si/Al ratio.

Keywords: CO_2 capture, natural zeolites, sorbents, carbon dioxide

1. Introduction

The growth of human resources, the high demand for fossil fuels due to the development of industry have led to an increase in anthropogenic emissions of carbon dioxide into the atmosphere. The largest sources of greenhouse gas emissions are primarily the combustion of coal, oil and natural gas. In this regard, many studies have been carried out and various approaches have been proposed to reduce carbon dioxide emissions. There are several technologies for capturing carbon dioxide, including physical absorption, chemical adsorption, membrane separation, etc. [1-3]. Among them, physical absorption and chemical adsorption are the most widely used methods in CO_2 capture. However, the physical absorption method is carried out at high pressure and low temperature, which is the main disadvantage of this method [4, 5].

One of the most effective ways to reduce the amount of carbon dioxide is its capture (adsorption), since this method is simple to perform and allows high CO_2 adsorption to be achieved [6, 7]. In addition, the regeneration of sorbents and the ease of temperature change during adsorption show the effectiveness of this method.

The literature contains adsorbents widely used to capture carbon dioxide, such as activated carbon [8], carbon materials [9] and mesoporous silica [10], zeolites [11-12], and other adsorbents [13]. Among these adsorbents, zeolites are effective sorbents in terms of textural and structural properties and high adsorption capacity in the process of carbon dioxide capture, as well as controlled physicochemical properties [14-15]. Various zeolite formulations, including synthetic zeolites and modified zeolites, have been studied in carbon dioxide capture. The cost of synthetic zeolite varies from 100 to $600 per ton, depending on its composition and processing, as well as its market price [16]. Natural zeolites are of great interest to researchers due to their absorbing properties of various substances. They have effective sorption qualities for capturing the greenhouse gas CO_2 and are also an alternative to synthetic sorbents due to their low cost, efficiency and availability. In this regard, natural zeolites are suitable for use in CO_2 adsorption processes and are universal sorbents in adsorption and catalysis processes.

Two large zeolite deposits are known in Kazakhstan – Tayzhuzgen, located in the Tarbagatay region of the East Kazakhstan region and Shankanay, located in the Kerbulak region of the Almaty region. The Tayzhuzgen deposit is larger than Shankanay, its

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reserves are 7.1 million tons. The natural zeolites of these deposits were used in agriculture, as additives in the food of animals and poultry, as well as to increase the yield of vegetables and grains [17, 18] in which the zeolites of the Tayzhuzgen and Shankanay deposits were studied as sorbents for the sorption of lead and cadmium ions from aqueous solutions.

In our work, natural zeolites from these deposits were first studied as sorbents for capturing carbon dioxide.

It is known that the flue gases of various enterprises mainly contain carbon dioxide, nitrogen, water vapor, etc. The flue gas released from the chimney has a high temperature, above or slightly below 300 °C [19]. In this regard, the purpose of this work is to study the sorption properties of natural zeolites of the Tayzhuzgen and Shankanay deposits in terms of carbon dioxide at high temperatures and their physicochemical characteristics.

2. Experimental part

The objects of study are natural zeolites of the Tayzhuzgen and Shankanay deposits, which are designated in the article as zeolite (T) and zeolite (Sh), respectively. Physico-chemical characteristics of natural zeolites were studied by X-ray phase analysis, scanning electron microscopy, Brunauer-Emmett-Taylor (BET) and FT-IR. The surface area of the studied samples was determined by the method of low-temperature nitrogen adsorption at a temperature of -196 °C on an automatic BEL Japan Inc. and thermal desorption of argon on a BELSORP-mini II instrument. Zeolite morphology was studied by scanning electron microscopy (SEM) on a JEOL JSM-6390 LA instrument with a JED 2300 energy dispersive X-ray detector radiation with CuKα radiation and a graphite monochromator on a diffracted beam.

Natural zeolites have been tested as sorbents for capturing carbon dioxide in the temperature range of 50-300 °C. Before testing, natural zeolites were mechanically crushed in a mortar and then sieved on a molecular sieve to a particle size of 0.25-0.5 mm. A small amount of water was added to the obtained samples of natural zeolites, the resulting paste was placed on a mini-form (Fig. 1), which produces granules in one technological cycle of exactly the same shape (cylindrical, Ø2.7 mm, h = 2.7 mm). The paste was calcined in a muffle furnace at a temperature of 600 °C for 5 h at a rate of 2 °C per minute.

