

KAUNAS UNIVERSITY OF TECHNOLOGY

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**THE INFLUENCE OF MINERALOGICAL COMPOSITION AND
TEXTURE ON FUNCTIONAL PROPERTIES OF SILICATE
STONE**

Summary of Doctoral Dissertation

Technological Sciences, Chemical Engineering (05T)

2017, Kaunas

The research was carried out between 2013 and 2017 at the Department of Silicate Technology, Faculty of Chemical Technology, Kaunas University of Technology and in the JSC “Matuizu duju silikatas” manufactory. The research was supported by Research Council of Lithuania.

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KAUNO TECHNOLOGIJOS UNIVERSITETAS

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INTRODUCTION

Relevance of the work

Autoclaved aerated concrete (AAC) is an artificial silicate stone, characterized by its environmental performance, durability, fire resistance, good heat and sound insulating properties, and is easily mechanically treated (cut, polished, milled, etc.). Thermal insulation properties of AAC are the best of all manufactured masonry units. In addition, the use of a thin layer of the mixture as a binder eliminates the appearance of "thermal bridges", wherefore construction costs are declined and savings of thermal energy make it possible to reduce heating costs. Such materials as cement, and/or ground lime as well as fine SiO_2 components containing material are used for the production of AAC. These raw materials are mixed with foamer and water, forming a semi-finished product, which is hardened in the autoclave under saturated steam pressure.

Calcium silicate hydrates (C-S-H) 1.13 nm tobermorite and xonotlite are the main compounds which formed during the reactions of AAC production. These C-S-H significantly influence the mechanical and transport properties of AAC products. The conditions of formation and stability of these calcium silicate hydrates are governed by the duration and temperature of AAC hydrothermal treatment; the CaO/SiO_2 molar ratio of initial mixture; the composition and purity of raw materials; used additives; stirring intensity and other factors. Depending on the the production of autoclaved aerated concrete, different SiO_2 components containing raw materials can be used.

Various raw material processing technologies, the composition of formation mixtures, and different hardening treatment are used during the manufacturing of products with particular characteristic. Therefore, calcium silicate hydrates with different basicity can form in the products. The literature data suggest that calcium silicate hydrates prepared under hydrothermal treatment act as adsorbents for the removal of heavy metal ions. However, the chomosorption properties of binding compounds which formed during AAC production investigated are only fragmentary.

Besides that, considerable attention should be given to the efficiency of technological process, which is influenced by the common problems of the production: slow increment of plastic strength of forming mixture, temperature, low rate of expansion, and cracks. Due to these difficulties, the production process is long, and the amount of waste material increases, therefore it is important to choose the right proportions of initial raw materials and optimize the manufacturing process without compromising the same/improved product characteristics.

The aim of this work is to examine the formation kinetic of calcium silicate hydrates in model and industrial systems in order to optimize the technological

parameters of autoclaved aerated concrete production and expand its rational application areas.

In order to achieve the aim of the work, we have to accomplish the following **goals**:

1. To determine the influence of synthesis parameters on the calcium silicate hydrates formation kinetics in porous and dense silicate stone and in unpressed samples as well as to evaluate the dispersibility, texture and stability of formed products.
2. To investigate the influence of the formation mixture components ratio and the mineralogical composition of new compounds on autoclaved aerated concrete properties.
3. To determine the optimal hydrothermal treatment conditions of autoclaved aerated concrete in industrial production line.
4. To evaluate the utilization possibilities of Cd^{2+} and Zn^{2+} ions containing compounds during the synthesis of calcium silicate hydrate.
5. To propose recommendations for the improvement of autoclaved aerated concrete technology in order to reduce the amount of binder material in products, cost and suggest new application areas.

Scientific Novelty of the Research

It was determined that the decrease of compressive strength of autoclaved aerated concrete products, which occurs by prolonging the duration of hardening, causes the new compound – gyrolite, which does not possess binding properties, formation.

It was proved that various materials containing soluble environmental aggressive transition metal ions can be intercalated, thereby dispose, into the crystal lattice structure of calcium silicate hydrates during the hardening treatment of products.

Practical Significance of the Scientific Research

It was found that the amount of cement in autoclaved aerated concrete formation mixture can be reduced from 21.0 to 16.2 %. This not only reduces production costs by 5%, but also around a 17.5% increase in the compressive strength, reducing the amount of waste. Increasing the hydrothermal treatment temperature from 180 to 200 ° C, most of the semi-crystalline C-S-H(I) recrystallize to 1.13 nm tobermorite, therefore the thermal shrinkage of products is reduced by approximately twice and the operating temperature increases from 450 °C to 600-650 °C. The obtained results were implemented in a plant UAB „Matuizu duju silikatas“, achieved economic effect is 1.7 kg/ m³ (production line capacity - 160 thousands m³/year).

Approval and Publication of Research Results

The results of the research have been presented in four scientific publications included into the Thomson Reuters Web of Knowledge database: one of them was published in the Science of Sintering; one was presented in the Advances in Applied Ceramics, two articles were delivered in the Romanian Journal of Materials. The results of the dissertation have been reported in six international conferences proceedings: „33rd Cement and Concrete Science Conference” (2013, UK, Portsmouth); “BaltSilica” (2014, Poland), “BaltSilica” (2016, Lithuania); „ESTAC11: the 11th European symposium on thermal analysis and calorimetry: (2014, Espoo, Finland); “Chemistry and Chemical Technology” (2016, Lithuania).

Structure and Content of the Dissertation

The dissertation consists of the introduction, a survey of the relevant scholarly literature, the experimental section, the results and discussion section, the conclusions, the list of references and publications on the dissertation topic as well as appendixes. The list of references includes 149 bibliographic sources. The main results are discussed on 111 pages illustrated in 11 tables and 80 figures.

Statements Presented for Defence

1. Qualitative and quantitative composition of monobasic calcium silicate hydrates, which formed during the production of autoclaved aerated concrete, is determined by its performance properties and the working temperature.
2. The hydrothermal treatment temperature has an influence on the microstructure of the formed porous stone – by increasing the temperature from 180 to 200 °C, the lamellar 1.13 nm tobermorite crystals are largely transformed to the fibrous crystals, while the diameter 0.6–0.0065 µm of the prevailing micropores increased from 49–52 to 54–58%.

EXPERIMENTAL

The following materials were used: ground quartz sand from the JSC „Matuizu plytinė“ field, grinding fineness of up to 250 m²/kg; ground lime (CaO) from the company “Lhoist Bukowa”, activity of 88.67 %; Cement CEM II/A-LL, 42.5 R mark produced by „Akmenės cementas”; gypsum with 89.97 % of CaSO₄·2H₂O produced by „Dolina Nidy”; aluminum pastes (5-6380/80 and 5-6355/80) with 80 % of pure aluminium („Benda-Lutz“); CaO (“Stanchem,” Poland, purity 97%) which was produced by burning calcium hydroxide at 950 °C for 30 minutes and ground to the specific surface area S_a of 1190 m²/kg, with the quantity of free CaO equal to 97.41%; fine ground SiO₂·nH₂O (“Reaktiv,” Russia, purity 98%) having a specific surface area S_a = 1529 m²/kg, with loss of ignition – 6.10%; silica gel, i.e., a waste product of AlF₃ production in the chemical plant of “Lifosa” (Kėdainiai, Lithuania) (with moisture content 60–65%), dried to <0.5 % of moisture, S_a = 964 m²/kg.

Other reagents Zn(NO₃)₂·6H₂O, CdO, acetone (C₃H₆O) and hydrochloric acid (HCl).

Preparation of forming mixture and formation of specimens. The forming mixture of autoclaved aerated concrete was prepared and mixed in the industrial production line. All the required amount of raw materials was weighed at the beginning of mixing. The components of the forming mixture were mixed in the high speed vertical propeller stirrer of 5 m³ volume by the speed of 1500 rpm. First, the required quantities of sand and return slurries were mixed with cold and hot water together (to achieve the temperature mean of 40 °C). Second, cement, lime and gypsum were added. Finally, aluminum paste suspension was placed. Overall, the mixing time was 3.05 minutes. After mixing, homogenized forming mixture was poured into the steel moulds (size 6220×1580×700 mm). The poured moulds were transported to the fermentation area (38 °C temperature) and kept in moulds for 160 min. Later, the heap of forming mixture was cut off. Samples were hardened in the industrial autoclave (180 °C, 9 or 15 h) of 221 m³ capacity. After hardening, the samples were cut into 100×100 mm cubes and dried to the constant mass in the conditioning oven at the temperature of 105 ± 5 °C.

