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IMPACT OF NATURAL ZEOLITE ON THE PROPERTIES AND RESISTANCE TO ALKALI SILICA REACTION OF MORTAR

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This research investigates the effect of the amount (from 2.5 % to 15 %) of natural zeolite on the alkali silica reaction resistance (ASR), density, ultrasonic pulse velocity and mechanical properties of mortar. Research revealed that physical and mechanical characteristics of mortar mix improve with higher amount of natural zeolite, however, the appropriate zeolite amount must be selected because overdosing leads to deterioration of mortar mix properties. The impact of natural zeolite and the amount used on the expansion of mortar specimens was tested using Rilem AAR-2 method. Altered composition mortar specimens were tested using SEM and X-ray structure analysis, their mineral composition was determined. Dependence between natural zeolite amount in mortar mix, mineral composition of hardened mortar and expansion was determined. Test results revealed that alteration of mortar composition by natural zeolite not only improves physical and mechanical properties of the mix but also increases alkali silica reaction resistance (ASR) of mortar. The tests revealed that alteration of mortar composition center mortar mix) increases durability and resistance to alkaline silica reaction.

INTRODUCTION

In today's construction industry, concrete is the main and most used material [1]. Concrete is a durable and very reliable building material. [2]. However, over time even concrete becomes vulnerable, some properties deteriorate [3]. The properties of concrete can be improved by using various mineral additives with different characteristics [4]. Concrete is made from four basic ingredients: coarse aggregate (gravel), fine aggregate (sand), portland cement, and water. When curing concrete, complex chemical reactions occur between aggregates and cement. Amorphous silica and carbonates contain most natural aggregates and they react with Na and K in the concrete mix [5]. The expansion of the alkaline and aggregate reaction (AAR) product causes cracks and degradation of mechanical properties, which can cause concrete to collapse [6].

ASR in concrete is one of the most dangerous reactions in concrete [7]. Silica or silicate, which drive ASR in concrete, are mainly supplied by reactive aggregates. Subsequently, silica reacts with alkali-hydroxides in concrete to form ASR gel [8]. This interaction is widely known as alkali aggregate reaction (AAR). There are two types of AAR depending on the minerals involved, namely alkali silica reaction (ASR) and alkali-carbonate reaction (ACR). Aggregates containing reactive silica minerals are responsible for the development of ASR in concrete [9].

Research on this reaction has taken more than half

a century. This allowed the researchers to investigate the reaction mechanism and make recommendations to reduce the harmful effects [10]. However, the reaction in concrete has not yet been completely stopped. Huge money is being spent all over the world to eliminate defects caused by ASR [11].

ASR cracks in concrete structure are caused by expansions of alkali silica reaction product (gel) [12]. ASR is a slow process that develops over years to decades and it is influenced by changes in environmental and loading conditions of the structure [13].

Mineral admixtures are siliceous materials in finely divided form that are added in relatively large amounts to concrete during mixing. Industrial by-products are the primary source of mineral admixtures. In Europe these mineral admixtures are normally called additives. Many researchers also call them supplementary cementing materials (SCM) [14].

Zeolites are crystalline aluminum hydrosilicates of alkali and alkaline earth metals with an open three-dimensional backing (tectosilicates) consisting of: (a) repeated fundamental building units of silicon-oxygen and aluminum-oxygen tetrahedras forming single 4/6-ring, double 4/6-ring, or larger symmetrical polyhedral SiO₄--AlO₄ framework, (b) charge-balancing, exchangeable mono and divalent alkaline and alkaline earth metal cations (Na⁺, Ca²⁺, K⁺, Mg²⁺, Sr⁺, Ba²⁺) present in the interconnected void spaces or spacious channels in the framework and in intraframe cavities, and (c) highly variable amounts of water molecules in the cavities that are weakly bonded to anionic framework atoms [15]. Zeolites are rich in active SiO_2 and Al_2O_3 . Silica dioxide and ash, like other pozzolanic materials, can improve concrete strength by means of Ca(OH)₂ reaction with pozzolans [16].

Most of the natural zeolites are formed by volcanic rocks in contact with fresh or seawater. Natural zeolites formed in the following geological environments: alkaline deserts, lake deposits, ash ponds, etc. In the 1950's, geologists discovered that million-ton deposits of volcanic tuff consisting mostly of zeolitic materials are not uncommon [17].

