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INVESTIGATION OF VARIOUS INFLUENCING FACTORS OF HYDROTHERMAL SYNTHESIS OF ANALCIME ZEOLITE

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Abstract. The analcime zeolite of potential practical importance has been obtained based on the natural mineral of Nakhchivan Autonomous Republic. Analcime has a wide range of application and therefore its optimal synthesis conditions have been determined. The influence of temperature and crystallization time, the concentration of alkaline solution and mineralizer on the process of synthesis of analcime has been studied. The optimal conditions established in this study for the synthesis of analcime zeolite with a 100% degree of crystallinity are as follows: temperature of 180°C, alkaline and mineralizer solution of 10-15% KOH and 5-10% KCl and processing time of 50 hours. It has been shown that the presence of the KCl mineralizer promotes the production of pure analcime with a 100% crystallinity, and the natural mineral of Nakhchivan represents a good source for the synthesis process.

Keywords: analcime, hydrothermal synthesis, natural mineral, zeolite, crystallization.

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Introduction

The study of the synthesis and properties of zeolites and their crystallization process based on natural minerals is promising and relevant research area. Many studies on the hydrothermal synthesis of zeolites based on kaolinite and the investigation of the effect of synthesis conditions on the crystallization process have been well summarized by Johnson, E.B.G. *et al*. [1].

Analcime presents potential practical importance among zeolites, due to its wide range of applications, as an adsorbent in the purification of water from heavy metals [2], as a catalyst in the oil industry [3,4] and in gas separation [5], as fertilizer in agriculture [6]. An analysis of the scientific literature showed that analcime can be obtained from various structural types of the starting components in hydrothermal conditions. Previously, it has been reported the synthesis of analcime from natural clinker [7], that resulted in a 95.2% degree of crystallinity in the presence and absence of tetramethylammonium hydroxide, and processing time of 24, 36 and 72 hours [8]. Analcime was also obtained from industrial waste (recycled glass, silica fume, siliceous concrete waste aggregates, sterile coal and foundry sand from the steel industry) [9], in the reaction process of potassium feldspar in Na_2SiO_3 solution [10]. Synthesis of analcime was also carried out by hydrothermal treatment of local pottery stone of 2 M NaOH at 60°C, 80°C and 120°C temperatures and processing time of 8, 12 and 24 hours [11]. Analcime was also synthesized from a perlite (volcanic glass) in sodium form [12], obtained from amorphous $SiO_2 \cdot nH_2O$ and Al containing components (gibbsite and two types of γ-Al_2O_3) [13] and synthesized by mixing aluminate and silicate solutions, which had been prepared separately by dissolving silica and aluminium raw materials in a sodium hydroxide solution while being stirred permanently [14].

Synthetic zeolites surpass their natural counterparts in their physicochemical properties. Similarly, synthetic analcime has better characteristics in comparison with natural one, thus having a wider range of application (as seen from the analysis of scientific literature). In this context, the aim of this paper consists in the synthesis and optimization of the conditions for obtaining of potential practical importance analcime zeolite with a 100% degree of crystallinity and phase purity based on the natural mineral of Nakhchivan. Additionally, the effects of the alkaline solution and mineralizer concentration, crystallization temperature, and processing time on the formation of analcime zeolite were studied in detail. It should be noted that the hydrothermal synthesis of analcime based on natural zeolite-containing tuff, in the presence of a mineralizer is carried out for the first time.

Experimental

Materials

Sodium hydroxide, potassium hydroxide, sodium chloride and potassium chloride (flake, 99% purity, Alfa Aesar GmbH & Co KG, Germany) have been used without further purification.

The natural samples have been obtained from the zeolite horizon in the north-west of the Kyukyuchai river of Nakhchivan Autonomous Republic, where zeolite content varies in the range of 75−80%. The samples have been washed thoroughly with distilled water and dried at a temperature of 100°C for three days.