AAC specimens hardening in the laboratory. The formed specimens in the industrial line were cut into (300×300×200 mm) samples and hardened in the laboratory autoclave of 20 l capacity in the specified mode (2.5 + (4-6-8-12-16) + 2.5), at water vapour temperature of 170, 180 and 200 °C. After hardening, the specimens were cut into 100×100 mm cubes and dried to the constant mass in the conditioning oven at the temperature of 105 ± 5 °C.

The temperature of AAC formation mixture. After the formation mixture was mixed and poured into mould, their initial temperature and time were measured. During the fermentation period, the temperature of the formation mixture were measured, firstly, each 5 min, and after 30 min from the beginning of the experiment – every 10 min. The experiment proceeded until the ending of

fermentation. Time was measured with chronometer with 1s accuracy and the temperature with a digital thermometer with 0.1 °C accuracy.

The plasticity of AAC formation mixture was measured at certain intervals, 5 psi, with a mechanical plastometer. The plastometer was graduated in pounds per square inch (psi), so the results were converted to SI units – MPa: 1 psi= 0.006896 kPa=6,896·10⁻⁶ MPa.

The expansion of AAC formation mixture. The height of forming mixture expansion was recorded every 1 min using mechanical stopwatch with the precision of 1 s and a metal ruler with the precision of 1 mm.

Optical micrographs of AAC cakes. Fujifilm cameras were used. Resolution - 12 Mega Pixels, Optical Zoom – 3 times.

The preparation of hardened AAC samples for the experiments of performance properties. The three cubes with a side length of 100 mm were cut from AAC samples for the experiments. One sample was cut from the product of the upper, middle and lower thirds according to expansion direction of formation mass.

The dry density of AAC samples was determined according to EN standard, calculating the constant weight (105 ± 5)°C after drying three samples (100×100 mm) and their weight by volume ratio.

AAC samples moisture content was determined according to EN standard, i.e. moisture content is calculated as the weight loss during drying process and the dried sample weight ratio.

Hydrothermal synthesis of low base calcium silicate hydrates has been carried out in unstirred suspensions in 25 ml volume PTFE cells which were placed in a stainless steel autoclave (“Parr instruments 4566B” Germany) under saturated steam pressure at 175, 180 and 200 °C temperature for 4, 8, 12, 16, 24, 48 and 72 hours. The products of the synthesis have been filtrated, rinsed with acetone to prevent the carbonization of material, dried at a temperature of 50 °C ± 5 and put through a sieve with an 80-µm mesh.

The XRD analysis was performed using the D8 Advance diffractometer (Bruker AXS, Karlsruhe, Germany) operating at the tube voltage of 40 kV and tube current of 40 mA. The X-ray beam was filtered with a Ni 0.02 mm filter to select the CuK α wavelength. Diffraction patterns were recorded in a Bragg-Brentano geometry using a fast counting detector Bruker LynxEye based on the silicon strip technology. The specimens of the samples were scanned over the range $2\theta = 3\text{--}70^\circ$ at a scanning speed of 6° min⁻¹ using a coupled two theta/theta scan type.

Simultaneous thermal analysis (STA) (differential scanning calorimetry and thermogravimetry) was carried out on a Linseis STA PTI1000 instrument with ceramic sample handlers and crucibles of Pt at a heating rate of 15 °C/min, the temperature ranging from 30 °C to 900 °C under the ambient atmosphere.

The dilatometric (DIL) analysis was performed by using the Linseis “L75H1600 Platinum series” with Al₂O₃ sample holder and pushrod. Cylindrical dilatometric samples of 5 mm diameter and 22 mm length were heated to 1000 °C at a heating rate of 10 °C/min to monitor the sample change under air atmosphere.

The porosity and pore size distribution of the samples were measured by mercury intrusion porosimetry (MIP, Micromeritics, AutoPore 9500 IV). Pressure greater than 400 MPa can be achieved by the calorimetry apparatus and allows the mercury to penetrate pores as fine as 0.003 μm in diameter.

The sample microstructure was investigated by using the field-emission scanning electron microscope SEM JSM-7600F (JOEL). The split surface of the samples was tested, using low accelerating voltage (10 kV).

The compressive strength of AAC samples was determined according to the requirements of the standard. The press MEGA 10-400-50 (FORM+TEST GmbH) was used. The loading rate of samples during compression was 50 N/s until the destruction of the sample. 3 samples of each batch were subjected to testing. The volume density of the samples was determined according to the requirements of the standard.

Fourier transform infrared (FTIR) spectra were obtained using a Perkin-Elmer FTIR Spectrum X system. Specimens were prepared by mixing 1 mg of the sample with 200 mg of KBr. The spectral analysis was performed in the range of 4000–400 cm⁻¹ with a spectral resolution of 1 cm⁻¹.

The texture of the synthesis products was measured by a BET surface area analyzer KELVIN 1042 Sorptometer (Costech Instruments). The analyzer determines the surface area, total pore volume and pore size distribution of a sample by employing the techniques of adsorbing the adsorbate gas (N₂) from a flowing mixture of adsorbate and an inert non-adsorbable carrier gas (He) at 77 K.

The concentration of Ca²⁺ and Zn²⁺ ions was determined by using a Perkin Elmer Analyst 400 atomic absorption spectrometer with the following parameters: Zn²⁺ wavelength = 213.86 nm; Ca²⁺ wavelength = 422.67 nm; hollow cathode lamp current (I) = 30 mA; type of flame was C₂H₂-air; oxidant air = 10 l/min; acetylene = 2.5 l/min.

RESULTS AND DISCUSSION

Dependence of autoclaved aerated concrete performance properties on its mineral composition

Binding materials, which are formed during hydrothermal treatment of AAC, significantly effect the physical and mechanical properties of AAC products, and this material cementitious properties, in turn, depends on hardening conditions and formation mixture composition. This binding material determines the composition and quantity of formed cementitious compounds and also influences the cost of product.

The influence of cement addition on the AAC semiproduct formation process and their properties

The amount of cement in the initial mixtures varied from 13.7 to 23.6 % (preselected point from 12.5 to 22.5 %) and the amount of sand slurry, respectively - from 52.3 to 42.6 %. Experiments were carried out in industrial technological lines using large quantities of raw materials, thus the significant weighing errors are obtained (Table 1).

Table 1. The composition of raw materials mixtures with different amount of cement

Raw material		The amount in mixtures M ₁ , ..., M ₅ , wt. %				
		M ₁	M ₂	M ₃	M ₄	M ₅
Ground quartz	Preselected point	52.5	50.0	47.5	45.0	42.5
	Error evaluation	52.3	50.0	47.3	44.9	42.6
Waste slurry	Preselected point	20.0				
	Error evaluation	18.8	18.7	18.8	18.8	18.9
Cement	Preselected point	12.5	15.00	17.5	20.0	22.5
	Error evaluation	13.7	16.2	18.6	21.1	23.6
Lime	Preselected point	12.5				
	Error evaluation	13.0	12.8	13.0	12.9	12.6
Gypsum	Preselected point	2.5				
	Error evaluation	2.3	2.3	2.3	2.3	2.3
Total amount		100.0	100.0	100.0	100.0	100.0

It was determined that an increased amount of cement has no significant influence on the temperature and expansion height of formation mixtures. The amount of cement in formation mixture has an influence on the compressive strength of formed semiproduct. The highest plasticity strength ($0,97 \cdot 10^{-3}$ MPa) of formation mixtures was after 90 min of filling mold when the amount of cement were 21.1 % and 23.6 % (Fig. 1).

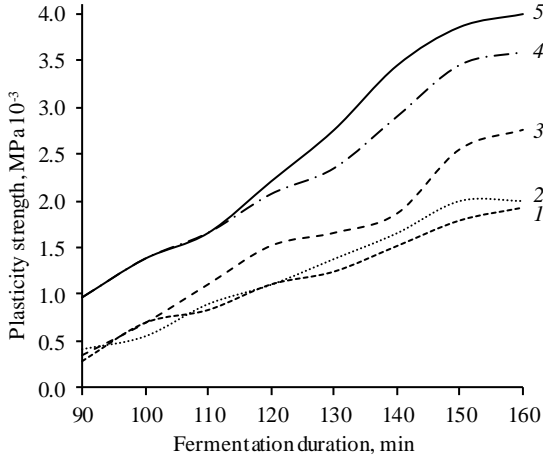


Fig. 1. The dependence of plasticity strength on fermentation duration when the amount of cement were, %: 1 – 13.7; 2 – 16.2; 3 – 18.6; 4 – 21.1; 5 – 23.6

By increasing the amount of additive from 13.7 % to 23.6 %, after 160 min the plasticity strength of cake grows more than twice ($1.93 \cdot 10^{-3}$ MPa ir $4.00 \cdot 10^{-3}$ MPa). The higher the plasticity strength, the lower the probability that the cake will crack during hydrothermal treatment, because the cured semi-finished product can sustain higher internal stresses.

