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CRYSTALLINE STRUCTURE AND SPECTROSCOPIC ANALYSIS OF LIA ZEOLITE

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Abstract: In this study, Na-A type zeolite with high crystallinity was synthesized hydrothermally from $NaAlO_2$ and Na_2SiO_3 precursors. The obtained Na-A zeolite was converted into Li-A zeolite through stepwise ion exchange using LiCl solution. During the three-stage exchange process, Na^+ ions were effectively replaced by Li^+ ions while maintaining the structural stability of the zeolite framework. The resulting products were thoroughly characterized by X-ray diffraction (XRD) and infrared (IR) spectroscopy. XRD results confirmed that Li-A zeolite retained high crystallinity and predominantly formed lithium aluminosilicate phase. IR spectroscopy revealed shifts in Si–O–Al and Si–O–Si vibrations, indicating the incorporation of Li^+ ions into the framework cavities. The final Li-A zeolite exhibited high ion-exchange capacity, stable morphology, and selective adsorption properties, making it a promising material for gas separation, catalysis, and electrochemical applications.

Keywords: Na-A zeolite, Li-A zeolite, ion exchange, XRD analysis, IR spectroscopy, crystal structure, selective adsorption, catalysis.

Introduction. LiA type zeolites are crystalline aluminosilicate substances with a three-dimensional spatial lattice consisting of large cavities and channels. Due to this unique structure, they possess ion-exchange, adsorption, and catalytic properties. Na-A zeolites are widely used in water softening, gas separation, and catalysis processes. In the petrochemical industry, they are used as effective catalysts in processes such as isomerization, cracking, and hydrocarbon synthesis [1-4]. On an industrial scale, the most effective synthesis method is the hydrothermal approach, which is usually carried out at temperatures below 300 °C and under autogenous pressure in autoclave conditions [5-6].

Literature analysis.

In recent years, due to the factors of environmental sustainability and economic efficiency, the use of cheap and local raw materials in zeolite synthesis has generated great interest on a global scale. While the synthesis of Na-A zeolite based on NaAlO2 and liquid glass provides high crystallinity and perfect morphology, the use of local resources reduces production costs and allows for the development of waste-free technologies [7-8]. In the process of hydrothermal synthesis, NaOH or KOH is used as a mineralizing agent, and cations of alkali and alkaline earth metals act as a structural guiding agent. An increase in the pH level of the mixture contributes to the faster dissolution of amorphous aluminosilicates, leading to the formation of silicate and aluminate lattices at high concentrations. These conditions stimulate rapid nucleation and crystal growth, facilitating the formation of an ordered crystal structure.

Crystallization time is one of the most important parameters of the synthesis process, and if sufficient time is not provided, an amorphous product may form [9-10].

In the synthesis of Na-A type zeolites, the initial composition, temperature, pressure, and crystallization time directly affect the degree of crystallinity, particle morphology, and size of the product [11-13]. At the same time, Li-A zeolite can be obtained by replacing Na+ ions with Li+ ions using LiCl salt during ion exchange. This approach expands the selective adsorption properties of zeolite and makes it a promising material for energy storage, gas separation, and electrochemical processes [14-15].

Today, about 20 different types of zeolites are produced on an industrial scale. The global annual demand for zeolites is estimated at 1.7-2 million tons, of which a significant portion is Na-A type zeolites [16]. This indicator is explained by the high efficiency of Na-A zeolite in ion exchange, adsorption, and catalytic processes. Li-A zeolite, obtained by ion exchange with the help of LiCl, opens up new possibilities of application due to its high selectivity and stability.

The main goal of this research is an in-depth study of the process of synthesizing Na-A zeolite based on NaAlO2 and liquid glass, as well as a scientific substantiation of the possibility of obtaining Li-A zeolite through ion exchange using LiCl salt. The results determine new technological approaches in the field of zeolite synthesis and open up important prospects for industrial application.

Result and discussion.