Synthesis

General procedure: Hydrothermal synthesis of analcime has been carried out in Morey autoclaves made of 45MNFT stainless steel with a volume of 18 cm^3, and the filling coefficient of F= 0.8. The hydrothermal crystallization experiments have been performed generating a temperature gradient ΔT= 0 and without stirring of the reaction mass. The solid−liquid ratio was set to 1:10. After crystallization was completed, the final material was separated from the initial solution, washed with distilled water from excess alkali, and dried at 80°C. For each experiment, 2 g of natural zeolite was used. The stage of preparation of the initial mixture consists of mixing a heat-treated sample of the natural mineral of Nakhchivan Autonomous Republic in alkaline solutions or an alkali+mineralizer solution at room temperature. After mixing the initial component and the alkaline solution or the alkali+mineralizer solution, the initial mixture was transferred to the autoclave and the crystallization process was performed at various temperatures. The crystalline structure of the original natural mineral was destroyed and recrystallized into a cubic analcime structure (with a 100% degree of crystallinity, crystallizes within 50 hours).

In order to optimize the process, synthesis of analcime has been carried out by varying the conditions: in solutions of KOH+KCl, KOH+NaOH (ratio= 1/1) and KOH+NaOH+KCl+NaCl (in the ratio of OH^-/Cl^-= 1/1); in the temperature range from 90 to 200°C; the alkaline solution concentration range from 5 to 20% and mineralizer of 3−15%; reaction time of 10−80 hours.

Characterization techniques

X-ray diffraction analysis

The X-ray diffraction measurements were performed using the Bruker 2D PHASER X-ray powder difrractomenter (Germany) (CuK_α radiation, 2θ= 5−50°), using NaCl, SiO_2 (quartz) and pure zeolites in internal and external standards, respectively. Samples have been placed on a front mounted plastic sample holder. The measuring conditions have been as follows: step size of 0.15 s/step, nickel filter as incident beam, slit aperture of 0.3° and scan 2θ range from 0.5° to 10°.

Thermogravimetric analysis

The thermogravimetric analysis of the samples has been carried out on a derivatograph Q-1500-D (Hungary) in the dynamic mode in the temperature range of 20-1000°C. Shooting mode: heating rate of 20°/min; paper speed of 2.5 mm/min; the sensitivity of differential thermal analysis (DTA), difference thermogravimetry (DTG) and thermogravimetry (TG) is 500 mv; ceramic crucibles; Al_2O_3 was used as the standard.

Elemental analysis

Elemental analysis of the starting material and the reaction conversion products has been carried out on a Launch of Trition XL dilution refrigerator multichannel X-ray spectrometer (U.K.). Measurement mode: Pd - anode, voltage of 25 kW, current strength of 70 MA, exposure time of 100 sec., sensitivity limit of 10^{-2}. For analysis, the samples have been prepared as follows: the analyte was diluted with $Li_2B_4O_7$ flux (ratio 1:10) at a temperature of 1250°C. The resulting glass has been crushed under the pressure of 20 t/cm^2 with a holding time of 1 min.

Scanning electron microscopy

Scanning electron microscopy (SEM) analysis of the starting materials and reaction products was performed on a Hitachi 3000 TM high-resolution microscope (Japan) (an increase of 30000 times). Low vacuum mode allowed exploring samples without pre-deposition. The sample was placed on a double-sided adhesive tape glued onto a metal disk and vacuum to a pressure of 10^{-4} Pa to obtain micrographs.

Results and discussion

An analysis of the scientific literature shows that analcime is obtained in the presence of a structure-forming organic agent [15,16], by mixing a large number of reagents [17,18], which is not profitable from a financial point of view. Thus, there is an interest in developing a more accessible and optimized method of synthesis of analcime. The present paper presents a synthesis process without a structure-forming organic agent, with a minimum number of reagents and by using the natural zeolite tuff of Nakhchivan, as an initial

material, due to its advantage to wide distribution and lower cost. In addition, the main disadvantages of the carried out studies lie in the complex synthesis process (*i.e.*, long aging and reaction time) and the use of expensive template, which make it costly and difficult to apply on a larger scale. Therefore, finding alternative cheap raw material with all the necessary components for zeolite by a simple and economic method is of great significance.

