WARSAW UNIVERSITY OF TECHNOLOGY Index 351733
FACULTY OF CIVIL ENGINEERING
COMMITTEE FOR CIVIL AND WATER ENGINEERING
POLISH ACADEMY OF SCIENCES ISSN 1230-2945

DOI: 10.24425/ace.2025.156191

ARCHIVES OF CIVIL ENGINEERING

Vol. LXXI ISSUE 4 2025

pp. 607 – 617

Research paper

Possibility of assessing cements with non-clinker constituents in terms of alkali reactivity with silica aggregate

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Abstract: This paper presents the results of a study investigating the influence of non-clinker constituents in cement on the risk of deleterious alkali-silica reaction (ASR). The cements selected for this study were CEM II types containing commonly used non-clinker constituents, such as ground granulated blast furnace slag (GGBS) and silica fly ash. Additionally, a binder was prepared by mixing CEM I cement with metahalloysite in a 3:1 ratio. As a reference, CEM I cements without non-clinker constituents were used. Expansion tests were conducted on mortars using standard reactive aggregate (borosilicate glass cullet) according to ASTM C 441. The results, evaluated against the criteria outlined in ASTM C 1157, demonstrated that the binders with non-clinker constituents met the "Option-R" requirement. The expansion of, mortars made with cements containing non-clinker constituents was significantly reduced compared to mortars made with pure Portland cement. The most significant effectiveness in this regard was achieved with the metahalloysite cement. The obtained expansion results correlated well with the microstructural changes in the mortars, which can be linked to the occurrence of ASR. The microstructure of the mortars was examined using scanning electron microscopy.

Keywords: alkali-silica reaction, borosilicate glass aggregate, cement, mineral additive

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1. Introduction

Given the widespread use of concrete as a construction material, durability is its crucial property and has been the subject of numerous research publications. Numerous factors influence concrete's durability, including its susceptibility to alkali-silica reaction (ASR), a form of internal concrete corrosion [1]. Improper selection of concrete constituents can lead to damage caused by the reaction of alkalis with aggregates. One approach to prevent ASR is to avoid using reactive aggregates. Consequently, standardized testing methods for this phenomenon typically evaluate aggregate reactivity by measuring the dimensional changes of mortar or concrete specimens prepared with the aggregates. These tests form the basis for determining aggregate reactivity according to ASTM standards [2, 3], PN-B 06714-46 [4], RILEM recommendations [5], and GDDKiA guidelines [6, 7]. The primary drawback of these methods is the extended duration required for their completion. Accelerated 14-day mortar tests are also less reliable than long-term concrete tests, which typically last at least six months [5, 8, 9]. Additionally, certain aggregates may exhibit delayed reactivity with alkalis, posing a risk of misclassification as non-reactive aggregates [9, 10].

When designing concrete composition, it is crucial to consider that the alkali-silica reaction (ASR) requires not only reactive aggregates but also the presence of alkalis, primarily derived from cement [11, 12]. Therefore, employing an appropriate cement can prevent this reaction from occurring, even when reactive aggregates are used.

Polish standards [13] specifically define categories of low alkali-active cement (NA) for such applications. For Portland cement (CEM I), the alkali content that prevents ASR degradation should not exceed 0.6% [1, 14], corresponding to 0.3–0.47% active alkalis [15]. Even stricter alkali content limitations are imposed when preventing alkali-carbonate reaction (ACR). In this case, the alkali content in CEM I cement should not exceed 0.4% [1].

Cement testing for ASR susceptibility focuses on alkali content without considering its impact on the reaction process. Hence, these tests are indirect measures of concrete's ASR vulnerability. Not all alkalis trigger ASR; only active alkalis in the concrete's pore solution are responsible [11,12]. Alkali content is typically reported as the cumulative sodium and potassium oxide content expressed as sodium oxide equivalent (Na_2O_{eq}), which is a simplification [11]. Literature [14] has also raised concerns about the effectiveness of this method in preventing ASR.

