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Effects of ultrasonic treatment on zeolite NaA synthesized from by-product silica

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ABSTRACT

The synthesis of zeolite NaA from silica by-product was carried out in the presence of 20 kHz ultrasound at room temperature. Zeolites obtained in this type of synthesis were compared to zeolites obtained by performing conventional static syntheses under similar conditions. The sonication effects on zeolite NaA synthesis were characterized by phase identification, crystallinity etc. The effects of different parameters such as crystallization time and initial materials preparation methods on the crystallinity and morphology of the synthesized zeolites were investigated. The final products were characterized by XRD and FT-IR. It was possible to obtain crystalline zeolite NaA from by-product silica in the presence of ultrasound.

1. Introduction

Zeolite is a family of hydrated aluminosilicate minerals that contain alkali and alkaline earth metals. Zeolites are ecologically clean, inert and non-toxic substances, fully suitable to use in many fields of industry, agriculture, household economy and environment protection as well as for the liquidation of consequences of ecological catastrophes and elsewhere [1].

Zeolites are usually synthesized under hydrothermal conditions, from solutions of sodium aluminate, sodium silicate, or sodium hydroxide [2]. In this research zeolitic material was synthesized by ultrasonic treatment and by conventional experiments.

Ultrasonic waves can propagate in different materials and for this reason ultrasonic waves can be used to investigate the properties of polymers, metals, liquids, gases, and to study the effect of the waves on the materials. Polymerization and de-polymerization reactions and many other phenomena may occur in cavitating liquids under certain conditions. Ultrasonic irradiation provides unique reaction conditions via acoustic cavitation [3].

Ultrasonic irradiation of aqueous liquids generates free radicals. This leads to decomposition of oxygen and water molecules and generation of H and HO radicals: $H_2O \rightarrow H^{\cdot} + HO^{\cdot}$ [4]. These radicals can recombine to return to their original form or combine to produce H_2 and H_2O_2. They can also produce HO_2 by combination with O_2. These strong oxidants and reductants are utilized for various sonochemical reactions in aqueous solutions. With a simple modification of reaction conditions, various forms of nanostructured materials can be synthesized.

In the synthesis of zeolite NaA and MCM-49 aging of the synthesis mixture is a prerequisite for fast crystallization. It was also found that the synthesis of an unaged mixture can be improved dramatically by adding an aged synthesis mixture to it. Aging can noticeably broaden the crystallization range of zeolites and shorten the crystallization time [5,6].

Synthesis of zeolite A from an optically clear sodium aluminosilicate solution was carried out in the presence of ultrasound. It was possible to obtain highly crystalline zeolite A in the presence of ultrasound [7]. Andac et al. [2] studied the preparation of zeolite 4A coatings on stainless steel substrates by direct synthesis at 60 °C from a clear aluminosilicate solution in the presence of ultrasound of 35 kHz. XRD and SEM analyses revealed that more nuclei formed at earlier times in the presence of ultrasound, allowing the preparation of thinner and continuous zeolite coatings in significantly shorter synthesis times.

The synthesis of MCM-22 zeolite under hydrothermal crystallization conditions has been performed by an ultrasonic-assisted

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aging procedure. The ultrasonic-assisted aging of the initial aluminosilicate gel can shorten the crystallization time of MCM-22. The acoustic cavitation caused by the ultrasonic treatment is believed to be responsible for these positive results due to its cracking of the crystal seeds and improving the solubility of silicate species [8].

The synthesis of zeolites from three samples of fly ash was carried out through a low-temperature (25–60 °C) hydrothermal process with a NaOH pre-fusion treatment preceded by sonication. XRD and SEM investigations demonstrate that the application of ultrasonic treatment facilitates the formation of zeolites at lower-temperature (25 °C) compared to the syntheses not preceded by sonication [9].

The synthesis of zeolites from fly ash was performed through a low-temperature hydrothermal process with seawater. Ultrasonic treatment reduced the crystallization temperature. The results confirm the previous findings obtained using distilled water. The crystallization efficiency is surprisingly lower when seawater is used [10].

In this research 5A zeolite nanocrystals were synthesized and the effects of the controlling parameters and effects of ultrasound on morphological and crystal size modifications were investigated. Kaolin and/or aluminate derived from kaolin were utilized as the main sources of Al. Nanoemulsion-ultrasonic method produces smaller, more uniform, and purer zeolite nanocrystals with greater ion exchange capacity than the conventional synthesis methods [11].

Sonochemical synthesis of zeolite A was conducted by ultrasonic irradiation of mixtures of metakaolinite and NaOH solution. The enhancement of nucleation and crystallization rate of zeolite A was achieved by ultrasound. Comparing the results with those of conventional methods, this heterogeneous reaction was particularly accelerated by the ultrasound. The use of ultrasound enables us to obtain well-dispersed fine zeolite A particles with the mean particle size of around 1 mm. The high solid concentration in the suspension, however, hindered the ultrasound from intense agitating, resulting in the decrease of zeolite A yield [12].

