



METHODS OF MITIGATING ALKALI REACTIVITY OF GRAVEL AGGREGATE

SPOSOBY OGRANICZENIA REAKTYWNOŚCI KRUSZYWA ŻWIROWEGO

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Abstract

Effectiveness of selected chemical admixtures and mineral additives to mitigate alkali-silica reaction was compared based on reactive gravel aggregate. Lithium compounds in the form of nitrate and lithium polysilicate were used as chemical admixtures. Natural pozzolans containing zeolite were used as mineral additive. Efficiency of the additive was enhanced by modification with ammonium ions. Linear changes of mortars with crushed gravel aggregates were studied with the accelerated and long-term methods. Additionally, scanning electron microscopy was used for microstructural observations. It was demonstrated that at elevated temperatures the application of lithium compounds provided better protection. Under conditions similar to those in the field, 20-30% of natural pozzolans proved to be more effective in inhibiting the expansion. Regardless of the method of protection applied, the presence of alkali-silica reaction products was detected in the microstructure of the mortars.

Keywords: alkali-silica reaction, lithium compounds, natural pozzolana, zeolite, expansion, reaction inhibition

Streszczenie

Na przykładzie reaktywnego kruszywa żwirowego porównano efektywność ograniczenia reakcji alkalia-kruszywo przy pomocy wybranych domieszek chemicznych i dodatków mineralnych. Jako domieszki chemiczne zastosowano związki litu w postaci azotanu i polikrzemianu litu. W przypadku dodatków mineralnych zastosowano naturalną pucolanę, zawierającą zeolit, której efektywność zwiększono poprzez modyfikację jonami amonowymi. Wykonano badania zmian liniowych zapraw z rozdrobnionym kruszywem żwirowym metodą przyspieszoną i długoterminową. Dodatkowo wykonano obserwacje mikrostruktury z wykorzystaniem elektronowego mikroskopu skaningowego. Wykazano, że w warunkach podwyższonej temperatury lepsze zabezpieczenie uzyskano po zastosowaniu związków litu. W warunkach zbliżonych do eksploatacyjnych zastosowanie 20-30% pucolany naturalnej skuteczniej hamowało ekspansję zapraw z kruszywem reaktywnym. Niezależnie od sposobu zabezpieczenia, w mikrostrukturze zapraw wykryto obecność produktów reakcji alkalia-krzemionka.

Słowa kluczowe: reakcja alkalia-krzemionka, związki litu, pucolana naturalna, zeolit, ekspansja, inhibicja reakcji

1 INTRODUCTION

The alkali-silica reaction causes significant swelling and deterioration of various elements of concrete infrastructure, such as dams, sidewalks, bridges, walls,

barriers and structural elements of nuclear power plants [1, 2] and represents a significant problem in the context of the durability of concrete all over the world. The reaction begins with the dissolution of

reactive silica due to the high pH value of the pore solution, which subsequently leads to the creation of alkali-silica gel. The absorption of more calcium ions causes gelation and/or crystallization, which leads to the creation of amorphous and/or crystalline products of the alkali-silica reaction. The products of the reaction can differ greatly, depending on the composition of the pore solution, the stage of reaction and the temperature [3, 4, 5]. The exact stages and mechanisms of expansion due to the alkali-silica reaction have not been fully identified and explained yet. It is generally assumed that expansion caused by the alkali-silica reaction is connected with the swelling of products in aqueous environments, because more damage has been observed in the presence of high relative humidity [6, 7]. However, recently published papers [3, 8] have revealed that neither amorphous, nor crystalline products of the alkali-silica reaction that contain Na, K, Ca and silica, experience swelling because of the presence of water. The absorption of water by the products of the alkali-silica reaction was lower than in the case of the C-S-H phase, which would suggest that expansion is not caused by swelling, but rather by some other mechanisms, which would suggest that more research is urgently needed.

