Analysis of Na-A zeolite based on kaolin using an X-ray diffractometer

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In this paper, the hydrothermal synthesis of Na-A zeolite synthesized from Angren kaolin and γ-Al_2O_3 mixtures is considered. Crystal sizes of Linde Na-A and synthesized Na-A zeolite were compared using X-ray phase analysis. Zeolite Linde Na-A has a lattice strain between crystals from 0.019 to 0.23, and a lattice strain between synthesized Na-A zeolite crystals from 0.019 to 0.24. The dislocation density of the samples was found to be between $10^{14} m^{-2}$ and $10^{17} m^{-2}$. Using IR Spectrometric analysis was determined to be compatible with data presented in the literature. In this study, Angren kaolin was selected as the source of aluminosilicate available in Uzbekistan. Today, the use of zeolites for technological purposes is widely introduced worldwide, they are used in the production industry for purification of various gases, and in the oil and gas industry as an adsorbent and catalyst, for the separation of hydrocarbons, and for the deep purification of gases.

Keywords: zeolite; kaolin; clay; crystal; nanostructure; metakaolin; deformation; hydrothermal; dislocation.

Рентгендік дифрактометрді қолдана отырып, каолин негізіндегі Na-A цеолитін талдау

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Бұл жұмыста Ангрен каолині мен γ-Al_2O_3 қоспасынан синтезделген na-A цеолитінің гидротермиялық синтезі қарастырылады. Linde Na-A және синтезделген na-A цеолит кристалдарының өлшемдері рентгендік фазалық талдауды қолданумен салыстырылды. Linde na-a цеолитінде 0,019-дан 0,23-ке дейінгі кристалдар арасындағы тор деформациясы және синтезделген na-A цеолит кристалдары арасындағы тор деформациясы 0,019-дан 0,24-ке дейін болады. Үлгілердің орналасу тығыздығы 10^{14} м$^{-2}$ және 10^{17} м$^{-2}$ аралығында болды. ИК-спектрометриялық талдау арқылы олардың әдебиетте берілген деректермен үйлесімді екендігі анықталды. Бұл зерттеуде Өзбекстанда қол жетімді алюминий силикаттарының көзі ретінде Ангрен каолині таңдалды. Бүгінгі таңда цеолиттерді технологиялық мақсатта қолдану бүкіл әлемде кеңінен қолданылады, олар әртүрлі газдарды тазарту үшін өндіріс салаларында, сондай-ақ газ өнеркәсібі адсорбент және катализатор ретінде, көмірсутектерді бөлу және газдарды терең тазарту үшін.

Түйін сөздер: цеолит; каолин; саз; кристалл; наноқұрылым; метакаолин; деформация; гидротермиялық; дислокация.

Анализ цеолита Na-A на основе каолина с использованием рентгеновского дифрактометра

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В данной работе рассмотрен гидротермальный синтез цеолита Na-A, синтезированного из смеси ангренского каолина и γ-Al_2O_3. Размеры кристаллов Linde Na-A и синтезированного цеолита Na-A сравнивали с применением рентгенофазового анализа. Цеолит Linde Na-A имеет деформацию решетки между кристаллами от 0,019 до 0,23 и деформацию решетки между синтезированными кристаллами цеолита Na-A от 0,019 до 0,24. Плотность дислокаций образцов оказалась в пределах 10^{14} м$^{-2}$ и 10^{17} м$^{-2}$. С помощью ИК-спектрометрического анализа было установлено, что они совместимы с данными, представленными в литературе. В данном исследовании в качестве источника алюмосиликатов, доступных в Узбекистане, был выбран Ангренский каолин. Сегодня применение цеолитов для технологических целей широко внедрено во всем мире, они широко применяются в производственной промышленности для очистки различных газов, а также в газовая промышленность в качестве адсорбента и катализатора, для разделения углеводородов и глубокой очистки газов.

Ключевые слова: цеолит; каолин; глина; кристалл; наноструктура; метакаолин; деформация; гидротермальный; дислокация.

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1. Introduction

There is no denying that zeolites are the great importance in commercial, technological and scientific applications. In recent years, the global zeolite market is estimated at several billion dollars [1]. More than 80% of the commercial demand for detergents, catalysts, absorbents and cleaning agents is zeolite materials. Currently, 241 completely ordered and 11 partially disordered structures of various zeolites have been identified [2]. The process of synthesis of zeolites is carried out by the hydrothermal method by creating conditions that lead to their crystallization.

