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# FROST RESISTANCE OF HARDENED CEMENT PASTE MODIFIED WITH SYNTHETIC ZEOLITE

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**Abstract.** The experiment was coducted using synthetic zeolite (modification A) obtained in the laboratory of Kaunas University of Technology by means of low-temperature synthesis (below 105 °C). Aluminium fluoride ($AIF_3$) production waste as well as sodium hydroxide NaOH and aluminium hydroxide $Al(OH)_3$were used to obtain a complex zeolite admixture. SEM analysis revealed that synthetic zeolite admixture was made of modification A zeolite and gypsite. SEM analysis results were confirmed by X-ray analysis. Three batches of specimens were made to determine the effect of synthetic zeolite admixture on the durability of hardened cement paste. The first batch was without synthetic zeolite admixture, the second batch and the third batch contained 5 wt% and 10 wt% of synthetic zeolite admixture, respectively. Synthetic zeolite admixture was found to have a significant effect on the durability of the hardened cement paste. The weight loss in the hardened cement paste containing 5 wt% of the admixture was three times lower, whereas the weight loss in the hardened cement paste containing 10 wt% of the admixture decreased up to 100 times.

**Keywords:** hardened cement paste, synthetic zeolite, freeze-thaw cycles, SEM, X-ray analysis.

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## Introduction

The deterioration of concrete structures exposed to salts is a significant problem and a big concern in North America and northern Europe. NaCl and $CaCl_2$ salts are widely used to ensure adequate traffic conditions in winter (Baltrėnas *et al.* 2009). However, they deteriorate the condition of concrete bridges, roads and landscaping elements. De-icing salts cause repeated freezing and thawing on concrete structures and cause adverse cracking. The cracks create conditions for chemical substances to penetrate into concrete structure and cause adverse effects, such as alkaline reactions and corrosion of reinforcement bars. The cost of bridge and road repairs due to salt-caused damage in the USA amounts to approximately USD 90–151 million. Although part of these costs can be attributed to insufficient maintenance of concrete structures, the main cause of deterioration in the roadworthiness of concrete bridges in the majority of northern countries is the use of de-icing salts.

The analysis of scientific, especially technical and practical literature on this issue has revealed that the mechanism of concrete degradation caused by cyclic freeze-thaw conditions and de-icing salts is not entirely clear. J. Marchand, E. Sellevold and M. Pigeon in their review on concrete degradation caused by cyclic freeze-thaw conditions and de-icing salts (Marchand *et al.* 1994) emphasize the agreement reached by the authors that the effect of de-icing salts on hardened cement paste and concrete is more physical than chemical. Chemical reactions between cement hydration products and salts are a secondary factor in

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concrete destruction process because weaker de-icing salt solutions (up to 5%) have a greater adverse effect compared to stronger solutions. The most frequently observed fact in the testing of concrete resistance to de-icing salt solutions is that concrete subjected to 3% NaCl solution has the lowest frost resistance. More tests in this field were made by Marchand, Kaufmann and other authors (Marchand *et al.* 1999; Kaufmann 2000). The evaluations of the effect of the type of salt solution on the destruction of concrete structures and the researcher opinions differ. K. Wang and J. Šelih state that $CaCl_2$ has the greatest destructive effect on cement-based materials (Wang *et al.* 2006; Šelih 2010). L. Sutter and his colleagues (Sutter *et al.* 2006) emphasize that the most hazardous effect of salt on the durability of bridge structures is caused by prolonged exposure to $MaCl_2$ ir $CaCl_2$. Concrete saturation and brine absorption rate depends on brine concentration (Marchand *et al.* 1994). Basing on the research work of other authors, these authors state that de-icing salts usually increase the saturation of the upper layer of concrete with water (brine), because the pressure of saturated gas above the brine is lower than above water. Besides, water vapour condenses easier in brine than in fresh water. Therefore, the general assumption is that in practice, de-icing salt solutions cause higher water absorption in concrete and subsequently a higher amount of ice. In the opinion of referred authors, such hygroscopic action of de-icing salts is not so evident in concrete frost resistance tests and for this reason the test methodologies need improvement.