American standards offer a distinct approach to testing cement for its non-alkali-silica reaction (ASR) properties. ASTM C 1157 [16, 17] classification includes an "Option R" designation for cements with low reactivity to alkali-silica reactive aggregates. This designation can be given to cements that have undergone testing according to ASTM C441 [18], which is a modification of ASTM C 227 [19], where the expansion of mortar bars at 14 and 56 days does not exceed the established limits. The test procedure [16, 18] involves replacing the aggregate with cement as the test specimen. It entails substituting the reference material, a standard cement (with the required alkali content), with a specified reactive aggregate in the form of crushed borosilicate glass. The rationale behind this approach is to enable the evaluation of the potential of any cement' to induce ASR, not just in relation to the specific reactive aggregate currently being tested. This is crucial considering the variability in aggregate reactivity due to the form of reactive silica [20] and morphological differences [21].

Despite some concerns regarding the use of borosilicate glass aggregate [14, 17, 22], it is used due to the high reactivity of its amorphous silica [19, 23, 24]. An advantage of borosilicate glass aggregate over natural reactive aggregate is the relative stability of its properties.

When analyzing the requirements for low-alkali cement (NA), it is observed that non-clinker constituents can contain a higher alkali content. This is partly because the alkalis introduced with non-clinker constituents may exhibit lower solubility, limiting their potential to participate in the alkali-silica reaction compared to alkalis present in Portland cement clinker [11, 12, 14, 15]. Active pozzolanic and/or hydraulic non-clinker cementitious components, which react with calcium hydroxide to form additional C-S-H or C-A-S-H phases [25, 26], contribute to pore filling and reduced concrete permeability, thereby limiting the effective diffusion of sodium and potassium ions. This is an additional factor in preventing the alkali-silica reaction [11, 27]. The C-S-H and C-A-S-H phases formed can also bind sodium and potassium ions from the surrounding pore fluid to their structure, further reducing their concentration. Thus, the use of active non-clinker components in cement is a popular and effective method for preventing the alkali-silica reaction. The effectiveness of ASR prevention using active components depends on their type and typically increases with their content in the cement [28].

The impact of mineral additive cement on alkali-silica reactions (ASR) is evaluated according to ASTM standards in a manner analogous to the "Options R" classification for cement, using Pyrex glass as a reference reactive aggregate [17]. The test method outlined in ASTM C 441 [18] involves determining the effectiveness of a pozzolanic or slag cement admixture in preventing excessive expansion caused by the alkali-aggregate reaction. The evaluation focuses on the expansion of mortar made with Portland cement, pozzolanic or hydraulic additives, and reactive borosilicate glass aggregate when stored under specific curing conditions.

The literature does not contain any studies on the influence of binder components on expansion caused by the alkali-silica reaction with the use of borosilicate glass for cements produced according to European standards.

This article explores the progression of the alkali-silica reaction (ASR) in multi-component cements in the presence of borosilicate glass aggregate. Mortar specimens with borosilicate aggregate were prepared using CEM II/A or B multi-component Portland cement containing non-clinker main constituents, i.e., granulated blast furnace slag (S) and silica fly ash (V). As a reference binder, CEM I Portland cement (derived from the same Portland clinker as the multi-component cements) was employed. Additionally, mortar specimens were prepared with CEM I Portland cement and metahalloysite, and the results were compared to those obtained for control mortars made with this cement.

2. Materials and methods

2.1. Materials

The tests were carried out using five binders, applying Portland cement CEM I (C1) and CEM I with the addition of 25% metahalloysite (binder M). Metahalloysite [29] was prepared by dehydroxylating halloysite at 750°C for two hours. Halloysite is an aluminosilicate clay mineral from the kaolinite group, occurring in Poland in one of the largest deposits in the world, the Dunino deposit near Legnica. Portland cement CEM I (C2) was also used, as well as cements based on its clinker with the addition of blast furnace slag CEM II/B-S 42.5 R-NA (binder S) and fly ash CEM II/A-V 42.5R-NA (binder V). The oxide compositions of the binders determined by the XRF method are presented in Table 1.

