Factors that influence analcime formation

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Navickas A. A., Špokauskas A. A., Štuopys A. Factors that influence analcime formation. *Geologija.* Vilnius. 2003. No. 42. P. 19–25. ISSN 1392-110X.

Natural conditions of analcime formation, determined by geochemists (prolonged duration of synthesis, high pressure and temperature) cannot be applied in industry. Usually, analcime is synthesised at high temperatures (over 180 °C) and in a wide range of pressures (0.15…100 MPa), mostly from aluminosilicate gels. Analcime as the dominating phase was found in the surface layers of the stratified synthesis products, though it can be not dominant in the synthesis batch volume. It was concluded that analcime is dominating in the surface layer of the synthesis products due to a high alkali concentration. Analcime with a low quantity of impurities was synthesised in the investigation, as the quantity of the mineral in the synthesis products depends on the nature of the raw materials. The mixture of NaOH solution, active γ-Al_2O_3 and silicic acid with the molar ratio 1–2:1:4–6 is optional for the synthesis conditions applied. γ-Al_2O_3 was preferable to gibbsite in the synthesis conditions. Using gibbsite in analcime synthesis we obtained much more other zeolites and impurities than using γ-Al_2O_3. When the molar ratio SiO_2 / Al_2O_3 increased from 4 to 6 in the mixture of raw materials, the analcime quantity in the synthesis batch increased too. The conclusion was made that the Na_2O / Al_2O_3 molar ratio in the mixture of raw materials must be higher than 1. γ-Al_2O_3 as one of the raw components is preferable to gibbsite in the selected synthesis conditions, because the output of analcime is much higher in that case.

Keywords: analcime, gibbsite, mordenite, synthesis products, zeolites

Received 03 January 2003, accepted 04 February 2003

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INTRODUCTION

Results of analcime synthesis are presented of in this paper. Usually analcime is attributed to zeolites, though in natural conditions it is often discovered together with feldspatoids. Some properties of analcime are similar to those of feldspatoids, that is why it is called feldspatoid too. The idealised formula of this cubic syngony mineral is $NaAlSi_2O_6 \cdot H_2O$ (Флейшер, 1990), but in nature it makes an isomorphic row of solid solutions up to wairacite (zeolite of Ca cationic form). Besides, its chemical composition is rich in potassium (Фрей, 1985). The Si / Al ratio varies from 1.8 to 2.8 (Брек, 1985) in the natural mineral and the content of water can increase, if the amount of SiO_2 grows in its lattice.

Natural analcime is found in cavities of basalt, serpentinite, dacite, riolites, and tuff rocks, in associations with other zeolites. Neither natural nor synthetic analcime is used as a molecular sieve or catalyst, because the channels of its crystal lattice (internal cavities of the analcime lattice) are relatively narrow. It couldn't be used as a molecular sieve because of a high density of the mineral (2.25…2.30 g/cm^3).

Natural and synthetic zeolites are in the focus of interests of ceramics industry in the last decades. Zeolites are used as a nonplastic material or peptizing agent of ceramics masses (Медведовский, 1993; Ковзун и др., 1993), as a raw material for faience majolica and other products (Lehnhänser et al., 1987). The mineral is often found in alkalis activated and polluted by clay siliceous mixes after their autoclave treatment, it occurs in concrete (mortar) articles modified with kaolin as a secondary component of the steam cured concrete binder with glass fibers and so on.

Some natural or synthesized zeolites are easily modified by the ion exchange method, therefore the minerals could be used as raw materials suitable for burning at lower temperatures in comparison with ordinary raw material. This is the way to obtain ceramics, for example, cordierite (US pat. 4814303) from zeolites. Often by this method ceramics for denture is produced. It is well known that leucite $KAlSi_2O_6$ improves the properties of this kind of ceramic (US pat. 5071801; US pat. 4798536; US pat. 4814303). Analysis of the patents shows that it is possible to obtain leucite by burning ion exchanged (modified) analcime. Leucite and analcime are often formed in the same natural conditions or are substituted by each other in the rocks. Thus, the synthesis of analcime has the practical value.