The contribution of preliminary radiation-oxidative treatment to the change in the electrophysical characteristics during thermal and radiation-thermal tests in the contact of zirconium with water is revealed [3]. There arises the formation of thin films of cobalt monosilicide (CoSi) deposited into the base surface of SiO_2/Si (111) using magnetron ion-plasma sputtering and subsequent thermal annealing. It was found that, in addition to the formation of CoSi silicide, also there are Co and Si atoms that do not form bonds on the surface [4-5].

Among zeolites, zeolite A or LTA is one of the first completely synthetic zeolites that has no natural analogues [6]. The X-ray diffraction (XRD) spectrum of the nano-ZrO_2 compound was drawn, the crystal structure was determined at room temperature and under normal conditions [7]. There arises the formation of thin films of cobalt monosilicide (CoSi) deposited into the base surface of SiO_2/Si (111) using magnetron ion-plasma sputtering and subsequent thermal annealing. It was found that, in addition to the formation of CoSi silicide, also there are Co and Si atoms that do not form bonds on the surface [8]. Other countries, including Korea, Ukraine and Iran, also produce large volumes of kaolin. Kaolin has the chemical composition $Al_2Si_2O_5(OH)_4$. Many researchers have synthesized Na-A zeolite by changing its chemical and physicochemical properties from kaolin [9-13]. Several different methods (microwave, vapor phase, hydrothermal and sol-gel methods) have been used in the synthesis of Na-A zeolite from kaolin. The most common methods for the synthesis of LTA-type zeolite membranes are primary synthesis and secondary (crystalline) growth. In combination with these methods, the microwave method was used to synthesize LTA-type zeolite membranes. Compared with the traditional hydrothermal synthesis method, microwave synthesis of zeolite membranes has advantages such as shorter synthesis time and higher membrane formation [14].

The synthesis of zeolites is influenced by several factors: molar ratioof reagents, synthesis temperature, synthesis time, source of raw materials, crystallization time. To synthesize LTA zeolites using structure-directed agents (CDA), researchers selectively modify certain synthesis processes.

Amongst different water purification, sorption is the best one due to its unique features; especially universal and inexpensive. A large number of publications are appearing in the literature every year, confirming its importance [15-16]. A temperature of 110°C was kept constant throughout the synthesis process of the zeolite NaA, and the durations and Na/Al ratios were altered. The of X-ray diffraction (XRD) analysis showed that high purity zeolite NaA with a high degree of structural order was produced in the following conditions: a Na/Al ratio of 1.64 for KC and KJ and synthesis durations of 18 h for KC and 20 h for KJ. According to the XRD results, which indicated the formation of the zeolites KA, MgA and CaA, the process of cation exchange between the Na from zeolite NaA and the K, Mg and Ca from the solutions was efficient. [10].This article examined the hydrothermal synthesis of Na-A zeolite synthesized from mixtures of Angren kaolin and γ-alumina with Linde Na-A zeolite.

2. Experiment

In this study, Angren kaolin was selected as the source of aluminosilicate available in Uzbekistan. Kaolin is a crystalline natural aluminosilicate in which the molar ratioof silicon oxide toalumina is 1.0-1.15 (Table 1). Kaolin has a structure including layers formed by repeating layers of silicon-oxygen tetrahedra and aluminum-oxygen octahedra.

Table 1 – Chemical composition of Angren kaolin

Substance	Content, %
Al_2O_3	31.20
CaO	1.24
Fe_3O_4	0.80
K_2O	0.28
MgO	0.72
Na_2O	4.69
P_2O_5	0.07
SiO_2	48.90
TiO	0.45
Other substances	11.67

The results obtained showed that the resulting films have a structure suitable for instrumentation. The paper also presents theoretical studies on the creation of photomasks to create a device design. Usingthe method of magnetron sputtering, a structure was created that can be used in thermal radiation receivers. The mechanism of structure formation has been developed [17].