The durability of hardened cement paste also depends on porosity. The pores of hardened cement paste are categorized by their form, size and nature into the following three groups: capillary, air and gel pores (Mehta, Monteiro 1993). The porosity also depends on the mineral compositions of clinker, the content of gypsum in the cement, the fineness of cement, alkaline content and also on the type and content of active mineral admixtures. Physical and mechanical properties of concrete as well as its frost resistance are also influenced by unpredictable cement storage time due to economic instability. Technologists of reinforced concrete plants are well aware of the fact that prolonged storage of Portland cement reduces the strength properties of products made of such cement and the frost resistance is affected much more, although no direct relationship between cement activity and concrete frost resistance has been established (high-strength frost resistant concrete can be produced from low-active cement).

Chemical and mineral composition of Portland cement, especially the content of calcium aluminates $C_3A$ and alkali in the cement, are the key factors that are taken into account in the analysis and prediction of concrete's resistance to the effect of de-icing salts (Jackson 1958; Pigeon *et al.* 1992). Prince and Gagne (2001) argue that the immigration of chlorides depends on solution concentration and composition of migrating ions; therefore it is hard to compare chloride diffusion rates obtained in different laboratories. The ion composition in the solution also depends on the chemical composition of the cement, especially on the alkaline content. Portland cement that is not good for producing frost resistant structures is described by Žiogas *et al.* (1996), and Muller *et al.* (1995) with colleagues. However, the authors relate the insufficient frost resistance of concrete with calcium hydroxide release during freezing, which is justified by higher solubility of this hydroxide at low temperatures. The authors advise to use Portland cement with low alite content ($C_3S$) in frost resistant concrete manufacturing. We assume, however, that higher release of free lime in concrete subjected to freeze-thaw cycles is a secondary characteristic that indicates the beginning of concrete destruction and the formation of new surfaces (cracks) in concrete matrix where calcium hydroxide is released by the cement particles that had not reacted before. In the discussion of the effect of Portland cement (clinker) fineness on concrete frost resistance Gumuliauskas (1998) states that the use of very fine cement (especially repeatedly grinded) to accelerate concrete setting is disputable because the initial effect may deteriorate the durability of such concrete as the long setting potential of concrete is exhausted and there are no remaining clinker minerals necessary to maintain the protection of reinforcing bars.

Many authors offer ungrounded and often controversial reasons of such destruction without adequate evaluation of concrete frost resistance enhancing elements well know to concrete technologists, for instance $SiO_2$ microparticles, ash, sodium silicate solution, air entraining admixtures ir plasticizers. Sodium silicate solution, as well as superplasticizer, contains nano-structures. Sodium silicate solution contains undissolved $SiO_2$ microparticles of 1–2 mm in size and sometimes even a small content of sodium silicate (0.03–1%) has a positive effect on cement hydration (Sandberg, Mosberg 1989; Pundiene *et al.* 2005; Kiricsi *et al.* 2006; Skripkiūnas *et al.* 2008). Highly active $SiO_2$ added to concrete mixes at 0.25–0.5 wt% is able to bind free $Ca(OH)_2$ (portlandite).

Zeolites are crystal hydro aluminium silicates of alkaline and alkaline earth metals with different Al/Si ratio. At present 180 zeolites have been synthesized and about 100 natural zeolites have been detected. Zeolite application is most efficiently developed in the following directions: mixes of binding materials, concretes and non-autoclaved silicate products. Zeolites have a high content of active $SiO_2$ and $Al_2O_3$. Silica dioxide and ash that are similar to other pozzolanic substances, can improve the strength of concrete through $Ca(OH)_2$ reaction with pozzolans. Zeolites, as other pozzolanic substances, add greater strength to concrete compared to cement (Su *et al.* 2000).

## 1. Research methodology

Dry admixtures were used to produce cement pastes for the tests. Cement and synthetic zeolite admixture were dosed by weight, water and chemical admixtures were dosed by weight. The plasticizing admixture was mixed into the cement together with water that was used to prepare the cement paste.

Cement pastes were made in the forced action mixer *Automix* from dry matter in accordance with the testing procedure provided by the standard LST EN 196-1. The mixing time was 90 seconds.

