Frost Resistance of Alkali-Activated Concrete—An Important Pillar of Their Sustainability

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Abstract: Sustainable development of concrete construction requires sustainable materials or sustainable binders. Specifically, alkali-activated materials (AAMs) are an interesting and wide group of materials. They have good strengths and are considered environmentally friendly materials because secondary materials are consumed during the preparation of AAMs. The durability of AAMs is also excellent. One of the most important parts of durability is frost resistance. The frost resistance of alkali-activated materials is usually very good. However, some studies showed opposite properties and poor frost resistance. The reason for this may be a different composition of the activator. The content of alkalis is often considered the main characteristic of alkali-activated materials. However, SiO2 content can play an important role too. This paper discusses the different results for the mechanical properties and frost resistance of different compositions of alkali activators made of sodium water glass with a silicate modulus modified with potassium hydroxide. The role of the activator content and the water-to-cement ratio in this phenomenon is discussed. The results of this article show that the strengths of AAMs are significantly affected by the curing method. Water curing reduced some of the strength of the specimens compared to foil-covered specimens. Frost resistance depends on the method of curing and on the composition of the activator; some concretes with high strengths showed very low frost resistance.

Keywords: alkali-activated concrete; curing; freezing and thawing; frost resistance; GBFS; strength

1. Introduction

Very briefly, sustainable development, according to the Brundtland definition, is “development that meets the needs of the present without compromising the ability of future generations to meet their own needs” [1]. To make concrete more sustainable, we can follow many arrangements. For example, in accordance with Naik, “sustainable concrete should have a very low inherent energy requirement, be produced with little waste, be made from some of the most plentiful resources on earth, be able to produce durable structures, have a very high thermal mass, and be made with recycled materials” [2]. A more detailed list of arrangements was presented by Aïtcin and Mindess [3], including “the use of higher strength concrete, making concrete more durable, replacing up to half of Portland cement with supplementary cementing materials”. Mehta [4] expressed the sustainability of concrete very briefly by categorizing it into three roles: consume less concrete, consume less cement, and consume less clinker. In essence, we need to produce high-performance concrete with minimal use of clinker and with good durability.

Alkali-activated materials (AAMs) meet the basic requirements of sustainable development. They can be considered one of the most promising alternatives to Portland cement. AAMs represent a very wide variety of materials [5–10]. Instead of geopolymers or geocements, which are prepared from sources without a significant content of Ca and show a different structure (i.e., a three-dimensional network instead of a C–S–H gel), slag alkaline concretes (with the possibility of replacing a part of the slag with fly ash or other
fillers but without cement clinker) or hybrid cements [9,11,12], with a very small portion of Portland clinker, are potential alternatives. This division is rough, but it shows the large potential varieties of AAMs.

From the point of view of the abovementioned sustainability requirements, it is possible to obtain AAMs with high strengths [13–15]. This can also be interpreted as “consuming less concrete”. Portland cement can be replaced completely or partially by more than half, that is, “consume less clinker”. They can be prepared using secondary materials, such as blast furnace granulated slag (GBFS); fly ash (FA); and other powders, such as fillers from quarries, fillers from recycled concrete, etc. [16–19]. There is also the possibility to use natural pozzolan [20]. This consumption of secondary materials together with the reduction in CO₂ pollution due to the elimination of cement makes AAM binders suitable for sustainable structures.

AAMs are also considered very durable [21–28], which is another very important pillar of sustainability, but there are also some problems, which are collected, for example, in [27]. Among the most important are, inter alia, rapid setting and poor processability, high shrinkage, complicated and not fully understood chemistry, and limited large-scale use. The latter means some uncertainty about the durability of AAMs. Problems with the frost resistance and durability of AAMs are discussed in Reference [7]. This paper especially focuses on the freezing and thawing resistance of AAMs. Cyr and Pouhet [29] discussed in great detail freezing and thawing resistance and the scaling resistance, but the results were very inconsistent; sometimes, good freezing and thawing resistance were recorded, while other times, they were not. Generally, the freezing and thawing resistance seems to be slightly better than that of ordinary cement-based concrete. In contrast, the scaling resistance of AAMs is slightly worse than that of OPC (ordinary Portland cement)-based concrete. Air entraining can significantly enhance both types of resistance. Matalkah and Soroshian [30] showed similar results; they also enhanced the scaling resistance by adding admixtures, especially organic-based ones. Bilek [31] found poor freezing and thawing resistance of AAMs based on activated slag. In particular, the substitution part of the slag with fly ash seemed to be very damaging. Many authors [6,7,9,26], in particular, showed good freezing and thawing resistance results together with explanations for their findings; however, not all studies were consistent, as some reported worse results.