Thereto synthesize Na-A zeolite, kaolin treated with oxalic acid solution was heated at 650°C for 4 h (thermal dehydration process) and converted into metakaolin (Table 2). To improve the Al/Si molar ratio in Na-A zeolite, γ-$Al_2O_3$was added to the resulting metakaolin in a mass ratioof 2.2/1. The prepared sample was mixed with 2M NaOH solution (Stable Temp Cole Palmer) at 50°C for 24 h. The prepared mixture is poured intoa Teflon container and placed in an autoclave. The sample was kept at a temperature of 90°C for 16 h. The synthesis product was washed several times with distilled water to remove excess alkali (pH-7) and redried at 60°C for 12 h.

Table 2 – Purified kaolin composition

| Substance	Content, %
Al_2O_3	37.4
Na_2O	5.5
SiO_2	55.6
Fe_3O_4	0.1
Other substances	1.4

3. Results and discussion

In this research work, the optimal activation temperature ranges from 550 to 850°C for different periods, with metakaolinization observed predominantly in the range of 650-750°C [18]. The metakaolin is heated to 650°C and then boiled for 4 h. The kaolin chunks were cooked at 550°C and then heated to room temperature. The optimal solution to this problem is to use the following methods:

$$2Al_2Si_2O_5(OH)_4 \xrightarrow{550\text{-}560°C} 2Al_2Si_2O_7 + 4H_2O$$
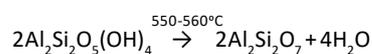
First, the kaolin sample is dried at 100°C for 6 h. Alkaline activator sodium hydroxide (NaOH), oxalic acid to purify excess compounds (Fe^{+3}) in local kaolin, γ-Al_2O_3 to improve the molar ratio of SiO_2/Al_2O_3 in the synthesis of Na-A zeolite, equipment for preparing the necessary solution processes was prepared. In the HERZOG-100P mill (GNR Nord, Germany) a sample of Angren kaolin AKF-78 is crushed to a size of 100 nm. 10 g of this crushed sample is placed in a magnetic stirrer (Cole Palmer, USA) at 100°C with 0.5 M $H_2C_2O_4$ solution. After a certain time, the mixture was cooled to room temperature, filtered and dried [19]. At this stage, a mechanism for purifying kaolin from additives, especially iron oxides, was implemented. In that:

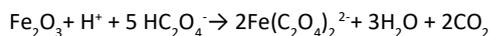– in solution, oxalic acid ($H_2C_2O_4$) dissociates to biooxalate ion($HC_2O_4^-$):

$$H_2C_2O_4 \rightarrow H^+ + HC_2O_4^-$$
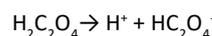
– in turn, the resulting biooxalate ion dissociates, leaving the oxalate ion ($C_2O_4^{2-}$):

$$HC_2O_4^- \rightarrow H^+ + C_2O_4^{2-}$$
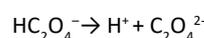
The biooxalate ion produced in this reaction is the ion responsible for dissolving iron:

$$Fe_2O_3 + H^+ + 5\ HC_2O_4^- \rightarrow 2Fe(C_2O_4)_2^{2-} + 3H_2O + 2CO_2$$

There crystals of hydrothermally synthesized Na-A zeolite based on Angren kaolin and a sample of Linde Na-A zeolite were studied using an X-ray diffractometer XRD-6100 (Shimadzu, Japan). As a result of the data obtained from the X-ray diffractometer, various X-ray diffraction peaks were recorded (Figure 1).

