

**OPTIMIZATION OF THE PROCESS FOR OBTAINING L-TYPE ZEOLITES FROM
ANGREN KAOLIN RAW MATERIAL****Turabov Bahodir Alikul ugli**

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Abstract

The article presents the possibility of obtaining microporous adsorbents using Angren kaolin. Structural changes in kaolin during extraction with an adsorbent were also studied. A scheme for obtaining L-sorbents is presented.

Keywords

Angren kaolin, metakaolin, adsorbent, aluminosilicate.

INTRODUCTION.

The structure of adsorbents is a series of structural and porous-textured with features such as pores, voids and channels of various sizes availability (from ultramicro to mesoporous), availability of exchange cations and different substances active centers on the surface, exfoliation into individual layers [1]. The crystal size and morphology depend on the composition of the starting material (water, alkali or oxide ratio), as well as the heating. The adsorption capacity of L adsorbents is controlled by adjusting the conditions (heating rate and temperature). Disk-shaped L adsorbents with sizes ranging from nanometers to micrometers have already been reported [2, 7].

The authors also tested adsorbent synthesis using microwave heating. Today, microwave heating is a method used in modern organic synthesis and production of nanoparticles and nanostructures. This method generally helps to reduce energy consumption, synthesis process productivity, reusability and environmental damage [3, 4]. The main characteristics of heating with a microwave oven is that by controlling the temperature accuracy, it is possible to ensure a uniform distribution of heat. This prevents temperature differences between the oven and the samples.

The above method aims to characterize the hydrothermal synthesis of adsorbent L by taking some of the optimal conditions reported in previous literature for conventional furnaces and applying them to microwave heating [2]. The effect of reaction conditions (heating rate, time, temperature and various conditions) on the size, structure and chemical properties of the formed crystals was studied. Thus, crystals of adsorbent L [5] were synthesized using the gel composition and the above-mentioned reaction conditions.

MATERIALS AND METHODS

Typically, the synthesis of adsorbents in the laboratory is carried out by hydrothermal means. In this approach, a silicon source, an aluminum source, and a hydroxide source are combined with water in a closed system and heated under autogenous pressure (up to 50-250°C). The hydrothermal reaction is characterized by the stepwise transformation of an amorphous aluminosilicate mixture and a crystalline aluminosilicate mixture into an adsorbent. This process lasts from several hours to several days [6].

RESULTS AND DISCUSSION.

Amorphous silica-alumina nanoporous materials are used in the chemical and petrochemical industries as carriers for metal and oxide systems. Their use in heterogeneous catalysis, like that of adsorbents, is associated with the presence of acid-base properties, which in

turn have a strong influence on the activity and selectivity of the catalyst. The sol-gel method usually produces disordered amorphous aluminosilicates or mixed oxide systems, depending on the degree of incorporation of aluminum into the silicate structure and the formation of the composite structure. In the literature, catalysts and ceramics, glasses and there are many publications on the sol-gel synthesis of aluminosilicates for the production of membranes. It has been proven that in the sol-gel synthesis of aluminosilicate gels, such as silanes, aluminum nitrate or aluminum chloride, it is possible to use kaolin in addition to traditional sources of aluminum and silicon. SiO₂-Al₂O₃ the use of aluminum alkoxides in the synthesis of aluminum is limited by the large difference in the activity of aluminum and silicon alkoxides. The amount of water required for the slow hydrolysis of tetraethoxysilane is sufficient for the rapid precipitation of aluminum hydroxide, and the two separate phases of aluminum and silicon oxides condense without the formation of Si-O-Al heterobonds. This problem can be solved in several ways. In the first case, the hydrolysis rate of the entire system is slowed down by the use of a minimum amount of water or lower temperatures. Agents that slow down the hydrolysis of aluminum, for example, 2-methoxyethanol or a complex chelating agent, acetylacetone, which leads to the formation of aluminum diketonates, are also used.

Angren kaolin aluminosilicate available in the Republic in research work was selected as the source. Its composition is as follows (Table 1):

Table 1.

Chemical composition of Angren kaolin

Substance	Al ₂ O ₃	CaO	Fe ₃ O ₄	K ₂ O	MgO	Na ₂ O	P ₂ O ₅	SiO ₂	TiO	Other substances
%	35,1	1,24	0,8	0,283	0,723	4,69	0,07	48,9	0,605	7,589

The kaolin composition was purified from additional compounds that negatively affect the synthesis of the adsorbent. In this case, in the first stage, first a 2% solution of sulfuric acid was used, and then 5% solutions of hydrochloric acid. This process was carried out by heating at a temperature of 200°C. In the second stage, 3% sodium hydroxide was used. solution was carried out at a temperature of 300°C (Table 2).