Table 1. Chemical composition of cements

Cement type	**Component, weight percent [%]**								
	SiO_2	**Al_2O_3**	**Fe_2O_3**	**CaO**	**MgO**	**TiO_2**	**SO_3**	**Na_2O_{eq}**	**LOI**
C1	20.0	5.0	2.9	63.2	1.8	0.3	2.6	0.7	3.2
C2	19.8	4.9	2.7	63.3	2.3	0.3	2.6	0.7	3.3
V	24.4	8.2	3.2	53.6	2.3	0.4	2.9	0.9	3.6
S	24.7	5.7	2.0	59.0	3.0	0.3	3.0	0.7	1.5
M	21.8	9.1	10.9	47.7	2.9	2.1	2.0	0.6	2.8

Both Portland composite cements comply with the requirements for low-alkali binders, with alkali contents in accordance with the PN-B 19707:2023 [13] standard amounting to 0.8% for CEM II/B-S and 1.2% for CEM II/A-V, respectively. Based on current knowledge, such reduced alkali levels are expected to decrease the potential for alkali–aggregate reaction (AAR) [11, 12]. The partial replacement of clinker with other main constituents may also contribute to a pH reduction of the concrete pore solution, thereby further limiting the progress of the alkali–silica reaction (ASR). The siliceous fly ash content in the CEM II/A-V Portland fly ash cement used was 12.79%, while the ground granulated blast furnace slag content in the CEM II/B-S Portland slag cement was 27.53%.

Figure 1 presents the diffractogram obtained for metahalloysite produced by calcining halloysite at 750°C for two hours.

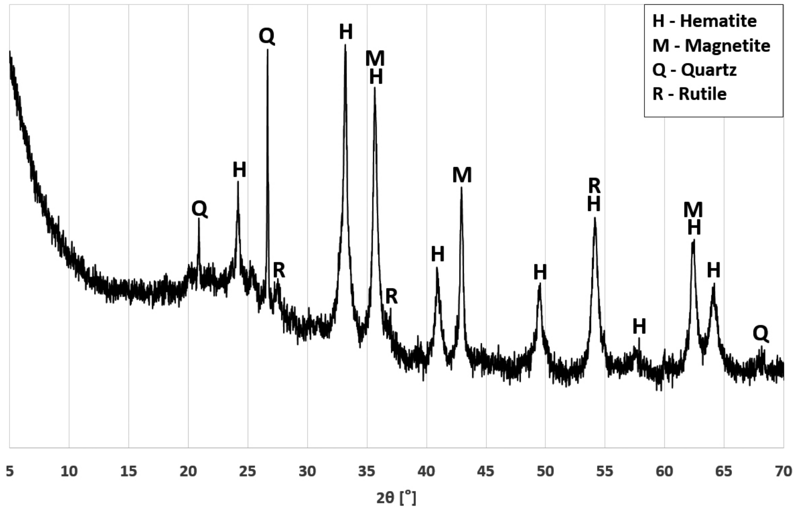


Fig. 1. XRD patterns of metahalloysite

The main constituents are quartz, as well as iron compounds (hematite and magnetite) and titanium (rutile), which distinguish it from the composition of other metahalloysites [30]. The observed background elevation, especially in the $2\ \theta$ range from 15°to 30°, combined with the low relative intensity of the peaks, may indicate the presence of a glassy phase. Metahalloysite tested in accordance with PN-EN 450-1 showed pozzolanic activity indices of 109% after 28 days and 108% after 90 days.

Borosilicate glass crushed to the gradation recommended in ASTM C 227 [19] was used as the aggregate.