According to XRD data, 27 peaks were determined in Linde Na-A zeolite crystals and 30 peaks in the synthesized Na-A zeolite crystals (Figures 1 and 2). In our opinion, in the process of obtaining hydrothermally synthesized Na-A zeolite based on Angren kaolin, additional crystalline peaks may appear due to the use of γ-Al_2O_3 and NaOH in the appropriate concentration.
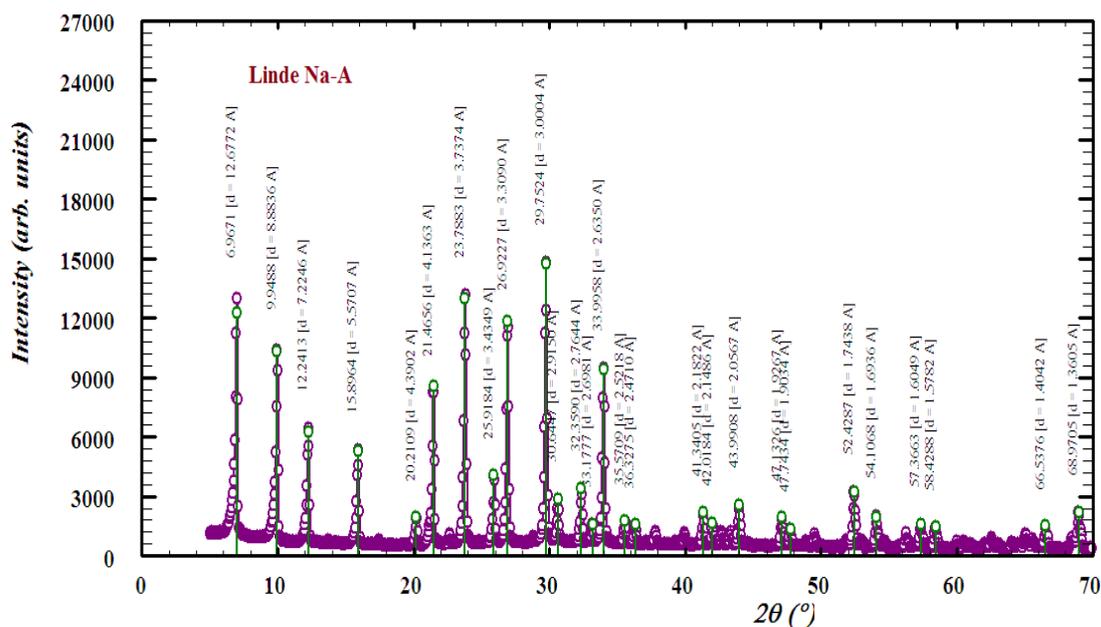

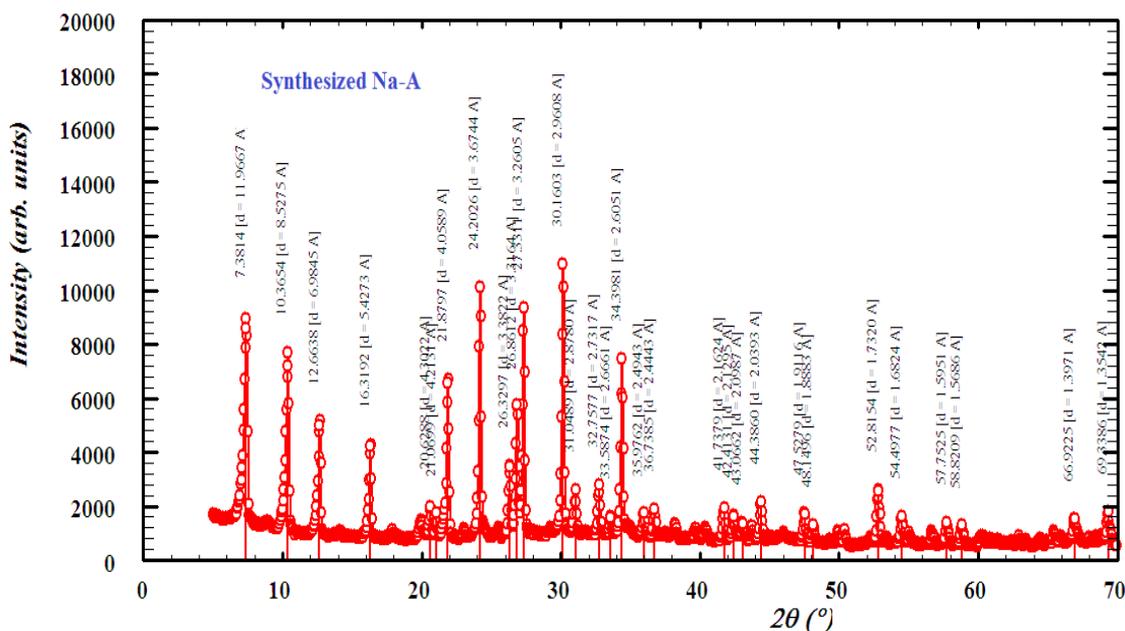Figure 1 – X-ray diffraction of the Linde Na-A zeolite crystals