Methods for measuring frost and thawing resistance also play an important role. The lowest temperature differs with various recommendations. In the Czech Norm CSN 73 1322 [32], the temperature is −20 °C and the freezing period lasts 4 h. The specimens are then stored in water at 20 °C. Usually, two sets of specimens are used: one is cycled to the required number of cycles (usually 100–200), and the other is the reference. The specimens are stored in water at 20 °C for 2 h. However, what is the correct time for testing this second set? Is it before cycling or after cycling of the cycled set? Is it correct to compare the properties of frosted concrete with the properties of concrete before the test? Cycling may continue for a relatively long time, and the concrete may have better (or worse) properties after this time interval. Specimens in the freezer are in water for some time (thawing) and without water for some time (freezing). Is it better to compare their properties with the properties of specimens stored in water at 20 °C or with those that are differently cured (without additional water)? A discussion of these questions form the most important part of this paper. The answers can contribute to a wider application of AAMs in sustainable construction.

2. Materials and Experimental Procedures

Ground granulated blast furnace slag (GBFS) produced in the Czech Republic was used in this paper. The slag had the chemical composition shown in Table 1. The specific surface area in accordance with the producer’s information was 420 m²/kg. The specific gravity of the slag was 2850 kg/m³.
Table 1. Chemical composition of ground granulated blast furnace slag (GBFS).

|       | CaO | SiO$_2$ | Al$_2$O$_3$ | Fe$_2$O$_3$ | MgO | Na$_2$O | K$_2$O | SO$_3$ | Cl$^-$ |
|-------|-----|---------|-------------|-------------|-----|---------|-------|--------|-------|
| GBFS  | 41.2| 35.6    | 8.3         | 0.5         | 2.3 | 0.1     | 0.5   | 2.0    | 0.1   |

The sodium water glass with a silicate modulus $M_s = 2.0$ was produced by the company Vodni sklo Brno. The water glass contained 32.0% Na$_2$O and 16.5% SiO$_2$, and the density of the solution was 1500 kg/m$^3$. A 50% solution of potassium hydroxide (KOH) with a density of 1500 kg/m$^3$ was used to modify the $M_s$. It was stated that the exchange of NaOH for KOH was very advantageous in terms of workability and efflorescence [33]. Significantly better cone flow was the main reason for the combination of the sodium water glass and KOH.

Natural sand 0/4 mm was used for the preparation of mortars. The density of this sand was 2600 kg/m$^3$. This sand is widely used for concrete production, including high-quality concrete with excellent frost resistance. Drinkable water was used as the mixing water for the production of mortars (fine-grained concrete).

Mortars with different (Na$_2$O + K$_2$O)/SiO$_2$ mass ratios (R/S ratio) were prepared. This ratio had a value of approximately 34% Na$_2$O and 66% SiO$_2$ (34/66) for the water glass only up to 100% K$_2$O + 0% SiO$_2$ (100/0) for a mixture activated only with the KOH solution. The dosage of GBFS was 450 g per 1 L of mortar, and the water-to-slag ratio was maintained at 0.50, i.e., 225 g of water including water from the water glass and KOH solution for 450 g of GBFS. The total dry mass of the alkalis in the activator $\sum$ (dry mass Na$_2$O + K$_2$O) was maintained at 6% of the mass of GBFS (i.e., 27 g for 450 g of GBFS). In the case of 34/66 mortar (water glass only), this represents 27 g of Na$_2$O from the water glass. This means that another 52.4 g is SiO$_2$. In this case, the total dry mass of the activator was 17.6%. In the case of the 100/0 mixture (only KOH), the total mass of K$_2$O was 6%, which also represents the total dry mass of the activator $\sum = 6%$. In all mortars, the rest up to 1 L was sand. Mortars were prepared from R/S ratios of 34/66, 40/60, 50/50, 60/40, 70/30, and 100/0 and with a total dry mass of the activator at 17.6%, 15%, 12%, 10%, 8.6%, and 6%, respectively.