2.2. Methods

The mortar expansion tests were conducted according to ASTM C441 [18]. This standard evaluates the effectiveness of mineral admixtures in preventing ASR-related expansion. Mortar bars were prepared with dimensions of 25×25×250 mm. The dry mortar ingredients were weighed in a 1:2.25 cement-to-aggregate ratio, and the water-to-binder (w/b) ratio was 0.47. For the first 24 hours after molding, the mortar specimens were stored at 20°C over water, and then demolded and cured in a climate chamber at 38°C and > 95% relative humidity. Linear expansion measurements of the specimens were performed using a Graff–Kaufman apparatus after demolding and at 14, 28, and 56 days. The mortar bar expansion results were evaluated against the criteria specified in ASTM C1157-08a [16]. After completing the expansion tests, ground-sections were prepared from the mortar bars and their microstructure was analyzed using a scanning electron microscope FEI Quanta FEG 250.

3. Results and discussion

The results of the linear change tests of mortars with various cement types and borosilicate glass aggregate are presented in Figure 2. The red dashed lines represent the maximum expansion according to the "Option-R" requirements contained in the standard [16].

The results of the linear change tests showed that mortar bars with CEM I cements (C1 and C2) exhibited very rapid expansion growth, which exceeded 0.02% after 3 days (allowable expansion limit at 14 days) and reached 0.06% after 10 days (maximum expansion at 56 days). It should be noted that the tested cements contained an average alkali content of 0.7% Na_2O_{eq}. The expansion of mortars with CEM I after 56 days is 0.156% and 0.146% for C1 and C2, respectively, which is a significant elongation, greater than often observed in the case of mortars with natural reactive aggregate [31–34]. Expansion of mortars with borosilicate glass and CEM I Portland cements occurs primarily during the first 14 days of sample curing, but does not stabilize until 56 days.

The mortar with C1 cement and metahalloysite (cement M) showed shrinkage during the tests, although the mortar with reference cement C1 showed the greatest expansion at all test dates.

The expansion of mortars with cements (V, S) was significantly lower compared to the reference mortar (C2). Initially, the expansion of mortar with blast furnace slag (S) was lower than that with silica fly ash (V). However, at later stages, the expansion rate with cement S was

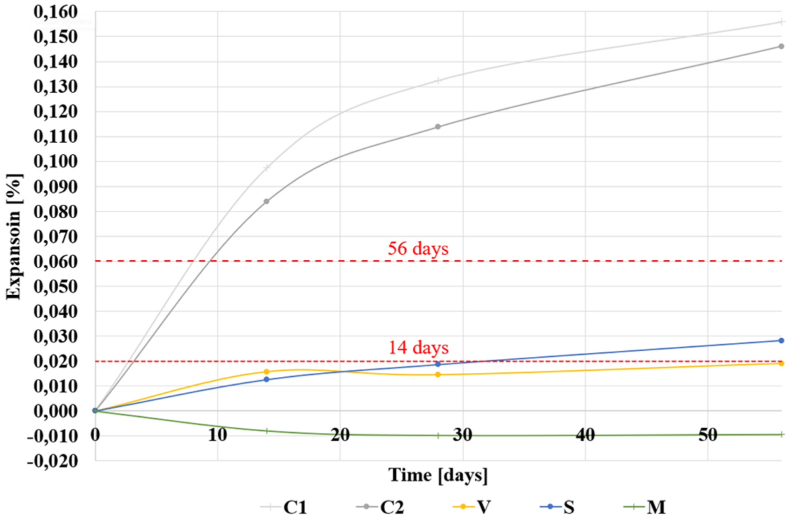
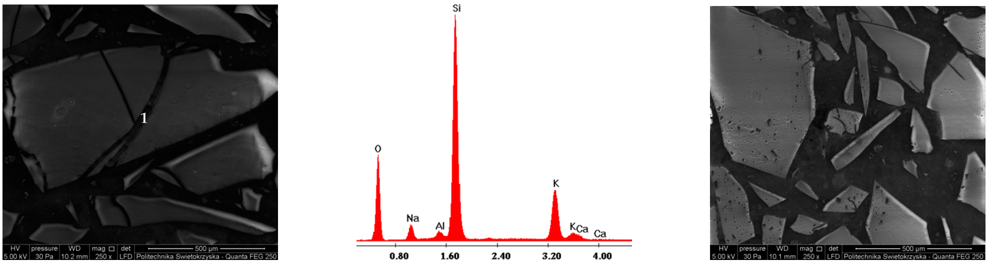


Fig. 2. Expansion of mortar bars with reference cements (C1 and C2) and CEM II cements (V, S, M) and borosilicate glass aggregate

slightly higher than that with cement V. Both cements meet the requirements of the standard [16] as the expansion after 14 days did not exceed the limit of 0.02% and 0.06% after 56 days.