The suppression of the alkali-silica reaction in existing structures is difficult and costly and may require the application of water insulation layers. The alkali-silica reaction may be allowed to continue, however, because the amount of water present in concrete may be sufficient for its continuation even in insulated concrete structures. In new structures, the use of non-reactive aggregate is a relatively cheap and effective method of avoiding the alkali-silica reaction. However, local aggregates are often used for environmental and economic reasons, and in some distant locations, especially in the case of massive structures such as dams, they may represent the only choice. The effective mitigation (limitation) of the alkali-silica reaction effects is of key importance in the context of the safe use of reactive aggregates in the production of durable concrete.

The use of mineral cement additives and chemical admixtures, such as lithium salts, in order to prevent concrete expansion resulting from the alkali-silica reaction, was first reported over 80 years ago, soon after the discovery of this effect in the 1940s. In the case of the production of concrete elements, an effective way to minimise the risk of their damage as a result of the alkali-silica reaction is to use cement with mineral additives (containing main components other than Portland clinker), in order to reduce its pH

and the concentration of sodium and potassium ions in the concrete's pore solution. Low soluble sodium and potassium content and a low pH value reduce the risk of the generation of alkali-silica reaction (ASR) products [5], reducing also the speed of reaction by inhibiting the dissolution of reactive phases of the silica [9]. Mineral additives in the form of waste products from other manufacturing processes, such as fly ash or blast-furnace slag, are sometimes difficult to obtain, therefore additives obtained from natural sources, such as zeolite or metakaolin, are used.

Contrary to mineral additives, which can be introduced only during the production of concrete, lithium salt solutions can be applied directly onto a concrete structure that demonstrates the effects of an alkali-silica reaction, and can also be used as a chemical admixture during the production of concrete. Different types of lithium salts have been tested in the past in order to reduce the effects of the alkali-silica reaction [10-13]. Certain problems with the application of lithium salts have been encountered, because the dosage of different types of lithium salts varies greatly depending on several factors, such as the mineral structure of reactive aggregates and the content of alkali in concrete [14, 15]. Additionally, the operating mechanisms of different types of lithium salts are complicated and produce different types of alkali-silica reaction products that contain lithium and lithium silicates [16-18].

The understanding of the mechanisms that alleviate the effects of the alkali-silica reaction through the use of mineral additives as well as lithium compounds, is important in the context of the optimum use of these materials, as well as the assurance of their long-term effectiveness in the protection of concrete structures. This has posed a significant challenge for many decades, mainly due to problems with the specification of the products of alkali-silica reaction generated in small quantities and volumes using conventional laboratory techniques. It is also difficult to characterise Li containing products of the reaction, mainly due to their chemical composition.

This article presents the results of tests aiming to establish the effectiveness of reducing the effects of alkali-aggregate reaction in reactive gravel aggregate using selected chemical admixtures and mineral additives. Lithium compounds in the form of lithium nitrate and lithium polysilicate were used as chemical admixtures. As for mineral additives, natural pozzolana with zeolite content was used and its effectiveness was enhanced by modifying it with ammonium ions.

Linear changes for mortars with fine-crushed gravel aggregate were investigated using the accelerated and long-term method. Additionally, their microstructure was analysed using a scanning electron microscope.

2 MATERIALS AND METHODS

Samples needed for the tests were prepared using CEM I 42.5R Portland cement and gravel aggregate with confirmed reactivity [19, 20]. Lithium nitrate and lithium polysilicate, together with natural Transcarpathian zeolite, were used in order to reduce the degradation of mortar as a result of the alkali-silica reaction. Some of the zeolite was modified by replacing exchangeable cations with ammonium cations. Ion exchange was carried out by immersing natural zeolite in a 2M solution of NH_4Cl for 24 hours [21]. During this time, some of the sodium and potassium ions were replaced with ammonium ions, in accordance with the cation selectivity sequence for clinoptilolite [22, 23]. Chlorine was removed by rinsing with distilled water.

The chemical composition of cement and zeolite has been listed in Table 1. As a result of the ion exchange, the content of Na_2O and K_2O in modified zeolite was reduced to 0.46% and 1.41%, respectively. The $\text{SiO}_2/\text{Li}_2\text{O}$ molar module of the used lithium polysilicate was 3.5, the density was 1.190 g/cm^3 . The molar ratio of lithium to the total content of sodium and potassium in the cement was 0.74. The lithium compounds were added to the mix with the mixing water.