Concrete specimens were formed in impermeable and moisture repellent forms with dimensions 40×40×160 mm. The specimens were left in moulds for 20 hours at the ambient temperature of 20 ± 2 °C, then removed from moulds and hardened in water at 20 ± 2 °C for 7 days. After curing in water the specimens were removed from the heating bath. All specimens except for the test specimen were covered with a 3 mm-thick rubber sheet. Rubber glue was used to glue the rubber sheet along the perimeter of the specimen. The edge of the rubber sheet was formed 20 mm above the specimen surface. Gaps between the specimen and the rubber sheet were sealed with silicone along the entire perimeter in order to prevent the brine from getting under the rubber. Prior to cyclic freezing and thawing, surfaces of all specimens, except for the test specimen, were thermally insulated, i.e. the specimens were placed into boxes made of 2.0 mm Styrofoam. Frost resistance after 7, 14, 21, and 28 freeze-thaw cycles was estimated by weight loss. Every 7th freeze-thaw cycle, starting at the 23rd hour until the 24th hour of the thawing phase, the scaling was collected from the test surface. The test surface was sprayed with water and cleaned with a brush (with stiff bristles) until no scaling remained. The scaling was collected into a container. The scaling was carefully poured into a filter and rinsed with at least one litre of tap water in order to remove the residual NaCl. The filtering paper with deposited scaling was dried for 24 h at 105 ± 5 °C. The weight of the dry scaling was measured with ±0.2 g precision taking into consideration the weight of the filtering paper. After weighing the scaling the weight loss was calculated per surface area (L) in $kg/m^2$.

$$L=M/A, \tag{1}$$

where: M is the weight of the peeled matter in kilograms after $n$ cycles; $A$ is the surface area of the test surface in square meters.

30–15 minutes prior to placing the specimens into the freezing chamber a 5–2 mm-thick layer of 3% NaCl was poured onto the test surface. The thickness of the brine layer was measured from the top surface of the specimen. To prevent the evaporation, the brine was covered with a polyethylene sheet. Cyclic freezing and thawing was conducted in the freezing chamber *RUMED 3001 bis 3601* with programmed limits of temperature change (Fig. 1).

One freeze-thaw cycle lasted for 24 hours. The temperature during each cycle was above 0 °C for at least 7 hours, however not longer than 9 hours.

JEOL scanning electron microscope (SEM) *JSM-6490 LV* with 3.0 nm resolution and magnification from 5 to 300 000 times was used for the analysis of synthetic zeolite microstructure. The specimens were covered with electrically conductive carbon in vacuum.

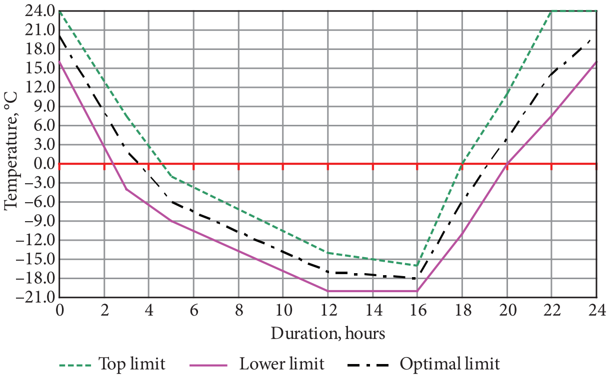

Fig. 1. The diagram of freeze-thaw cycle temperature changes in the freezing chamber

The test was performed at accelerating voltage of 20 kV. A computerized optical microscope OLYMPUS with magnification up to 1000 times was used for the test.

X-ray diffraction analysis was done by diffractometer *ДРОН-6.* Test specifications: CuK radiation, Ni filter, detector step 0.02°, 0.5 s intensity measuring span, 30 kV anode voltage, and 20 mA current.

## 2. Materials

Portland cement CEM I 42.5 R complying with EN 197-1:2001 requirements. Portland cement characteristics are presented in Table 1. The compressive strength after 2 days and after 28 days was 28 ± 2 MPa and 54 ± 3 MPa respectively, the initial set was 160 minutes, w/c ratio was 25.3%, the specific surface area was 3700 $cm^2/g$. Chemical properties of the cement: loss on ignition 1.4%, content of insoluble residue 0.5%, sulphate ($SO^3$) content 2.8%, chloride ($Cl^-$) content 0.005%, hydration heat 365 J/g.