The ratios 80/90 and 90/10 were found to be very poor from the point of view of very quick setting; see for example Reference [28]. Mortars with total dry mass of the activator $\sum = 10%$ and 15% were prepared for R/S = 34/66 and R/S = 60/40, and mortars with $\sum = 10%$ and 17.6% were also prepared for R/S = 100/0; see Table 2.

Table 2. Scheme of the experiment: mixtures are designated with an X.

| (Na$_2$O + K$_2$O)/SiO$_2$ | 34/66 | 40/60 | 50/50 | 60/40 | 70/30 | 100/0 |
|---------------------------|-------|-------|-------|-------|-------|-------|
| Total dry mass of activator |       |       |       |       |       |       |
| 17.6%                     | X     | X     |       |       |       |       |
| 15%                       | X     | X     |       |       |       |       |
| 12%                       |       |       | X     |       |       |       |
| 10%                       | X     |       |       | X     |       |       |
| 8.6%                      |       |       |       |       | X     |       |
| 6%                        |       |       |       |       |       | X     |

Mortars were mixed in a laboratory mixer at a volume of 1 L. The water glass contents together with the KOH solution and water were poured into the mixing contents, and the slag was added. Mixing started immediately, and after 30 s, sand was added over the next 15 s. The total mixing time was 180 s. After mixing, the workability of the mortars was tested using a truncated cone with a height of 60 mm and with lower and upper bases of 100 mm and 70 mm, respectively. The cone flow was measured after 15 jolts of the jolting table. Then, 40 × 40 × 160 mm prisms were prepared and compacted. The prisms were unmolded after hardening (usually after 22 h), and two prisms were tested.
The obtained properties were volume density, three-point bending $f_b$, and compressive strength on fragments $f_c$. Other prisms were enveloped with polyethylene foil to prevent water exchange with the environment. This method of curing seems to be better for AAMs than water curing. Two prisms at 24 h and the next two at 7 days of age were tested. For testing, a FORM+TEST Prufsysteme machine with maximum relative accuracy deviation lower than 0.2% was used. These strengths were considered indicative only. At the age of 28 days, three of the specimens were tested, another three were placed in water, and another three were used for freezing and thawing (F&T) cycles in an automatic freezer KD-20–T3.1. As mentioned above, one F&T cycle represented storage at $-20 \, ^\circ C$ for 4 h, and then the space in the freezer was filled with water $+20 \, ^\circ C$ for 2 h. The selected number of cycles was 125, which was recommended for medium frost-resistant concretes in the Czech Republic. Usually, prisms of size $100 \times 100 \times 400 \, mm$ are used for the test according to Reference [32], but in our case, only fine-grained concrete or mortar was used and the dimensions were reduced to $40 \times 40 \times 160 \, mm$. As one cycle lasted 6 h, four F&T cycles were performed over 24 h. This meant that the age of the specimens at the end of cycling was 60 days. At this age, three prisms were also pulled out of water and tested. This means that frosted prisms and water-cured prisms were tested after cycling. Three-point bending strength and compressive strength on fragments of the prisms were performed as above.

The frost resistance of concrete is usually evaluated by the frost resistance index, which is the ratio of the properties of cycled specimens/the properties of normally cured specimens. Suitable properties are bending strength, compressive strength, dynamic modulus of elasticity, and volume density of the specimen. In accordance with the presented course of the experiment, it is possible to express the following indices with respect to the comparative specimens before cycling or with respect to the comparative specimens after cycling. The latter were additionally cured in water.

$$I_{BW} = \frac{f_b \text{ of specimens cured 28 days in foil and additionally in water}}{f_b \text{ of specimens cured 28 days in foil}} \quad (1)$$

$$I_{BB} = \frac{f_b \text{ of specimens cured 28 days in foil + F&T cycles}}{f_b \text{ of specimens cured 28 days in foil before cycling}} \quad (2)$$

$$I_{BA} = \frac{f_b \text{ of specimens cured 28 days in foil + F&T cycles}}{f_b \text{ of specimens cured 28 days in foil and additionally in water}} \quad (3)$$

3. Results and Discussion

First, the results obtained on pastes are mentioned. In previous papers in [34], it was shown that the start of setting depends very significantly on the silicate modulus of the activator or, in terms of this paper, on the R/S-ratio (see Figure 1).