Such zeolitic crystals, by virtue of their unique structures, get filled up with water which can be driven off by heating. As such, the dried up crystals possess a honeycomb-like structure consisting of openings or pores of the order of a few atoms in width ($\sim 2 \text{ to } 10 \text{ Å}$) [18, 19].

Natural zeolites, such as Clinoptilolite (i.e., popularly known as clino zeolites) and Chabazite, have applications in the fields of water treatment, fertilizers for soil amendment and plant growth by better retention of nutrients. Clinoptilolite has been widely used in agriculture for soil improvement and as feed additives because it has a higher amount of acid resistant silica (viz., Si/Al = 1:5). However, such zeolites are contaminated by other minerals (e.g., Fe⁺⁺, SO₄⁻⁻, Quartz, other zeolites, and amorphous glass) and hence they may not be suitable for several important commercial applications where uniformity and purity are essential [20].

The use of zeolite increases the hydration rate of the cement and affects the physical and mechanical properties [21].

ASR depends on several factors: activators, cement, reactivity level of aggregates, humidity and temperature. The product of ASR lead to expansion, causing degradation of the concrete, particularly in civil engineering structures subjected to wet environments such as dams, massive bridge parts railroad sleepers and prestressed units [22].

Fine particles of concrete mix are very important and have a great influence on the water demand, properties of fresh and hardened concrete, so it is necessary to pay attention to this when designing the composition of concrete mix [23]. Properties of concrete mix and hardened concrete (physical, mechanical, rheological, durability, etc.) can be increased by using various additives [2].

ASR impact can be mitigated by using the right type of cement containing pulverised flue ash or blast furnace slag [24]. The rate and extent of the reaction depends on the reactivity level, amount, composition and particle size of the aggregates.

Mineral additives also have a significant effect on the properties of the concrete mix: water demand, heat of hydration and reactivity [4]. The durability of concrete structures is one of the most important properties that can be improved by using natural zeolite as a mineral additive [25]. The effect of AAR in concrete can be minimized by the use of pozzolanic additives [26, 27].

The analysis of the literature found research mortar that replaces cement with natural zeolite resistance to alkali silica reaction. The aim of our work was to investigate the effect of zeolite content on the alkali silica reaction resistance and physical and mechanical properties of the mortar.

Studies have shown that it is effective to replace cement in the mortar with 12.5 % to 15 % natural zeolite due to increased resistance to alkaline corrosion.

EXPERIMENTAL

Mortar specimens were made from Portland cement CEM I 42.5 R. The chemical composition of the cement is presented in Table 1.

Sand of fraction 0/4 was used as fine aggregate. Polycarboxylate-based plasticising admixture with pH of 4.6. The mortar was modified with natural zeolite additive. The chemical composition of zeolite is presented in Table 2.

7 bathes of mortar specimens were moulded for laboratory tests. The specimens contained different amounts of zeolite in the cement mix ranging from 0 % to 15 % by weight of cement. The compositions of mortar mixtures proportions are presented in Table 3. The dimensions of moulded specimens were $40 \times 40 \times 160$ mm. Density, compressive strengths and flexural strengths, ultrasonic pulse velocity of the specimens were measured after 28 days of curing.

Table 1. Chemical composition of cement.

Chemical composition	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	K ₂ O	SO ₃	Na ₂ O	Other
Amount (%)	63.31	20.70	6.10	3.36	1.0	0.80	0.30	4.44

Table 2. Chemical composition of natural zeolite.

Chemical composition Amount (%)	SiO ₂	Al ₂ O ₃	CaO	K ₂ O	FeO ₃	MgO	Na ₂ O	TiO ₂	Other
	68.15	12.18	3.95	2.80	1.30	0.90	0.75	0.20	9.77

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Table 3. Mortar mixture proportions. (wt. %)								
Natural zeolite in the cement mix	Portland Sand cement		Water	W/C	Plasticizing admixture			
0	26.88	60.48	12.63	0.47	0			
2.5	26.21	60.48	12.63	0.47	0			
5.0	25.54	60.48	12.63	0.47	0			
7.5	24.87	60.48	12.63	0.47	0.05			
10.0	24.19	60.48	12.63	0.47	0.10			
12.5	23.52	60.48	12.63	0.47	0.10			
15.0	22.85	60.48	12.63	0.47	0.10			

The density of the specimens was measured according to EN 12390-7:2009, the compressive strength was measured according to EN 12390-3:2009/AC:2011, the flexural strength was measured according to EN 12390-5:2009, and ultrasonic pulse velocity was measured according to EN 12504-4:2004.