The duration of fermentation reaches 35–39 min, gas of H_2 released intensively. At this stage, the expansion of cake ends e. i. cavity of H_2 gas appears on the surface of cake and the height has not increased. It should be noted that increasing the amount of cement, intense outgassing of H_2 occurs three minutes faster and has a positive influence on the quality of the surface of the cake, which is evaluated visually (Fig. 2).



Fig. 2. The optical microscopy micrographs of cake after the intense outgassing of H_2 when the amount of cement were, %: a – 13.7; b – 23.6.

It was determined that after 9 h of hydrothermal treatment at 180 °C temperature, the increment of the amount of cement addition from 21.1 to 23.6 % has a negative

influence on cake quality i.e., the negligible cracks were observed on the surface of blocs, which were formed from M₄ and M₅ formation mixtures (Fig. 3, a). One of the main reasons were that cake gained such high plasticity strength too fast during the fermentation process, when gas of H₂ were still released. The release of H₂ gas from the hardened cake was difficult, therefore the pressure of gas increased which causes crack in approximately 5 cm below the top of the cake.

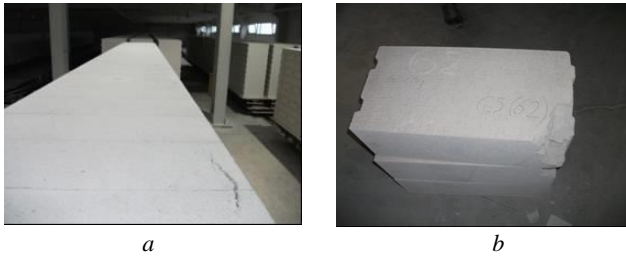


Fig. 3. The optical microscopy micrographs of product with 23.6 % amount of cement additive: *a* – block view from top, *b* – one block

It was found that the compressive strength and density of samples reduced by increasing the amount of cement from 16.2 to 23.6 % (Fig. 4.). The maximum compressive strength (3.27 MPa) typical for samples produced from the mixture M₂ and a density – for the samples produced from the mixture – M₁. By evaluating/comparing mechanical properties of the formation mixtures and products, the most suitable composition for the AAC production are formation mixtures M₁ and M₂. In addition, from these two mixtures, M₂ is better because it characterizes better formability and formed products are stronger and lighter (better heat-insulating properties).

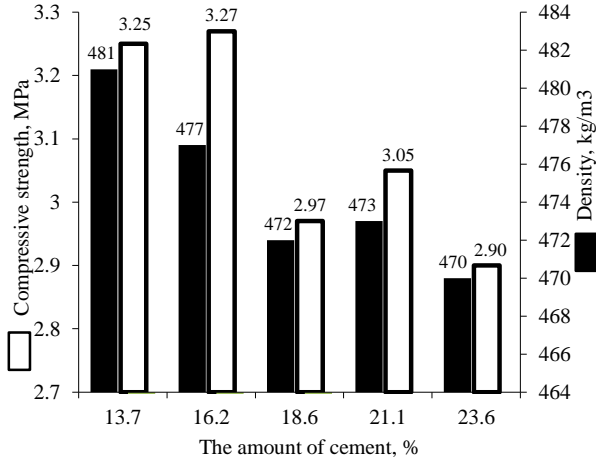


Fig. 4. The influence of the amount of cement on AAC samples compressive strength and density

XRD analysis data showed that the additive of cement has no significant influence on the mineralogical composition of AAC samples (Fig. 5.). The main unreacted SiO_2 component – quartz ($d - 0.426; 0.335; 0.228; 0.182$ nm), calcium silicate hydrate - 1.13 nm tobermorite ($d - 1.144; 0.547; 0.281; 0.251; 0.184; 0.167$ nm) and semi-crystalline C-S-H(I) ($d - 0.304; 0.278; 0.183$ nm) are dominant in all samples (Fig. 5). It should be noted that the amount of quartz coherently decreases by adding larger amounts of cement.

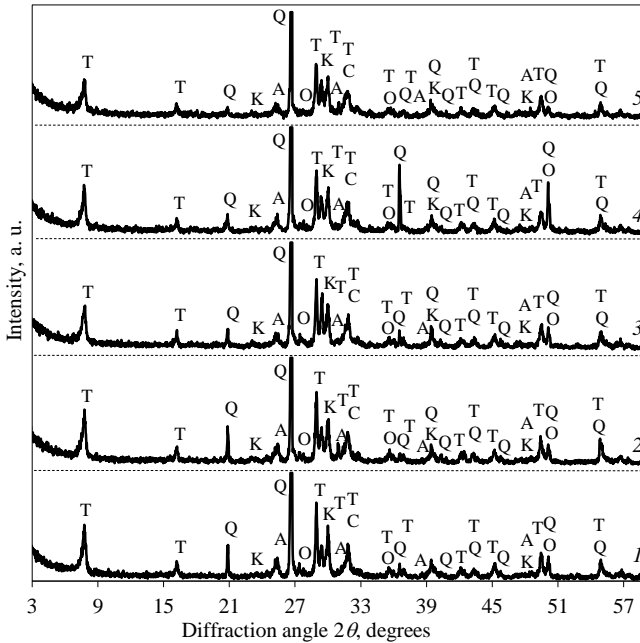


Fig. 5. XRD analysis patterns of AAC samples after 9 h hydrothermal treatment when the amount of cement: 1 – 13.7 %; 2 – 16.2 %; 3 – 18.6 %; 4 – 21.1 %; 5 – 23.6 %. Indexes: T – 1.13 nm tobermorite; Q – quartz; A – anhydrite; K – calcite; O – ortoclaze; C – C-S-H(I)

The recommendations of formation mixture composition for industrial plant of AAC were proposed according to these results: cement - 16.2 %, lime - 12.8% gypsum - 2.3 % sand slurry - 50.0 %, aluminum paste - 0.11% sludge waste - 18.7 %.

The influence of lime addition on the AAC semiproduct formation process and their properties

The 5 mixtures were prepared to examine the influence of lime content on the AAC product properties (Table 2). The amount of lime in the formation mixtures varied from 7.7 to 17.5 %, and sand slurry respectively – from 50.0 to 40.2 %. The amounts of other formation mixture components does not vary.

The formation mixture which had the highest amount of lime additive (M_{10}) fell after 27 min from the beginning of filling. This happens because the initial temperature of formation mixture was too high, and cake formed faster than released H_2 gas. For these reasons, the structure of semiproduct was destroyed and it deformed.

Table 2. The composition of formation mixture with different amount of lime additive

Raw material		The amount in mixtures M_6, \dots, M_{10} , wt. %				
		M_6	M_7	M_8	M_9	M_{10}
Ground quartz	Preselected point	50.0	47.5	45.0	42.5	40.0
	Error evaluation	50.0	47.4	45.1	42.4	40.2
Waste slurry	Preselected point	20.0				
	Error evaluation	18.9	18.9	18.7	18.6	19.0
Cement	Preselected point	20.0				
	Error evaluation	21.1	21.1	21.2	21.2	21.1
Lime	Preselected point	7.5	10.0	12.5	15.0	17.5
	Error evaluation	7.7	10.3	12.8	15.5	17.5
Gypsum	Preselected point	12.5				
	Error evaluation	2.3	2.3	2.3	2.3	2.3
Total amount		100.0	100.0	100.0	100.0	100.0

In the AAC production the temperature of cutted cakes is about 85 °C, so $M_{10(2)}$ and M_9 samples temperature was too high - 93.7 and 92.0 °C, and M_6 : too low - 74.3 °C (Fig. 6). Thus, these compositions of formation mixtures are not suitable for the production of AAC.

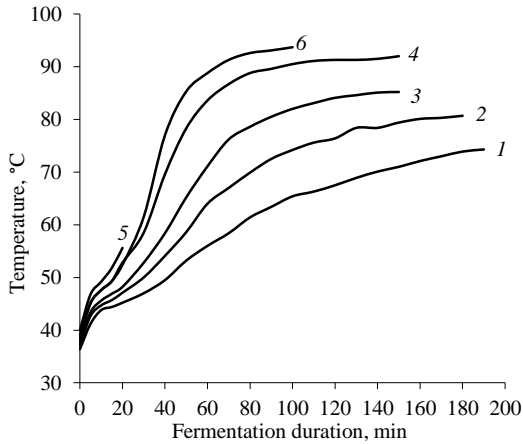


Fig. 6. The dependence of cake temperature on fermentation duration when amount of lime, %: 1 – 7.7; 2 – 10.3; 3 – 12.8; 4 – 15.5; 5 – 17.5: $M_{10(2)}$; 6 – 17.5: $M_{10(2)}$

Plasticity strength of sample $M_{10(2)}$ are shown in the Figure 7. The pattern with number 5 showed the plasticity strength which was measured in stright place of cake and the pattern with number 6 showed the plasticity strength in dense area (index – $M_{10(2)S}$).