Pal et al. [13] proposed that NaP zeolite nano crystals were synthesized by sonochemical method at room temperature with crystallization time of 3 h. NaP zeolites are directly formed by ultrasonic treatment without the application of autogenous pressure and also hydrothermal treatment. The effect of ultrasonic energy and irradiation time showed that with increasing sonication energy, the crystallinity of the powders decreased but the phase purity remained unchanged. With increasing irradiation energy and time, the crystallinity of the synthesized zeolite samples increased slightly.

In the present investigation, we report the synthesis of NaA zeolite at room temperature and short crystallization time by sonochemical method. For comparison, the same synthesis was carried out by conventional hydrothermal method. NaA zeolite was synthesized from AlF_3 production waste.

2. Experimental procedure

2.1. Materials

The following reagents were used: NaOH, $Al(OH)_3$ and H_2O. Along with the reagent materials, by-product silica (AlF_3 production waste) was used as Si and Al source.

In the majority of cases the raw materials for the formation of natural zeolites are unstable semi-crystal substances, which within a wide interval of temperatures are impacted by alkaline solutions. Such substances can be volcanic glasses and ashes, amorphous forms of SiO_2, or production residue. One of those materials is by-product silica – amorphous, finely divided substance, called silica gel. The X-ray diffraction analysis showed that by-product silica (Fig. 1) contained amorphous $SiO_2{\cdot}nH_2O$ (rise from 10° up to 30°) and $AlF_3{\cdot}3.5H_2O$. As $SiO_2{\cdot}nH_2O$ is in amorphous state, reflections of by product silica are not visible in the XRD pattern.

High fluoride concentrations were found after the elemental analysis of amorphous silica (Table 1). Basing on the law of mass conservation we may state that silica gel used for the synthesis contained: 63.85% SiO_2, 18.38% H_2SiF_6, 9.27% AlF_3, 8.98% H_2O. Presumably some unreacted H_2SiF_6 in the investigated silica has been left in addition to these compounds. The determination of H_2SiF_6 in heterogeneous systems is problematic.

In order to compare the intensity of zeolite formation in ultrasonication, part of the aforementioned suspensions were hydrothermally treated, i.e. suspension specimens were heated at 100 °C temperature.

Mixtures of initial materials (molar ratio: $Na_2O{:}SiO_2{:}Al_2O_3$ H_2O = 2:2:1:10) after 24 h of aging, or without aging (Table 2), were irradiated with ultrasonic waves for 5, 10, 20 and 30 min.

After ultrasonication or hydrothermal treatment the solid matter of the suspension was separated by means of filtering, whereas Na_2SiF_6, NaF and excess NaOH were removed by washing

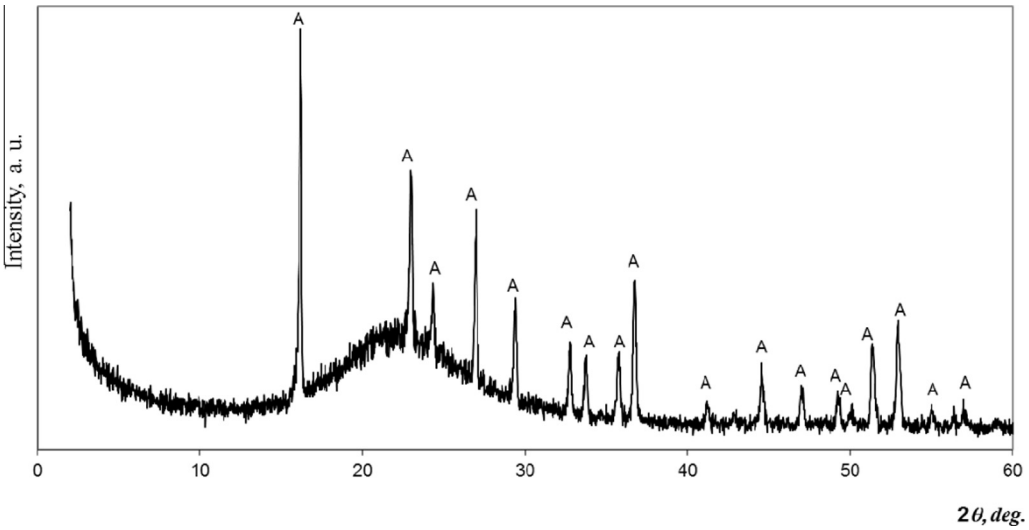

Fig. 1. X-ray diffraction pattern of by-product silica. *Note:* A – $AlF_3{\cdot}3.5H_2O$.

Table 1
By-product silica element composition.

Element	Wt (%)	At (%)
Oxygen	41.98	52.09
Aluminum	2.98	2.19
Silicon	33.43	23.64
Fluorine	20.84	21.79
Calcium	0.30	0.15
Iron	0.46	0.16
Total	100	100

Table 2
Plan of the experiment.