CONDITIONS OF NATURAL AND SYNTHETIC ANALCIME FORMATION

For a long time analcime was believed to have no industrial value, therefore no attention was given to its synthesis. In many investigations it is often treated as a secondary phase of zeolite synthesis. The natural conditions of this mineral formation were studied by geologists (Гогишвили и др., 1968; Михайлов, 1980). Unfortunately, the conditions of analcime formation established by them cannot be reproduced in industry because of prolonged synthesis duration, high pressures and temperatures. Data of geological investigations, summarized by D. Breck in his monograph (Брек, 1985), lead to the following conclusions:

– analcime is usually found in the old basic rocks not rich in SiO_2;

– dense and less hydrated zeolites, such as analcime, are formed at high temperatures and pressure;

– sometimes analcime forms in water solutions rich in dissolved silica and aluminum oxide (the case of sedimentary rocks).

The data generalized by D. Breck and other investigators are presented in Table 1. According to D. Breck, analcime is often synthesized from chemical reagents such as aluminum silicate gel at a high temperature (over I50 …180 °C) and in a wide range of pressures (0.15 …100 MPa).

Aluminum silicate gel or other chemical reagents are not the only kind of raw material suitable for analcime synthesis. It is well known that analcime can be obtained by treatment of natural or technical glass, slag (Štuopys, 1997; Skvara et al., 1997), clays (Валюс, 1982), catalyst wastes (Štuopys, 1996). The chemical and mineral structure of these raw materials is unstable, they are highly reactive, but they are polluted with different pollutants too; therefore not only analcime prevails in the synthesis products. This fact determined the raw materials used in this investigation, – analcime was synthesized from pure reagent mixtures. The synthesis conditions were chosen according to data summarized

Table 1. **Conditions of analcime synthesis**
1 lentelė. **Analcimo sintezės sąlygos**

No.	Molar ratio of reactants in synthesis mixtures mol/Al_2O_3			Reagents	Conditions of synthesis	Analcime composition mol/Al_2O_3		
	Na_2O	SiO_2	H_2O			Na_2O	SiO_2	H_2O
1	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*
1.	–***	4	~80	Solution of sodium silicate, sodium aluminate	282 °C, 46 h	–	–	–
2.	>1	4	–	$Al_2O_3 \cdot 3H_2O$, NaOH, silicic acid	180 °C	1	4	2
3.	1	2–6	–	Glass, $Na_2O \cdot Al_2O_3 \cdot SiO_2$	200 °C	1	2..6	1..2
4.	1	2–10	–	Silicic acid, $NaAlO_2$	300 °C, 24h	–	–	–

Table 1 (continued)
1 lentelė (tęsinys)

1	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*
5.	1	2.5–13	–	Gel $Na_2O \cdot Al_2O_3 \cdot SiO_2$	120–180 °C, 19-103 days	–	–	–
6.	–	~9	–	Volcanic glass and NaOH solutions	120–150 °C, 4 h–25 days	~1	~4–4.2	~2.5..2.9
7.*	~3–18	~20–100	~300–1000	Technical glass and NaOH solutions	180 °C pressure of the saturated steam, 4–8 h	–	–	–
8.	1	2	–	Jadeite $NaAlSi_2O_6$	250–650 °C, p = 1200–1900 MPa, hydrothermal treatment of the mineral	–	–	–
9.	–	–	–	Glass $(NaAlSi_3O_8)_x \cdot (CaAl_2Si_2O_8)$ (1-x) · · 4.25 SiO_2 x = 0; 0.2; 0.4; 0.6; 0.8; 1	200–450 °C, p = 200 MPa, hydrothermal crystallization of the glass	–	–	–
10.**	4.5	4.5	368	Aluminum sulfate $(Al_2(SO_4)_3 \cdot 16H_2O)$, sodium metasilicate $(Na_2O \cdot SiO_3 \cdot 5H_2O)$, triethanolamine $(N(C_2H_4OH)_3)$	200 °C, 24 h	~1	~4.3	2
11.	pH > 10	8–10	–	Solutions of sodium metasilicate, aluminate and NaOH	254 °C and 293 °C	–	3.7–6.3	–
12.	1…4	4–6	–	Solutions of sodium metasilicate, aluminate and NaOH	180 °C	–	–	–