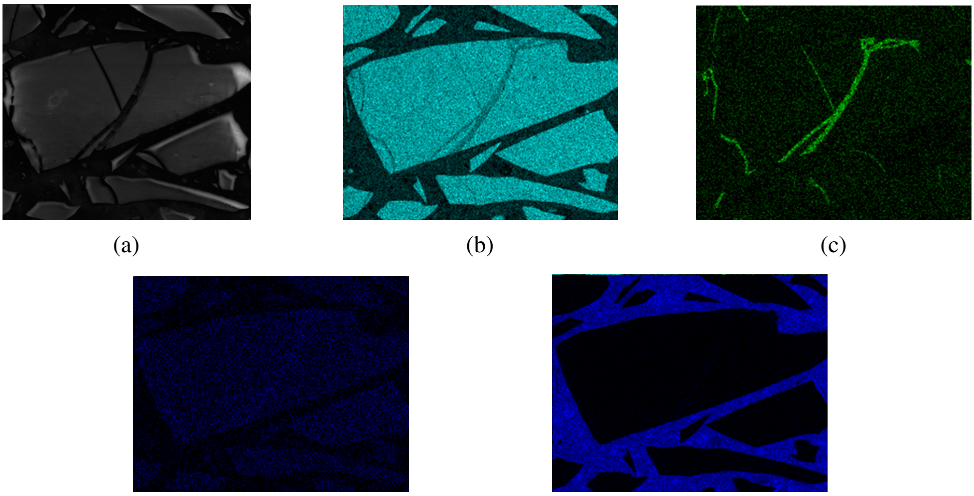
The expansion test results for mortar bars with borosilicate glass aggregate and multi-component Portland cements CEM II/A,B demonstrate that the use of active non-clinker main constituents (silica fly ash V, granulated blast furnace slag S, metahalloysite) effectively limits expansion below the limit values specified in the standard.

Figures 3 to 6 present the microstructure of mortars with reference cements, multi-component cements CEM II/A, B and after 56 days of testing according to ASTM C441 [18].



(a) (b) (c)

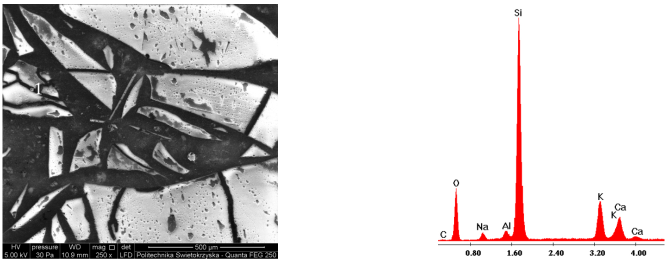
Fig. 3. Comparison of: (a) the microstructure of mortar made with CEM I cement (C1), along with (b) the microanalysis of alkali–silica reaction products at point 1 to (c) the microstructure of mortar made with cement containing metahalloysite (M)



(a) (b) (c)

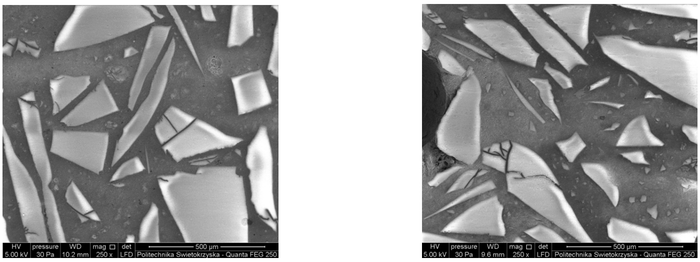
(d) (e)