Method sequence number	Synthesis conditions
1	Initial dry materials mixed, diluted with required amount of water and irradiated with ultrasonic waves (power 210 W)
2	$Al(OH)_3$ complete diluted in NaOH solution (sodium aluminate obtained), the required amount of by-product silica and water added. The liquid irradiated with ultrasonic waves (power 210 W)
3	Initial dry materials mixed, diluted with required amount of water and aged for 24 h. Afterwards the liquid irradiated with ultrasonic waves (power 210 W)
4	Initial dry materials mixed, diluted with required amount of water and irradiated with ultrasonic waves (power 250 W)
5	$Al(OH)_3$ diluted in NaOH solution (sodium aluminate obtained), the required amount of by-product silica and water added and subjected to hydrothermal treatment
6	Initial dry materials mixed, diluted with required amount of water and subjected to hydrothermal treatment

the synthesis products with water. Afterwards the product was dried at 100 °C temperature.

2.2. Experimental techniques

The X-ray powder diffraction data were collected using DRON-6 X-ray diffractometer of Bragg–Brentano design using Ni-filtered Cu Kα radiation and graphite monochromator. Operating voltage of 30 kV and emission current of 20 mA were used in the experiments.

IR spectra were recorded with Perkin Elmer FT-IR System spectrometer. 1 mg of the substance was mixed with 200 mg of KBr and compressed in a forming press under vacuum for the IR analysis.

Particle size distribution and specific surface area were determined by "Mastersizer 2000" instrument from Malvern. Red light was produced by helium-neon laser and blue light was obtained from a solid phase source.

The structure of zeolitic material was studied by scanning electron microscope. A high resolution scanning electron microscope FEI Quanta 200 FEG with a Schottky field emission gun (FEG) was used for the research. Chemical compositions of by-product silica were investigated by an energy-dispersive X-ray spectrometer (EDS) with silicon type drift droplet detector.

Zeolite A was produced by using sonication treatment, with ultrasonic dispergation device BANDELIN electronic UW3400, or by heating the suspension in the kiln SNOL 200/200. Ultrasonication procedure lasted from 5 to 30 min using 20 kHz and 210 or 250 W power ultrasonic waves.

3. Results and discussions

In order to identify the crystalline phase and crystallinity (%) setting, the X-ray analysis data was used. It is the method of standard addition using diffracted X-ray intensity. Crystallinity (%) was calculated according to the Eq. (1). Commercial zeolite A was used as a standard. The total sum of the intensity of major peaks in XRD patterns is a semi-quantitative index of the crystallinity of these samples [11,14,15].

$$\%\,\text{Crystallinity} = \left(\frac{\sum \text{intensity of XRD peaks of product}}{\sum \text{intensity of XRD peaks of standard}}\right) \times 100 \quad (1)$$

The percentage of crystallinity was taken as the sum of the unknown materials divided by the sum of the peak heights of the standard material that had been assumed to be 100% crystalline.

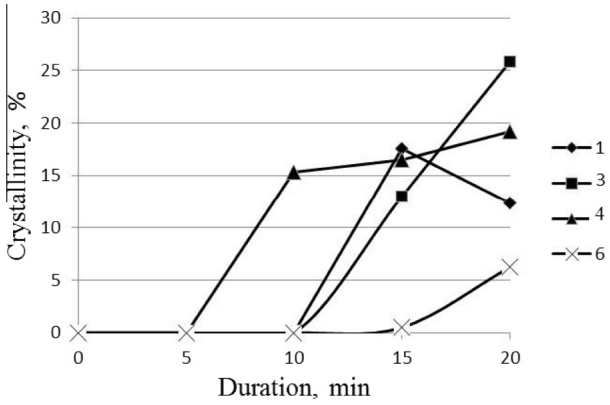

Fig. 2. Zeolite Na-A crystallinity dependence on synthesis type and duration parameters.

Fig. 2 illustrates the crystallinity curves of zeolite present in the specimens calculated from Eq. (1); the following diffraction angles were used: 2θ = 7.2, 10.3, 12.5, 16.1, 21.7, 24, 26.1, 27.3, 30, 30.9, 32.6, 33.4, 34.2, 35.7, 36.5, 44.2, and 47.4.

Method 1 ultrasonic synthesis analysis results (Table 2) revealed that the target product zeolite NaA is not formed during the synthesis that lasts 5–10 min (Fig. 3). Synthesis with duration extended to 15 min yields zeolite NaA with 17.59% crystallinity calculated from Eq. (1), whereas when the synthesis time is extended up to 20 min, part of zeolite NaA transforms into hydrosodalite, and zeolite NaA crystallinity degree drops to 12.36%.