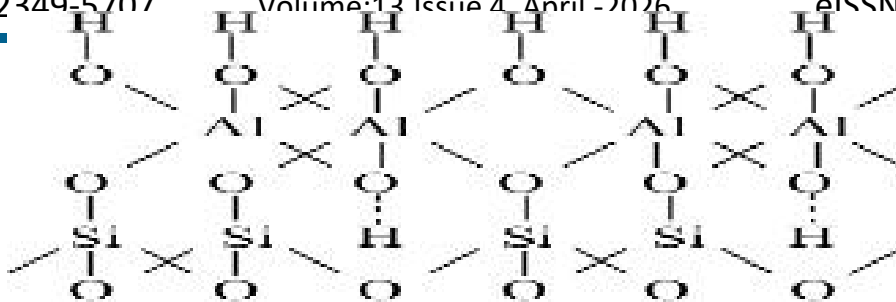
Table 2.

Purified kaolin composition

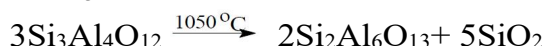
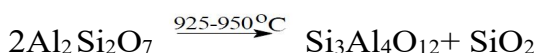
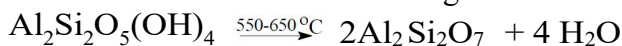
Substance	Al ₂ O ₃	Na ₂ O	SiO ₂	Fe ₃ O ₄	Other substances
%	38,4	5,5	54,6	0,1	1,4

When heated in air, kaolin undergoes several stages of transformation. First, 550- at 650°C, endothermic dehydration produces a disordered (amorphous) phase of metakaolin (aluminum disilicate). The structure of metakaolin is a mixture of amorphous silicon and aluminum oxides. Metakaolin is thermally stable up to 925°C. At higher temperatures, it transforms from an amorphous state to a crystalline state, i.e., a defective aluminosilicate spinel. Increasing the heating temperature to 1050°C leads to the transformation of spinel into mullite and cristobalite. Kaolin crystal is a natural aluminosilicate in which the molar ratio of silicon oxide to aluminum oxide is 1.0 - 1.15. Kaolin has a layered structure formed by repeated layers of silicon- oxygen tetrahedra and aluminum-oxygen octahedra (Figure 1)

Figure 1. Kaolin structure.



The thermal transformation diagram of kaolin is given below;



Studying the adsorption properties of adsorbents provides useful information on structural characterization and practical application possibilities. Adsorption measurements can provide information on various factors related to the structure of a given adsorbent (e.g., channel dimensions, pore volume, cation arrangement, etc.).

Of particular interest is the study of the adsorption of various substances on L-type adsorbents. The adsorption of these adsorbents depends on the type and number of cations present in the structure. Due to the large space between the adsorption sites, the adsorbent is convenient for modeling adsorption behavior. L grade purification of gases from harmful compounds, separation of substances mainly from adsorbents used for the maximum dimensions of channels and cavities in adsorbents are 1-1.5 nm can reach Negative charges of aluminum-oxygen tetrahedra of the framework Alkaline and alkaline earth metal cations cover this crystal lattice located in the spaces, they occupy a relatively small part of the volume. Hydrothermal crystallization of aluminosilicate gels of adsorbents (on the basis of resulting from mixing solutions of aluminum and silicon oxides).

Adsorbents made from natural kaolin have some difficulties in synthesis due to the presence of foreign elements such as Fe, Ti, Mn, Ca, Mg, and Ni in the kaolin composition. occurs. Because these elements can negatively affect the hardness and catalytic properties of the adsorbent, as well as the production efficiency.

Kaolin is usually used after treatment at temperatures ranging from 500-900°C. When treated, metakaolin contains more reactive phases, and less of the AlO₆ octahedral structure is retained due to water loss. As a result of the research, NaXL adsorbent was synthesized from local natural kaolin.

The synthesis steps of an aluminosilicate-based material (NaX type zeolite) are shown. γ-Al₂O₃ is obtained from technical alumina and metakaolin from kaolin, which are mixed with a 12% NaOH solution to form an aluminosilicate mixture. The mixture is stirred at a ratio of 1:2 at 60°C for 24 hours to form a gel. The resulting mass is washed in distilled water, filtered and dried at 75–120°C. Then the material is hydrothermally treated in an autoclave at 140–250°C for 48 hours. As a result, a crystalline NaX type zeolite is obtained. For this purpose, in the initial stage, kaolin was treated at temperatures up to 900°C and metakaolin was obtained. Metakaolin was mixed with NaOH solution and γ - Al₂O₃ solution to obtain a molar ratio of components – (SiO₂/Al₂O₃)=3:1 . Compounds with ratios (NaOH/Al₂O₃)=1.1:1 and (H₂O/NaOH)=8:1 were used for the reaction mixture . The obtained sample was heated in a closed vessel and under autogenous pressure for 24 hours at a temperature was processed. The adsorbent NaXL solution was obtained from activated kaolin by mechanical and chemical processing. To neutralize the resulting solution medium the solution was rinsed in distilled water and dried at 120°C for 48 hours.

CONCLUSION

The raw material base for obtaining microporous adsorbents with silicon and aluminum oxide using local Angren kaolin was studied. Methods for obtaining microporous adsorbents using enriched AKF-78 Angren kaolin and alumina were selected. The use of the hydrothermal method for obtaining microporous adsorbents is a favorable condition for bonding silicon and aluminum together.

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