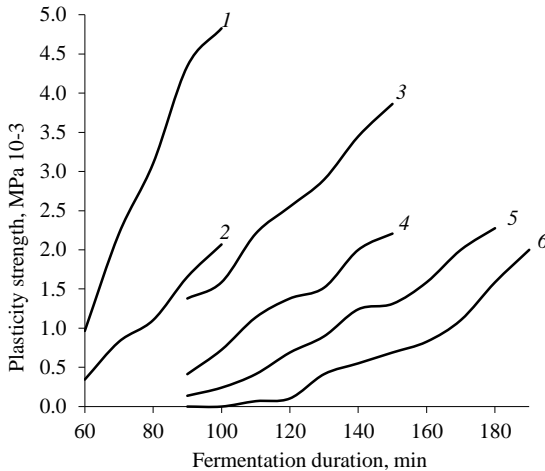


Fig. 7. The dependence of plasticity strength on fermentation duration when amount of lime, %: 1 – 7.7; 2 – 10.3; 3 – 12.8; 4 – 15.5; 5 – 17.5: $M_{10(2)}$; 6 – 17.5: $M_{(2)S}$

The intense release of H₂ gas into the surface of cake depends on the formation mixture composition – the more lime it has, the shorter duration of gas release (Table 3). In the cake M₁₀₍₂₎ duration is twice shorter in M₆.

Table 3. Cake fermentation temperature and duration when released H₂ gas is intense

Sample	M ₆	M ₇	M ₈	M ₉	M ₁₀	M ₁₀₍₂₎
Amount of lime, %	7.7	10.3	12.8	15.5	17.5	17.5
Duration until the outgassing of H ₂ , min	61	49	43	40	27	31
Cake temperature, °C	56.2	58.4	61.5	69.6	67.5	63.0

The highest compressive strength is typical for samples produced from the mixture which has 12.8 % lime addition, and the lowest – samples with highest lime additive, i. e. 17.5 % (Table 4).

Table 4. The samples compressive strength, mass, moisture content and density dependence on quantity of lime additive

Sample	M ₆	M ₇	M ₈	M ₉	M ₁₀₍₂₎
Compressive strength, MPa	2.84	3.02	3.10	3.01	2.59
Mass of cutted samples, g	631.73	624.98	640.05	626.05	622.33
Mass of dried samples, g	479.25	471.83	477.70	470.63	467.65
Moisture content, %	31.81	32.46	33.99	33.03	33.08
Density, kg/m ³	469	463	466	470	464

Thus, after this experiment, the formation mixture of AAC samples remain the same as was recommended and mentioned above: cement - 16.2 %, lime - 12.8 % gypsum - 2.3 % sand slurry - 50.0 %, aluminum paste - 0.11 % sludge waste - 18.7 %.

The influence of hardening conditions of autoclaved aerated concrete on tobermorite formation

The average values of the compressive strength of AAC samples are shown in Fig. 8. It was found that after 8 hours of the hydrothermal treatment at 170 °C temperature, the compressive strength of sample was equal to 2.58 MPa (Fig. 1). After increasing the hydrothermal treatment temperature to 180 °C, the compressive strength increased and reached the maximum value after 8 h of curing (3.97 MPa). Meanwhile, the further increment of the reaction temperature to 200 °C led to lower values of the mentioned parameter (~ 30 %). The further decrease in the compressive strength was also observed by prolonging the isothermal curing from 8 to 16 h. On the other hand, the density of AAC samples was very similar and varied in the 470–481 kg/m³ range.

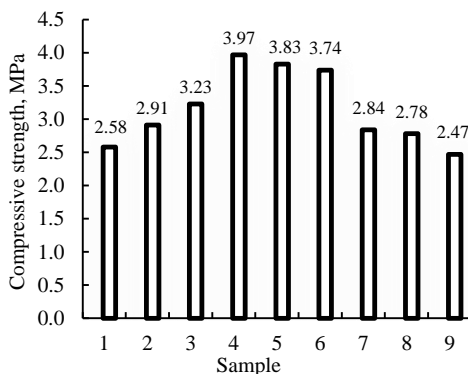


Fig. 8. The influence of hardening conditions on the compressive strength of AAC samples, when the isothermal temperature and duration of hardening were: 1 – 170 °C, 8 h; 2 – 180 °C, 4 h; 3 – 180 °C, 6 h; 4 – 180 °C, 8 h; 5 – 180 °C, 12 h; 6 – 180 °C, 16 h; 7 – 200 °C, 8 h; 8 – 200 °C, 12 h; 9 – 200 °C, 16 h

In order to estimate the mineralogical composition of AAC samples, XRD, STA and SEM analysis were performed. It was determined that a moderate amount of 1.13 nm tobermorite forms already after 8 h of the hydrothermal treatment at 170 °C temperature in the AAC sample (Fig. 9, curve 1). A similar amount of the mentioned compound also remains after 4 and 6 hours of the autoclaving treatment at 180 °C temperature (Fig. 9, curves 2 and 3). However, after extending the duration of the treatment to 8 h the amount of formed 1.13 nm tobermorite noticeably increased (Fig. 9, curve 4). In addition, a large amount of other compound, namely, semi crystalline calcium silicate hydrate C-S-H (I) with high binding properties remained in the sample (Fig. 10, a and b, curve 1).

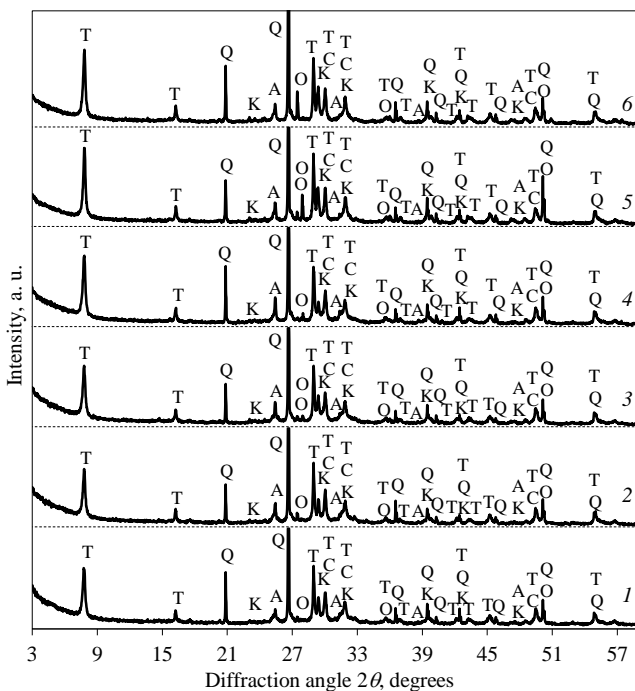


Fig. 9. X-ray diffraction patterns of AAC samples, when the isothermal temperature and duration of hardening were: 1 – 170 °C, 8 h; 2 – 180 °C, 4 h; 3 – 180 °C, 6 h; 4 – 180 °C, 8 h; 5 – 180 °C, 12 h; 6 – 180 °C, 16 h. Indexes: T – 1.13 nm tobermorite; Q – quartz; A – anhydrate; C – calcite; O – orthoclase; I – C-S-H (I)

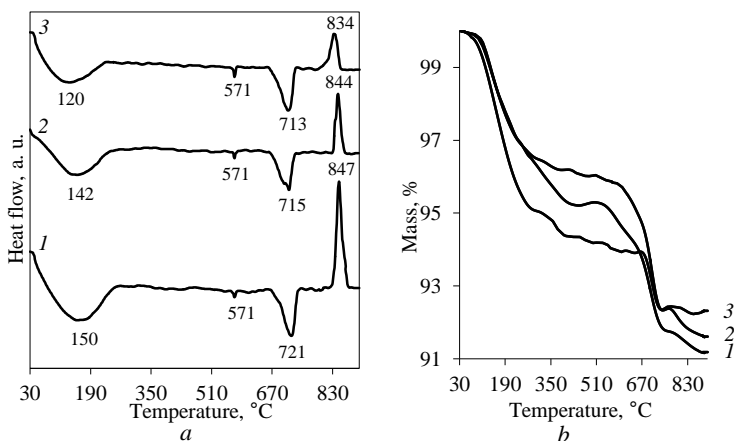


Fig. 10. DSC (a) and TG (b) curves of AAC samples, when duration of the hydrothermal treatment at 180 °C is: 1 – 8 h; 2 – 12 h; 3 – 16 h

The quantity of 1.13 nm tobermorite narrowly but coherently decreased after extending the duration of the hydrothermal treatment to 12 and 16 h: the area and intensity of the main 1.13 nm tobermorite diffraction peak (d -spacing – 1.133) slightly decreased: from 12.7 to 11.5 a. u. and from 41.2 to 38.5 a. u., respectively, as well as the heat of exothermic effect assigned to C-S-H (I) recrystallization to wollastonite (847 – 834 °C) decreased from 82 to 23 J/g (Fig. 10, curves 1 and 3). In addition, under all hydrothermal treatment conditions, these compounds were also identified: anhydrite (d -spacing – 0.350; 0.284; 0.233; 0.187); CaCO₃ (d -spacing – 0.385; 0.303; 0.187); orthoclase (d -spacing – 0.324; 0.319; 0.182) and quartz (d -spacing – 0.426; 0.334; 0.182; 0.167) (Fig. 9).