Because Polish cements have higher levels of potassium content, K_2SO_4 was used to raise the content of $\text{Na}_2\text{O}_{\text{eq}}$ in the cement to 1.1%.

6 mortar batches were made using the described materials and their chemical composition has been listed in Table 2. In the case of samples containing zeolite as an additive, their w/s ratio was increased from the normative 0.47 to 0.49, due to the high water demand of zeolite which resulted in the lower flowability of mortar.

Table 1. Chemical composition of cement and zeolite, % w/w

Materials	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	TiO_2	MnO	P_2O_5	LOI	Cz. n.r.
Cement	20.20	4.80	3.00	61.70	1.80	2.70	0.78	0.15	0.45	–	–	3.00	1.00
Zeolite	67.07	12.40	0.90	2.09	0.72	–	2.80	2.05	0.19	0.04	0.014	–	–

LOI – Loss on ignition, Cz.n.r. – non soluble residue

Table 2. Composition and indication of mortars

Indication of samples	Content [g]				[Li/Na+K]	w/s
	Cement	Sand	Zeolite	Modified zeolite		
CEM	525	1175	0	0	0	0.47
Z20	420	1175	105	0	0	0.49
Z30	367.5	1175	157.5	0	0	0.49
Zm20	420	1175	0	105	0	0.49
LiN*	525	1175	0	0	0.74	0.47
LiS**	525	1175	0	0	0.74	0.47

*LiN – samples with lithium nitrate **LiS – samples with lithium polysilicate

The main measurable effect of the alkali-aggregate reaction, both in concrete as well as in mortar, is expansion. In order to examine the efficiency of its reduction through the application of lithium compounds and the addition of zeolite, mortar bars measuring $25 \times 25 \times 250 \text{ mm}$ were prepared with the application of reactive gravel aggregate. They were then tested in order to establish the rate of expansion using the accelerated method (according to the methodology of standard ASTM C 1260) and using the

long-term method (according to the methodology of standard ASTM C 227) [24, 25]. Additionally, during the tests, a sample was taken from each mortar after 28 days in the case of samples tested in accordance with procedure described in standard ATM C1260, and after 360 days in the case of samples tested in accordance with procedure described in standard ATM C227, in order to examine their microstructure under a scanning electron microscope. Such tests were carried out on mortar fracture samples.

3 RESULTS

3.1. Results of accelerated tests according to the methodology of ASTM C1260

The results of linear changes in mortars tested in accordance with ASTM C1260, demonstrated in Figure 1, confirm that the used gravel aggregate contributes to the degradation of mortar as a result of the alkali-silica reaction. Simultaneously, all of the applied methods of mitigating this reaction have contributed to the reduction of expansion. Lithium compounds were the most effective in this regard. All of them had a similar effect that limited the expansion to no more than 0.025%. After 12 days in the case of lithium nitrate, and after 16 days in the case of lithium polysilicate, the expansion of the samples was stabilised.

In the case of all other mortars, the stabilisation of the volume of their samples was not observed during the entire 30-day testing period. Mortars with different kinds of zeolite additives demonstrated significant differences in terms of the rate of expansion. The most effective use of zeolite in order to reduce expansion entailed the replacement of 30% of cement in the mortar with zeolite. Such addition of zeolite reduced the expansion of mortar during the normative 16-day testing period to a level of less than 0.1%. This allowed us to conclude that mortar was not subject to the negative effects of an alkali-silica reaction. Other 20% additives of natural and modified zeolite only facilitated the reduction of expansion to a level of less than 0.2% during this period. This would allow us to classify such mortars as potentially exposed to the negative effects of an alkali-silica reaction.

On this basis we can also observe the effectiveness of modification of zeolite with ammonium ions, which improve the efficiency of zeolite as an expansion-inhibiting factor in the context of the alkali-silica reaction. Modified zeolite reduces expansion by more than 30% in comparison with non-modified zeolite used in the same quantity.