Na-A type zeolites are characterized by high ion exchange capacity and a perfect crystalline structure. They are widely used in water softening, gas separation, and catalysis processes. By replacing Na+ ions with Li+ ions during ion exchange, it is possible to obtain Li-A zeolite, which is a promising material for selective adsorption and electrochemical application.

Na-A zeolite synthesis.

The synthesis of Na-A zeolite was carried out by gel formation from NaAlO2 and Na2SiO3 solutions. A NaAlO2 solution was prepared in an amount of 23.8 mmol, and Na2SiO3 was added to the solution in an amount of 52.1 mmol. To ensure a Si/Al ratio of ≈ 1, a NaAlO2 solution was gradually added to the Na2SiO3 solution and stirred at 40°C for 2 hours. The resulting gel was crystallized under hydrothermal conditions at 120°C for 24 hours. The crystalline solid phase was washed (pH ≈ 7) and dried at 100 °C for 6 hours. As a result, Na-A zeolite with a high degree of crystallinity was obtained.

Li-A zeolite synthesis (through ion exchange)

The ion-exchange capacity of zeolite Na-A was tested using a LiCl solution. A LiCl solution was prepared in an amount of 58.8 mmol and had a concentration of 1 M. 20 mmol of Li+ ions were used for each exchange cycle.