Significant changes in the mineralogical composition were observed, when the temperature of the isothermal curing was increased to 200 °C (Fig. 11). After 8 h of the isothermal curing, the intensities of the main diffraction peak characteristic to 1.13 nm tobermorite, in comparison with 180 °C temperature, decreased.

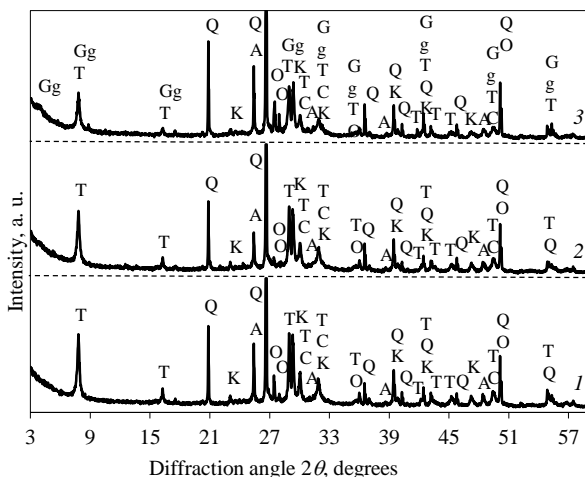


Fig. 11. X-ray diffraction patterns of AAC samples, when duration of the hydrothermal treatment at 200 °C is: 1 – 8 h; 2 – 12 h; 3 – 16 h. Indexes: T – 1.13 nm tobermorite; Q – quartz; A – anhydrite; C – calcite; O – orthoclase; I – C-S-H (I)

Furthermore, visible changes can be seen after 16 hours of hardening because traces of a new compound of calcium silicate hydrate gyrolite gel (*d*-spacing – 2.225; 1.130; 0.184; 0.167) (Fig. 11, curve 3) were formed. This compound does not have binding properties.

In order to estimate the morphology of formed compounds, an SEM analysis was performed (Fig. 12 a, b). It was found that after 8 h of curing at 180 °C the surface of pores in the AAC samples is covered with plate shape crystals (Fig. 12, a), as well as structure material (between pores). This is in good agreement with XRD analysis data, as under the conditions mentioned above, the intensity of the main diffraction peak of 1.13 nm tobermorite was the highest one.

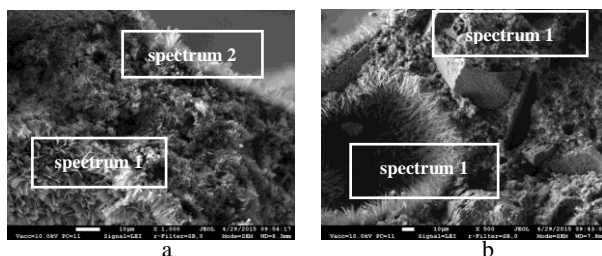


Fig. 12. SEM micrographs of AAC sample cured 8 h at 180 °C temperature (a), and 16 h at 200 °C temperature (b)

Meanwhile, by increasing the temperature and duration of treatment (200 °C, 16 h), the form of crystals was changed (Fig. 12, b). Platelike shape crystals

together with fibrous crystals and amorphous aggregates were identified in the structure material (Fig. 12, b). The latest, presumably are associated with gyrolite gels.

Finally, thermal shrinkage of AAC samples was investigated (Fig. 13). All samples are shrinking similar to 800 °C temperature (1.05 – 1.36 %) but after increment of burning temperature significant differences were determined. For instance, shrinkage (in the range of 800 – 1000 °C temperature) of samples treated at 200 °C temperature for 16 h is 1.70 %, whereas samples autoclaved at 180 °C for 8 h – 3.51 % (Fig. 13, curves 1 and 6).

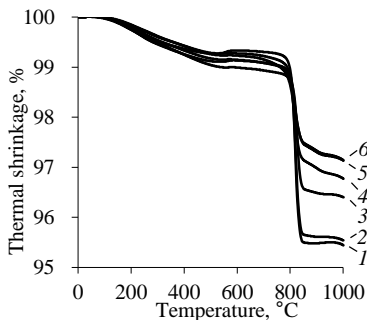


Fig. 13. Thermal shrinkage of AAC, when the isothermal temperature and duration of hardening were: 1 – 180 °C, 8 h; 2 – 180 °C, 12 h; 3– 180 °C, 16 h; 4 – 200 °C, 8 h; 5 – 200 °C, 12 h; 6 – 200 °C, 16 h

Therefore, thermal shrinkage of samples hardened at lower temperature increased twice. This could be explained as follows: a lower amount of C-S-H (I) is formed in the AAC samples cured at higher temperature and later compound intensively shrinks with recrystallization to wollastonite at higher than 800 °C temperature. Thus, AAC samples with lower thermal shrinkage could be used for structures, requiring greater resistance of the products.

The utilization of Cd²⁺ and Zn²⁺ ions containing compounds during hydrothermal treatment of calcium silicate hydrate

The XRD analysis data showed that in the pure CaO–SiO₂·nH₂O–H₂O system after 4 h of hydrothermal treatment at 200 °C temperature, a semi-crystalline calcium silicate hydrate C-S-H(I) are formed (Fig. 14., curve 1). It was found that an insoluble CdO additive at the beginning of synthesis has no significant influence on the mineralogical composition of formed products because the diffraction peaks characteristic to C-S-H(I) and CdO ($d = 0.235; 0.166; 0.142$ nm) are observed in the XRD curve (Fig. 14., curve 2). Meanwhile the soluble Zn(NO₃)₂·6H₂O additive influences the formation of high crystallinity Z-phase with intercalated Zn²⁺ ions, because in XRD pattern peaks typical to Z-phase ($d = 0.157; 0.834; 0.280; 0.180$) (Fig. 14., curve 3) are identified.

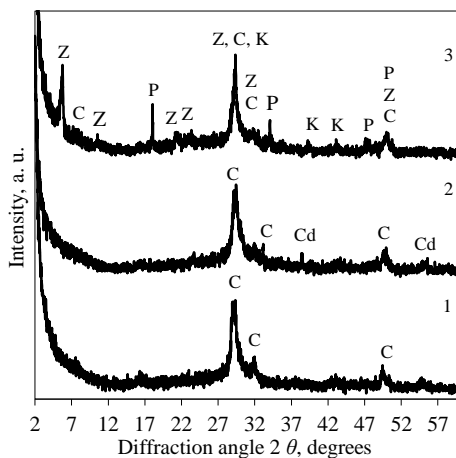


Fig. 14. XRD patterns of hydrothermal synthesis products after hydrothermal treatment at 200 °C for 4 h. Indexes: 1 – pure mixtures, 2 – with CdO additive, 3 – with $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ additive; C – C-S-H(I), Cd – CdO, Z – Z-phase, P – portlandite, K – calcite

The previous results were confirmed by STA analysis data. Two endothermic effects were observed in DSC curve: the endothermic effect at 141 °C can be attributed is related to the loss of interlayer water from the crystal structure of Z-phase (Fig. 15, curve 2), the second - at 451 °C is related with portlandite dehydration. The exothermic effect at 854 °C was assigned to semi-crystalline C-S-H(I) recrystallization into wollastonite (Fig. 15., curve 2). Furthermore, the endothermic effect at ~725 °C can be assigned to the decomposition of calcium carbonate (Fig. 15., curve 2).

