Freeze-thaw resistance with de-icing salts of alkaliactivated slag concrete: the influence of activator type and dosage and comparison to the ordinary Portland cement concrete

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Freeze-thaw resistance with de-icing salts of alkali-activated slag concrete: the influence of activator type and dosage and comparison to the ordinary Portland cement concrete

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Abstract

This paper presents the results of an experimental study of the freeze-thaw resistance with de-icing salts (resistance to scaling) of three slag-based alkali-activated (AA) concrete mixes and the reference OPC concrete mix. The scaling resistance was tested on the samples subjected to the 7, 14, 28, 42 and 56 freezing and thawing cycles with 3% NaCl solution as freezing medium. The results showed that the increased sodium silicate content enhances the scaling resistance of the AA concrete. OPC concrete exhibited much higher scaling resistance up to 42 cycles, after which it showed severe damage, while the scaling of AA concrete mixes became more moderate after 7 cycles of testing.

Key words: freeze-thaw resistance, scaling, alkali - activated materials, slag, durability

Otpornost alkalno-aktiviranog betona na bazi zgure na smrzavanje i odmrzavanje sa solima za odmrzavanje: utjecaj vrste i količine aktivatora i usporedba s betonom na bazi običnog portlandskog cementa

Sažetak

U radu su prikazani rezultati eksperimentalnog ispitivanja otpornosti tri mješavine alkalno-aktiviranog (AA) betona na bazi zgure i referentne betonske mješavine s običnim portlandskim cementom (OPC), na smrzavanje i odmrzavanje sa solima za odmrzavanje (otpornost na ljuštenje). Otpornost na ljuštenje ispitana je na uzorcima izloženim 7, 14, 28, 42 i 56 ciklusa smrzavanja i odmrzavanja s 3 %-tnom otopinom NaCl kao medijem za smrzavanje. Rezultati su pokazali da povećani sadržaj natrijeva silikata povećava otpornost na ljuštenje AA betona. OPC beton pokazao je mnogo veću otpornost na ljuštenje do 42 ciklusa, nakon čega su primijećena ozbiljna oštećenja, dok je ljuštenje AA betona postalo umjereno nakon 7 ciklusa ispitivanja.

Ključne riječi: otpornost na smrzavanje i odmrzavanje, ljuštenje, alkalno-aktivirani materijali, zgura, trajnost

1 Introduction

The need for sustainable construction sets the requirements for developing alternative binders to cement, since the production of cement clinker is responsible for 8% of anthropogenic CO_2 emissions [1]. Thus, the partial or complete cement replacement in concrete mixes by alternative binders became the central point in the research area of cementitious materials. One type of alternative binders that gains a lot of research attention are alkali-activated materials (AAMs): a cement-free binders, based on aluminosilicate rich powder precursors activated by alkaline solutions. Waste materials and industrial by-products such as slag, fly ash or metakaolin can be used as precursors, while alkaline hydroxides, silicates, carbonates and sulphates are used as activators, separately or combined [2].

The potential benefits of AAMs application are not only in the reducing the CO_2 emissions, but also in reducing the exploitation of natural resources as raw materials and utilization of waste materials in significant amount, compared to partial replacement of OPC in traditional concrete [2, 3].

The AAMs can be designed to have comparable properties to ordinary Portland cement (OPC) concrete but the durability of AAMs is hard to predict, since their properties depend on the chemical composition of the precursors and activators, which defers among the different types of materials used [4]. Even though there are examples of structural application of AAMs [5], there are no standardized testing methods for evaluation of the AAMs long-term performance [2, 5]. Hence, there is still a need for extensive research to reliably assess the long-term performance of AAMs.