Investigation of the starting material

The zeolite tuff of the Nakhchivan deposit of Kyukyuchay was used as a starting material. According to X-ray diffraction and elemental analyzes, it was found that 78.5% of the zeolite tuff consisted of mordenite ($Ca_2Na_2K_{2.8}Al_{8.8}Si_{39.2}O_{96}{\cdot}34H_2O$), 19.5% of quartz ($SiO_2$) and 2% of anorthite ($Ca_{0.86}Na_{0.14}Al_{1.94}Si_{2.06}O_{8.01}$).

According to elemental analysis, it can be argued that the zeolite tuff of the Nakhchivan Autonomous Republic of Kyukyuchay deposit was distinguished by phase purity, *i.e.* the bulk of the sample was concentrated on mordenite. Impurities were present in small quantities.

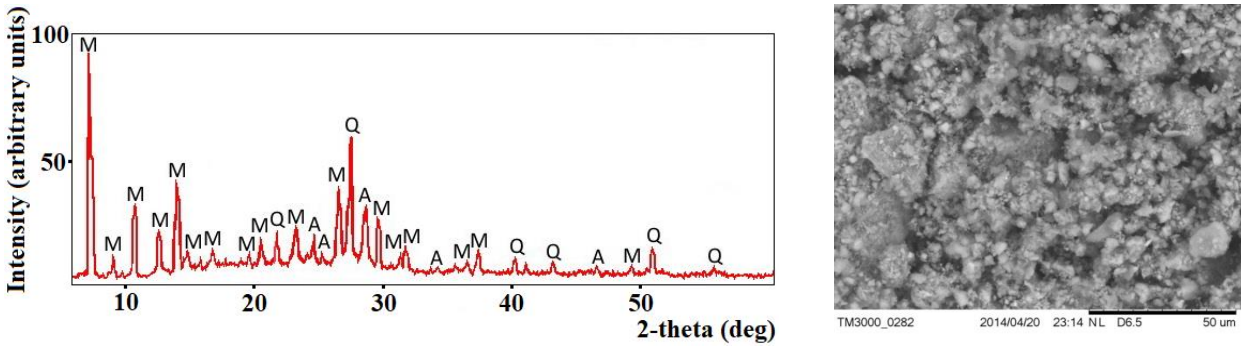
Comparison of X-ray data obtained for the zeolitic tuff of Nakhchivan (Figure 1*(a)*) with literature data showed that the studied sample of zeolite consisted mainly of mordenite [19]. The peaks in the diffractogram with interplanar distances d= 3.34 Å, 2.45 Å, 2.28 Å, 2.12 Å indicate the α-quartz content. Also, a small amount of anorthite (4.30 Å, 3.60 Å, 3.40 Å, 3.19 Å) was found in the sample composition.

SEM image of the Nakhchivan zeolitic tuff is presented in Figure 1*(b)* showing that the sample is characterised by an indeterminate surface relief with microcrystals of different sizes on the surface, possibly due to its mineral composition.