Zeolites are usually synthesized under hydrothermal conditions from solutions of sodium aluminate, sodium silicate, or sodium hydroxide [1]. Such conditions are typical of those found in the Earth's crust where some zeolites are found naturally. According to numerous studies [16,17], the most feasible zeolite synthesis methods involve components solved in water where zeolitization reactions take place in colloid systems. Literature sources report that zeolite NaA can be synthesized from some aluminum sources [18]. A disadvantage of using salts is that, after pH adjustment or addition of alkali silicate solutions, alkali salts are formed which have a strong electrolytic effect on gel formation. For example, such salts may cause sodalite to be crystallized instead of zeolite A type materials. Generally, sodium aluminate was used as an aluminum source to synthesize various zeolites.

The analysis of method 2 (Table 2) syntheses results revealed that zeolite NaA is not formed in these reactions (Fig. 4). Presumably, under the investigated zeolite NaA synthesis conditions it would be unfeasible to dissolve $Al(OH)_3$ and amorphous silica gel in NaOH in advance. From manufacturing viewpoint it enables to avoid an additional phase of sodium aluminate production.

24-h aging of the mix of initial substances before the synthesis has a great influence on the crystallinity of the minerals of the final product zeolite. Aging relates to the appearance of crystallization centers, the maximum size of crystals and shorter crystallization

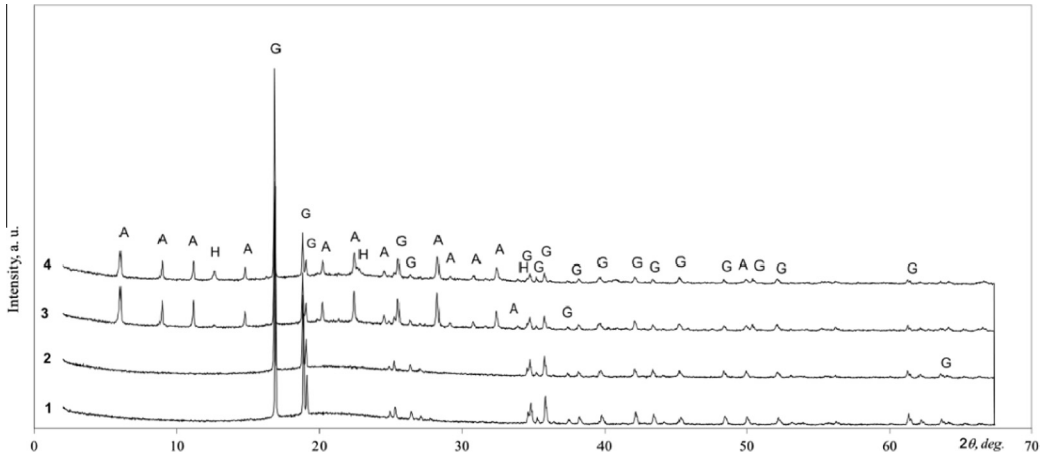

Fig. 3. X-ray diffraction patterns of method 1 synthesis products (Table 2), with ultrasound treatment time: 1 – 5 min; 2 – 10 min; 3 – 15 min; 4 – 20 min. *Notes:* G – $Al(OH)_3$; A – zeolite NaA; H – hydrosodalite.

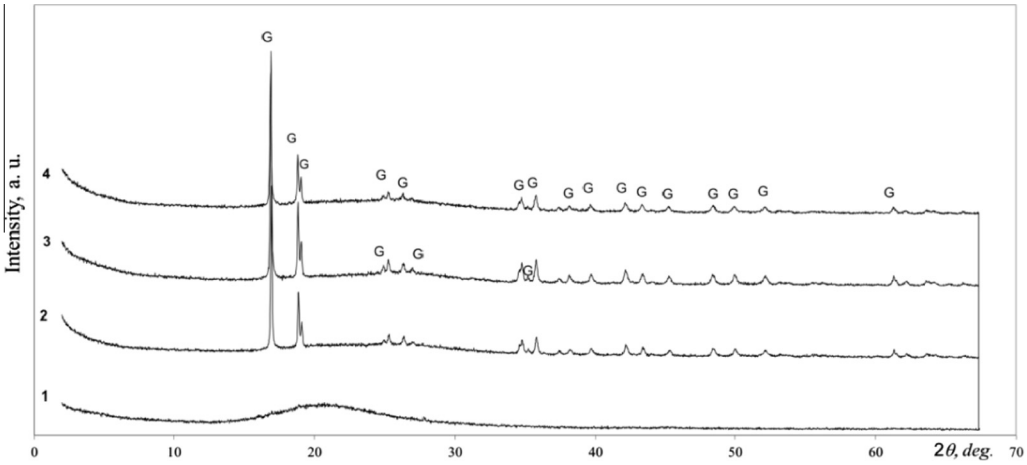

Fig. 4. X-ray diffraction patterns of method 2 synthesis products (Table 2), with ultrasound treatment time: 1 – 5 min; 2 – 10 min; 3 – 15 min; 4 – 20 min. *Notes:* G – $Al(OH)_3$.