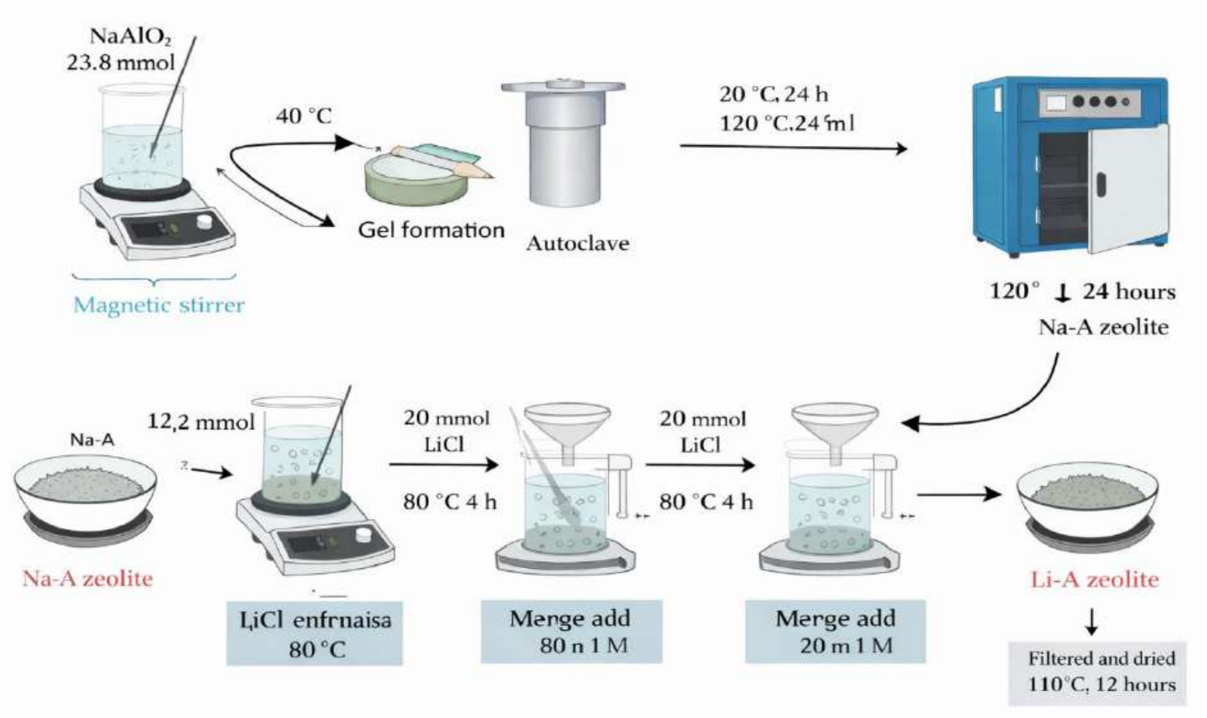

Figure 1. Synthesis scheme of zeolite LiA obtained by the hydrothermal method

- First substitution: 1 g of Na-A zeolite containing 12.2 mmol of Na+ ions was added to a solution containing 20 mmol of Li+ ions and stirred at 80°C for 6 hours. Most Na+ ions were replaced by Li+ ions.
- Second substitution: The washed zeolite was added to another 20 mmol of Li+ ion solution and stirred at 80°C for 4 hours. The ion exchange process continued, but the rate decreased slightly.
- Third substitution: Zeolite was again added to 20 mmol of Li+ ion solution and stirred at 80°C for 4 hours. In the final stage, the ion exchange centers were filled, and the process slowed down.

The final product was dried at 110 °C for 12 hours (Fig. 1).

In the process of ion exchange, Na+ ions were gradually replaced by Li+ ions. In the first cycle, ion exchange proceeded quickly and efficiently, while in the second and third cycles the process slowed down, which is explained by the filling of ion exchange centers in the zeolite framework. The high crystallinity of Na-A zeolite accelerated the ion exchange process and ensured the effective placement of Li+ ions in the framework. The final Li-A zeolite was highly enriched with Li+ ions, and the crystalline framework was preserved. Na-A zeolite, synthesized on the basis of NaAlO2 and Na2SiO3, was successfully converted into Li-A zeolite through stepwise ion exchange using a LiCl solution. The three-stage exchange process ensured the effective placement of Li+ ions in the zeolite framework.

The final Li-A zeolite, characterized by high crystallinity, stable morphology, and selective ion exchange properties, is recommended as a promising adsorbent in the processes of gas separation, catalysis, and energy storage.

The Na-A zeolite sample, synthesized on the basis of NaAlO2 and liquid glass, was converted into Li-A zeolite through stepwise ion exchange using a LiCl solution. The obtained products were thoroughly analyzed in terms of structure, morphology, and adsorption.

To determine the crystalline structure, X-ray phase analysis (XRD) was performed using the SHIMADZU XRD-6100 diffractometer. Measurements were carried out under the conditions of a Cu-Kα radiation source ($\lambda = 1.5406$ Å), a voltage of 40 kV, and a current of 30 mA. The diffraction peaks confirmed the phase corresponding to the LTA type zeolite. After ion exchange, the degree of crystallinity in the Li-A zeolite was preserved, which indicates structural stability during the penetration of Li+ ions into the zeolite framework.

IR spectroscopic analysis recorded vibrations characteristic of Si-O-Al, Si-O-Si, and sodalite cells. Intense and distinct peaks were observed in Na-A zeolite, while in Li-A zeolite, the peaks were slightly shifted, reflecting a change in the equilibrium in the framework as a result of ion exchange.

Li-A zeolite, obtained using LiCl from Na-A zeolite, synthesized on the basis of NaAlO2 and liquid glass, is characterized by high crystallinity, a stable morphology, and a developed porous structure. XRD, IR, and BET analyses scientifically substantiated that Li-A zeolite is a promising adsorbent in industrial-scale gas separation, catalysis, and ion exchange processes.

Results and their analysis. RRD Analysis. The Na-A zeolite sample, synthesized on the basis of NaAlO2 and liquid glass, was converted into Li-A zeolite through stepwise ion exchange using a LiCl solution. The crystalline structure of the obtained product was studied in depth using X-ray phase analysis (XRD).