One of the most important durability properties for concrete in cold regions is resistance to freezing and thawing. There are two types of damages caused by frost action: the internal structure damage and scaling of the concrete when de-icing salts are used, described as peeling of the concrete surface [6]. The deterioration mechanism of freeze-thaw action is a combination of several processes. When frozen, the volume of water in pore structure increases by approximately 9%. The volume expansion of the ice causes an increase in hydraulic pressure, forcing unfrozen water away. If the free space in the capillary pores is insufficient for water to be displaced from freezing front, the restrained expansion of the ice will induce additional tensile stresses due to the unrelieved hydraulic pressure. This will induce local cracking of the concrete [6, 7].

In addition to the volumetric changes of the ice, the hydraulic pressure may be enhanced by the ice growth due to unbalanced thermodynamic conditions between gel water and ice, i.e., diffusion of gel water into the capillary pores, and osmotic pressure gradients [7, 8]. The pore solution in concrete is not a pure water but a solution of various chlorides, alkalis and sodium hydroxide, especially when de-ic-

ing salts are used [6]. The freezing point of the pore solution is lower than the freezing point of pure water (i.e., lower than 0°C). Additionally, the size of the pores determines the freezing temperature of the pore solution. The smaller the pores, the lower the freezing point of the pore solution. The water in gel pores in concrete with a diameter of about 2 nm, practically never freezes. Most of the ice will form in capillary pores, with diameter in the range 0.01 – 1 μm [6–8]. Since the water in the pores freezes at different temperatures, not all water in concrete pores freezes at the same time [6–8]. The differences in the salt concentration of the pore solution will cause unfrozen water in the gel pores to migrate from the gel into the capillary pores, where it can freeze and form additional ice that will expand [9]. In [2] it is observed that the freeze-thaw resistance is mainly determined by physical and mechanical processes and thus, the same deterioration mechanisms can be expected in AAMs as in OPC concrete. Therefore, water to binder ratio, pore size and structure and degree of saturation will govern these mechanisms. It is reported that concretes with saturation below 80-90% are resistant to frost action, even without air-entraining agents [7]. Lower water to binder ratio ensures less free water that can be frozen in concrete and decrease the amount of capillary pores in concrete, which is favourable for permeability, freeze-thaw resistance and strength [8]. Favourable pore structure can also be attained by creating air-voids as additional space for the ice expansion with air-entraining agents. [8, 10].

The resistance to scaling is of great interest for the durability of the structure elements treated with de-icing salts due to freezing and thawing exposure. This property is evaluated by simulating the field conditions by subjecting the sample surface to a solution of de-icing salts and exposing samples to repeated freezing and thawing [2, 7, 11]. The resistance to scaling is reported to be generally lower than without de-icing salts, both in OPC concrete and AAMs [2, 6]. However, there is a scarcity in results on scaling of AAMs and the results are not consistent [12]. Apart from the mentioned ones mutual to the OPC concrete, there are additional factors important for the freeze-thaw resistance of the AAMs. The type and amount of precursor plays a major role in the resistance to frost action of the AAMs. It is reported in the literature that the AAMs made with fly-ash are much more susceptible to scaling than made with slag, due to its lower hydration kinetics [5, 8, 13]. Furthermore, the type and dosage of the activator is very important as well. Although sodium hydroxide enhances the kinetics of alkaline activation [14], sodium silicate has been reported as the most reliable activator for obtaining less porous structure and higher frost resistance [5, 8, 10, 15]. The chemistry of alkaline activator will also determine the freezing point of the pore solution [5]. The lower freezing points of the pore solution than in OPC concrete are promising and the reason for this can also be the higher content of gel pores in AA concretes, while OPC systems have more capillary pores [8, 16].

This paper presents a comparative analysis of the freeze-thaw resistance with de-icing salt of slag-based alkali-activated (AA) concrete and reference OPC concrete. The activator dosage differs in all three AA concrete mixes to evaluate its effect on scaling resistance of the AA slag concrete.