Synthetic zeolite admixture (modification A) obtained by means of low temperature synthesis. Aluminium fluoride ($AIF_3$) production waste as well as sodium hydroxide NaOH and aluminium hydroxide $Al(OH)_3$ were used to obtain a complex zeolite admixture. Synthetic zeolite contained: $SiO_2$ – 28.6%, $Al_2O_3$ – 37.9%, $Na_2O$ – 14.1%. Dried and sieved synthesis products were modified by the solution of soluble salt calcium chloride ($CaCl_2$) to obtain the synthetic zeolite of modification A. Composition of modified synthetic zeolite: $SiO_2$ – 29.3%, $Al_2O_3$ – 36.5%, $Na_2O$ – 7.3%, CaO – 7.5%. Modified zeolite is a mix of zeolite A ($Na_{96}Al_{96}Si_{96}O_{384}\cdot 2164H_2O$) and calcium zeolite $(Ca, Na)\cdot Al_2Si_{2,5}O_9\cdot 6,4H_2O$.

Superplasticizer: polycarboxylic ester based superplasticizer *Muraplast FK 63.30.* A yellowish liquid with density 1.06 $g/cm^3$, viscosity 750 mPas and pH 4.8–6.8. Water – complying with LST EN 1008:2002 technical requirements.

Table 1. Compositions of hardened cement paste, $1m^3$

| Synthetic zeolite, % 0 5 10 | | | |
|---|---|---|---|
| Synthetic zeolite, kg | 0 | 0.058 | 0.116 |
| Portland cement, kg | 1.647 | 1.565 | 1.482 |
| Water, kg | 0.445 | 0.445 | 0.445 |
| Superplasticizer, ml | 8.2 | 8.2 | 8.2 |
| V/K | 0.27 | 0.27 | 0.27 |
| V/C | 0.27 | 0.28 | 0.30 |

## 3. Analysis of the test results

SEM images of synthetic zeolite revealed that synthetic zeolite crystals had the form of a cube about 1μm in size. The images are presented in Figure 2. Image (a) in Figure 2 is enlarged 5000 times, image (b) is enlarged 10 000 times and image (c) is enlarged 20 000 times. Small cubes of synthetic zeolite are seen in the images. Plate-shaped gypsum crystals are also seen in the image increased 10 000 times.

X-ray diffraction pattern obtained by means of diffractometer *ДРОН-6* is presented in Figure 3. The X-ray images produced similar results as SEM images. Patterns of modification A zeolite and gypsite were mainly identified.

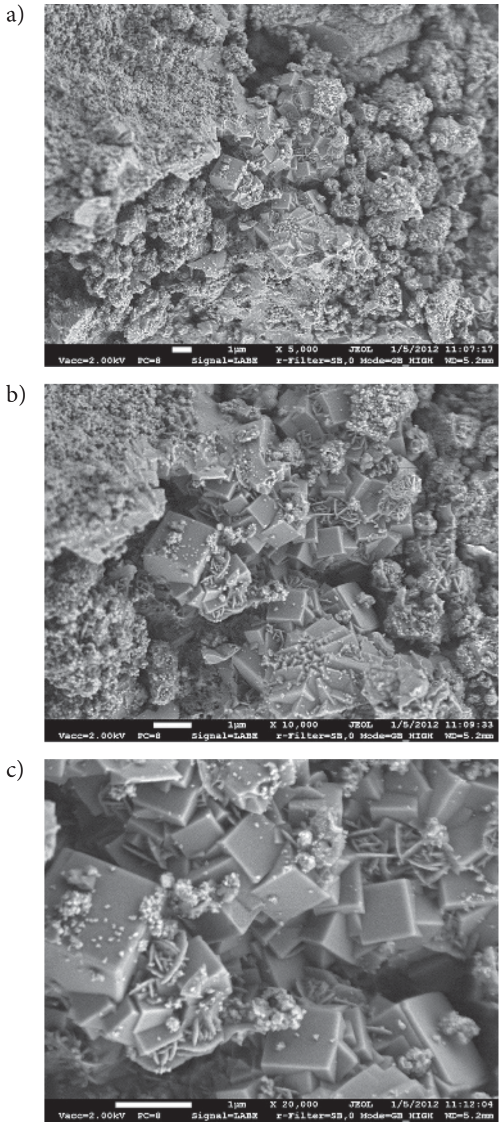

Fig. 2. SEM images of modification A synthetic zeolite produced from $AIF_3$ production waste: a – the view enlarged 5000 times; b – the view enlarged 10 000 times; c – the view enlarged 20 000 times