* With other zeolites.
** Unconfirmed data of the patent, there were organic compounds in the initial mixture.
*** Not shown or nameless data.

by S. Zhdanov and E. Jegorov (Жданов и др., 1968). They stated that:

– analcime together with wairacite (Ca ionic analogue of analcime) are zeolites, which form at comparatively high temperatures;

– analcime is formed at a lower temperature if there is more Na_2O in the raw material;

– analcime can be synthesized in conditions in which usually another zeolite – mordenite – is formed, if the amorphous raw materials are changed by crystalline raw materials.

In agreement with the data of the literature and on the basis of the used equipment possibilities, analcime was synthesized in an autoclave, at a comparatively low temperature (180 °C). In most of quoted works it is shown that the highest temperature of analcime synthesis under saturated steam pressure is 300….350 °C. The molar ratio of sodium, silicon and aluminum oxides was chosen the same as in the 2nd, 11th and 12th synthesis cases according to Table 1. The quantity of water in the synthesis mixtures was equal to the average quantity indicated by D. Breck as typical for industrial zeolite synthesis (Брек, 1985).

CONDITIONS OF ANALCIME SYNTHESIS

1. Raw materials. Pure analytical grade silicic acid $SiO_2 \cdot nH_2O$ was used in the synthesis mixtures. Mass losses after its burning at 1000 °C (exuding water) reached 20% by mass. They were evaluated

by counting the ratio of the components in autoclave treated mixtures.

The other raw materials were pure analytical gibbsite (hydroargilite) $Al_2O_3 \cdot 3H_2O$ or α-$Al(OH)_3$. Water content in gibbsite was established during heating at 1000 °C. Its mass losses after heating reached to 34% by mass, *i.e.* almost the same as should be according to the stoichiometric proportion of the chemical combination.

Aluminum oxide (γ-Al_2O_3) was obtained by heating gibbsite at 550 °C for 3 h. A presumption was made that the solubility of aluminum oxide with a defective spinel structure should be better than of gibbsite (Нечипоренко и др., 1986). XRD analysis of γ-Al_2O_3 and gibbsite proved that the combinations were free of admixtures, which could be found by X-ray diffraction analysis methods.

The pure grade sodium hydroxide was used as the alkaline component in this research. It has more than 97% NaOH by mass (sodium carbonate was up to 1.5% and other admixtures were less).

2. Synthesis mixtures. Dry reagents (raw materials of the synthesis) were sifted through a sieve, mesh size 80 μm, then thoroughly mixed and poured into stainless dishes. Sodium hydroxide solutions of necessary concentrations were poured into these dishes later and the mixtures were mixed again. The total amount of the dry mixture components were about 30…40 g (depending on the ratio between aluminum oxide and silica in a mixture, see Table 2). The volume of NaOH solutions was always more than of dry components and it flooded dry components of each mixture. The dishes with the mixtures of raw materials were put into an autoclave 1.5 h following the moment of alkali solution infusion. The initial H_2O / Al_2O_3 molar ratio of the mixtures was always the same and equalled 115.

3. Synthesis conditions. The mixtures of the raw materials were not mixed during autoclave treatment; therefore the conditions of analcime synthesis were similar to natural ones. These peculiarities determined the segregation of the synthesis products. Analcime was synthesized in an autoclave 0.05 m^3 in volume at a temperature of 180 ± 5 °C and under saturated steam pressure. The suspensions of raw materials were treated for 18 h at a maximum temperature (the duration of the isothermal cycle of the autoclave treatment t_{iz}) and this maximum temperature and pressure were attained within 1.5 h.