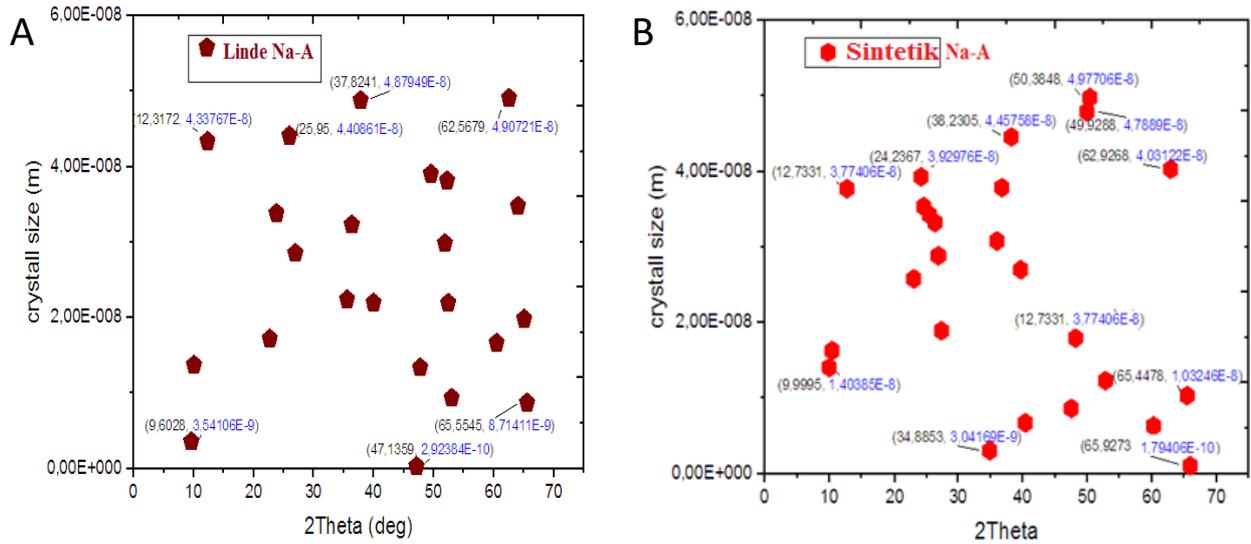Figure 2 – X-ray diffraction pattern of synthesized Na-A zeolite crystals

Figure 3 – Crystal size of the Linde Na-A and synthesized Na-A zeolites

Using X-ray diffraction, the crystal size of the samples (Scherrer equation) was determined (Equation 1):

$$D = \frac{0.9\lambda}{\beta\,Cos\,\theta} \qquad (1)$$

The broadening in the XRD peaks can be attributed to both the limited crystallite size and the presence of microstrain within the crystal lattice. We use the Williamson-Hall method to separate these contributions. Microstrain expansion: lattice distortion or deformations within crystals. The total broadening observed in an XRD peak (β_{total}) is the sum of the broadening due to crystallite size (β_D) and the broadening due to deformation ($\beta\varepsilon$). Williamson-Hall Method The Williamson-Hall method quantifies the contributions of crystallite size and microstrain to peak broadening. provides a separation method. General expansion:

$$\beta_{total}\cos\theta = \beta_D\cos\theta + \beta\varepsilon\sin\theta \qquad (2)$$

Here: β_{total} – is the observed broadening of the diffraction peak (FWHM in radians), β_D – is the broadening due to finite crystallite size, $\beta\varepsilon$ – expansion due to microstrain. The Scherrer equation (Equation 3) for crystal volume expansion:

$$\beta_D = \frac{k\lambda}{D\cos\theta} \qquad (3)$$

and considering the strain expansion (Equation 4):

$$\beta\varepsilon = 4\varepsilon\tan\theta \qquad (4)$$

where: D – crystallite size, ε – microstrain. Then the Williamson-Hall equation becomes Equation 5:

$$\beta_{total}\cos\theta = \frac{K\lambda}{D} + 4\varepsilon\sin\theta \qquad (5)$$

The total broadening (β_{total}) observed in the XRD peak is the sum of the broadening due to crystal size (β_D) and the broadening due to microstrain strain ($\beta\varepsilon$).