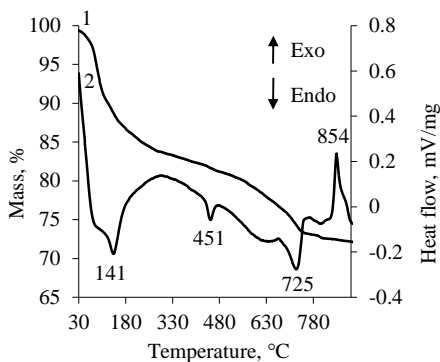


Fig. 15. STA (1 – TGA curve; 2 – DSC curve) curves of hydrothermal synthesis products when $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ additive were used after hydrothermal treatment at 200 °C for 4 h

After prolonging the duration of hydrothermal treatment to 8 h, the traces of Z-phase are identified in pure mixtures and in the mixtures with CdO additive (Fig. 16., curves 1 and 2). Meanwhile in the system with $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ additive together with Z-phase also unreacted $\text{Ca}(\text{OH})_2$ remained (Fig. 16., curve 3). These results confirmed the fact that Zn^{2+} ions are intercalated into the structure of Z-phase and C-S-H(I) and replaced Ca^{2+} ions in their lattice therefore portlandite is identified in the synthesis products.

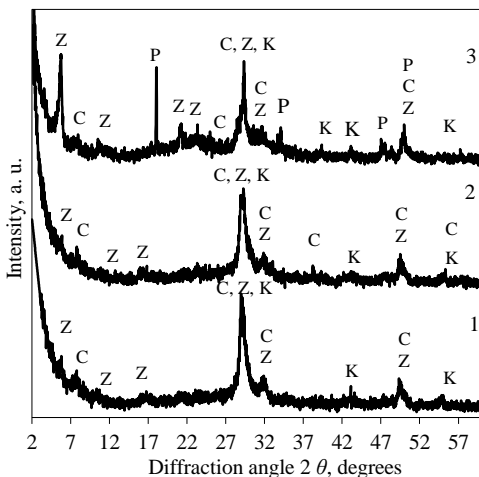


Fig. 16. XRD patterns of hydrothermal synthesis products after hydrothermal treatment at 200 °C for 8 h. Indexes: 1 – pure mixtures, 2 – with CdO additive, 3 – with $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ additive; C – C-S-H(1), Cd – CdO, Z – Z-phase, P – portlandite, K – calcite

It should be highlighted that in the mixtures with CdO, the additive Z-phase remain stable longer than in the pure system, because the small peak ($d - 1.563$) characteristic to this compound is observed after 72 h of synthesis (Fig. 17., curve 2).

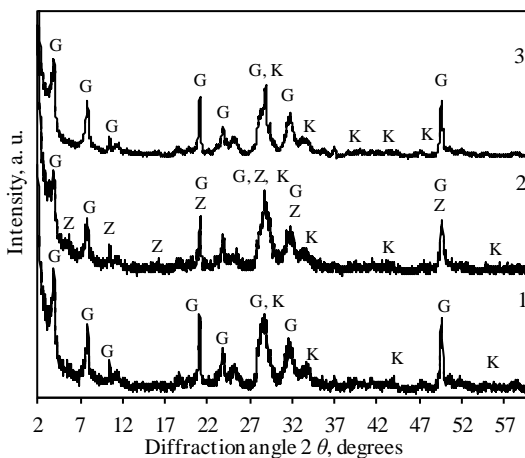
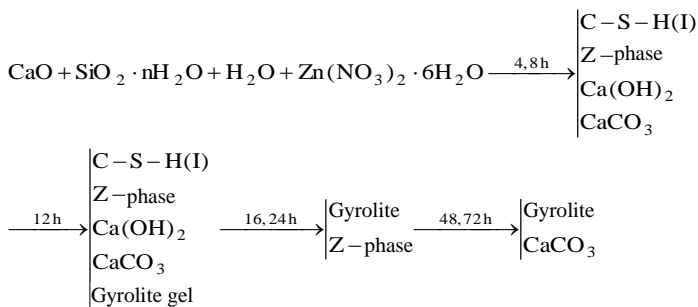
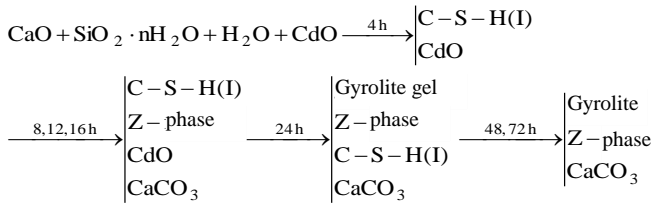


Fig. 17. XRD patterns of hydrothermal synthesis products after hydrothermal treatment at 200 °C for 72 h. Indexes: 1 – pure mixture, 2 – with CdO additive, 3 – with $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ additive; Z – Z-phase, P – portlandite, K – calcite, G – gyrolite

Thus, the duration of Z-phase synthesis is shorter twice when soluble $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ additive are used. compounds Thus, the sequence of which formed in a $\text{CaO-SiO}_2\text{-H}_2\text{O}$ system with $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ additive looks as follows:



Meanwhile the compounds formation mechanism in the system $\text{CaO-SiO}_2\text{-nH}_2\text{O-CdO-H}_2\text{O}$ at 200 °C temperature occur in the following sequence:



Technological recommendations for AAB production without waste

It is recommend to return back to the production line the formed AAC waste (which formed during the production of AAC), before milling this waste with sand. Therefore, the required amount of raw materials are weighed 10 and transported into mixer 11. The formation mixture are poured into form and are kept 160 min in the fermentation camera 13. Then, cake is cut into the correct size of products in the cutting line. Prepared products 18 are hardened in autoclave 19 for 8 h at 180 °C temperature. Obtained products are packed 21 and transported into storage. The waste which remains on the hardening platform is crushed with a branchial breaker and then transported to the waste disposal bunker. After this treatment these wastes can be used as the initial raw material.

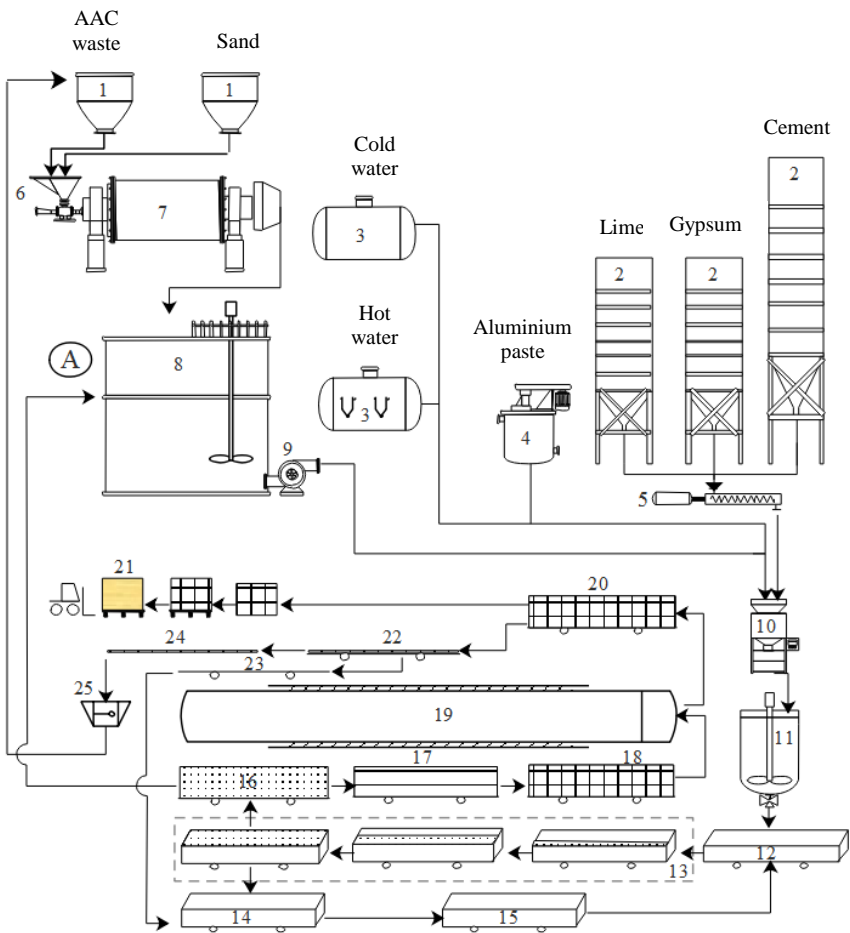


Fig. 18. Principal technological scheme of AAB production without waste:

1 – bunker of raw material, 2 – silos of raw material, 3 – the reservoir of water, 4 – the reservoir/mixer of aluminum suspension, 5 – screw feeder, 6 – vibro-feeder 7 – ball mill, 8 – the reservoir of slurry, 9 – pump, 10 – the weight dispenser, 11 – mixer, 12 – metallic form, 13 – fermentation camera, 14 – hardening plate, 15 – form plastered with olive, 16 – cake, 17 – cake cut horizontally, 18 – cake cut horizontally and vertically, 19 – autoclave, 20 – hardened cake, 21 – packed production, 22 – platform of hardening with AAC waste, 23 – platform of hardening, 24 – hardened AAC waste, 25 – branchial breaker. A – waste formed after cutting