The sorption properties of natural zeolites were studied in a laboratory flow unit (Scheme 1). The installation consists of a reactor, a thermocouple, an electric furnace, a control unit and a control unit for supplying initial gases. For experiments, CO_2 was used as an adsorbate (Ikhsan Technogaz LLP, purity 99.99%), as well as He carrier gas for purging the line from the reactor to the chromatograph (Ikhsan Technogaz LLP, purity 99.99%). The reactor was made of quartz glass with an inner diameter of 10mm and a length of 25 cm. The sorbent was weighed in a volume of 2 ml and placed in a fixed bed reactor. Adsorption of carbon dioxide on natural zeolites (T) and (Sh) was carried out in the temperature range of 50-300 °C for 30 min, desorption – at a temperature of 600 °C.

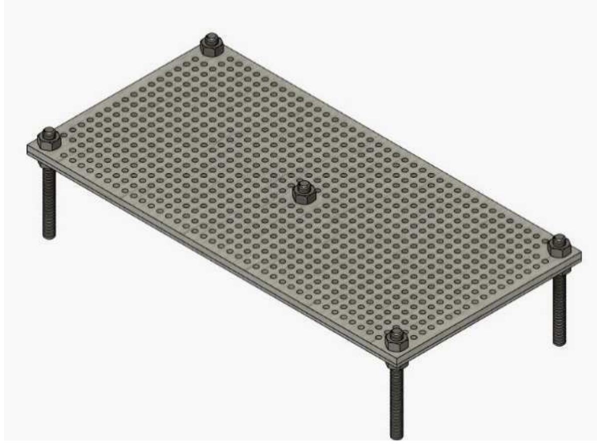


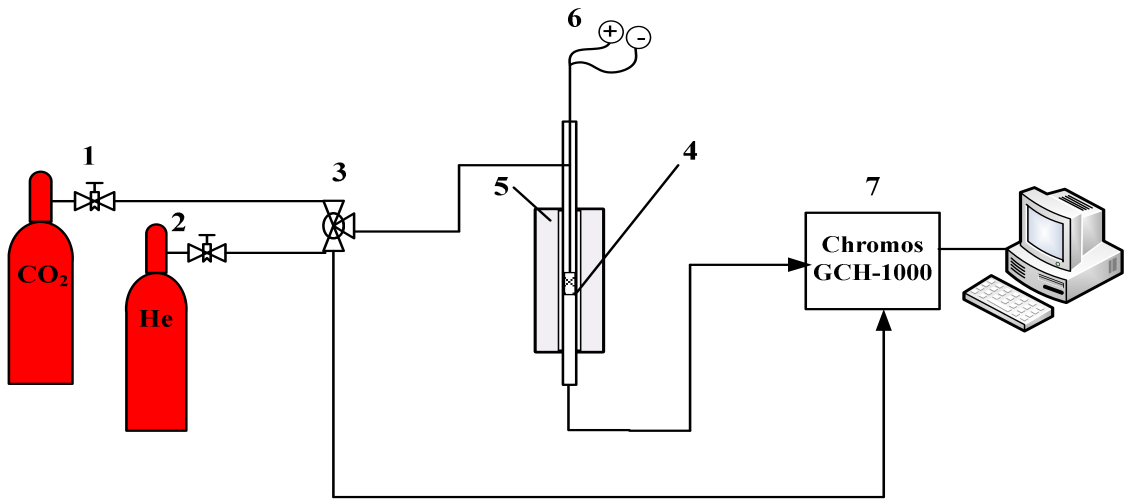
Fig. 1. Mini-form for obtaining zeolites in the form of granules.

3. Results and discussion

The elemental composition of natural zeolites of the Tayzhuzgen and Shankanay deposits, expressed in the form of oxides, was determined using X-ray fluorescence spectroscopy, the results are presented in Tables 1 and 2. The elemental composition of the zeolite of the Tayzhuzgen deposit is presented in a number of works by Kazakh scientists [20, 21], the elemental composition of our sample differs slightly from these data.

The chemical composition of the zeolite of the Tayzhuzgen deposit contains mainly oxides of silicon (72.2%), aluminum (12.9%), calcium (5.6%), zinc (1.2%), indium (7.7%), and iron (0.4%). The zeolite of the Shankanay deposit contains 16.9% aluminum oxide, 49.5% silicon oxide, 15.2% calcium oxide, 14.7% iron oxide, and a small amount of potassium oxide (3.7%).