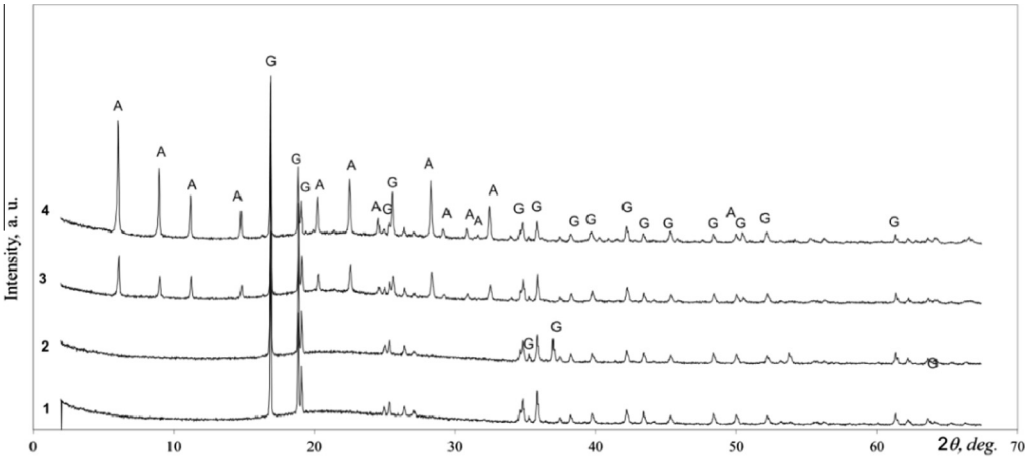

Fig. 5. X-ray diffraction patterns of method 3 synthesis products (Table 2), by using 24 h aging and with ultrasound treatment time: 1 – 5 min; 2 – 10 min; 3 – 15 min; 4 – 20 min. *Notes:* G – $Al(OH)_3$; A – zeolite NaA.

time [19]. X-ray diagrams of method 3 synthesis (Fig. 5) show that there is optimal synthesis time enough for the formation of zeolite NaA. After 20 min of ultrasonic synthesis the crystallinity of the zeolite is 25.81%, which is significantly higher compared to the crystallinity of the specimens (Fig. 2) obtained by the synthesis method 1 of the same length. When ultrasonic treatment time is shortened to 15 min, the crystallinity of zeolite NaA is about 2% lower (15.23%) compared to the crystallinity obtained in the synthesis without aging (17.59%).

The power of ultrasonic waves also has a great influence on sonochemical reactions. It was found that the increase of ultrasonic irradiation power from 210 to 250 W leads to faster zeolite NaA

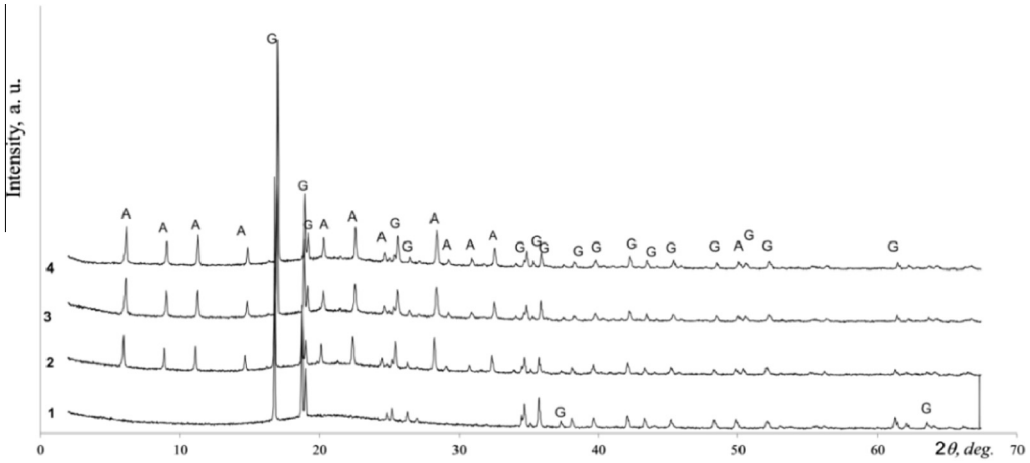

Fig. 6. X-ray diffraction patterns of method 4 synthesis products (Table 2) with ultrasound treatment time: 1 – 5 min; 2 – 10 min; 3 – 15 min; 4 – 20 min. *Notes:* G – $Al(OH)_3$; A – zeolite NaA.