Phase analysis of natural zeolite was done by X-ray diffraction meter SmartLab (Rigaku) with a rotating Cu anode X-ray generator tube. The X-ray diffraction patterns were recorded in the angular range $5 - 75^{\circ}$ (2 θ), detector step 0.02°, detector movement speed 1° per minute. Database of crystal structures PDF- 4+ (2016) was used for the analysis

The microstructure of mortar specimens and natural zeolite was tested with the scanning electron microscopy (SEM) device SEM JEOL 7600F. Electron microscopy parameters: Power 10 kV and 20 kV, distance to specimen surface 7 - 10 mm. Characteristics of the microstructure was identified by testing the specimen splitting surface. The image was obtained by recording the signal of secondary electrons. Before testing the splitting surface was coated with electrically conductive thin layer of gold by evaporating the gold electrode in the vacuum using the instrument QUORUMQ150R ES. The specimens of the tested materials were photographed by the digital camera Sony ILCE 5000.

The resistance of mortar sample to ASR was tests in accordance with the standardised Rilem AAR-2 methodology. According to "RILEM AAR-2" method the specimens ($40 \times 40 \times 160$ mm) hardened in water of 80 °C temperature was kept for 14 days in 1 N NaOH solu-tion at 80°C temperature and afterwards their expansion is measured (expansion limit 0.054 %). The methodology prescribes a longer conditioning time for specimens with aggregates containing slowly reactive rocks. In our case the time of soaking specimens in 1N NaOH solution was extended to 56 days (expansion limit 0.1 %).

RESULTS AND DISCUSSION

Mineral structure of natural zeolite determined by X-ray diffraction analysis is presented in Figure 1. The analysis revealed that the prevailing element in natural zeolite heulandite $(Na_{2.6}Mg_{0.36}Al_{3.32}Si_{14.68}O_{36}$ $(H_2O)_{10.32})H$ representing 92.2 % of natural zeolite. X-ray image also showed the presence of muscovite ((K, Na)(Al, Mg, Fe)_2(Si_{3.1} Al_{0.9}) O_{10}(OH)_2)M and anorthite (Ca Al₂ Si₂ O₈)A representing 0.7 and 7.1 % respectively in natural zeolite.



 $\begin{array}{l} \mbox{Figure 1. XRD spectrum of natural zeolite:} \\ \mbox{H-heulandite } (Na_{2.6}Mg_{0.36}Al_{3.32}Si_{14.68}O_{36}(H_2O)_{10.32}); \\ \mbox{M-muscovite } ((K, Na)(Al, Mg, Fe)_2(Si_{3.1}Al_{0.9})O_{10}(OH)_2); \\ \mbox{A-anorthite } (CaAl_2 Si_2O_8). \end{array}$

Images of natural zeolite microstructure are presented in Figure 2. Images obtained by the scanning electron microscope (SEM) revealed that natural zeolite particles have irregular shape and their size is approx 11 μ m in average. The image magnified 5000 times (Figure 2b) shows that particles of natural zeolite are made of irregular and angular crystals distributed in different directions, the size of the crystals is up to 0.5 μ m.

Figure 3 represents the results of density tests of altered composition mortar with natural zeolite. The diagram shows that the highest density of 2111 kg·m⁻³ was obtained in mortar specimens containing 10 % and the lowest density of 2031 kg·m⁻³ is observed in the specimen without zeolite additive. Density increase 3.94 % (80 kg·m⁻³) until the amount of natural zeolite in the mix reaches 10 %. With the increase of natural zeolite amount up to 12.5 %, the density decreases (2109 kg·m⁻³) and further increase of zeolite amount up to 15 % the density decreases even more (2101 kg·m⁻³).