After autoclave treatment the synthesized products were cooled for the next 6 h. During cooling, the autoclave was closed. The synthesis products were pulled out when the pressure in the autoclave became equal to atmospheric. After that the products were examined, washed out with distilled water and dried up at 105 °C. XRD analysis samples were pulverized in a pestle and sifted through a 80 μm mesh size sieve.

Table 2. **Chemical composition (molar ratios) of the raw material mixtures**
2 lentelė. **Žaliavos mišinių cheminė sudėtis (molio santykis)**

Na_2O	Al_2O_3*	SiO_2	Notation of mixture*
1	1	4	A0
1	1	5	B0
1	1	6	C0
2	1	4	A1
2	1	5	B1
2	1	6	C1
3	1	4	A2
3	1	5	B2
3	1	6	C2
4	1	4	A3
4	1	5	B3
4	1	6	C3

* Mixtures with gibbsite (hydroargilite) are marked with a supplemental symbol – a dash, for example A0', B3' etc.

4. Identification of synthesis products. Synthesized products were investigated with a DRON-2 diffractometer (copper anode, CuK_α radiation, nickel filter, excitation voltage 30 kV at 12 mA, goniometer splits 0.5 and 1.0 mm wide, external standard – quartz powder, scanning angle velocity – 2 °/min). The reproducibility of each XRD pattern was verified by recording the XRD pattern several times after sample holder rotation. The recorded XRD patterns were ciphered using (ICDD, 1999) database.

SYNTHESIS RESULTS

The synthesized minerals (products) of each synthesis bath are enumerated in Table 3. XRD patterns of analcime synthesis products are shown in Fig. 1.

Examination of the synthesis products showed their segregation. The segregation of the synthesis products was more visible in mixtures with gibbsite. The synthesis mixtures with γ-Al_2O_3 were less stratified (see Table 3). XRD analysis of each layer of the synthesis products showed that analcime is always formed in surface layers (sometimes together with other zeolites and feldspatoids, such as zeolite Pc, hydrosodalite, cancrinite and others). Usually analcime is a dominating phase in the surface of synthesis products, though it could be not dominant in the whole synthesis batch volume. It might be that analcime prevails in the surface layer of the synthesis products due to a higher alkali concentration.

Table 3. **Mineralogical composition of the autoclave treated products**
3 lentelė. **Autoklavu apdorotų produktų mineraloginė sudėtis**

Notation of mixture *	Mineralogical composition of the products **
A0'	Biomite, gibbsite, hydrosodalite, mordenite, nepheline hydrate, amorphous phase
A1'	Analcime, hydrosodalite, zeolite Pc, traces of cancrinite
A2'	Analcime, hydrosodalite, less cancrinite and traces of biomite
A3'	Cancrinite, hydrosodalite, analcime
B0'	Gibbsite, biomite, traces of nepheline hydrate and amorphous phase
B1'	Analcime (dominant), traces of hydrosodalite, zeolite Pc and biomite
B2'	Biomite, analcime, cancrinite and hydrosodalite
B3'	Analcime, hydrosodalite, less cancrinite
C0'	Gibbsite, biomite, large quantity of amorphous phase
C1'	Mordenite, analcime, zeolite Pc and biomite
C2'	Analcime, less hydrosodalite, traces of biomite and amorphous phase
C3'	Analcime, hydrosodalite, less cancrinite and amorphous phase
A0	Analcime, traces of zeolite Pc and biomite
A1	Analcime, less zeolite Pc and biomite
A2	Analcime, less hydrosodalite
A3	Analcime, less hydrosodalite
B0	γ-Al_2O_3 and traces of analcime (beginning of crystallization)
B1	Analcime, less hydrosodalite and traces of zeolite Pc
B2	Analcime, biomite, traces of zeolite Pc
B3	Analcime, less hydrosodalite
C0	Analcime, low quantity of biomite and zeolite Pc
C1	Analcime, traces of biomite
C2	Analcime, zeolite Pc, biomite
C3	Analcime, less hydrosodalite and traces of zeolite Pc

* The synthesis mixture marks are the same as in Table 2.
** All synthesized minerals are enumerated by their relative amount in the synthesis products.