CONCLUSIONS

1. It was found that by increasing the hydrothermal treatment temperature from 180 to 200 °C, most of the semi-crystalline C-S-H(I) recrystallize to 1.13 nm tobermorite, therefore the thermal shrinkage of products is reduced by approximately twice and the operating temperature increases from 450 °C to 600-650 °C.
2. It was determined that the amount of cement in the autoclaved aerated concrete, formation mixture can be reduced from 21.1 to 16.2 %, thus reducing the formation of cracks in formation mixture and thereby increasing the compressive strength of the product upto 6.7 %.
3. It was found that in the AAC samples which were hydrothermally treated 8 h at 180 °C the main formed products – 1.13 nm tobermorite and C-S-H (I) – had the highest compressive strength. Meanwhile, by increasing the hardening temperature and duration, the lower amount of the mentioned compounds and new compound – gyrolite – which does not show the binding properties, resulting in smaller values of the compressive strength, are formed.
4. It was estimated that at the beginning of calcium silicate hydrates synthesis, both soluble and insoluble transition metal ion change the calcium silicate hydrate formation mechanism in the $\text{CaO-SiO}_2\cdot n\text{H}_2\text{O-H}_2\text{O}$ system. Therefore, the duration of low base calcium silicate hydrates synthesis is shortened about 2 times when the initial mixture is mixed with soluble zinc ion addition. Meanwhile, insoluble in the reaction medium, cadmium oxide became unstable only after 24 h of hydrothermal treatment and then fully interfered into the structure of the synthesis products (gyrolite gel and Z-phase).
5. The recommendations of formation mixture composition for industrial plant of autoclaved aerated concrete were proposed: cement - 16.2 %, lime - 12.8 % gypsum - 2.3 % silica flour - 50.0 %, aluminum paste - 0.11 % sand slurry - 18.7 %, W/S=0.675. The achieved economic effect was 1.7 EUR/m³.

LIST OF SCIENTIFIC PUBLICATIONS ON THE THEME OF THE DISSERTATION

Publications in Journals Included into the Institute for Scientific Information (ISI) Database

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2. Baltakys, Kęstutis; Eisinas, Anatolijus; Baltakys, Marius; Šiaučiūnas, Raimundas. The incorporation of insoluble heavy metal oxide in the structure of Z-phase during hydrothermal treatment // *Advances in Applied Ceramics*. ISSN 1743-6753. 2014, vol. 113, no. 8, p. 466–471. [ISI Web of Science].
3. Iljina, Aliona; Baltakys, Kęstutis; Baltakys, Marius; Šiaučiūnas, Raimundas. Neutralization and removal of compounds containing fluoride ions from waste silica gel // *Romanian Journal of Materials*. ISSN 1583-3186. vol. 44, no. 3, p. 265–271. [ISI Web of Science].
4. Baltakys, Marius; Šiaučiūnas, Raimundas; Eisinas, Anatolijus. Influence of hardening conditions of autoclaved aerated concrete on tobermorite formation // *Romanian Journal of Materials*. ISSN 1583-3186. [ISI Web of Science]. (priimtas spaudai).

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1. Baltakys, Marius; Šiaučiūnas, Raimundas. The dependence of autoclaved aerated concrete properties on its mixture composition // *Proceedings of the BaltSilica 2014*, Poznan, Poland, 1-3 June, 2014 / Editors: A. Wawrzynczak, I. Nowak. Poznan: faculty of Chemistry, Adam Mickiewicz University, 2014, ISBN 9788362783014. p. P-6.
2. Šiaučiūnas, Raimundas; Baltakys, Marius; Bizimavičiūtė, Laura. The peculiarities of tobermorite and xonotlite formation in the dense samples // *Proceedings of the BaltSilica 2014*, Poznan, Poland, 1-3 June, 2014 /

- Editors: A. Wawrzynczak, I. Nowak. Poznan: faculty of Chemistry, Adam Mickiewicz University, 2014, ISBN 9788362783014. p. P-4.
3. Šiaučiūnas, Raimundas; Baltakys, Marius; Eisinis, Anatolijus. Dependence of thermal behaviour of aerated autoclaved concrete on its mineral composition // ESTAC11: the 11th European symposium on thermal analysis and calorimetry, August 17-21, 2014, Espoo, Finland. [S.l.]: Jyväskylä by Grano Ltd, 2014. p. 54.
 4. Baltakys, Marius; Šiaučiūnas, Raimundas. The influence of structure and mineral composition on properties of autoclaved aerated concrete // Chemistry and chemical technology: international conference of Lithuanian Society of Chemistry: Lithuanian Academy of Science, Vilnius, Lithuania, April 28-29, 2016: book of abstracts / Fizinių ir technologijos mokslų centras, Vilniaus universitetas, Lietuvos mokslų akademija, Kauno technologijos universitetas. [S.l.: s.n, 2016], ISBN 9786099551135. p. 253.
 5. Baltakys, Marius; Šiaučiūnas, Raimundas. AAC adsorption properties for zinc ions // BaltSilica 2016: book of abstracts of the 7th Baltic conference on silicate materials, 26-27 May 2016, Kaunas, Lithuania / Kaunas University of Technology, Riga Technical University, Adam Mickiewicz University in Poznan. Kaunas: Kauno technologijos universitetas. ISSN 2243-6057. 2016, p. 84.

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REZIUMĖ

Temos aktualumas

Autoklavinis aktytas betonas (AAB) – tai dirbtinis silikatinis akmuo, pasižymintis ekologiškumu, ilgaamžiškumu, atsparumu ugniai, geromis šilumą ir garsą izoliuojančiomis savybėmis bei yra lengvai mechaniškai apdorojamas (pjaustomas, šlifuojamas, frezuojamas ir t. t.). AAB šilumos izoliacinės savybės yra geriausios iš visų gaminamų mūro gaminių. Plono mišinio sluoksnio, kaip jungiančios medžiagos, panaudojimas eliminuoja „terminių tiltelių“ atsiradimą, todėl mažėja statybos išlaidos, o sutaupyta šilumos energija sudaro galimybę sumažinti išlaidas šildymui. Jam gaminti naudojamos hidraulinės rišamosios medžiagos – cementas ir / arba kalcitinės kalkės bei smulkios SiO₂ komponento turinčios medžiagos. Šios žaliavos sumaišomos su porodariu ir vandeniu, suformuojamas pusgaminis, kuris sukietinamas autoklave esančiame dideliame sočių vandens garų slėgyje.

Pagrindiniai junginiai, susidarantys vykstančių reakcijų metu gaminant autoklavinį aktytą betoną, yra kalcio hidrosilikatai: C-S-H(I), 1,13 nm tobermoritas bei ksonotlitas. Jie nulemia gaminių eksploatacines savybes. Šių kalcio hidrosilikatų susidarymas priklauso nuo AAB hidroterminio apdorojimo trukmės ir temperatūros, pradinio mišinio molinio santykio (CaO/SiO₂), žaliavų granulimetrinės sudėties ir grynumo, naudojamų priedų, maišymo intensyvumo ir kitų faktorių. Autoklavinio aktyto betono gamybai gali būti naudojamos įvairios, SiO₂ komponento turinčios žaliavos. Gaminant skirtingų savybių gaminius pasirenkamos įvairios žaliavų apdorojimo technologijos, formavimo mišinių sudėtys, kietinimo režimai, todėl gaminiuose gali susidaryti skirtingo baziškumo kalcio hidrosilikatai. Mokslinėje literatūroje yra duomenų, jog kalcio hidrosilikatai gali būti naudojami sunkiųjų ir radioaktyviųjų metalų jonais užterštiems vandenims valyti. Tačiau AAB gamybos metu susidarantių rišamųjų junginių adsorbcinės savybės sunkiųjų metalų jonams yra tirtos fragmentiškai.