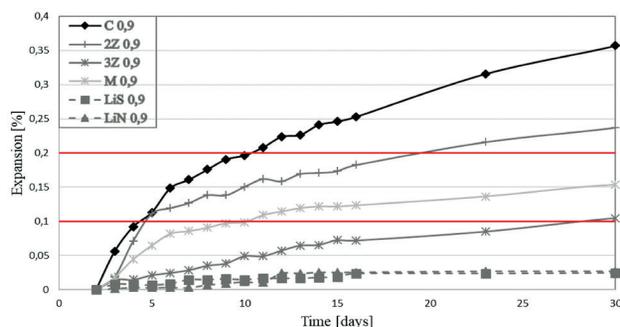


Fig. 1. The expansion of mortars during testing in accordance with ASTM C1260

3.2. Results of long-term tests according to the methodology of ASTM C227

Figure 2 shows the results of long-term testing of expansion. These results allow us to conclude that all implemented methods of the reduction of expansion caused by the alkali-silica reaction gave satisfactory results. Whether lithium compound additives were used, or mineral additives in the form of natural and modified zeolite were applied, the expansion of mortars never exceeded the 0.04% threshold. In the course of these tests, unlike during the accelerated tests, zeolite additives proved to be more effective than lithium compound admixtures in limiting the expansion of mortars. A 30% natural zeolite additive and a 20% modified zeolite additive proved to be the most effective in inhibiting the expansion of mortars. In both cases, the shrinkage of samples was observed instead of their expansion. This test allows us to conclude that by modifying zeolite by way of an ion exchange, it is possible to reduce its quantity in the binder, which can also contribute to the improvement of its consistency [26-28].

The 20% natural zeolite additive proved to be less effective. In this case, no significant changes in the measurements of the tested samples were observed. Lithium compound-based chemical admixtures were also less effective. In both cases expansion was observed and its rate was similar to the one observed for lithium admixtures.

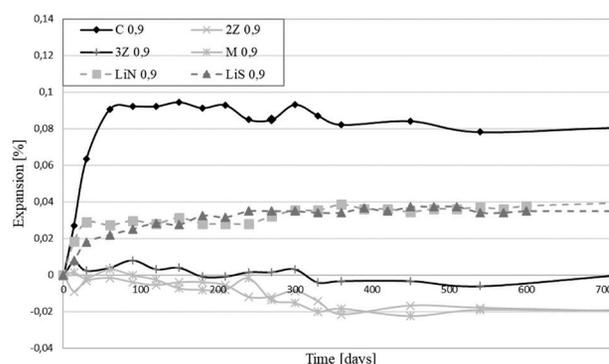


Fig. 2. The expansion of mortars during testing in accordance with ASTM C227

3.3. Microstructure analysis

Short-term research of the microstructure of all mortars containing zeolite has revealed the presence of damages that could be attributed to the alkali-silica reaction (Fig. 3). They demonstrated visible signs of local cracking that could be the result of the expansion of alkali silica gel. In the case of sample Zm20 (with a 20% additive of modified zeolite), the damage

mechanism shown in Figure 3c has been thoroughly detailed in the Czapik paper [20]. Apart from visible cracking, the alkali-silica reaction generates reaction products of variable swelling and stress-inducing characteristics. They can be observed on the enclosed drawings (Fig. 3a, 3b), inside air voids situated near areas of intense cracking. In the case of mortar with a lower natural zeolite content (Fig. 3a), they form agglomerates of compacted gel [29]. The fibrous character of the gel, which is typical for alkali-silica reaction products, is demonstrated by the sample with higher zeolite content (Fig. 3b). The reason for this could be that reaction products generated in smaller quantities have more space to fill up freely and will thus generate less stress in the mortar, which leads to lower expansion of the samples. Alkali-silica reaction products concentrated in voids are fractured, which can be considered as typical for these products [30].