Investigation of the synthesized analcime

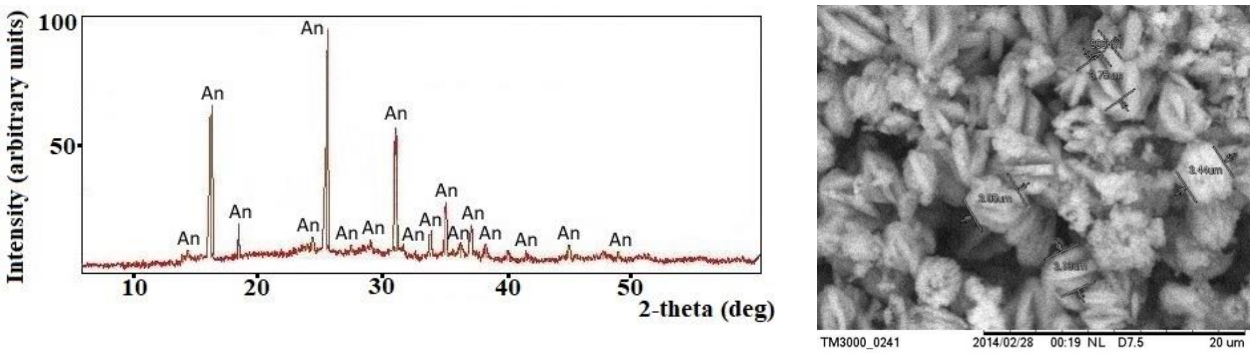
Analcime was synthesized in two alkaline solutions (KOH and KOH+NaOH), in the presence and absence of a mineralizer (KCl, KCl+NaCl), at different temperatures and crystallization times. The obtained results have shown that analcime with a high degree of crystallinity was obtained in solutions KOH+KCl, KOH+NaOH, KOH+NaOH+KCl+NaCl, at temperatures of 100°C and 180°C, and a processing time of 50 hours. The concentration of alkaline solutions and mineralizers ranged from 10 to 15% and 5 to 10%, respectively.

X-ray diffraction patterns of the analcime and its micrograph are shown in Figure 2*(a)* and *(b)*, respectively. Table 1 presents the data of X-ray diffraction analysis.



(a) ***(b)***

Figure 1. The X-ray diffraction pattern of Nakhchivan zeolitic tuff* *(a)* and its SEM image *(b)*. *(M − mordenite, Q − quartz, A – anorthite)



(a) ***(b)***

Figure 2. The X-ray diffraction pattern of analcime (An) with 100% degree of crystallinity *(a)* and its SEM image *(b)*.

Table 1

X-ray diffraction data of the obtained analcime.

d_{exp}, Å	I_{rel}	*hkl*	d_{calc}, Å
5.60	60	211	5.60
4.85	20	220	4.86
3.67	10	321	3.67
3.43	100	400	3.43
2.92	55	332	2.93
2.80	10	422	2.80
2.69	20	431	2.69
2.51	15	521	2.51
2.43	10	440	2.43
2.22	10	611	2.22
2.17	10	620	2.17
2.11	10	541	2.12
1.98	10	444	1.98
1.90	20	640	1.90
1.86	10	633	1.87

According to the X-ray phase analysis, analcime crystallizes in the cubic crystal system with the unit cell parameter a = 13.73 Å, which is in good agreement with earlier [20]. The presented XRD pattern (Figure 2*(a)*, Table 1) relates to analcime obtained under optimum conditions with a 100% degree of crystallinity. A comparison of the experimental values of the interplanar distances (*d*, Å) to literature data [20] and calculated ones [21] shows that the obtained diffraction patterns indicate the phase – analcime with a 100% degree of crystallinity.

Using the thermogravimetric analysis (Figure 3), the region of dehydration and thermostability of the analcime has been established. The DTA curve is characterized by one endothermic and one exothermic effect. The endothermic effect corresponds to the dehydration of the sample with a maximum of 350°C, at which the weight loss along the TG curve is 13%.

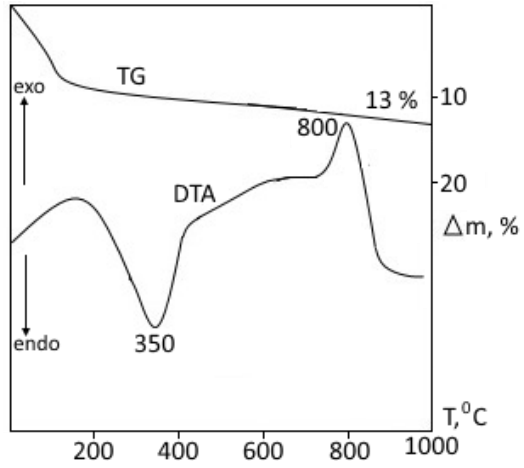


Figure 3. Thermogram of synthesized analcime zeolite obtained under optimal conditions.