It is known [22] that an important factor in the study of the structure of zeolites is the ratio of silicon oxide and aluminum (Si/Al). High-silicon zeolites



Scheme 1. Schematic diagram of a flow type installation: 1 – source gas CO_2; 2 – carrier gas helium; 3 – gas mixer; 4 – sorbent; 5 – oven; 6 – thermocouple; 7 – chromatograph.

Table 1

Chemical composition of zeolite (T)

Compound	Al_2O_3	SiO_2	CaO	ZnO	In_2O_3	Fe_2O_3
Concentration, wt.%	12.9	72.2	5.6	1.2	7.7	0.4

Table 2

Chemical composition of zeolite (Sh)

Compound	Al_2O_3	SiO_2	CaO	K_2O	Fe_2O_3
Concentration, wt.%	16.9	49.5	15.2	3.7	14.7

with a high Si/Al ratio have the maximum absorption effect. According to the data presented in Tables 1 and 2, the Si/Al ratio for the zeolite of the Tayzhuzgen deposit is 5.6, while for the zeolite of the Shankanay deposit it is 2.9.

The specific surface area of the samples was estimated by the low temperature nitrogen adsorption (BET) method (Table 3).

As can be seen from Table 3, the specific surface of the natural zeolite of the Tayzhuzgen deposit is 11.12 m^2/g, the specific surface of the zeolite of the Shankanay deposit is 5.61 m^2/g.

To obtain data on the morphology of the catalyst particles, the samples were analyzed using scanning electron microscopy. Figure 2 shows SEM micrographs of natural zeolites.

Table 3

Specific surface area of natural zeolites

Samples	Specific surface, m^2/g
Zeolite (Sh)	5.61
Zeolite (T)	11.12

It is known [23] that zeolites mainly have a complex microsurface relief formed by microcrystals and aggregates, represented in most cases by a finely dispersed mass. On the micrographs of samples of natural zeolites from the Tayzhuzgen and Shankanay deposits, one can observe accumulations of crystals of a monometric shape. A finely dispersed crystalline structure is observed on the surfaces of zeolite samples.

The phase composition of natural zeolites (T) and (Sh) was studied by X-ray phase analysis, the results are shown in Figure 3.

On the diffraction patterns of zeolites, there is practically no amorphous background and broadening of peak lines, which indicates a high degree of crystallinity and homogeneity of the sample. According to XRD results, zeolites from the Tayzhuzgen and Shankanay deposits are classified as clinoptilolite aluminosilicates.

Zeolites from the Tayzhuzgen and Shankanay deposits were also evaluated using FT-IR spectroscopy. FT-IR spectra of samples of natural a)

b)

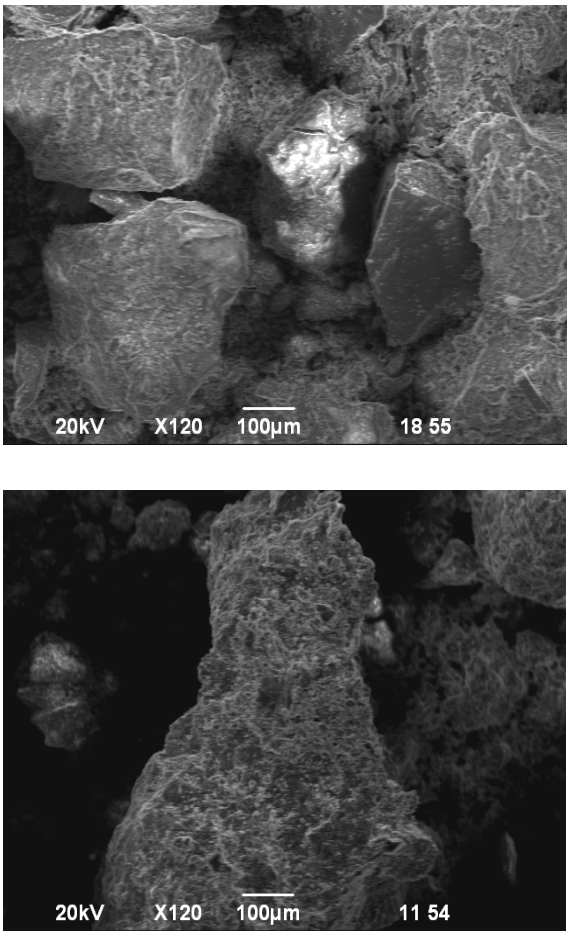


Fig. 2. SEM micrographs: a) zeolite (T) and b) zeolite (Sh).