The introduction of lithium nitrate into mortars effectively reduces the harmful reaction of aggregate with alkali, which is confirmed by the low number of fractures observed in the microstructure of the mortars. However, the presence of highly porous reaction

products on the surface of the grains (pt 1; Fig. 4a) and fine gels (pt 2; Fig. 4a) has been observed, which may be proof of the process of dissolution of silica and thus the reaction of alkali from the cement (Fig. 4a). The products of the reaction can also have a fibrous form, but this is limited mainly to the surface of the grains and can generate only low stresses (Fig. 4b). None of the analysed samples have revealed the presence of reaction products in the form of crystalline lithium silicate that could protect the grains of reactive silica against reaction with the alkali. However, it was not possible to definitely determine that reaction products observed on the surface of the aggregate grains do not contain any lithium ions. The limitations of the EDS technique did not allow for the detection of lithium compounds, which greatly restricted the analysis of test results.

Mortars containing lithium polysilicate demonstrated fractures of the aggregate grains. Figure 4c demonstrates the separation of carbonate envelopes of bioclasts filled with chalcedony, which has been indicated as one of the minerals responsible for the reactivity of the analysed polymineral aggregate, from fine grains [20, 31].

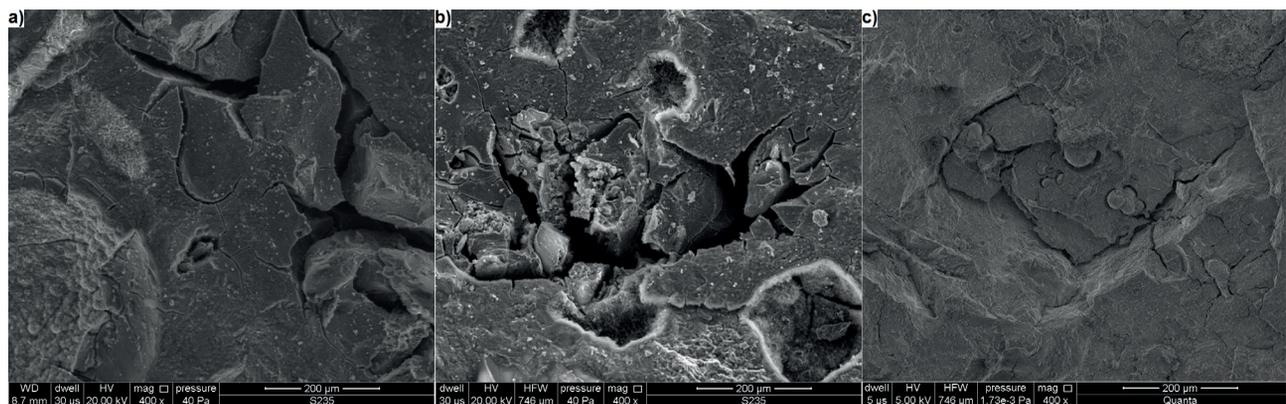


Fig. 3. The microstructure of mortars with zeolite after tests acc. to ASTM C 1260, a) Z20 (20% of zeolite), b) Z30 (30% of zeolite), c) Zm20 (20% of modified zeolite)