This line broadening can be used to measure crystal size and lattice strain. The size (D) of Linde Na-A zeolite crystals was established from $2.92 \cdot 10^{-10}$ m to $4.8 \cdot 10^{-8}$ m. The size (D) of the synthesized Na-A zeolite crystals was established from $1.79 \cdot 10^{-10}$ m dan $4.9 \cdot 10^{-8}$ m (Figure 3).

The lattice stress or strain in Linde Na-A crystals and synthesized Na-A zeolites is determined using Equation 6 [21-22]:

$$\varepsilon = \frac{\beta}{4 \times tg\theta} \qquad (6)$$

It has been established that the lattice strain of Linde Na-A crystals varies from 0.019 to 0.23. Change in lattice deformation of the synthesized Na-A zeolite crystals from 0,019 dan 0.24 (Figure 4).

The crystals of Linde Na-A and the synthesized Na-A zeolites have different sizes, and the deformation of the crystal lattice is caused by distortion of the crystal lattice, which leads to its dislocations. The dislocation density is determined using Equation 7 [23]:

$$\delta = \frac{1}{D^2} \qquad (7)$$

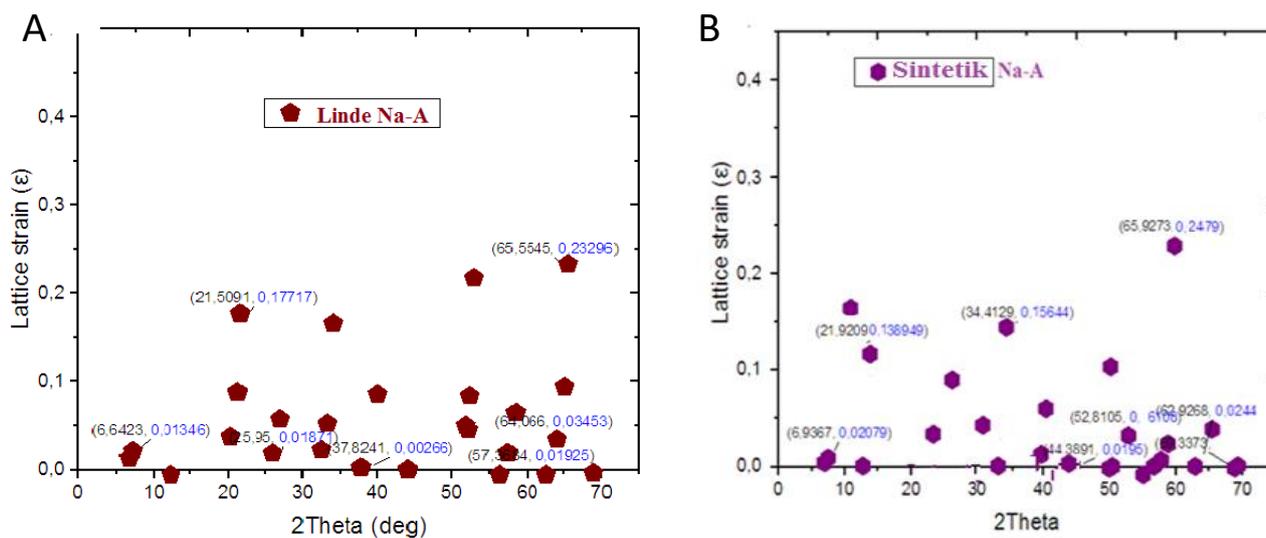**Figure 4** – Lattice strain **of** the Linde Na-A and synthesized Na-A zeolites