Other zeolites, such as hydrosodalite and zeolite Pc, were synthesized during autoclave treatment, too. It's known that hydrosodalite formation is stimulated by various anion admixtures and high alkali concentrations. When gibbsite was used in synthesis mixtures, large quantity of other minerals in the synthesis products were found. The quantity of accessory phases after autoclave treatment of mixtures with γ-Al_2O_3 was lower. We can conclude that various anions – admixtures of gibbsite, such as carbon, chloric groups, etc., surplus of Na_2O in the synthesis mixtures stimulate formation of such feldspatoids as cancrinite. Part of these volatile admixtures evaporated when we heated gibbsite, turning it into γ-Al_2O_3. Otherwise, there are too little carbon groups in sodium alkali, that is why cancrinite is not formed from mixtures with γ-Al_2O_3.

Mordenite is a zeolite rich in SiO_2. During this investigation it was formed because of a relatively large amount of SiO_2 dissolved in the liquid phase of the synthesis suspensions (mixtures) prepared with gibbsite. The solubility of gibbsite and the amount of its main constituent, Al_2O_3, in the suspensions are respectively lower. That's why zeolite with a high SiO_2 / Al_2O_3 molar ratio was synthesized from the mixtures with gibbsite. The SiO_2 / Al_2O_3 molar ratio of mordenite is equal to 5…6 and the SiO_2 / Al_2O_3 molar ratio of analcime is about 2.

The amount of analcime in the synthesis products was evaluated by the intensity of its XRD peak d = 1.74 Å. Though the intensity of this peak is taken the gauge of only 60 units in the standard analcime XRD pattern, in XRD patterns obtained by us it was a single intensive peak. This peak is typical only of the XRD pattern of analcime and the peaks belonging to another mineral do cover it. Dependence of this peak intensity on the synthesis conditions is shown on Fig. 2.

It can be stated that when the SiO_2 / Al_2O_3 molar ratio in the synthesis mixtures grows, the content of analcime in the synthesis products increases too. This rule is valid for mixtures with gibbsite and with