The results for two concepts are demonstrated here. The full line represents the results for the total dry mass of the activator $\Sigma = Na_2O + K_2O +SiO_2 = 10\%$ and the different R/S ratios. The dashed line shows the results for $R = Na_2O + K_2O = 6\%$ and the different total dry mass of activator $\Sigma$. However, the general course of the lines is similar. There is an optimal start setting for $R/S = 60/40$. The neighboring ratios 50/50 or 70/30 are also acceptable for mixing alkali-activated concretes with an optimal time of workability. In addition, $R/S = 100/0$—activation with hydroxide only—appears to be very good in terms of workability time. The ratios 80/20 and especially 90/10 are really unsuitable.

All of the results for these workabilities and strengths (mean value and standard deviation) are shown in Table 3. The workabilities of mortars are presented in Figure 2. It can be seen that the best cone flow was achieved for $R/S = 60/40$ and that the worst was achieved for $R/S = 100/0$. This is quite interesting because the most activator was for ratio 34/66, but the workability was significantly worse. Workability also depended on the composition of the activator. The poor workability for $R/S = 100/0$ can be explained in terms of the lowest activator content and the absence of water glass in the activator.
Sixty days means 28 days in foil and 32 days (during cycling) in water at 20 °C. The compressive strengths of the mortars with dry mass Na₂O + K₂O = R = 6% and different total dry mass of activator are shown in Figure 3.

Table 3. Results of the tests.

| R/S in activator | 34/66 | 40/60 | 50/50 | 60/40 | 70/30 | 100/0 |
|------------------|-------|-------|-------|-------|-------|-------|
| R = N + K (%)    | 6     | 6     | 6     | 6     | 6     | 6     |
| ∑ (%)            | 17.6  | 15    | 12    | 10    | 8.6   | 6     |
| Cone flow (mm) (%) | 168 | 191 | 167 | 202 | 191 | 119 |
| 24 h.            |       |       |       |       |       |       |
| f_b1 (MPa)       | 1.7 ± 0.1 | 2.75 ± 0.1 | 4.35 ± 0.05 | 3.4 ± 0.05 | 4.1 ± 0.5 | 1.3 ± 0.2 |
| f_c1 (MPa)       | 8.2 ± 0.8 | 13.3 ± 1.0 | 15.4 ± 0.6 | 30.6 ± 0.4 | 19.1 ± 0.5 | 3.7 ± 0.4 |
| 7 days           |       |       |       |       |       |       |
| f_b7 (MPa)       | 6.4 ± 0.2 | 5.1 ± 0.1 | 9.1 ± 0.3 | 8.6 ± 0.4 | 6.1 ± 0.3 | 3.5 ± 0.2 |
| f_c2 (MPa)       | 60.0 ± 1.5 | 64.2 ± 2.0 | 73.0 ± 2.0 | 65.2 ± 2.3 | 44.9 ± 1.5 | 7.9 ± 0.1 |
| 28 days          |       |       |       |       |       |       |
| f_b28 (MPa)      | 9.1 ± 0.3 | 9.4 ± 0.3 | 9.3 ± 0.2 | 9.0 ± 0.3 | 8.3 ± 0.4 | 3.5 ± 0.2 |
| f_c28 (MPa)      | 91.0 ± 5.0 | 91.7 ± 6.4 | 84.1 ± 4.6 | 74.4 ± 3.8 | 57.3 ± 2.0 | 11.7 ± 0.9 |
| 60 days          |       |       |       |       |       |       |
| f_b60 (MPa)      | 6.3 ± 0.2 | 8.15 ± 0.1 | 8.1 ± 0.2 | 8.3 ± 0.1 | 7.9 ± 0.2 | 4.6 ± 0.5 |
| f_c60 (MPa)      | 108.2 ± 12 | 103.1 ± 20 | 94.8 ± 4.2 | 86.5 ± 1.4 | 62.5 ± 5.3 | 16.3 ± 1.3 |
| l_bSW (%)        | 69    | 87    | 87    | 92    | 95    | 131   |
| l_cSW (%)        | 119   | 112   | 113   | 116   | 109   | 139   |
| 125 cycles       |       |       |       |       |       |       |
| f_b (MPa)        | 5.5 ± 0.1 | 9.2 ± 0.3 | 8.5 ± 0.2 | 8.3 ± 0.5 | 5.6 ± 2.5 | disintegration (45 c.) |
| f_c (MPa)        | 104.5 ± 4.6 | 102.4 ± 2 | 91.5 ± 5.3 | 84.4 ± 1.8 | 56.1 ± 2.5 |  |
| l_bB (%)         | 60    | 98    | 91    | 92    | 67    | 0     |
| l_cB (%)         | 115   | 112   | 109   | 113   | 98    | 0     |
| l_bA (%)         | 87    | 113   | 105   | 100   | 70    | 0     |
| l_cA (%)         | 97    | 99    | 97    | 98    | 90    | 0     |