The analysis was carried out using a MiniFlex 300/600 goniometer and a D/teX Ultra2 detector under the conditions of a Cu-Kα radiation source ($\lambda = 1.5406$ Å). The measurement parameters were determined as follows: voltage 40 kV, current 15 mA, scanning range 5-70° (2θ), step 0.02°, speed 10°/min. These conditions made it possible to record diffraction peaks with high accuracy. (WPPF) determined the phase composition of Li-A zeolite:

Lithium aluminosilicate - 47.1%
Li element (in ionic form) - 25.0%
Styshovitis - 2.51%
Aluminum oxide (Al2O3) - 3.0%
Aluminum silicate - 0.104%

Main phase: Lithium aluminosilicate (≈ 47%) indicates the formation of Li-A zeolite as the dominant phase after ion exchange. This confirms the deep penetration of Li+ ions into the framework and the preservation of a stable crystalline structure of the zeolite.

A high proportion of Li+ ions: the presence of a Li phase of about 25% indicates the effectiveness of ion exchange. This means that most of the Na+ ions are replaced by Li+ ions, and the ion exchange centers of the zeolite are fully utilized. Side phases: small phases, such as Styshovit (2.5%) and Al2O3 (3%), were recorded as secondary products formed during the synthesis process. Due to their low content, they do not significantly affect the main adsorption and ion-exchange properties of the zeolite. Aluminosilicate residues: Al-silicate phase, recorded in the amount of 0.1%, refers to amorphous residues that have not fully crystallized during the synthesis process (Fig. 2).

Diffraction peaks: the XRD profile showed sharp peaks corresponding to LTA type zeolite. The shift of peaks after ion exchange indicates a change in the coordination equilibrium of Li+ ions in the framework. XRD analysis confirmed the high degree of crystallinity of zeolite Li-A, the formation of lithium aluminosilicate as the main phase, and the effective placement of Li+ ions in the framework. The phase composition showed the complete implementation of the ion exchange process and scientifically substantiated the high efficiency of Li-A zeolite in selective adsorption, gas separation, and electrochemical applications.