2   Materials, sample preparation and test methods

2.1   Raw materials and mix design

Three AA concrete mixes were made with ground granulated blast furnace slag (GGBFS) as a precursor and a combination of sodium hydroxide and sodium silicate as activators. GGBFS was supplied by Ecocem Benelux. The sodium hydroxide solution was made by dissolving pellets in water and Geosil 34417, produced by Woellner was used as sodium silicate solution. The chemical composition of GGBFS (Table 1) was obtained by X-ray fluorescence. This analysis was conducted in accordance with ISO/TS 16996:2015 standard [17], by Central laboratory for chemical analysis at HEP, Croatia, using the Rigaku NEX CG instrument and the Rigaku RPF-SQX software, on the samples with particle size 50 µm, in the helium environment.

Table 1. Chemical composition of GGBFS

Oxide	SiO_2	Al_2O_3	CaO	Fe_2O_3	MnO	TiO_2	MgO	K_2O	Na_2O	SO_3
wt% GGBFS	31.1	13.7	40.9	0.401	0.31	1.26	9.16	0.685	0	2.31

The reference OPC concrete mix was designed in accordance with HRN EN 206 [18] to achieve the requirements for exposure class XF4. CEM I 42.5 R supplied by Holcim, Croatia, and Masterglenium Sky 629 superplasticizer manufactured by Master builders solutions were used to achieve the desired consistency.
Crushed dolomite aggregate with a maximum particle size of 16 mm was used in all mixes. No air entraining agents were used. The detailed mix designs of the concrete mixes are presented in Table 2 and Table 3.

Table 2. Mix design of AA concrete mixes

Mix	w/b	GGBFS [kg/m³]	m (NaOH)* [kg/m³]	m (Na₂SiO₃)* [kg/m³]	Na₂O wt%** GGBFS	Ms	Additional water [kg/m³]	Fine aggregate [kg/m³]	Coarse aggregate [kg/m³]
S1	0.42	375.0	15	4.46	3.5	0.22	145.04	715.0	1072.0
S2	0.42	375.0	15	10.09	4.1	0.42	140.30	706.0	1058.0
S3	0.42	375.0	6.65	21.10	3.5	1.0	135.87	709.0	1063.0
*solid; **weight percent of GGBFS									

Table 3. Mix design of OPC concrete mix

Mix	w/b	Cement [kg/m³]	Water [kg/m³]	Superplasticizer [kg/m³]	Fine aggregate [kg/m³]	Coarse aggregate [kg/m³]
OPC	0.44	340.0	149.6	4.34	797.0	1177.0

The calculations for mix proportioning were performed for the absolute volume of 1m³ of concrete. In the AA concrete mixes, the GGBFS content and w/b ratio were kept constant. The w/b ratio represents the ratio of total water to total solids, where the total water is calculated as the sum of additional tapped water and the water in activator solutions. The total solids are the sum of GGBFS and solids in the activators. The starting point for the mix design was the RILEM TC 247-DTA round robin test [12], where the freeze-thaw resistance of slag-based AA concrete was tested, among other properties. The mix S2 had the same amount of GGBFS and the activators as the RILEM mix S3a, while the w/b ratio was higher (i.e., 0.42 in S2 and 0.382 in RILEM mix). In the mixes S1 and S3, the amount of activators was varied in order to investigate their effect on freeze-thaw resistance of AA slag concrete. In Table 2, the Na_2O content (i.e., the alkali content) was calculated as the sum of the alkalis in sodium hydroxide and sodium silicate. The silica modulus (Ms) is calculated as the SiO_2/Na_2O ratio, where the SiO_2 is the amount of silica in sodium silicate.

2.2 Sample casting and curing

Four 150 mm cubes were casted for each mix, for the freeze-thaw cycling. Samples were demolded after 24 hours. The OPC concrete samples were cured for 7 days in water. Later, samples were kept in the curing chamber until 21 days old, when the sample preparation for freeze-thaw test begun. Due to their sensitivity to water curing [2], AA concrete samples were cured sealed with polymeric films to prevent the moisture loss, for 21 days. 150 mm cube OPC samples for compressive strength tests were cured for 28 days in humidity chamber in accordance with HRN EN 12390-3 [19]. AA concrete samples were cured sealed, as described.