Figure 4 represents the dependence between the compressive strength of mortar and natural zeolite amount. The compressive strength after 7 days of curing



a) ×1000

b) ×5000

Figure 2. SEM images of natural zeolite: a) magnification ×1000, b) magnification ×5000



Figure 3. Dependence between density and natural zeolite amount in mortar.

control samples are 46.78 MPa. Increasing the natural zeolite content to 12.5 % increases the compressive strength of mortar. The highest compressive strength, which is 6.01 MPa above the compressive strength of the control specimen, was registered in the specimen containing 12.5 % of mineral additive. The difference between these two compressive strength values is 12.85 %. After 7 days of curing the compressive strength in the specimen containing the maximum tested amount of natural zeolite, namely 15 %, decreased down to 52.56 MPa.

After 28 days of curing the compressive strength increases with the increase of mineral additive up to 12.5 %. The highest compressive strength was (56.41 MPa) was observed when 12.5 % of natural zeolite was added to the mix and it was 3.61 MPa (8.31 %) higher than in control specimen. The compressive strength of the specimen containing the maximal amount of natural zeolite (15 %) was 55.59 MPa or 0.82 MPa lower than the samples are 12.5 % natural zeolite additive.

Figure 5 represents the dependence between the flexural strength and natural zeolite amount in specimens cured for 7 and 28 days. After 7 days of hardened the flexural strength gradually increase with the increase



Figure 4. Dependence between the compressive strength and natural zeolite amount in mortar.

of natural zeolite amount until the amount of mineral additive reaches 12.5 %. The highest flexural strength, which is 0.38 MPa above the value of the control specimen, was registered in the specimen containing 12.5 % of natural zeolite. The difference between the flexural strength values was 6.39 %. The flexural strength of the specimen containing the maximum tested amount of natural zeolite, which is 15 % by weight of cement, decreased down to 6.29 MPa.



Figure 5. Dependence between the flexural strength and natural zeolite amount in mortar.

After 28 days of hardened the flexural strength increases in specimens containing up to 12.5 % of natural zeolite. The flexural strength of specimen without mineral additive was 6.91 MPa. The highest flexural strength (7.19 MPa) was observed when 12.5 % of natural zeolite was added to the mix and it was 0.28 MPa (4.05 %) higher than the value of the control sample. The flexural strength of the sample containing the maximum amount of natural zeolite (15 %) are 7.18 MPa or 0.01 MPa decreasing than the flexural strength of the specimen have 12.5 % of mineral additive.

Figure 6 represents the impact of the mineral additive on ultrasonic pulse velocity in mortar specimens. After 7 days of curing a very insignificant impact was observed in mortar specimens containing 2.5 % of natural zeolite. The ultrasonic pulse velocity in mortar was 4100 m·s⁻¹. A higher increase in ultrasonic pulse velocity was observed when cement was replaced with 7.5 % of mineral additive. The value of ultrasonic pulse velocity reached 4120 m·s⁻¹ showing an increase of 0.56 % The highest ultrasonic pulse velocity of 4151 m·s⁻¹ was registered in specimens containing 10 % of natural zeolite, and this meaning was 0.63 % higher compared to specimens without the mineral additive. Ultrasonic pulse velocity in mortar tend to decrease with higher amount of natural zeolite in the mix. Mortar specimens containing 12.5 % and 15 % of natural zeolite additive had ultrasonic pulse velocity values of 4119 m·s⁻¹ and $4115 \text{ m} \cdot \text{s}^{-1}$.



Figure 6. Dependence between the ultrasonic pulse velocity and natural zeolite amount in mortar.

Ultrasonic pulse velocity tests were also conducted with specimens after 28 days of curing. With the increase of mineral additive up to 5 % and 7.5 %, ultrasonic pulse velocity increased to 4258 m·s⁻¹ and 4267 m·s⁻¹ or 0.33 % and 0.54 % respectively compared to the control specimen. The ultrasonic pulse velocity value of 4270 m·s⁻¹ was registered in the specimen composition altered by 10 % of natural zeolite and this value was 0.61 % higher than in control mortar specimens. Ultrasonic pulse velocity in mortar tend to decrease with

higher amount of natural zeolite in the mix. Ultrasonic pulse velocity in mortar specimens containing 12.5 % and 15 % of natural zeolite decreased to 4265 m s⁻¹ and 4261 m s⁻¹.

After the amount of natural zeolite in mortar exceeded, density, ultrasonic pulse velocity, compressive and flexural strength decrease. It can be explained by the too low amount of cement (overdosing of mineral additives), which does not ensure a strong dependence between the aggregate and cement matrix.