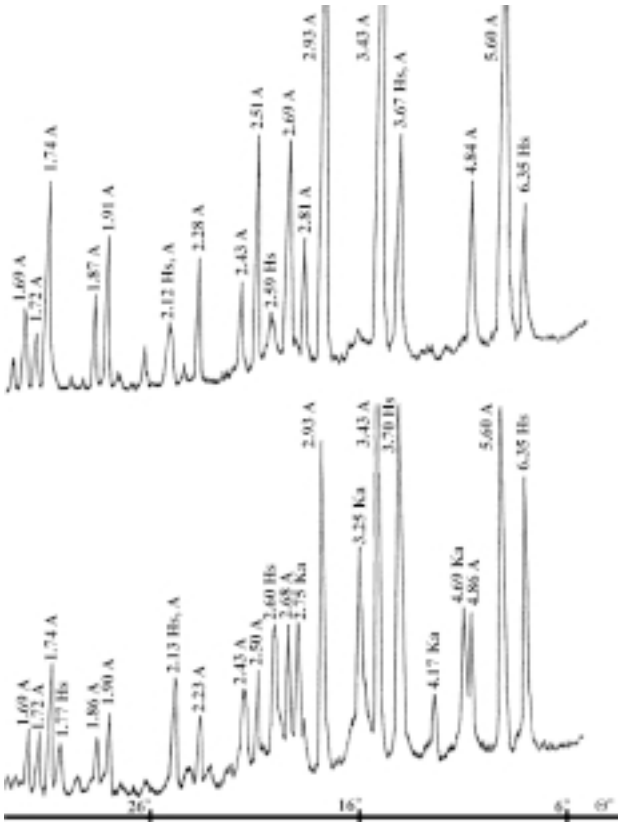

Fig. 1. XRD patterns of the analcime synthesis products. Above – XRD pattern of the products obtained after autoclave treatment of a mixture of γ-Al_2O_3, $SiO_2 \cdot nH_2O$ and sodium alkaline solution. Below – XRD pattern of the analogous synthesis products, where γ-Al_2O_3 was substituted by gibbsite. In the both synthesis mixtures the molar ratio SiO_2 / Al_2O_3 equalled 5 and the molar ratio Na_2O / Al_2O_3 equalled 4. The symbol of analcime peaks in XRD pattern is letter A, the symbol of hydrosodalite is Hs and of cancrinite Ka. The dimension of the d-spacing in the XRD patterns is ångströms

1 pav. Analcimo sintezės produktų rentgenogramos. Viršuje – produktų, gautų iš γ-Al_2O_3, $SiO_2 \cdot nH_2O$ ir natrio šarmo tirpalo mišinio, rentgenograma. Apačioje – produktų, gautų apdorojus autoklave gibsito, $SiO_2 \cdot nH_2O$ ir natrio šarmo tirpalo mišinį, rentgenograma. Abiejuose mišiniuose SiO_2/ Al_2O_3 molio santykis buvo lygus 5, o Na_2O / Al_2O_3 – 4. Analcimo smailės rentgenogramose pažymėtos A raide, hidrosodalito – Hs, kankrinito – Ka. Tarpplokštuminiai atstumai rentgenogramose surašyti angstremais

γ-Al_2O_3. Dependence of analcime level on the initial content of Na_2O in the synthesis mixtures was not obvious in this research.

Traces of analcime were found in the synthesis products when the raw material molar ratio Na_2O / / Al_2O was equal to 1. These mixtures are marked with the symbol "0" in Table 2. This enables us to draw a conclusion that the amount of Na_2O must be in excess in comparison with the amount of Al_2O_3.

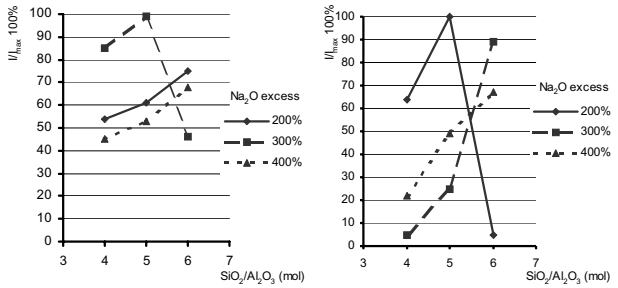

Fig. 2. Dependence of analcime peak d = 1.74 Å relative intensity on the molar ratio SiO_2 / Al_2O_3 in the mixture of raw materials. I_{max} is the highest intensity of this peak, and it was obtained when analcime was synthesized from the same initial raw materials; *a* – mixtures with γ-Al_2O_3, *b* – mixtures with gibbsite

2 pav. Analcimo smailės (d = 1,74 Å) intensyvumo priklausomybė nuo SiO_2 / Al_2O_3 santykio pradinėje žaliavoje. I_{max} – didžiausias šios smailės intensyvumas, gautas sintezavus analcimą iš tos pačios pradinės žaliavos; *a* – mišiniai su γ-Al_2O_3, *b* – mišiniai su gibsitu

CONCLUSIONS

Based on the results of the investigations described above, the following conclusions may be made:

1. Analcime with very little admixtures under the selected conditions of autoclave treatment can be obtained from mixtures of NaOH solution, active γ-Al_2O_3 and silicic acid, if the molar ratio in the raw materials is equal to 1–2:1:4–6. The use of γ-Al_2O_3 for analcime synthesis is preferable, because zeolitization of the synthesis mixture with this component occurs easier and produces relatively more analcime.