2.3 Test methods

Testing of freeze-thaw resistance with de-icing salts was conducted in accordance with the "slab test" procedure, prescribed in the standard HRN EN 12390-9 [20]. When the samples were 21 days old, 50 mm thick slab specimen were sawn from the 150 mm cubes, for each mix. After that, the slabs were placed in rubber sheets and prepared for the testing, as described in HRN EN 12390-9. The 3 days long pre-saturation of the samples' surface was conducted when the concrete was 25 days old, by pouring 3mm deep layer of de-ionized water. The NaCl water solution was prepared with NaCl concentration of 3% by mass and poured in 3 mm deep

layer on the top surface of samples, when they were 31 days old. Samples were then placed in the freezing chamber and subjected to repeated freezing and thawing cycles. One cycle of freezing and thawing was 24 hours long. The samples were examined after 7, 14, 28, 42 and 56 cycles. The test surface was rinsed with water and brushed with brush to collect the scaled concrete into the vessel. After each examination, the NaCl solution was poured again for the next set of freeze-thaw cycles. Scaled material was collected in vessel and dried to the constant mass at 110°C. After measuring the dried mass of scaled material, the results are expressed as cumulative mass after defined number of cycles [20].

Prior to the sample casting, fresh properties of the concrete mixes were tested, i.e., consistency, density and air content, as described in HRN EN 12350-2 [21], HRN EN 12350-6 [22] and HRN EN 12350-7 [23], respectively.

Compressive strength tests were conducted after 28 days of casting, in accordance with HRN EN 12390-3 [19].

3 Results and discussion

The results of control testing of fresh state properties and compressive strength of the samples are presented in Table 4. The results of measured scaled mass for AA concrete and OPC concrete mixes after freeze-thaw cycles are shown in Figure 1. The results from RILEM TC 247-DTA round robin test on freeze-thaw resistance with de-icing salts of slag-based AA concrete are plotted on the graph as well. The test method applied in RILEM round robin test is different only in the number of cycles after which the measurements were conducted.

Table 4. Fresh state properties and compressive strength of S1, S2, S3 and OPC mixes

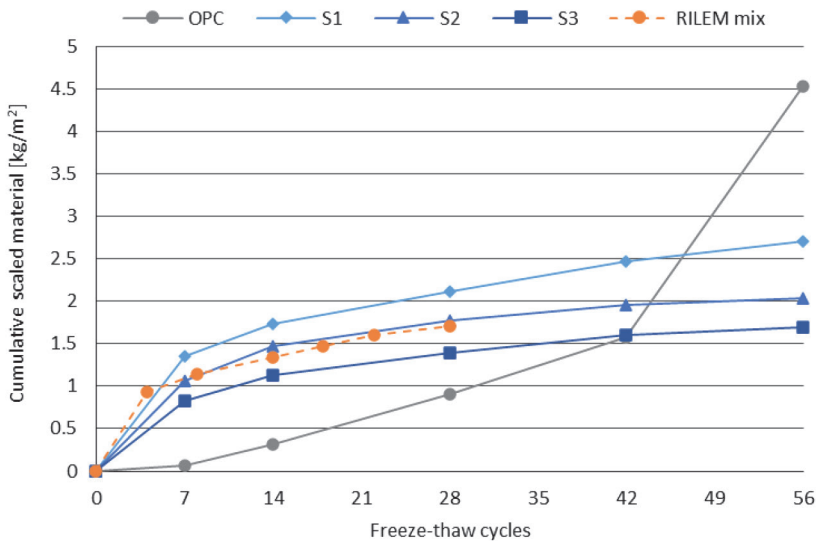| Mix	Slump [mm]	Density [kg/m³]	Air content [%]	Compressive strength [MPa]
S1	25.0	2474.5	2.2	49.35
S2	54.0	2464.7	1.2	53.76
S3	73.0	2470.9	1.3	70.45
OPC	67.0	2534.6	1.8	70.39