Figure 7 illustrates the dependence between expansion and natural zeolite amount. After 14 days of testing, expansion in specimens containing 2.5% and 5% of natural zeolite were lower compared to the expansion of control specimens (0.076 %) and are 0.071 % and 0.069 % respectively. Expansion of altered composition mortar specimens with 7.5 % and 10 %, which are 0.062 % and 0.058 % respectively, also exceeded the limit of 0.054 %. When natural zeolite amount was increased to 12.5 %, the expansion did not exceed the permissible limit after 14 days and was equal to 0.049 %. The average expansion limit in specimens with the maximum amount of zeolite of 15 % was 0.043 %.



Figure 7. Dependence between the expansion of altered composition mortar conditioned in 1 N NaOH solution of 80 $^{\circ}$ C for 56 days and the amount of natural zeolite.

Expansion tests of specimens after 56 days revealed the increase of expansion in all specimens with natural zeolite. After 56 days of testing the expansion in specimens containing 2.5 % of natural zeolite was 0.109 %. Expansion decreased to 0.107 % and 0.103 % in specimens containing 5 % and 7.5 % mineral additive respectively. The expansion slightly exceeds 0.1 % limit and reaches 0.101 % when 10 % of natural zeolite added to the mix. The expansion of altered composition specimens with 12.5 % and 15 % of mineral additive decrease to 0.096 % and 0.091 % respectively after conditioning the specimens in 1N NaOH solution of 80 °C for 56 days.

The expansion of altered composition specimens with 12.5 % of natural zeolite after 14 and 56 days

conditioning in 1 N NaOH solution of 80 $^{\circ}$ C did not exceed the expansion limits of 0.054 % and 0.1 %.

To estimation of the impact of natural zeolite on alkali-silica reaction resistance of mortar X-ray diffraction evaluation was done. The mineral structure of control hardened sample and altered composition sample with 12.5 % and 15 % amounts of natural zeolite was investigated.

Figure 8 represents the structure of control specimen. Figure 9 represents the structure of control sample. It was examined using SEM (scanning electron microscope).

Product of alkali silica reaction – an alkaline gel was found around the aggregate particles. Cracks in the cement matrix and aggregates were caused by the expansion of alkaline gel. All of these damages in the microstructure of the mortar reduce durability, physical and mechanical properties.

Figure 10 represents the mineral structure of mortar specimen containing the limit amount of natural zeolite of 12.5 % exposed to alkaline environment. The intensity of X-ray image is much higher compared to the X-ray image of mortar specimen without natural zeolite. Like



Figure 8. XRD spectrum of mortar without natural zeolite conditioned in 1 M NaOH solution of 80 °C for 56 days: Q – quartz (SiO₂), C – calcite (CaCO₃), A – anorthite (CaAl₂Si₂O₈), M – microcline (K(AlSi₃)O₈).

in mortar without natural zeolite, quartz (SiO_2) is the prevailing element.

Figure 11 represents the microstructure of altered composition mortar with 12.5 % of natural zeolite conditioned in 1 M NaOH solution of 80 °C temperature for 56 days.

By replacing part of the cement with natural zeolite, the contact area between the aggregate and cement matrix is much stronger, with verv few cracks. The alkaline



Figure 9. SEM images of mortar without natural zeolite conditioned in 1 M NaOH solution of 80 °C for 56 days.



Figure 10. XRD spectrum of altered composition mortar with 12.5 % of natural zeolite conditioned in 1 M NaOH solution of 80 °C for 56 days: Q – quartz (SiO₂), C – calcite (CaCO₃), A – anorthite (CaAl₂Si₂O₈), M – microcline (K(AlSi₃)O₈), V – vesuvianite (Ca₁₀MgFeAl₄Si₉O₃₄(OH)₄).

gel accumulates, but the structure of the gel is less massive than in mortar without natural zeolite.

Tests with altered composition mortar specimens with 15 % of natural zeolite were done to evaluate the impact of the maximum amount of additive. Figure 12 represents the results of XRD analysis.

Figure 13 represents SEM images of sample with 15 % of natural zeolite and conditioning the sample for 56 days in 1M NaOH solution of 80 °C. The contact area in the aggregate and cement matrix is sturdy in all points of altered composition mortar with 15 % of natural zeolite, no major damage is visible.