Figure 1. Start of setting for pastes with ∑ = R + S = 10% (full line) and for R = 6% (dashed line).
Previous research by the authors [31] solved also the strengths of mortars with different R/S ratios for a constant dry mass of activator $\Sigma$ and a constant water-to-slag ratio. Strengths were recorded to be the best for an R/S ratio at around 60/40. The strengths for R/S = 100/0 were very low, the values ranged around 1/3 to 1/2 of the values for R/S = 40/60. This was probably because the solubility of silicon ions from GBFS is worse than that of calcium ions [35]. Calcium ions are not able to form a C–S–H gel without another activator. Silica gel forms on water glass in water glass because its fineness is very high. The importance of water glass presence was reduced in comparison to mortars with ratios R/S = 70/30 ($\Sigma$ = 8.6%) and R/S = 100/0 ($\Sigma$ = 6%). The compressive strength of the mortar with R/S = 100/0 was nearly five-times lower than that with R/S = 70/30 and eight-times lower than that with R/S = 40/60. This was probably because the solubility of silicon ions from GBFS is worse than that of calcium ions [35]. Calcium ions are not able to form a C–S–H gel without silicon ions from water glass.

The maximum early compressive strengths were reached for R/S = 60/40. At the age of 1 day, the results of strengths were independent of the total dry mass of the activator. However, continuously, at the age of 7 days and especially at the age of 28 days, the total mass of activator $\Sigma$ played an increasingly significant role and the highest strength was reached when the highest $\Sigma$ and the strengths (bending and compressive) decreased continuously with the decreasing amount of activator $\Sigma$. The importance of water glass presence was reduced in comparison to mortars with ratios R/S = 70/30 ($\Sigma$ = 8.6%) and R/S = 100/0 ($\Sigma$ = 6%). The compressive strength of the mortar with R/S = 100/0 was nearly five-times lower than that with R/S = 70/30 and eight-times lower than that with R/S = 40/60. This was probably because the solubility of silicon ions from GBFS is worse than that of calcium ions [35]. Calcium ions are not able to form a C–S–H gel without silicon ions from water glass.

Figure 2. Cone flow of mortars with $R = 6\%$ and different R/S and $\Sigma$.

Figure 3. Compressive strengths of specimens stored for 28 days in foil.
the ratio 60/40. These results also generally agree with that of other authors collected in Reference [15]. The results for a constant R/S ratio and a different $\sum$ in the present paper show that a higher dry mass of the activator $\sum$ increases the compressive strengths. However, Figure 4 shows that this tendency is different for different R/S. In the case of R/S = 34/66 and 100/0, there is a significant increase in compressive strength with increasing $\sum$, but in the case of R/S = 60/40, the effect of $\sum$ is poor.

![Figure 4](image_url)

**Figure 4.** Compressive strengths for $\sum = 17.6\%$, 15\%, 10\%, and 6\% and R/S = 34/66, 60/40, and 100/0.

After 28 days, the specimens were stored in water (for similarity to cycled specimens). Figure 5 shows the compressive strengths at the age of 60 days when every three sets of specimens were tested.

![Figure 5](image_url)

**Figure 5.** Compressive strengths of specimens stored 28 days in foil and of specimens stored 28 days in foil and another 32 days in water.