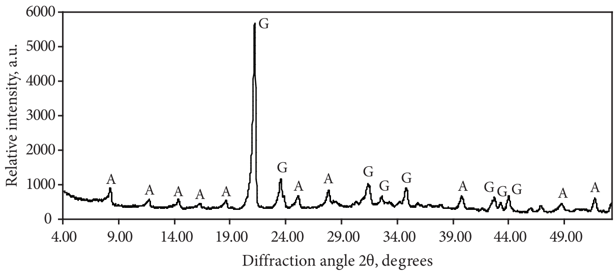

Fig. 3. X-ray of synthetic zeolite admixture: A – zeolite A; G – gypsite ($Al(OH)_3$)

SEM and X-ray analysis of synthetic zeolite was followed by freeze-thaw tests of hardened cement paste containing synthetic zeolite admixture.

Three batches of cement paste were tested. The first batch had no zeolite admixture; the second batch contained 5 wt% of synthetic zeolite and the third batch contained 10 wt% of synthetic zeolite.

The weight loss was measured every 7th freeze-thaw cycle.

The diagram in Figure 4 illustrates the change in weight loss in later freeze-thaw cycles. It should be noted that specimens without synthetic zeolite, i.e. specimens containing cement CEMI 42.5R, superplasticizer and water survived 21 freezing cycles. The diagram illustrates that in the first 7 cycles the weight loss gradually increased and reached 0.32 kg/m$^2$. After 14 cycles in the freezing chamber the total weight loss was 0.99 kg/m$^2$; the biggest weight loss of 10.61 kg/m$^2$ was observed after 21 freeze-thaw cycles. The specimens collapsed after 21 freeze-thaw cycles.

Figure 5 illustrates the results of weight loss in specimens containing 5% of synthetic zeolite. The weight loss curve in the diagram is very similar to the curve in Figure 4, only the different weight loss should be noted. We may see that after 7, 14 and 21 freeze-thaw cycles the weight loss was 0.19 kg/m$^2$, 0.29 kg/m$^2$, and 2.93 kg/m$^2$ respectively. The tests revealed that synthetic zeolite admixture added at 5 wt% reduces the weight loss in hardened cement paste about three times. Specimens without zeolite admixture as well as specimens with zeolite admixture collapsed after 21 freeze-thaw cycle.

Much better results were obtained in the tests of hardened cement paste where 10 wt% of cement was replaced by synthetic zeolite admixture. The specimens remained undestroyed after 28 freeze-thaw cycles and the weight loss was only 0.09 kg/m$^2$. Figure 6 illustrates the weight loss in hardened cement paste with 10 wt% of synthetic zeolite admixture.

As seen from the diagram, the biggest weight loss in specimens with 10 wt% of zeolite admixture occurred during the first 7 freeze-thaw cycles. The weight loss amounted to 0.073 kg/m$^2$. During 14 freeze-thaw cycles the weight loss was only 0.01 kg/m$^2$ between cycle 7 and cycle 21, and in the remaining 7 freeze-thaw cycles only 0.007 kg/m$^2$ of scaled mass was collected.