The surface dislocation density of Linde Na-A crystals was found to be between $10^{14}\,m^{-2}$ and $10^{15}\,m^{-2}$. The synthesized Na-A zeolites were found to have a dislocation density between $10^{14}\,m^{-2}$ and $10^{17}\,m^{-2}$ (Figure 5).
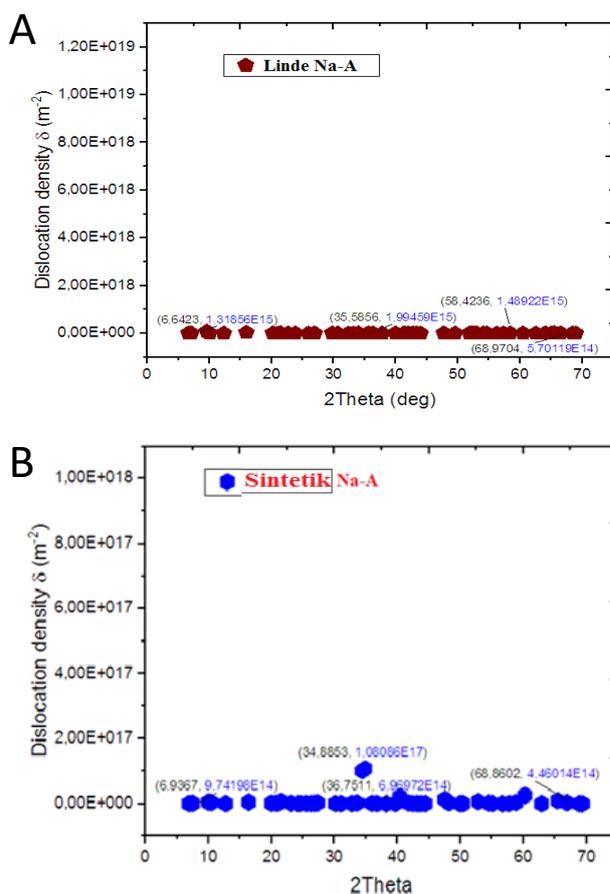

Figure 5 – Dislocation density **of** the Linde Na-A and synthesized Na-A zeolites

In the research work, 3 parameters (crystal size, dislocation density, inter-lattice deformation values) are presented. Crystalline size is a quantity measured on the basis of X-ray diffraction data with the Scherrer equation in all zeolite synthesis studies and provides conclusions about the crystallite size of the zeolite. The next is the dislocation density, which represents the displacement or approximation of the atoms located in the crystal nodes relative to each other. It is possible to conclude about the size and dimensions of the pores through this size. The values of this parameter are not significantly different between the obtained synthetic microporous Na-A zeolite and Linda Na-A zeolite. Interlattice deformation means that the distance between the atoms in the crystal nodes decreases and expands relative to each other. These quantities were measured using a special program based on X-ray phase analysis data of Linde Na-A and synthesized Na-A zeolites.

The sample synthesized in the research and Linde NaA zeolites were tested for infrared absorption and transmittance using Fourier-transform infrared (FT-IR) ALPHA II spectrometer (Bruker, USA) (Figure 6A) The vibration at 466.86 cm^{-1} in the synthesized NaA zeolite is close to that of Si-O or Al-O. The asymmetric vibration of the branched Si-O-Si bond has lower intensity compared to the symmetric vibration of Si-O-Si recorded at 719.40 cm^{-1}. In addition, the highest intensity peaks at 1001.15 cm^{-1} correspond to strong vibrational asymmetric stretching vibrations of Si-O-Si. Here, it was found that Si-O-Si bonds have less symmetric vibration than asymmetric vibration. The peak at 566.09 cm^{-1} indicates a double ring, which is a secondary building block in the A-type zeolite structure.