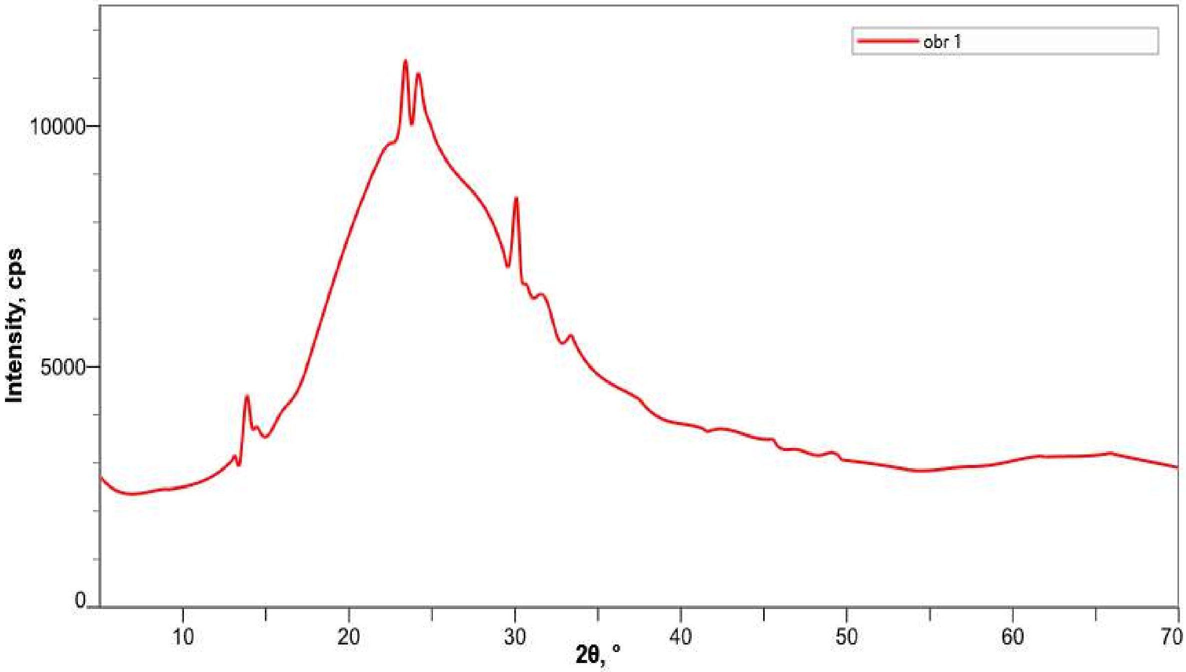

Figure 2. XRD image of synthesized LiA and zeolite crystals

IR analysis. Infrared (IR) spectroscopy is one of the most important analytical methods for studying the crystalline structure, ion-exchange properties, and degree of hydration of LiA-type zeolites. With the help of this method, the vibration frequencies of the Si-O and Al-O bonds inside the frame are determined, which makes it possible to assess the ordering of the zeolite, the arrangement of ions, and the distribution of water molecules. Frame oscillations. The peaks observed in the IR spectrum of LiA zeolite in the range of 460-500 cm−1 correspond to the bending vibrations of the tetrahedral Si-O-Al bonds. These peaks confirm the presence of connections that make up the main skeleton of the zeolite frame. Intensive peaks, observed in the range of 660-700 cm−1, are characteristic of the oscillations of the secondary ring in the frame and indicate a high degree of ordering of the crystalline structure of the zeolite.

A wide peak, observed in the range of 990-1050 cm−1, corresponds to the Si-O-Si stretching vibrations, which confirms the strength and high degree of symmetry of the frame. Influence of water molecules and cations. The peak observed in the IR spectrum of LiA zeolite at around 1630 cm−1 corresponds to the deformation vibrations of water molecules. This peak indicates the presence of water molecules in the zeolite cavities. A wide peak in the range of 3400-3600 cm−1 represents O-H stretching vibrations associated with hydrated cations. This confirms that Li+ ions are located in the framework cavities and interact with water molecules (Fig. 3).