The exothermic effect, detected at a temperature with a maximum of 800°C, according to X-ray diffraction analysis, refers to the destruction of the crystal structure of analcime and the formation of cristobalite and albite.

The dehydration-rehydration properties of analcime have been studied and it has been found that the sample dehydrated in the temperature range of 190-420°C completely rehydrated within 48 hours. Dehydration is reversible, which is typical for zeolites. According to the X-ray diffraction analysis, no structural changes occur after dehydration.

The study of reaction mechanism during zeolite synthesis

The reaction mechanism for the synthesis of analcime from the natural mineral of Nakhchivan under hydrothermal conditions encompasses several stages: mixing the initial component in an alkaline solution (KOH, KOH+NaOH) or in an alkali solution+mineralizer (KOH+KCl, KOH+NaOH+KCl+NaCl), dissolution of the starting component, hydrothermal reaction at various temperatures and crystallization time. The process of crystallization of zeolites is complex, as it depends on many factors (temperature and reaction time, concentration of solvent or mineralizer, *etc.*). In a simple case, the crystallization process (under hydrothermal conditions) of analcime from the natural mineral of Nakhchivan in an alkaline medium and the presence of a mineralizer can be achieved in 3 stages: the induction period, the nucleation of crystals and their growth, previously described in the scientific literature [22-24].

Zeolites are very sensitive to changes in the synthesis conditions. It is a known fact that the effect of the molar ratio of SiO_2/Al_2O_3 (Si/Al) in the gel mixture, aging condition, alkalinity, and crystallization time and the temperature, all have a major impact on the entire synthesis process, influencing the degree of crystallinity and phase purity of the product [25-28]. The results of hydrothermal synthesis of a new synthetic aluminosilicate (SUZ-4) patented by the British Petroleum Company at temperatures of 150°C, 165°C, 180°C and crystallization time of 24, 18, 12 hours are presented, and it was found, that highly crystallined SUZ-4 is obtained at high temperature and crystallization time conditions [29]. The effect of crystallization time on the synthesis of zeolite of faujasite (Y) from Elefun kaolinite clay was investigated and the results of the analysis indicated that the maximum crystallization time for the synthesis of zeolite Y was 48 hours [30].

Effect of reaction temperature and processing time

Crystallization of analcime in solutions of KOH+KCl, KOH+NaOH, KOH+NaOH+KCl+NaCl has been studied at temperatures of 90°C, 100°C, 150°C, 180°C, 200°C and a processing time of 10, 30, 50, 80 hours; the X-ray diffraction patterns of crystallization products in the obtained solutions are presented in Figure 4.

It was found that in a KOH+KCl solution at a temperature of 100°C for 10-50 hours, no noticeable structural changes of the starting material (natural mineral of Nakhchivan) occur, that is, the structure of the zeolite-containing tuff of Nakhchivan is stable at 100°C for 50 hours. A further increase in temperature to 150°C for 50 hours contributes crystallization products consisting of a mix of analcime and mordenite (Figure 4*(a)*). At 180°C, the initial component is completely recrystallized into pure analcime with a 100% degree of crystallinity. Further increasing the temperature to 200°C and the processing time above 50 hours (up to 80 hours) promotes crystallization in addition to analcime, chabazite and clinoptilolite (Figure 4*(b)*).

In KOH+NaOH solution, analcime with a maximum degree of crystallinity (87%) was obtained at 100°C and 50 hours. At 90°C and 50 hours of treatment, the initial component − mordenite and analcime crystallized with a low degree. Increasing the temperature above 100°C promotes the crystallization of other products, namely at 150-200°C for 50 hours, phillipsite and chabazite were obtained (Figure 4*(c)*).