In addition, two IR characterised bands of zeolite hydration water are observed in the resulting Na-A sample. In zeolites, water molecules are associated with cations, and the hydrogen atom is somewhat associated with oxygen ions of the framework. The binding of water molecules to the zeolite cation or too oxygen ions in the framework depends on the open structure of the structure. The observed broadband vibration at 3444.0 cm^{-1} indicates that it is characteristic of hydrogen (OH) bound to framework oxygen ions. There is also an intense line characteristic of a water molecule in the vibrational mode of the spectrometer at 1640.87 cm^{-1}. Therefore, it can be seen that similar structural units and chemical parts are formed from the peak vibrations in the obtained Na-A.
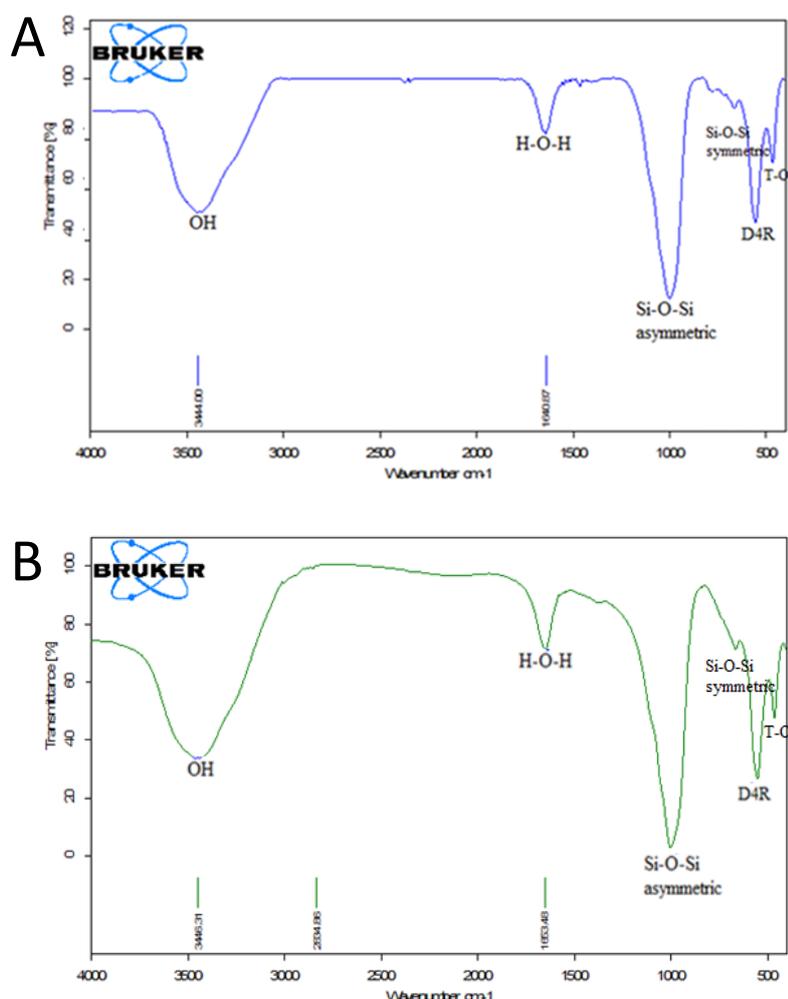

Figure 6 – A) Obtained NaA zeolite; B) Spectra of Linde NaA zeolite

Figure 6 shows infrared transmission spectrometers of Synthesized and Linde Na-A zeolites. As for their differences, the transmittance spectrometer represents the infrared transmittance of the zeolite, while the absorbance spectrometer indicates the level of infrared absorption. This conductivity spectrometer shows that it is 88% in synthetic Na-A zeolite and 75% in Linde Na-A zeolite. Absorption spectrometers show that synthetic Na-A zeolite is 12% and Linde Na-A zeolite is 25%, respectively.

The Linde Na-A zeolite particles exhibit a sharp, high-intensity peak at 1000.3 cm^{-1}, similar to the IR spectrum of the Na-A zeolite synthesized in this study (Figure 6B). This strong peak indicates a T-O-T (T=Al or Si) asymmetric vibration. The sharp band vibration at 460 cm^{-1} is close to the Si-O vibration. Symmetric vibration of Si-O-Si was recorded at 662 cm^{-1}. Symmetric stretching of the T-O-T bond has a lower intensity compared toasymmetric stretching of the T-O-T bond. A sharp peak at 560 cm^{-1} indicates the presence of a double ring, which is a secondary building block.

4. Conclusion

Today, the use of zeolites for technological purposes is widely introduced worldwide, they are widely used in the production industry for purification of various gases, and in the oil and gas industry as an adsorbent and catalyst, for the separation of hydrocarbons, and for the deep purification of gases. Currently, zeolites are synthesized on the basis of raw materials such as bentonite, kaolin, nephilin. Synthetic zeolites obtained from kaolin are of the most important practical importance in cleaning oil and gas industry products from additives. This article examines the hydrothermal synthesis of Na-A zeolite synthesized from mixtures of Angren kaolin and γ-alumina with Linde Na-A zeolite. Crystal sizes of Linde Na-Aand synthesized Na-A zeolite were compared using X-ray phase analysis. Zeolite Linde Na-A has a lattice strain between crystals from 0.019 to 0.23, and a lattice strain between synthesized Na-A zeolite crystals from 0.005 to 2. The dislocation density of the samples was found to be between 10^{14}-10^{17}. Using IR spectrometric analysis was determined to be compatible with data presented in the literature.

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