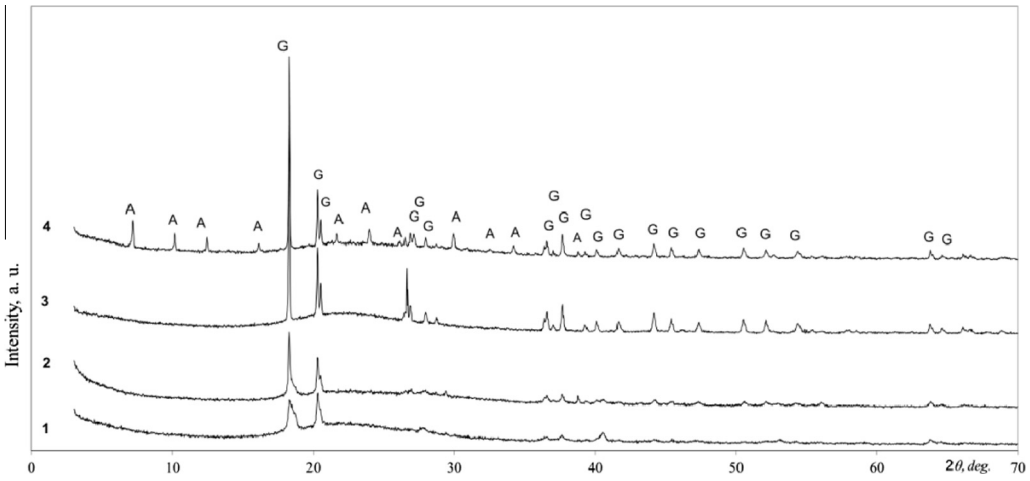

Fig. 7. X-ray diffraction patterns of hydrothermal synthesis products: 1 – the duration of hydrothermal treatment 5 min, method 5; 2 – the duration of hydrothermal treatment 20 min, method 5; 3 – the duration of hydrothermal treatment 5 min, method 6; 4 – the duration of hydrothermal treatment 20 min, method 6. *Notes:* G – $Al(OH)_3$; A – zeolite NaA.

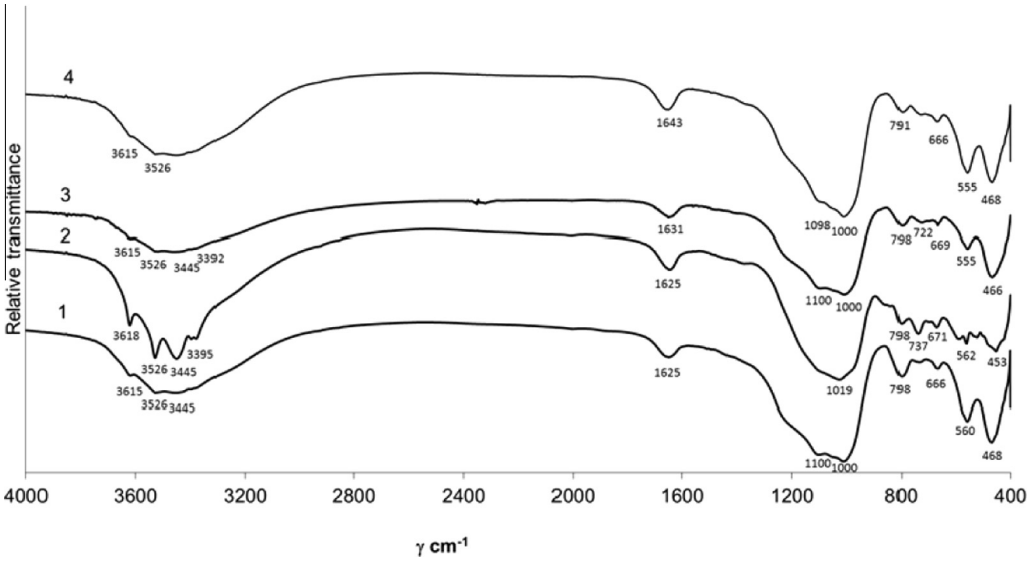

Fig. 8. IR curves of synthesis products. The duration of ultrasonic treatment was 20 min. *Notes:* 1 – method 1, 2 – method 2, 3 – method 3, 4 – method 4.

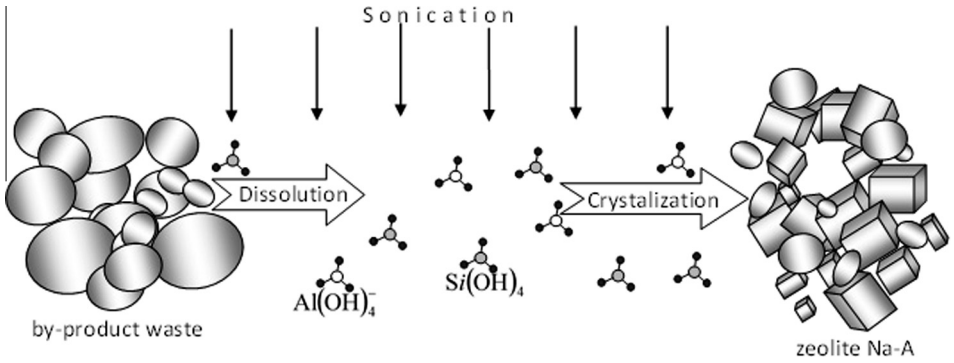

Fig. 9. The scheme of zeolite formation.