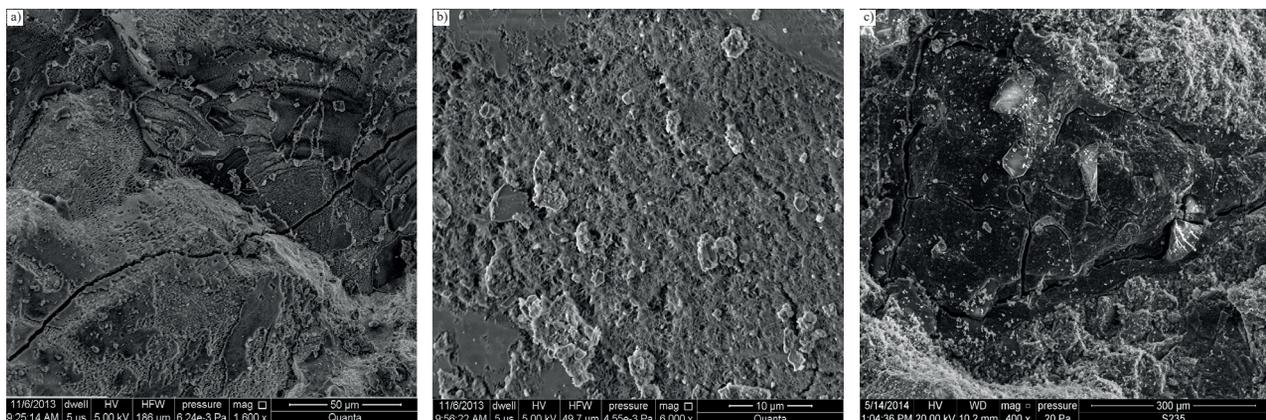


Fig. 4. The microstructure of mortars with lithium compounds after tests acc. to ASTM C1260, a-b) LiN, c) LiS

The microstructure of mortars with lithium nitrate, stored in conditions as defined by standard ASTM C227, indicates the presence of centres of corrosion, which has been confirmed by the appearance of microfractures and the presence of alkali silica gel in the area of reactive grains and partially in the cement paste. Figure 5a demonstrates the presence of sponge-like gel in the reactive grains (pt 1), next to the solid, amorphous gel (pt 2). The visible differences in appearance are a result of the texturisation of the gel with time, mainly because of the higher content of

calcium in the reaction products [32]. Mortars with lithium polysilicate demonstrate the presence of highly fractured gels resulting from the total reaction of aggregate grains (Fig. 5b).

Another characteristic feature was the presence of fractures caused by the reaction of only the reactive minerals in the non-reactive matrix, as visible in Figure 5c. The presence of reaction products that cause the fracturing of the grains and the matrix of the cement paste proves that harmful degradation of the gravel aggregate takes place in the presence of lithium compounds.

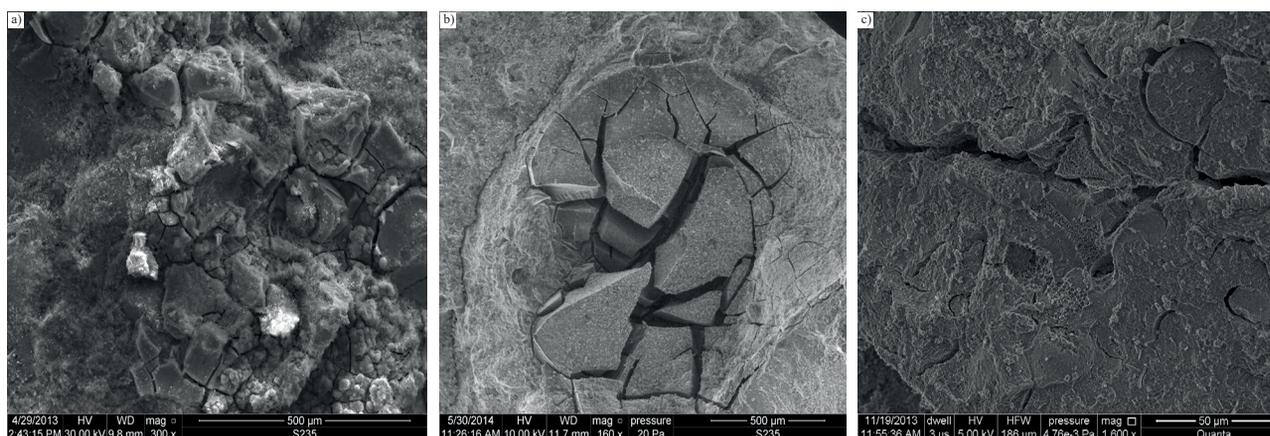


Fig. 5. The microstructure of mortars with lithium compounds after tests acc. to ASTM C227, a) LiN, b-c) LiS