XRD microstructure analysis and quantitative analysis of mineral composition revealed that the amount of quartz decreases with the increase of natural zeolite amount. Quartz is the main source of silica in the mix leading to deteriorating ASR through reaction with alkali present in the cement. Mineral composition of mortar specimens, in which cement was altered by 2.5 %, 5 %, 7.5 %, 10 %, 12.5 % and 15 % of natural zeolite, was tested to determine the impact of natural zeolite in the decrease of quartz in mortar mix. The results of the tests presented in Figure 14.

The test results showed that higher amount of natural zeolite in mortar mix leads of the change of mineral composition of the specimens: the amount of quartz and calcite decreases, the amount of microcline increases and the amount of anorthite remains similar. It means that the amount of pure SiO_2 in mortar mix can be reduced by increasing the amount of natural zeolite because during cement hydration and other chemical reactions silica transforms into microcline (K(AlSi₃)O₈, the amount of which increases. This mineral has much stronger chemical bonds than quartz (SiO₂) and thus the chemical reaction between silica and Na and K alkali is localised. Subsequently, the amount of ASR product alkaline gel reduces and there is less expansion of mortar.



Figure 12. XRD spectrum of altered composition mortar with 15 % of natural zeolite conditioned in 1 M NaOH solution of 80 °C for 56 days: Q – quartz (SiO₂), C – calcite (CaCO₃), A – anorthite (CaAl₂Si₂O₈), M – microcline (K(AlSi₃)O₈), V – vesuvianite (Ca₁₀MgFeAl₄Si₉O₃₄(OH)₄).



Figure 11. SEM images of altered composition mortar with 12.5 % natural zeolite conditioning the sample for 56 days in 1M NaOH solution of 80 °C.



Figure 13. SEM images of altered composition mortar with 15 % natural zeolite conditioning the sample for 56 days in 1 M NaOH solution of 80 °C.



Figure 14. Dependence between the mineral composition of altered composition specimens, expansion and natural zeolite amount in the mix after conditioning the specimens for 56 days in 1 M NaOH solution of 80 °C.

CONCLUSIONS

Images obtained by the scanning electron microscope (SEM) revealed that natural zeolite particles have irregular shape and their size is approx 11 μ m in average. X-ray diffraction microscopy analysis revealed that the prevailing element in natural zeolite is heulandite (Na_{2.6}Mg_{0.36}Al_{3.32}Si_{14.68}O₃₆(H₂O)_{10.32})H making up to 92.2 %. X-ray image also showed the presence of muscovite ((K, Na)(Al, Mg, Fe)₂(Si_{3.1} Al_{0.9})O₁₀(OH)₂)M and anorthite (CaAl₂Si₂O₈) A representing 0.7 and 7.1 % respectively in natural zeolite.

Studies have shown that the use of 15 % natural zeolite in mortar increased the compressive strength to 52.56 MPa after 7 days of hardened and to 55.59 MPa after 28 days of hardened. The highest compressive strength was achieved by replacing the cement with 12.5 % natural zeolite.

Research revealed that mechanical and physical characteristics of mortar mix improve with higher amount of natural zeolite (12.5 %), however, the appropriate

zeolite amount must be selected because overdosing leads to deterioration of mortar mix properties.

The use of 15 % natural zeolite in mortar reduces ASR induced expansion. ASR caused expansion of mortar depends on natural zeolite amount in mortar mix. The highest expansion (0.113) of mortar specimens was achieved without the use of natural zeolite. The opposite effect obtained with the use of 15 % natural zeolite - the expansion reached 0.043.

It means that the amount of pure SiO_2 in mortar mix can be reduced by increasing the amount of natural zeolite because during cement hydration and other chemical reactions silica transforms into microcline (K (AlSi₃)O₈, the amount of which increases. This mineral has much stronger chemical bonds than quartz (SiO₂) and thus the chemical reaction between silica and Na and K alkali is localised. Therefore, the amount of alkali silica reaction product – alkaline gel – reduces and lower expansion of mortar is observed.

The tests have shown that altered composition mortar with natural zeolite has better physical and mechanical properties, is more resistant to ASR and can be used in construction works.

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