The compressive strength of specimens stored in water increased during the F&T cycles, and this increase was nearly the same for all mixtures. The strengths represented...
a 113–139% increase with respect to the 28-day strength of the specimens in foil. This development was similar to the development of conventional Portland cement-based concrete. The bending strengths showed an opposite tendency (Figure 6). Except for the mortar activated only with KOH, R/S = 100/0, all bending strengths of the specimens stored in water for 28 up to 60 days were lower.

![Bending strengths of specimens stored for 28 days in foil and of specimens stored for 28 days in foil and another 32 days in water.](image)

**Figure 6.** Bending strengths of specimens stored for 28 days in foil and of specimens stored for 28 days in foil and another 32 days in water.

The situation in terms of indices is presented in Figure 7. All values of index $I_{bW}$ are lower than 100% with the exception of R/S = 100/0. Water worsens the bending strengths of the mortars. This means that, during frost-resistance tests, not only do the F&T cycles degrade the bending strength of the specimens but also it is degraded by the presence of only water. This is important from for the results in terms of indices $I_{bB}$ and $I_{bA}$.

![Indexes $I_{bW}$, $I_{bB}$, and $I_{bA}$ for different R/S ratios.](image)

**Figure 7.** Indexes $I_{bW}$, $I_{bB}$, and $I_{bA}$ for different R/S ratios.

It is obvious that, after 125 F&T cycles, three of the mixtures, R/S = 34/66, 70/30, and 100/0, do not reach the limit $I_{bB} = 75\%$ for frost-resistant concrete. Interestingly, these
are mixtures with the highest and the lowest total dry masses of the activator $\Sigma$. In the cases of $R/S = 70/30$ and especially $R/S = 100/0$ ($I_{bB} = 0$), the reason can be generally lower strengths—compressive strengths 57.3 and 11.7 MPa, respectively. Specimens with $R/S = 100/0$ disintegrated after 45 F&T cycles. However, specimens with $R/S = 34/66$ show a very high compressive strength (91 MPa) but were not frost resistant from the point of view of $I_{bB}$. On the other hand, all values of $I_{bA}$ were sufficiently high from the point of view of frost resistance except for $R/S = 100/0$. Some $I_{bA}$ values were higher than 100%, which meant that the cycled beams were in better condition than the “optimal” cured specimens. Probably, low temperatures retard some of the degradation processes inside the water-cured specimens.

The following figures show the influence of the dry mass of activator $\Sigma$ on the above-mentioned indices $I_{bW}$, $I_{bB}$, and $I_{bA}$.

The results presented in Figure 8 show that mortars with a higher total dry mass of activator cured for 28 days in foil and another 32 days in water at $+20^\circ C$ showed intense degradation of the bending strengths. The $I_{bW}$ index increased as the total dry mass of activator $\Sigma$ decreased. For the highest total dry mass of the activator $\Sigma = 17.6\%$, $I_{bW}$ reached the same value for all mixes regardless of the alkali/silica $R/S$ ratio—only approximately 60%. This was a very significant reduction in bending strength. It was only for $\Sigma = 10\%$ that the values of $I_{bW}$ were more than 80%, and they increased with an increasing number of alkalis in the activator. The compressive strengths were high for all tested mortars, and their $I_{bW}$ was above or close to 100%.

![Figure 8. Indexes $I_{bW}$ (Formula (1)) for mortars with different $\Sigma$.](image)

What is reason for this phenomenon? A decrease in bending strengths and an increase or stable value of compressive strengths can usually be explained by the occurrence of microcracks. It is well known that alkali-activated materials show higher shrinkage and higher microcracking than Portland cement-based concretes. Water can heal microcracks and can increase bending strength, but why the bending strength of a water-cured specimen decreases is not easy to explain.

Figure 9 shows the influence of the dry mass of the activator on the frost resistance of mortars in terms of the above indices $I_{bB}$ and $I_{bA}$. Figure 9 shows that a high total dry mass of activator $\Sigma$ was not suitable. All presented mixtures showed an index $I_{bB}$ of less than 75% for $\Sigma = 17.6\%$. At lower $\Sigma$, 15% and 10%, the $I_{bB}$ of mortars with $R/S = 34/66$ and 60/40 meet the requirements for frost-resistant concrete, with indices higher than 75%. There was a very significant increase in indexes between $\Sigma = 17.6\%$ and 15%. Mortars
activated only with KOH showed the opposite tendency: only at \( \Sigma = 17.6\% \), the specimens were not disintegrated during F&T cycles. In other cases, they disintegrated after 45 cycles.