Figure 1. Cumulative scaled material for S1, S2 and S3 mixes after 7, 14, 28, 42 and 56 cycles and after 4, 8, 14, 18, 22 and 28 cycles for RILEM mix [12]

When comparing AA concrete mixes, sodium silicate content had the predominant effect on resistance to scaling, which is in line with the results in the literature [5,8,15]. The mix S1 had the lowest amount of sodium silicate and the highest cumulative scaled mass after each measurement. Mix S3 had the highest sodium silicate content and the highest resistance to scaling. Additionally, after 28 cycles, mixes S2 and S3 had a very small increase in scaled material. The mix S2 showed almost the same results as the corresponding RILEM mix. It can be concluded that the small increase in w/b ratio for mix S2 compared to RILEM mix had almost no effect on the freeze-thaw resistance. The increase in sodium silicate content in the mixes resulted in the formation of denser binder matrix and higher compressive strength, leading to the highest resistance to scaling of the mix S3, followed by S2, followed by the mix S1 [10, 15].

Up to the 42nd cycle, all three AA concrete mixes exhibited much lower resistance to scaling than OPC concrete. However, after 7 cycles, the increase in scaled material for AA concrete became moderate, which can be seen in reduction of steepness of the cumulative curves (Figure 1), while the OPC concrete exhibited steep increase in scaled material. After 42 cycles, the mix S3 and OPC mix showed the same freeze-thaw performance. After 56 cycles, OPC mix had much more severe damages compared to all three AA concrete mixes, with 4.53 kg/m² of cumulative scaled material. Figure 2 shows the samples after 56 testing cycles. The scaling process of all three AA mixes and OPC mix was followed by falling out of aggregate particles which was

less prominent for AA concrete and increased for OPC concrete with increasing the number of test cycles. The aggregate particles can be seen above the AA concrete samples surfaces due to scaling of the binder (Figure 2), but the samples kept their integrity throughout the whole test period. The mix S1 (Figure 2-A) had deeper damages at the edges of the sample. Although there are parts of the samples with no scaling, OPC samples showed cracking and severe damage of the binder and falling out of the aggregate particles (Figure 2-D).
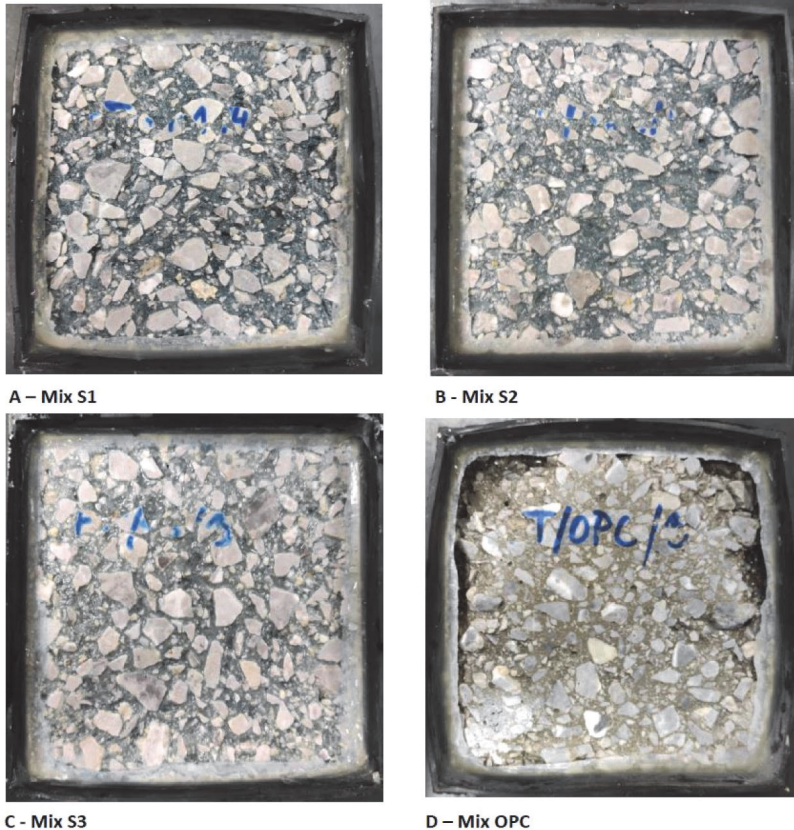
A – Mix S1