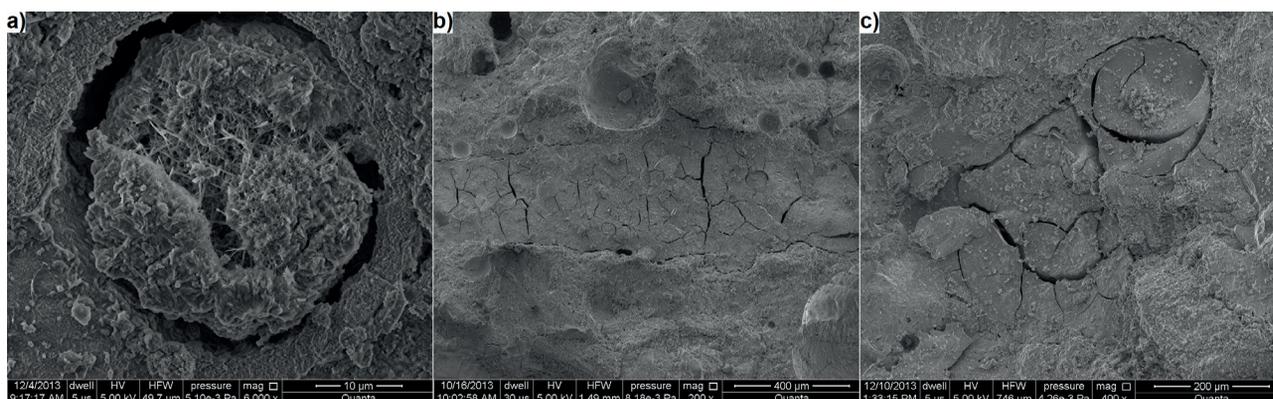


Fig. 6. The microstructure of mortars with zeolite after tests acc. to ASTM C 227, a) Z20, b) Z30, c) Zm20

The microstructures of all mortars with a zeolite additive tested using the long-term method demonstrated the presence of microstructural changes caused by the alkali-silica reaction, despite the fact that the effect of expansion was avoided (Fig. 6).

The presence of gel clusters (Fig. 6a), that may have formed in the place of fine reactive grains, has been observed. In this case, fibrous products of the alkali-silica reaction were present under the external envelope of compacted gel. In other locations, highly fractured aggregate grains of the size of more than 200 μm have been observed (Fig. 6b, 6c). Whereas

large aggregate grains can cause the degradation of samples in the course of the alkali-silica reaction, small grains may limit the alkali-silica reaction as a result of pozzolanic activity. The microstructure of the tested samples with modified zeolite also revealed the presence of highly fractured, compacted alkaline gel (Fig. 6c).

4. CONCLUSIONS

The results of the performed tests have revealed:

- differences in the effectiveness of the use of a zeolite additive or admixtures containing lithium

- compounds in the limitation of the effects of the alkali with gravel aggregate reaction, depending on the implemented testing method (short-term or long-term);
- in the case of the short-term method, lithium compounds – both lithium polysilicate as well as lithium nitrate – were the most effective in reducing the extension of the length of mortar bars. A zeolite additive was less effective in limiting mortar expansion, however the use of a 30% additive may be considered as an effective way of limiting the effects of alkali-silica reaction. A 20% additive of non-modified and modified zeolite has also visibly reduced expansion, but it still exceeded the first threshold value (0.1%);
 - in the case of the long-term method, a zeolite additive, irrespective of its content and modification, has proved to be a fully effective way of limiting expansion caused by the alkali-silica reaction. In the case of lithium-containing compounds, expansion was limited to a level considered in the ASTM C227 standard as safe;
 - irrespective of the type of lithium compound, the results of expansion tests using the short- and long-term methods were similar;
 - in the case of samples containing zeolite, the results of expansion achieved using the short- and long-term methods were different;
 - the analysis of the microstructure of samples, both in the case of zeolite as well as lithium-containing additives, has revealed the presence of the products of the alkali-silica reaction, but these products did not cause any extensive expansion of the mortar samples;
 - in the context of the conditions of long-term tests, which more closely resemble real operating conditions, the application of a zeolite additive can be considered as a more effective way of limiting the alkali-silica reaction than lithium salt-based admixtures. However, the explanation of the observed differences in the effectiveness of these materials, tested using different methods, still requires further research.

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