2. The presence of gibbsite (hydroargilite) in the analcime synthesis bath changes the mineral composition of the synthesis products in comparison with mixtures without gibbsite. Relatively large amounts of different zeolites and feldspatoids (zeolite Pc, hydrosodalite, cancrinite, and nepheline hydrate) are synthesized when gibbsite is used.

3. The amount of analcime in the synthesis products depends on the initial molar ratio SiO_2 / Al_2O_3 of the raw materials. If the molar ratio grows from 4 to 6, the quantity of analcime in the synthesis products is growing too. This regularity is evident if γ-Al_2O_3 is used in the synthesis mixtures.

4. The influence of Na_2O levels (within the limits studied) on the output of analcime is not very obvious, but it is evident that the molar ratio Na_2O / Al_2O_3 in the mixture of raw materials must be higher than 1.

5. Judging by the mineral composition of the new formations, the amount of secondary synthesis products such as minerals–admixtures (hydro-

sodalite, cancrinite, zeolite Pc and others) depends on the Al_2O_3 form in the initial raw material mixtures. The amount of these secondary products will be lower in case of using the active Al_2O_3 (γ-Al_2O_3) form. The advantage of this form of aluminum oxide over gibbsite could be proven by the fact that secondary minerals such cancrinite and mordenite are not formed in the synthesis mixtures with γ-Al_2O_3.

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VEIKSNIAI, TURINTYS ĮTAKOS ANALCIMO SUSIDARYMUI

S a n t r a u k a

Geochemikų nustatytos gamtinės analcimo susidarymo sąlygos neracionalios gamyboje – ilga sintezės trukmė, didelis slėgis, aukšta temperatūra. Analcimas dažniausiai sintetinamas aukštoje temperatūroje (per 180°C) ir įvairiame slėgyje (0,15...100 MPa). Šiuose tyrimuose aprašoma analcimo sintezės (180°C temperatūra, sočiųjų vandens garų slėgis, 19,5 valandos sintezės trukmė) rezultatų priklausomybė nuo žaliavų atmainos ir santykio pradiniuose mišiniuose. Sintezės metu žaliava nuolat nepermaišoma, tuo ji panaši į gamtinę. Dažniausiai tokiomis sąlygomis gautų sintezės produktų paviršiuje vyrauja analcimas, nors visoje naujadarų masėje jo gali būti ir nedaug. Padaryta išvada, kad paviršiniame sintezės produktų sluoksnyje analcimas vyrauja dėl didesnės natrio šarmo koncentracijos. Aprašytomis analcimo sintezės sąlygomis γ-Al_2O_3, kaip viena iš žaliavų, yra pranašesnis už gibsitą.

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УСЛОВИЯ, ВЛИЯЮЩИЕ НА ФОРМИРОВАНИЕ АНАЛЬЦИМА

Р е з ю м е

Условия формирования анальцима, установленные геохимиками, нерациональны в производстве – слишком велики продолжительность синтеза, давление и температура синтеза. Обычно анальцим синтезируется из алюмосиликатного геля в условиях высоких температур (свыше 180°C) и давлений (0,15–100 МПа). В данных исследованиях установлена зависимость результатов синтеза анальцима (при температуре 180°C, в условиях давления насыщенного водяного пара, при общей продолжительности синтеза 19,5 ч) от вида и соотношения компонентов сырьевых материалов. Смесь сырьевых материалов в автоклаве не перемешивалась, поэтому можно утверждать, что условия синтеза анальцима близки к существующим в природе. Как правило, на поверхности продуктов синтеза при данных условиях анальцим является преобладающей фазой, хотя в целой массе продуктов синтеза его может быть и немного. По-видимому, на расслоение продуктов синтеза влияют изменения концентрации щелочи натрия. Установлено, что в данных условиях синтеза как один из компонентов сырьевой смеси предпочтительно использовать γ-Al_2O_3, а не менее активный гиббсит.