B - Mix S2

C - Mix S3

D – Mix OPC

Figure 2. The example of samples exposed to 56 freeze-thaw cycles with de-icing salts: A - S1, B - S2, C - S3, D - OPC

Neither AA concrete or OPC concrete satisfied the durability requirements of XF2 and XF4 exposure classes, which are in Croatian national annex [24] set as 0.5 kg/m^2 of scaled material after 28 cycles and 56 cycles, respectively. Even though AA mixes had much lower resistance to scaling than OPC mix after 7 days of exposure, the fact that scaling of AA mixes became moderate after 7 cycles led them to perform better

after 56 cycles than OPC. Therefore, mitigating the fast scaling of the AA concrete in the first 7 cycles could possibly result in enhanced resistance to freezing and thawing with de-icing salts.

Reasons behind the difference in the behaviour between AAM and OPC during these initial cycles of freezing and thawing still need to be understood. One of the reasons could be carbonation of the initial layer of concrete. Previous research indicates the correlation between the scaling curve and carbonation curve, for the OPC concrete with 50% cement replacement with slag. The decrease of the surface scaling for OPC concrete with partial cement replacement coincided with the carbonation depth occurred during curing [12]. Since the carbonation causes the increase of the capillary pore volume [25], it is possible that the first, carbonated layers of the samples were more susceptive to frost impact than non-carbonated layers. In the case of present study, however, samples were sealed cured, avoiding the possibility to have carbonation in this first layer of concrete. Next reason could be the pre-saturation of the samples. Indeed, the study of Ahani and Nokken [13] on the effect of pre-saturation and curing conditions on the OPC concrete with 35% of the slag indicated that the scaling resistance is higher when the samples are pre-saturated for 7 days with the freezing medium than for non-saturated samples and even from saturated OPC samples. The pre-saturation of the sample surfaces with the NaCl solution could reduce the osmotic pressure caused by the disbalance of ions between the pores of the concrete and NaCl solution [13]. The extended moisture curing was also found to be beneficial for the compressive strength development [3] and therefore scaling resistance of OPC concrete with cement replacement [13] and AA concretes, depending on the type of precursor [12]. The future research will focus on attempt to address these issues in presented mixes and confirm or exclude the possible reasons for low freeze-thaw resistance with de-icing salts of the presented AA concrete mixes, especially in early stage of testing.

4 Conclusion

This paper presents the results of the experimental study on the effect of activator type and dosage on freezing and thawing resistance with de-icing salts of alkali activated slag concrete. Three AA concrete mixes with different dosage of sodium hydroxide and sodium silicate were prepared and subjected to the scaling resistance test at 7, 14, 28, 42 and 56 cycles, as well as the reference OPC concrete mix. The comparison between AA concrete mixes showed that increasing the sodium silicate content increased the scaling resistance. The reference OPC mix had significantly higher resistance to freeze-thaw cycling with de-icing salts than AA mixes up to 42 cycles. After the 56th cycle, the OPC concrete exhibited severe damage, i.e., disintegration of the binder and cracking, and finally showed lower resistance to

scaling. An important result of the test is the difference between the shapes of the cumulative scaling curves of the OPC and AA mixes. After a high cumulative mass of scaled material up to 7 cycles, the scaling of AA mixes became moderate, in contrast to the OPC concrete, where it became more evident after each testing cycle. Future studies will investigate the effects of carbonation, saturation, and curing conditions as possible reasons for the rapid scaling after the first 7 cycles. The moderation of the AA concrete scaling over time may be promising regarding its long-term durability. Therefore, mitigating the increased scaling in the early stage of testing could be a solution for improving the freeze-thaw resistance with de-icing salts of the AA concrete.

Acknowledgement

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