Due to degradation of the specimens during additional water curing, the indices \( I_{BA} \) were quite high for \( R/S = 34/66 \) and \( 60/40 \)—see Figure 10. Their values were affected by the lower strengths of the reference: additionally water-cured beams. The general trend was an increase in \( I_{BA} \) with decreasing \( \Sigma \). The only exception was a mortar with \( R/S = 100/0 \)—its \( I_{BA} \) was much lower than 75% but higher than \( I_{BB} \).

The presented results show two basic problems. The first one is the reduction in bending strengths during storage in water between 28 and 63 days. The reduction in strengths is quite significant, and the reason is unclear. Compressive strengths increased or remained stable during water curing. The most intensive decrease in bending strengths was observed for mixtures with the highest content of SiO\(_2\) in the activator (see also Figure 7). With increasing content of Na\(_2\)O + K\(_2\)O, the decrease in bending strengths was not so significant and, in the case of mortars activated only with hydroxide, the bending strength...
increased during water curing. There was also a permanent reduction in the dry weight of the activator. The maximum was for R/S = 34/66—17.6% by weight of the slag—and the minimum was for R/S = 100/0—6% by weight of the slag. It was likely that excess of the activator was leached from the specimens and that the occurrence of microcracks decreased bending strength. However, the impact of this process on the compressive strength was not clear. This phenomenon requires further study. Previous results of the authors also showed a higher shrinkage of mortars with a higher dry mass of the activator and with a higher content of SiO₂ in the activator. This may be the reason for the occurrence of microcracks.

The penetration water inside the specimens can play an important role. This can explain the worse frost resistance of the specimens with higher shrinkage but not the poorer strengths of water-cured specimens. Some healing of microcracks by water is more likely.

The second problem is frost resistance. Specimens exposed to cycling showed better bending strengths than specimens stored in water. This phenomenon is sometimes also observed in the case of Portland cement-based concrete, especially high-performance concrete. Collepardi et al. [36] explained this by a better structure of the C–S–H gel crystallized at a lower temperature. However, this did not seem to be a suitable explanation for such low temperatures.

The frost resistance of non-air-entraining alkali-activated concretes and mortars can also be affected by a higher amount of non-freezable water [37]. These materials do not contain portlandite, which contains a relatively high amount of chemically bound water in Portland cement-based concretes. In accordance with the micro-mechanism of frost degradation presented by Setzer [38] or other mechanisms based on thermal contraction and dilatation of ice, presented by Kaufmann [39], the materials contain more C–S–H gel and gel water can show poorer frost resistance. However, the results differ in the present case.

That many of other authors showed good frost resistance is also very interesting. Often, the AAMs are air-entrained and the frost resistance is enhanced [29,30]. The other aspect can be a different method of testing frost resistance. Use of water-cured comparative specimens instead of foil-enveloped specimens can also be one of the causes. In every case, further investigation is necessary to determine the causes of the observed results.

4. Conclusions

Alkali-activated materials are considered very suitable from the point of view of sustainable development. This article presented some results which showed that the materials meet many of the requirements—high strength, utilization of secondary materials, low carbon trace, minimum CO₂ emissions, etc. However, these requirements can only be fulfilled if the rules for optimal design of the materials are observed. The rules concerning frost resistance are very important. Just as in other areas (strengths, setting, etc.), good frost resistance can be achieved if

- the optimal ratio between the alkalis and silica in the activator—activation with hydroxide or with the water glass with a high silicate modulus (low R/S)—was not suitable. The optimal R/S was between 50/50 to 70/30;
- the optimal amount of activator—dry mass of the activator higher than 15% seems to be deleterious from the point of view of frost resistance, despite the fact that the strengths of these materials are very high.

The curing results of alkali-activated materials were rather unexpected. Storing 28-day-old specimens in water degrades bending strength, frost resistance, and generally, durability. This aspect requires more attention. This also very significantly affects the results of frost-resistance measurements, and a suitable method must be precisely specified.

As water-cured specimens show poorer bending strengths, a precise specification of the testing procedure for frost resistance of AAMs should be made.

Research aimed at a better and deeper understanding of the above aspects is underway, especially with respect to long-term development and occurrence of microcracks.
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