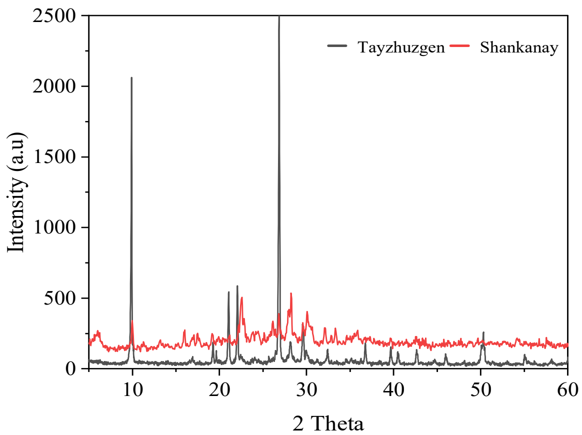


Fig. 3. X-ray patterns of natural zeolites (T) and (Sh).

zeolites are shown in Fig. 4. FT-IR spectra of natural zeolites are in the range of 500-4500 cm^{-1}.

The spectra of both zeolites show intense peak at 1028 cm^{-1}, which correspond to internal deformation and antisymmetric vibrations of Si–O bonds inside SiO_4 tetrahedra. In addition, absorption bands are observed in the region of 3800-3400 cm^{-1}, which correspond to symmetric and antisymmetric O–H

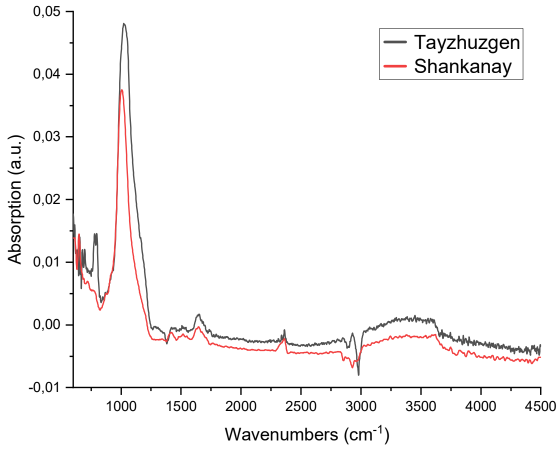


Fig. 4. FT-IR zeolites (T) and (Sh).

stretching vibrations. The bands at 613 cm^{-1} and 700 cm^{-1} indicate the presence of the Al-O bond, the bands at 801.35 cm^{-1} and 1649 cm^{-1} can be related to the bending vibration of the $Al\text{-}O_4$ bond [24].

The CO_2 sorption properties of natural zeolites from the Tayzhuzgen and Shankanay deposits were tested under dynamic conditions in a flow mode. Carbon dioxide adsorption was carried out at various temperatures of 50, 100, 150, and 300 °C, CO_2 desorption at 600 °C. The results obtained are shown in Figure 5.

As can be seen from Figure 5, the CO_2 sorption capacity of natural zeolites (T) and (Sh) increases with adsorption temperature. The sorption capacity of the zeolite from the Tayzhuzgen deposit increases from 22 to 27.4%, from the Shankanay deposit from 12.4 to 18.3%. The natural zeolite of the Tayzhuzgen deposit demonstrates a higher sorption capacity for CO_2 in the entire temperature range compared to the zeolite of the Shankanay deposit.

4. Conclusion

Thus, the physicochemical characteristics and CO_2 adsorption properties of natural zeolites from the Tayzhuzgen and Shankanay deposits were studied.

The specific surface area, morphology, chemical and phase composition of zeolites have been studied. It was determined that the specific surface of the natural zeolite of the Shankanay deposit is 5.61 m^2/g, of the Tayzhuzgen natural zeolite – 11.12 m^2/g. A finely dispersed crystalline structure is observed on the surfaces of zeolite samples. It is shown that the zeolites of the Tayzhuzgen and Shankanay deposits belong to clinoptilolite aluminosilicates. The molar ratio of SiO_2/Al_2O_3 for the zeolite of the Shankanay