Fig. 4. Comparison of: (a) the microstructure of mortars from CEM I (C1) cement and the distribution of (b) silicon, (c) potassium, (d) sodium, and (e) calcium



(a) (b)

Fig. 5. Microstructure of mortars: (a) with CEM I cement (C2), (b) analysis of alkali-silica reaction products at point 1



(a) (b)

Fig. 6. Microstructure of mortars: (a) with the fly ash (V) and (b) with granulated blast furnace slag (S)

All figures show sharp-edged borosilicate glass particles embedded in the cement paste matrix. The paste adheres well to the glass, and the interfacial transition zone is not clearly visible. Unreacted and partially reacted cement constituents, Portland clinker grains, fly ash (V mortar), granulated blast furnace slag (S mortar), and metahalloysite (M mortar) are observed in the cement matrix of each mortar.

As a result of the alkali-silica reaction, damage to the borosilicate glass aggregate grains is observed. The cracks that occur cut through the aggregate grains or are concentrated at their surface. They are often filled with compacted and also cracked products of this reaction [35]. The aggregate grains in mortars with CEM I cements are the most damaged. The cracks that occur in the aggregate most often have a width exceeding 30 μm, and in the cement paste matrix up to 0.5 μm. As evident from the elemental distribution mapping (Fig. 4) and the elemental composition analysis at selected points within the cracks (Figs. 3b and 5b), the crack-filling material exhibits a significantly elevated potassium content. Potassium is the primary alkali element present in cement, and therefore, its presence is primarily responsible for the occurrence of the alkali-silica reaction. Additionally, a clear signal from silicon, originating from the glass, is detected within the cracks. However, compared to pure glass, these signals are notably weaker. The sodium signals within the large cracks in Fig. 4d are slightly elevated, but this is not a distinctive feature. In other cracks, no significant differences in sodium content were observed relative to the surrounding (glass grains), and sometimes a minimal decrease was noted. Furthermore, no elevated calcium content was detected within the cracks, indicating the calcification of alkali-silica reaction products [36].

Based on the findings above, the material filling the cracks can be identified as potassium silicate gel.

In mortars with S and V cements (Fig. 6), damaged aggregate grains are also visible, but they are smaller, up to 6.5 μm wide, and less often filled with alkali-silica reaction products. On some aggregate grains in the mortar with slag, needle-like products appear. In the mortar with the binder containing metahalloysite (binder M) (Fig. 3c), cracked aggregate grains are sporadically visible, mainly to a small extent, at the surface. These cracks are usually not filled with alkali-silica reaction products. Based on the above observations, it can be stated that the microstructure changes caused by the alkali-silica reaction correspond to the expansion test results.

4. Conclusions

The conducted research has demonstrated the following:

- the adopted testing method based on ASTM C441 and the evaluation of results against the criteria in ASTM C1157 effectively assess the ability of multi-component cements to mitigate ASR-induced expansion,
- evaluating the impact of cement on ASR effect using ASTM C1157 guidelines provides clear results in a short timeframe, making it potentially useful for concrete mix design involving potentially reactive aggregates, mm
- the method can serve as a preliminary or screening test to assess the effectiveness of non-clinker main constituents in cement for preventing excessive ASR expansion,

- borosilicate glass aggregate proves to be a suitable reference material for studying the influence of cement on ASR progression due to its high reactivity with alkalis,
- the use of multi-component cements CEM II/A,B incorporating active pozzolanic and/or hydraulic non-clinker main constituents effectively prevents mortar degradation caused by ASR expansion. The most significant reduction in ASR effects was achieved using a multi-component Portland cement containing metahalloysite,
- The use of cements with non-clinker main constituents may enable broader utilization of aggregates (fine and coarse) with potential alkali reactivity in concrete compositions. However, this requires confirmation through further studies conducted on concrete.
- microstructural observations of mortars using a scanning electron microscope revealed a correlation between mortar linear changes and the presence of cracks and their filling with ASR reaction products.