In a solution of KOH+NaOH+KCl+NaCl at a temperature of 100°C and after 50 hours of processing, no changes occur, while a further increase in temperature to 150°C leads to the appearance of mordenite and analcime in the reaction products. In this solution, analcime with a maximum degree of crystallinity of 92% has been obtained at 180°C after 50 hours. Increasing the temperature to 200°C and the processing time to 80 hours, leads to the formation of analcime and chabazite in the reaction products (Figure 4*(d)*).

Effect of alkaline solution concentration

The effect of alkaline solution concentration has been studied in solutions of KOH and KOH+NaOH; the X-ray diffraction patterns of crystallization products are shown in Figure 5.

The obtained results have shown that experiments in the natural mineral – KOH+KCl system at a 5% concentration of KOH, crystallized mordenite and analcime. Pure analcime with a 100% degree of crystallinity has been obtained in the KOH concentration range of 10-15%. A further increase in the concentration of KOH (20%) promoted the crystallization of chabazite, clinoptilolite (Figure 5*(a)*), and above 20% (30%) faujasite and hydrosodalite were obtained (Figure 5*(b)*).

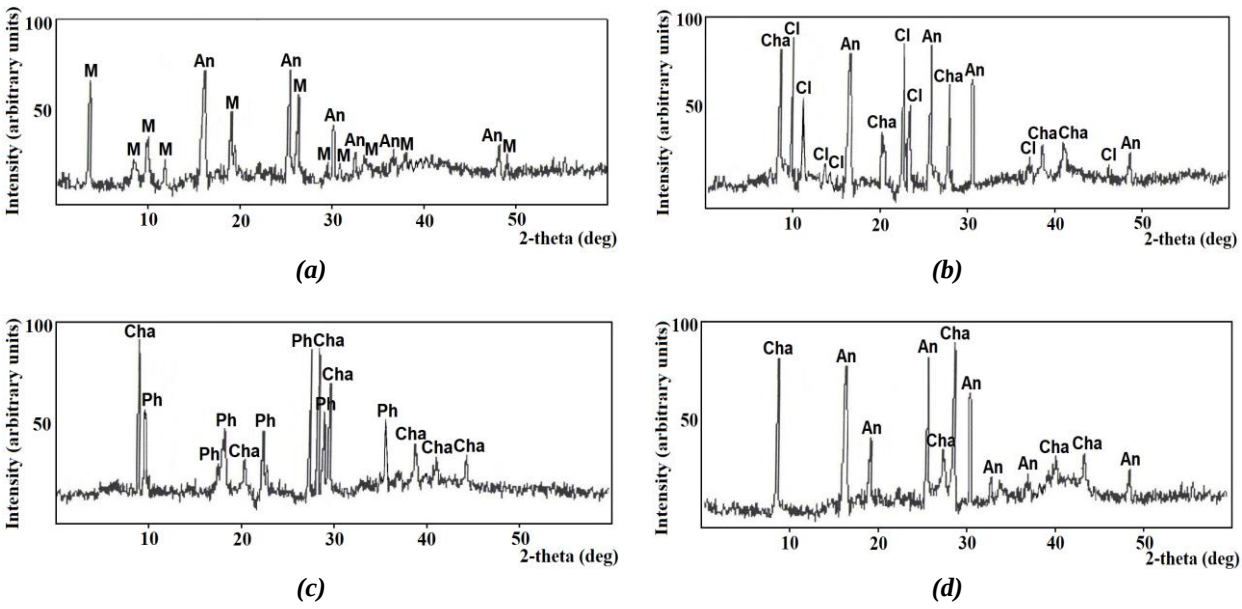


Figure 4. X-ray diffraction patterns of crystallization products* obtained in: KOH+KCl at 150°C for 50 hours *(a);* KOH+KCl at 200°C for 80 hours *(b);* KOH+NaOH at 150-200°C for 50 hours *(c);* KOH+NaOH+KCl+NaCl at 200°C for 80 hours *(d).*
***(M − mordenite, Ph − phillipsite, An − analcime, Cha − chabazite, Cl − clinoptilolite)**