Pagal *Europos Parlamento ir Tarybos direktyvą* iki 2020 m. būtina pasiekti bendrą tikslą – atsinaujinančių išteklių energija sudarytų ne mažiau kaip 20 % bendrojo, ES šalyse suvartojamo, energijos kiekio. Natūralu, kad ir Lietuvoje naujai statomiems gyvenamiesiems namams nuo 2017 01 01 įsigaliojo reikalavimas, pagal kurį sienų šiluminė varža turi būti ne mažesnė nei 8,3 m²·K/W. Šiluminė varža priklauso nuo medžiagos šilumos laidumo koeficiento ir izoliacinio sluoksnio storio. Norint tenkinti šiuos reikalavimus reikia pasirinkti tinkamas statybines medžiagas. Dėl šios priežasties AAB mūro gaminiai tapo itin populiarūs statybų rinkoje ir šiuo metu yra plačiai naudojami, nes gaminant AAB mūro gaminius, dėmesys yra kreipiamas į svarbiausias galutinio produkto savybes – gniuždomąjį stiprį ir tankį. Šios savybės nulemia ir kitas eksploatacines savybes, kurios turi didelę įtaką šilumos laidumo koeficiento vertei. Taip pat didelis

dėmesys skiriamas technologinio proceso efektyvumo didinimui, kuriam įtakos turi gamyboje pasitaikančios problemos: lėtas formavimo masės plastiškojo stiprio didėjimas, jos įšilimas, mažas išsipūtimo greitis bei formavimo masėje atsirandantys įtrūkimai. Dėl minėtų sunkumų gamybos procesas vyksta ilgai ir padidėja broko kiekis. Norint optimizuoti patį gamybos procesą nepabloginant, o pagerinant gaminio savybes, svarbu parinkti optimalius pradinių žaliavų santykius.

Darbo tikslas ir uždaviniai

Darbo tikslas – ištirti kalcio hidrosilikatų susidarymo kinetiką modelinėse ir pramoninėse sistemose autoklavinio akytojo betono gamybos technologiniams procesams optimizuoti ir praplėsti jo panaudojimo sritis.

Siekiant užsibrėžto tikslo reikėjo išspėsti šiuos uždavinius:

1. nustatyti sintezės parametrų įtaką kalcio hidrosilikatų susidarymo kinetikai akytame ir tankiame silikatiniam akmenyje bei nepresuotame bandinyje, taip pat produktų dispersiškumą, tekstūrą bei stabilumą;
2. ištirti formavimo mišinio komponentų santykio ir naujadarų mineralinės sudėties įtaką autoklavinio akytojo betono savybėms;
3. nustatyti autoklavinio akytojo betono gamybos optimalias hidroterminio apdorojimo sąlygas pramoninėje gamybos linijoje;
4. ištirti Cd^{2+} ir Zn^{2+} jonų turinčių junginių utilizavimo galimybes kalcio hidrosilikatų sintezės metu;
5. parengti rekomendacijas autoklavinio akytojo betono gamybos technologijos tobulinimui, siekiant sumažinti rišamosios medžiagos kiekį gaminiuose, jų savikainą ir pasiūlyti naujas panaudojimo sritis.

Mokslinis naujumas

Nustatyta, kad autoklavinio akytojo betono gaminių stiprumo sumažėjimą, vykstantį ilginant kietinimo trukmę, sukelia naujo junginio – girolito, kuriam nebūdingos rišamosios savybės, susidarymas.

Įrodyta, kad gaminių kietinimo metu į susidarančių kalcio hidrosilikatų kristalų gardelės struktūrą galima įterpti ir taip utilizuoti medžiagas, kurių sudėtyje yra aplinkai agresyvių tirpių pereinamųjų metalų jonų.

Praktinė vertė

Nustatyta, kad autoklavinio akytojo betono formavimo mišinyje cemento kiekį galima sumažinti nuo 21,1 iki 16,2 %. Tai ne tik leidžia apie 5 % sumažinti gaminių savikainą, bet ir ~17,5 % padidinti jų gniuždomąjį stiprį, sumažinti broko kiekį. Pakėlus hidroterminio apdorojimo temperatūrą nuo 180 iki 200 °C, didžioji dalis pusiau kristalinio C-S-H(I) persikristalيزuoja į 1,13 nm tobermoritą, todėl

gaminių terminė susitrauktis sumažėja apie du kartus ir jų darbinė temperatūra padidėja nuo 450 °C iki 600–650 °C. Remiantis gautais darbo rezultatais patobulinta UAB „Matuizų dujų silikatas“ gamybos technologija, dėl kurios gaunamas ekonominis efektas yra 1,7 EUR/m³ (gamybinės linijos pajėgumas – 160 tūkst. m³/metus).

Darbo aprobavimas ir publikavimas

Disertacinio darbo tema paskelbtos 4 mokslinės publikacijos leidiniuose su citavimo indeksu, įtrauktuose į *Thomson Reuters Web of Science* duomenų bazę: *Science of Sintering*, *Advances in Applied Ceramics* bei dvi publikacijos žurnale *Romanian Journal of Materials* (1 iš jų priimtas spaudai).

Disertacijoje atliktų tyrimų rezultatai paskelbti šešiuose pranešimuose, kurie skaityti penkiose tarptautinėse konferencijose: „33rd Cement and Concrete Science Conference“ (2013 UK, Portsmouth); „6th BaltSilica“ (2014, Lenkija); „7th BaltSilica“ (2016, Lietuva); „ESTAC11: the 11th European symposium on thermal analysis and calorimetry“ (August 17–21, 2014, Espoo, Suomija); „Chemistry and Chemical Technology“ (2016, Lietuva).

Darbo apimtis

Disertaciją sudaro įvadas, literatūrinių duomenų analizė, metodinė dalis, tyrimų rezultatai ir jų aptarimas, išvados, 149 literatūros šaltinių sąrašas, 4 publikacijų disertacijos tema ir 6 pranešimų mokslinių konferencijų medžiagoje sąrašas. Pagrindinė medžiaga išdėstyta 120 puslapių, įskaitant 11 lentelių ir 80 paveikslų.

Ginamieji disertacijos teiginiai

1. Autoklavinio akytojo betono gamybos metu susidarančių vienbazių kalcio hidrosilikatų kokybinė ir kiekybinė sudėtis nulemia jo eksploatacines savybes ir panaudojimo temperatūrą.
2. Hidroterminio apdorojimo temperatūra nulemia susidarančio akytojo akmens mikrostruktūrą. Ją pakėlus nuo 180 iki 200 °C dauguma plokštelių formos 1,13 nm tobermorito kristalų virsta pluoštiniais kristalais, o vyraujančių 0,6–0,0065 μm skersmens mikroporų kiekis padidėja nuo 49–52 iki 54–58 %.

IŠVADOS

1. Ištirta, kad padidinus hidroterminio apdorojimo temperatūrą nuo 180 iki 200 °C, didžioji dalis pusiau kristalinio C-S-H(I) pereina į 1,13 nm tobermoritą, todėl akytojo silikatinio akmens terminė susitrauktis sumažėja apie du kartus ir jų darbinė temperatūra padidėja nuo 450 °C iki 600–650 °C.
2. Nustatyta, kad autoklavinio akytojo betono formavimo mišinyje cemento kiekį galima sumažinti nuo 21,1 iki 16,2 %, tokiu būdu sumažinant formavimo mišinyje atsirandančius įtrūkimus ir taip padidinant gaminių gniuždomąjį stiprį 6,7 %.
3. Nustatyta, kad hidrotermiškai apdorojant AAB bandinius 8 h 180 °C temperatūroje, susidarę produktai – 1.13 nm tobermoritas ir C-S-H(I) – pasižymi didžiausiu gniuždomuoju stipriu. Tuo metu, didinat kietinimo temperatūrą ir trukmę, susidaro mažesnis minėtų junginių kiekis bei susidaro naujas junginys – girolitas, kuriam nebūdingos rišamosios savybės, todėl gaunamos mažesnės gniuždomojo stiprio vertės.
4. Ištirta, kad sintezės pradžioje tirpiųjų ir netirpiųjų pereinamųjų metalų jonų reakcinėje terpėje priedai keičia kalcio hidrosilikatų susidarymo $\text{CaO-SiO}_2\text{-nH}_2\text{O-H}_2\text{O}$ mišiniuose mechanizmą. Todėl mažabazių kalcio hidrosilikatų sintezės trukmė sutrumpėja ~ 2 kartus, kai į pradinius mišinius įmaišoma tirpaus cinko jonų turinčio priedo. Tuo metu, hidroterminėmis sąlygomis netirpus reakcinėje terpėje kadmio oksidas tik per 24 h tampa nestabilus ir iki galo įsiterpia į sintezės produktų (girolito gelio ir Z-fazės) struktūrą.
5. Rekomenduotina formavimo mišinio sudėtis pramoninei autoklavinio akytojo betono gamybai yra tokia: cementas – 16,2 %, kalkės – 12,8 %, gipsas – 2,3 %, maltas smėlis – 50,0 %, aliuminio pasta – 0,11 %, šlamo atliekos – 18,7 %, $V/K = 0,675$. Gautas ekonominis efektas yra 1,7 EUR/m³.

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