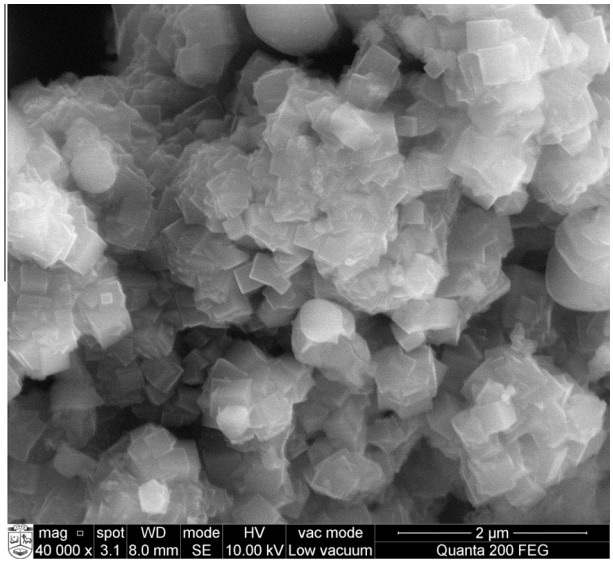

Fig. 10. SEM images of method 3 synthesis products (Table 2), by using 24 h aging and with 20 min ultrasound treatment time.

formation and shortens the synthesis time from 15 to 10 min (Fig. 6).

To evaluate the efficiency of zeolite NaA syntheses, part of the syntheses were done under hydrothermal conditions (Table 2, method 5, 6). After 20 min the reaction of initial materials with $Al(OH)_3$ produced zeolite NaA with low 6.22% crystallinity degree (Fig. 7).

FTIR spectroscopy was used to confirm zeolite crystallinity and phase purity. Infrared spectra absorption bands can be attributed to two types of vibrations: (1) oscillations inside TO_4 tetrahedrons that make up the primary units of the internal structure; (2) vibrations through external links of tetrahedrons. The second type depends on the zeolite structure, on the nature of tetrahedron binding into secondary units of the structure, and also on the properties of the structure formed at zeolite cavity inlets.

Fig. 8 illustrates that all studied IR curves are absorption bands distributed in the area ranging from 4000 to 400 cm^{-1}. The characteristics of zeolite structure are determined in 1100–400 cm^{-1} band width. Na-A absorption bands typical of synthetic zeolites are within 1011–1111 cm^{-1} area of asymmetric atomic vibrations, 737–761, 772, 667–6720 cm^{-1} area of symmetric atomic vibrations, 558–565 cm^{-1} area of double frame rings and 473–477 cm^{-1} area of Si(Al)–O deformation vibration [1].

In addition to absorption bands typical of zeolite Na-A, IR spectrum of the specimen (Fig. 8, 1 cr.) also reveals insignificant peaks typical of hydrosodalite. The absorption bands typical of hydrosodalite are as follows: in the 100 cm^{-1} of asymmetric atomic vibration, in the 722 and 666 cm^{-1} of symmetric atomic vibration, in the 468 cm^{-1} of Si(Al)–O deformation vibration.

Absorption bands 3618, 3516, 3451, 961, 734, 660 in specimen 8 (Fig. 8, 2 cr.) are attributed to unreacted gibbsite $Al(OH)_3$ [20,21].

Sonication energy facilitates the formation of active radicals which are responsible for rapid crystallization of zeolite phase (Fig. 9).

Firstly, ultrasound affects the depolymerization–polymerization equilibrium through increasing concentration of soluble species needed for the formation of zeolites. Secondly, ultrasound increases the nucleation rates because the cavitation bubbles themselves act as nuclei.

The zeolite sample with the highest crystallinity was examined by scanning electron microscopy (Fig. 10).

SEM images show that crystals of zeolite obtained during the ultrasound treatment of AlF_3 production waste have a cubic character.

4. Conclusions

In this paper we have reported the synthesis of zeolite NaA crystals by sonochemical method at room temperature and short crystallization time. Here ultrasonic irradiations were used instead of hydrothermal treatment for the synthesis of zeolite. Zeolite NaA was synthesized from by-product silica. Some zeolites NaA were synthesized using conventional, i.e. hydrothermal synthesis.

1. We have proved that zeolite Na-A can be synthesized from suspensions prepared with by-product silica under conventional conditions by irradiating the suspension with 200 W ultrasonic waves. The highest degree of zeolite crystallinity (25.81%) is obtained when the synthesis is performed with suspensions aged for 24 h.
2. We found that it was not feasible to dissolve $Al(OH)_3$ and amorphous silica gel in sodium hydroxide solution in advance for zeolite NaA synthesis under the tested conditions.
3. We can state that with the increase of ultrasonic wave power from 210 to 250 W the time needed to obtain zeolite NaA of similar crystallinity shortened from 15 to 10 min. Therefore, it is advisable to use the maximum ultrasonic power.
4. From the comparison of hydrothermal and sonochemical synthesis of zeolite NaA we may claim that with the same reaction time the sonochemical synthesis is more effective because higher crystallinity zeolites NaA prevailed among the sonochemical reaction products.