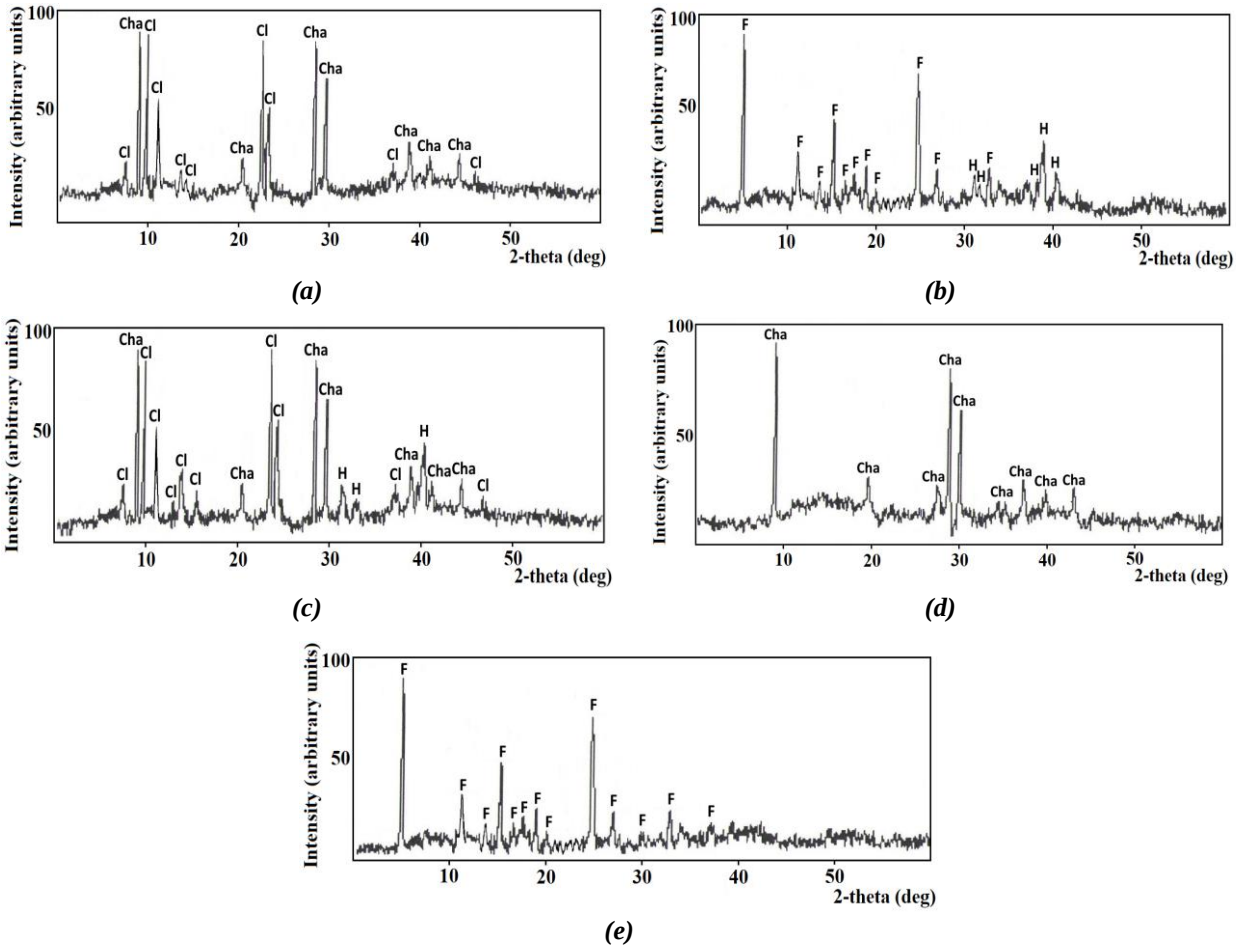


Figure 5. X-ray diffraction patterns of crystallization products* obtained in: 20% KOH and 15% KCl *(a)*; in KOH+KCl at 30% KOH *(b)*; at 30% KOH+NaOH *(c)*; in KOH+NaOH+KCl+NaCl at 20% KOH+NaOH and 15% KCl+NaCl *(d)*; in KOH+NaOH+KCl+NaCl at 30% KOH+NaOH *(e)*.
***(Cha − chabazite, Cl − clinoptilolite, F − faujasite, H – hydrosodalite)**