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Możliwość oceny cementów z nieklinkierowymi składnikami głównymi pod względem reaktywności alkaliów z kruszywem krzemionkowym

Słowa kluczowe: cement, dodatki mineralne, reakcja alkalia-krzemionka, szkło boro krzemianowe

Streszczenie:

W artykule przedstawiono wyniki badań wpływu obecności nieklinkierowych składników w cemencie na zagrożenie wystąpienia niszczącej reakcji alkalia-krzemionka. Zwykle badania nad przyczynami zachodzenie tego procesu degradacji betonu koncentrują się na określeniu reaktywności kruszywa. Badania wpływu nieklinkierowych składników często przeprowadza się stosując lokalne, naturalne kruszywa, których odmienne właściwości petrograficzne mogą wpływać na wyniki badań. W prezentowanej pracy zastosowano cementy CEM II z dwoma popularnie stosowanymi nieklinkierowymi składnikami, granulowanym żużlem wielkopiecowym, oraz popiołem lotnym krzemionkowym. Ponadto zastosowano spoiwo składające się cementu CEM I i metahaloizytu w stosunku 3:1. Spoiwem zastosowanym do wykonania próbek odniesienia były cementy CEM I, bez składników nieklinkierowych. Przeprowadzono badania ekspansji zapraw z wybranymi spoiwami i wzorcowym kruszywem reaktywnym ze szkła boro krzemianowego zgodnie z metodyką normy ASTM C 441. Wyniki badań odniesione do kryteriów zawartych w normie ASTM C 1157 wykazały, że spoiwa zawierające składniki nieklinkierowe odznaczają się niską reaktywnością względem kruszyw podatnych na reakcję alkalia-krzemionka – spełniają wymagania „Option-R”. Ekspansja zapraw z cementami ze składnikami nieklinkierowymi ulega wydatnemu obniżeniu, w stosunku do próbek z czystego cementu portlandzkiego z tego samego klinkieru. Największa efektywność w tym względzie, spośród przebadanych spoiw uzyskano dla cementu z dodatkiem metahaloizytu, z którym próbki zaprawy wykazały po 56 dniach skurcz wynoszący 0,009%. Z uwagi na ekspansje zapraw, wynoszącą po 56 dniach ponad 0,145%, odnosząc się do wymagań normy ASTM C 33/33M kruszywo, ze szkła boro krzemianowego można uznać, ze bardzo reaktywne. Uzyskane wyniki ekspansji są dobrze skorelowane z mikrostrukturą wykonanych zapraw. Obserwując mikrostrukturę zapraw pod skaningowym mikroskopem elektronowym stwierdzono obecność defektów, które można powiązać z zachodzeniem reakcji alkalia-krzemionka. Objawiają się one, przede wszystkim w postaci spękań ziaren kruszywa ze szkła boro krzemianowego, wypełnionych zbitymi produktami reakcji. Jakościowo oceniając, w największym stopniu spękane są próbki z cementu CEM I, gdzie gęsto rozchodzące się rysy w ziarnach kruszywa mają największe rozwarcie oraz obserwuje się spękania samej matrycy cementowej. W próbach, w których ekspansja jest najmniejsza ziarna kruszywa ze szkła boro krzemianowego prawie nie ulegają degradacji. Podsumowując można stwierdzić, że badając ekspansje zapraw z kruszywem ze szkła boro krzemianowego w okresie 56 dni, reakcja-alkalia krzemionka zachodząca w obecności spoiw z wybranymi składnikami nieklinkierowymi nie nosi znamion potencjalnej szkodliwości. Tym samym wykorzystując cementy z tymi dodatkami możliwe by było stosowanie do produkcji betonu nawet bardzo reaktywnego kruszywa bez narażania go na przedwczesną degradację.

Received: 2024-12-19, Revised: 2025-06-24