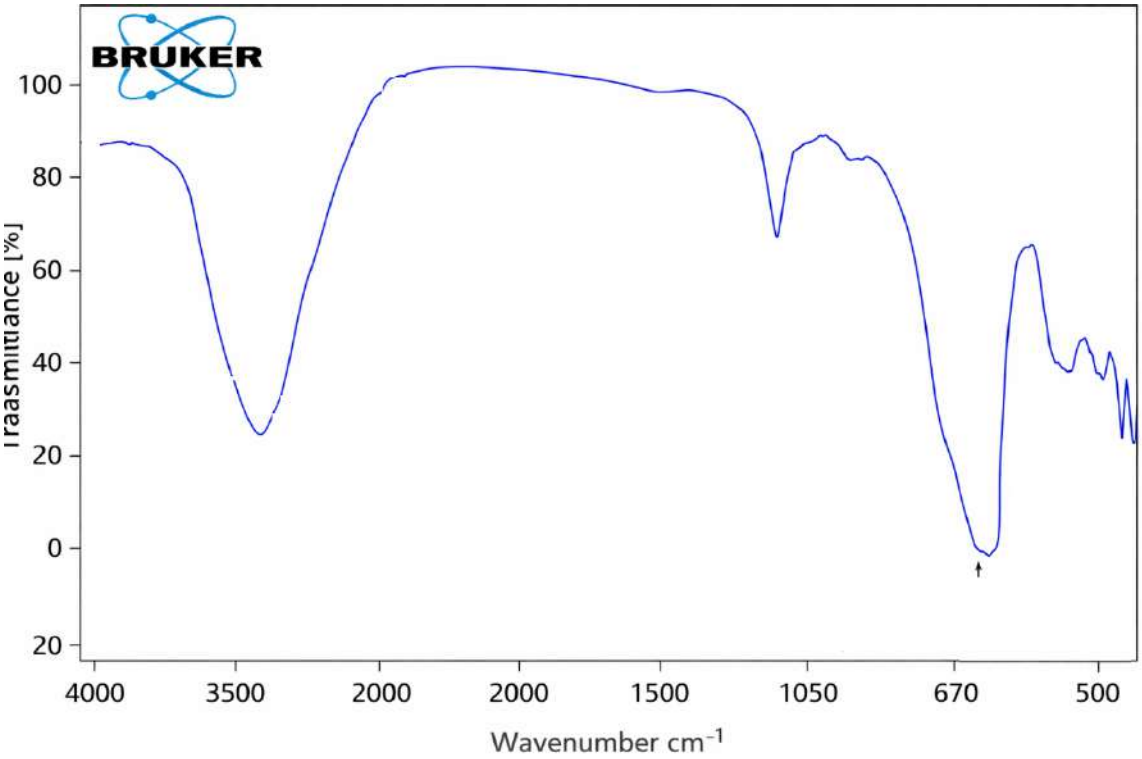

Figure 3. FTIR spectra of synthesized LiA and zeolite crystals

Specific effect of the Li+ ion. Since Li+ ions have a smaller radius compared to Na+ ions, a significant shift in the frame oscillation frequencies is observed. This shift increases the ion-exchange capacity of LiA zeolite. The arrangement of Li+ ions in the frame cavities improves the adsorptive and catalytic properties of the zeolite. Therefore, LiA zeolites demonstrate high efficiency in gas separation, water softening, and catalytic processes.

IR analysis provides important information about the frame structure, ion-exchange properties, and degree of hydration of LiA zeolite. The Si-O-Al and Si-O-Si oscillations indicate the orderedness of the frame, while the O-H oscillations confirm the presence of water molecules and Li+ ions. The observed shifts in frame oscillations due to the small radius of Li+ ions indicate the high ion-exchange capacity of the zeolite.

This makes it possible to use LiA zeolite as a promising material in the processes of adsorption and catalysis.

Conclusion. In this study, the hydrothermal synthesis of Na-A zeolite and its conversion into Li-A zeolite through ion exchange using Li+ ions were successfully carried out. A gel based on NaAlO2 and Na2SiO3 was mixed at 40°C for 2 hours and crystallized at 120°C for 24 hours. The crystalline solid phase was washed at a neutral pH and dried at 100°C. As a result, Na-A zeolite with a high degree of crystallinity was obtained.

The ion exchange process was carried out in three stages using a LiCl solution. At each stage, 20 mmol of Li+ ions were used. In the first stage, the main part of the Na+ ions was replaced by Li+ ions. In the second and third stages, the exchange process continued, but slowed down, which is associated with the filling of ion exchange centers. The final product was dried at 110°C for 12 hours, and Li-A zeolite enriched with Li+ ions was obtained.

XRD analysis played an important role in determining the crystalline structure of Na-A and Li-A zeolites. In Na-A zeolite, LTA-type diffraction peaks were clearly visible, confirming the high degree of crystallinity. After exchange with Li+ ions, the shift of the diffraction peaks and changes in intensity showed local deformations in the lattice structure as a result of ion exchange.

With the help of IR spectroscopic analysis, the molecular vibrational properties of zeolites were assessed. The presence of a frame structure was confirmed by Si-O-Al bending vibrations in the range of 460-500 cm−1, Si-O-Si tensile peaks in the range of 1050 cm−1, and ring vibrations in the range of 660-700 cm−1. Deformation vibrations of water molecules at 1630 cm−1 and peaks of O-H stretching in the range of 3400-3600 cm−1 showed that Li+ ions are located in cavities in a hydrated state.

Li-A zeolite, obtained by the synthesis of Na-A zeolite and its exchange with Li+ ions, has a high degree of crystallinity, a stable frame structure, and effective ion-exchange properties. XRD and IR analyses deeply revealed the structural and functional properties of these zeolites. The research results create a scientific basis for the use of Li-A zeolite as a promising material in adsorption, catalysis, gas separation, and environmental purification technologies.

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