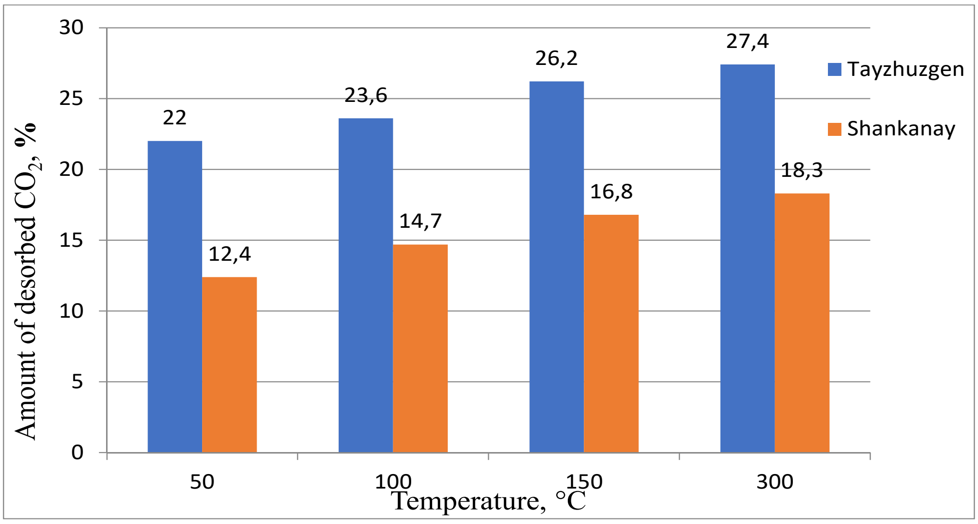


Fig. 5. Sorption capacity for CO_2 of natural zeolites (T) and (Sh).

deposit is 2.9, while for the Tayzhuzgen zeolite it is 5.6. It has been established that the zeolite of the Tayzhuzgen deposit has a better sorption capacity compared to the zeolite of the Shankanay deposit. The sorption capacity for CO_2 of the zeolite of the Tayzhuzgen deposit at a temperature of 300 °C is 27.4%, while the sorption capacity of the zeolite of the Shankanay deposit is 18.3%. The high sorption capacity of the zeolite from the Tayzhuzgen deposit is due to its specific surface area and high SiO_2/Al_2O_3 ratio.

The results of the study showed that the zeolite of the Tayzhuzgen deposit can be used as a sorbent for capturing CO_2 at high temperatures. Further research will be aimed at increasing the sorption capacity of natural zeolite by modifying it with oxides of alkali and transition metals.

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Физико-химические характеристики и сорбционные свойства природных цеолитов по диоксиду углерода

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АННОТАЦИЯ

Физико-химические характеристики природных цеолитов Тайжузгенского и Шанканайского месторождения исследованы методами БЭТ, СЭМ, РФА и ИК-Фурье спектроскопией. Сорбционные свойства природных цеолитов по диоксиду углерода исследованы в проточном режиме в интервале температур 50-300 °С. Среди исследованных образцов наиболее активным в процессе улавливания CO_2 оказался природный цеолит Тайжузгенского месторождения, при температуре 300 °С адсорбционная емкость по углекислому газу составила 27,4%. Высокая адсорбционная емкость по CO_2 природного цеолита Тайжузгенского месторождения обусловлена его удельной поверхностью и высоким соотношением Si/Al.
Ключевые слова: улавливание CO_2, природные цеолиты, сорбенты, диоксид углерода.

Табиғи цеолиттердің физика-химиялық сипаттамалары және олардың көмірқышқыл газы бойынша сорбциялық қасиеттері

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АҢДАТПА

Тайжүзген және Шаңқанай кен орындарының табиғи цеолиттерінің физика-химиялық сипаттамалары БЭТ, СЭМ, РҚТ және ИҚ-Фурье спектроскопия әдістерімен зерттелді. Табиғи цеолиттердің көмірқышқыл газы бойынша сорбциялық қасиеттері 50-300 °С температура диапазонында ағындық режимде зерттелді. Зерттелген үлгілердің ішінде CO_2 сорбциялау процесінде Тайжузген кен орнының табиғи цеолиті ең белсенді болды, 300 °С температурада көмірқышқыл газы бойынша адсорбциялық сыйымдылығы 27,4% құрады. Тайжүзген кен орнының табиғи цеолитінің CO_2 бойынша жоғары адсорбциялық сыйымдылығы оның меншікті бетінің ауданы мен Si/Al қатынасының жоғары болуына байланысты.
Түйін сөздер: CO_2 ұстау, табиғи цеолиттер, сорбенттер, көмірқышқыл газы.