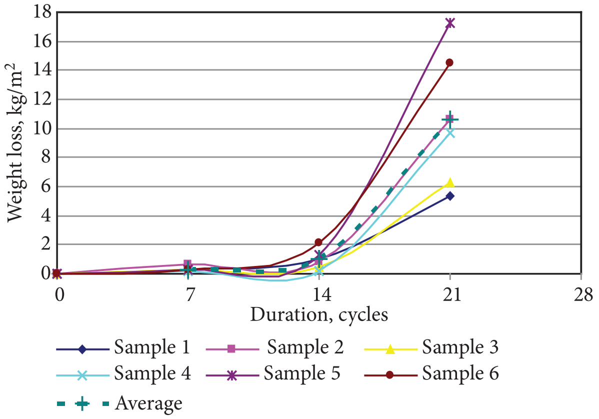

Fig. 4. Weight loss in hardened cement paste without SZ admixture

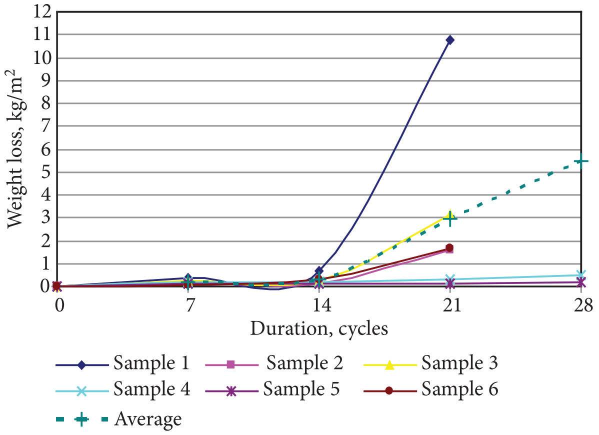

Fig. 5. Weight loss in hardened cement paste with 5 wt% SZ admixture

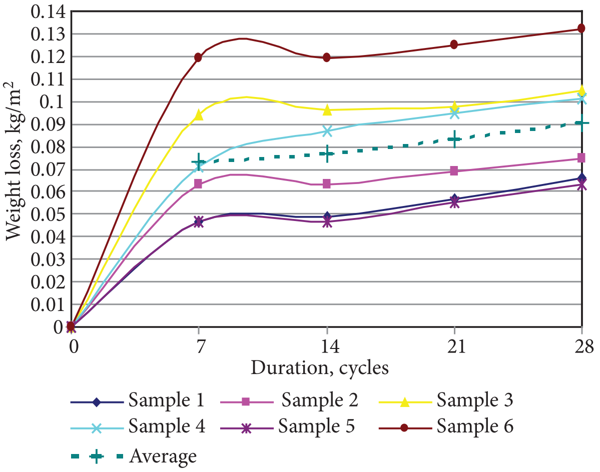

Fig. 6. Weight loss in hardened cement paste with 10 wt% SZ admixture

## Conclusions

1. SEM analysis of synthetic zeolite admixture revealed that zeolite particles have the shape of a cube and are 1μm in size.
2. X-ray images of synthetic zeolite admixture revealed that this admixture is made of modification A zeolite and gypsite ($Al(OH)_3$).
3. Weight loss after 28 freeze-thaw cycles reduces 100 times when 10 wt% of synthetic zeolite is added to the cement paste.
4. The test results have proved that synthetic zeolite may be used in concrete structures that are subject to higher requirements for freeze-thaw and de-icing salt resistance.

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# SINTETINIU CEOLITU MODIFIKUOTO CEMENTINIO AKMENS ATSPARUMAS ŠALČIUI

**G. Girskas, D. Nagrockienė, G. Skripkiūnas**

**Santrauka.** Tyrimams atlikti buvo naudojamas sintetinis ceolitas (A modifikacijos), gautas atliekant žematemperatūrę (iki 105 °C) sintezę. Kompleksiniam ceolitiniam priedui gauti naudota aliuminio fluorido ($AIF_3$) gamybos atlieka, taip pat natrio hidroksidas NaOH ir aliuminio hidroksidas $Al(OH)_3$. Darbe SEM analizės būdu buvo nustatyta, kad sintetinio ceolito priedas sudarytas iš A modifikacijos ceolito ir gipsito. Atlikti rengenografiniai tyrimai taip pat patvirtino SEM analizės duomenis. Norint nustatyti sintetinio ceolitinio priedo įtaką cementinio akmens ilgaamžiškumui, buvo suformuotos trys partijos bandinių. Pirmoji partija buvo be sintetinio ceolitinio priedo, antroji – su 5 %, trečioji – su 10 % priedo. Nustatyta, kad sintetinis ceolitinis priedas turi didelį poveikį cementinio akmens ilgaamžiškumui. Cementinio akmens su 5 % priedo masės nuostoliai sumažėja apie tris kartus, o akmens su 10 % priedo cemento masės nuostoliai sumažėja iki 100 kartų.

**Reikšminiai žodžiai:** cementinis akmuo, sintetinis ceolitas, užšaldymo ir atšildymo ciklai, SEM, rengenografinė analizė.

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