In the natural mineral – KOH+NaOH system, analcime with a maximum degree of crystallinity 87% has been obtained in the concentration range of 10–20%. Mordenite and analcime were present below 10% in the crystallization products, and above 20% - clinoptilolite, chabazite and hydrosodalite were obtained (Figure 5*(c)*).

The study of the natural mineral – KOH+NaOH+KCl+NaCl system has shown that 10-15% turned out to be the best concentration of KOH+NaOH to produce analcime with a maximum degree of crystallinity (92%). Below the 5% concentration of the alkaline solution, mordenite and analcime were present in crystallization products. At a concentration of alkaline solution above 15% chabazite crystallizes (Figure 5*(d)*), at 30% faujasite was obtained (Figure 5*(e)*).

Effect of mineralizer concentration

The effect of the mineralizer concentration has been studied in solutions of KCl and KCl+NaCl; the diffraction pattern of crystallization products is shown in Figure 5*(d)*. The experimental results suggest that in the natural mineral–KOH+KCl system, at a KCl concentration of 3%, mordenite and analcime appear in the reaction products. The optimal condition for the synthesis of pure analcime with a 100% degree of crystallinity is a KCl concentration of 5-10%. Increasing the concentration to 15% promotes the crystallization of chabazite+clinoptilolite (Figure 5*(a)*).

Studies performed in the natural mineral–KOH+NaOH+KCl+NaCl system have shown that at a concentration of KCl+NaCl (1:1) of 5-10%, pure analcime was obtained with a degree of crystallinity of 92%; at 3%, mordenite+analcime were present in the synthesis products, and increasing the concentration of alkaline solution up to 15%, chabazite was obtained (Figure 5*(d)*).

Optimal synthesis conditions

Having studied the process of crystallization of analcime, the optimal conditions for its synthesis with a high degree of crystallinity were established. The analcime obtained under

optimal conditions differed in phase purity and a high degree of crystallinity. Analcime with a 100% degree of crystallinity has been obtained under the following optimal conditions: the temperature of 180°C, the KOH and KCl concentrations of 10−15% and 5−10%, respectively, and processing time of 50 hours. Moreover, at 100°C, in KOH+NaOH solution with concentrations of 10−20%, at the crystallization time of 50 hours, and at 180°C, in KOH+NaOH with 10−15% and KCl+NaCl concentration of 5−10%, analcime with 87 and 92% degree of crystallinity has been obtained, respectively.

Conclusions

The natural mineral of Nakhchivan Autonomous Republic has been used for the synthesis of potential practical importance zeolite of analcime, the effect of temperature, alkaline solution and mineralizer concentrations, processing time on crystallization have been investigated. The obtained results have shown that analcime with a high degree of crystallinity (87, 92 and 100%) can be obtained at temperature of 180°C, alkaline solution KOH and mineralizer KCl concentrations of 10-15% and 5-10%, respectively, processing time of 50 hours.

Moreover, it was shown that a change in the synthesis conditions (temperature, alkaline solution and mineralizer concentration, processing time) can greatly affect the degree of crystallinity, the phase purity of the obtained zeolite.

The optimal conditions (temperature of 180°C, alkaline solution KOH and mineralizer KCl concentrations of 10-15% and 5-10%, respectively, processing time of 50 hours) for the synthesis of analcime zeolite with the high degree of crystallinity have been established. Also, it has been attested that the presence of the KCl mineralizer promotes the production of pure analcime with a high degree of crystallinity.

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