5. The XRD and IR analysis confirms the formation of zeolite NaA from by-product silica at room temperature. So sonochemical synthesis of zeolite NaA equipment and power cost is lower compared to hydrothermal synthesis.

References

[1] D. Brek, Zeolite molecular sieves (in Russian), Mir, Moscow, 1976.
[2] Ö. Andaç, Ş. Murat Telli, M. Tatlier, A. Erdem-Şenatalar, Effects of ultrasound on the preparation of zeolite A coatings, Microporous Mesoporous Mater. 88 (2006) 72–76.
[3] J.H. Bang, K.S. Suslick, Applications of ultrasound to the synthesis of nanostructured materials, Adv. Mater. 22 (2010) 1039–1059.
[4] V. Augugliaro, M. Litter, L. Palmisano, J. Soria, The combination of heterogeneous photocatalysis with chemical and physical operations: a tool for improving the photoprocess performance, J. Photochem. Photobiol., C 7 (2006) 127–144.
[5] H. Kita, K. Horii, Y. Ohtoshi, K. Tanaka, K.I. Okamoto, Synthesis of a zeolite NaA membrane for pervaporation of water/organic liquid mixtures, J. Mater. Sci. Lett. 14 (1995) 206–208.
[6] J. Wu, B. Wang, N. Li, S. Xiang, Effect of aging with ultrasound on the synthesis of MCM-49 zeolite, Chin. J. Catal. 27 (2006) 375–377.
[7] Ö. Andaç, M. Tatlıer, A. Sirkecioğlu, I. Ece, A. Erdem-Şenatalar, Effects of ultrasound on zeolite A synthesis, Microporous Mesoporous Mater. 79 (2005) 225–233.
[8] B. Wang, J. Wu, Z.Y. Yuan, N. Li, S. Xiang, Synthesis of MCM-22 zeolite by an ultrasonic-assisted aging procedure, Ultrason. Sonochem. 15 (2008) 334–338.
[9] C. Belviso, F. Cavalcante, A. Lettino, S. Fiore, Effects of ultrasonic treatment on zeolite synthesized from coal fly ash, Ultrason. Sonochem. 18 (2011) 661–668.
[10] C. Belviso, F. Cavalcante, S. Fiore, Ultrasonic waves induce rapid zeolite synthesis in a seawater solution, Ultrason. Sonochem. 20 (2013) 32–36.
[11] K. Shams, H. Ahi, Synthesis of 5A zeolite nanocrystals using kaolin via nanoemulsion-ultrasonic technique and study of its sorption using a known kerosene cut, Microporous Mesoporous Mater. 180 (2013) 61–70.
[12] W. Kim, D. Choi, S. Kim, Sonochemical synthesis of zeolite A from metakaolinite in NaOH solution, Mater. Trans. 51 (2010) 1694–1698.
[13] P. Pal, J.K. Das, N. Das, S. Bandyopadhyay, Synthesis of NaP zeolite at room temperature and short crystallization time by sonochemical method, Ultrason. Sonochem. 20 (2013) 314–321.
[14] R. Anuwattana, P. Khummongkol, Conventional hydrothermal synthesis of Na-A zeolite from cupola slag and aluminum sludge, J. Hazard. Mater. 166 (2009) 227–232.
[15] K. Shams, S.J. Mirmohammadi, Preparation of 5A zeolite monolith granular extrudates using kaolin: investigation of the effect of binder on sieving/adsorption properties using a mixture of linear and branched paraffin hydrocarbons, Microporous Mesoporous Mater. 106 (2007) 268–277.
[16] A. Hartmann, V. Petrov, J.C. Buhl, K. Rübner, M. Lindemann, C. Prinz, A. Zimathies, Zeolite synthesis under insertion of silica rich filtration residues from industrial wastewater reconditioning, Adv. Chem. Eng. Sci. 4 (2014) 120–134.
[17] H.M. Aly, M.E. Moustafa, E.A. Abdelrahman, Influence of aluminum source on the synthesis of nanosized ZSM-5 zeolite, Der Chemica Sinica 2 (2011) 166–173.
[18] X. Zhang, D. Tong, W. Jia, D. Tang, X. Li, R. Yang, Studies on room-temperature synthesis of zeolite NaA, Mater. Res. Bull. 52 (2014) 96–102.
[19] P.M. Slangen, J.C. Jansen, H. Van Bekkum, The effect of ageing on the microwave synthesis of zeolite NaA, Microporous Mater. 9 (1997) 259–265.
[20] P.A. Schroeder, Infrared Spectroscopy in clay science CMS Workshop Lectures, Teaching Clay Science 11, the Clay Mineral Society, CO, Aurora, 2002. pp. 181–206.
[21] S. Musić, Đ. Dragčević, S. Popović, N. Vdović, Chemical and microstructural properties of Al-oxide phases obtained from $AlCl_3$ solutions in alkaline medium, Mater. Chem. Phys. 59 (1999) 12–19.