# INFLUENCE OF CURING CONDITIONS ON ALKALI-ACTIVATED MORTARS INTENDED FOR CONCRETE REPAIR

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## Abstract

Repair mortars are costly materials with high level of Portland cement and various additives which questions their eco-efficiency. In this respect, cement free material solutions such as alkali-activated mortars based on ground granulated blast furnace slag (BFS) are gaining interest for structural repair. The aim of this research is to study blast furnace slag as a precursor for producing ambient cured alkali-activated repair mortars. To achieve this purpose, a total of four mixtures were prepared using four different molar ratios of the silicate solution. The fresh and hardened properties of the produced mortars including flow retention, dry density, compressive and flexural strength are studied comparing ambient-cured and sealed-cured specimens. Furthermore, the tensile bond strength between the repair mortars and a grit blasted concrete substrate was verified (after 7 days sealed + 21 days ambient curing). The results demonstrate, for the tested configurations, the feasibility of the alkali-activated repair mortar, including a good adherence. Given the limited scope of the tests, more work is needed to confirm the observations further.

Keywords: Alkali-activated materials, Geopolymers, Repair mortars, Tensile pull-off bond strength, Curing conditions

## **1. INTRODUCTION**

Over the last century the development of concrete as a building material and its usage exploded, changing the landscapes of the entire planet. Most of the contemporary infrastructure is made of concrete and designed for a life span of 50 to 100 years. A vast amount of these structures reach the end of service life, in a condition more suitable for repair or upgrading, rather than for replacement. Furthermore, concrete structures often experience various environmental attacks, accidental events, lack of maintenance and other actions, which can result in need for repair during their service life. For all of these reasons, in recent years, developed countries often are investing more in rehabilitation and repair than in new construction.

Alkali-activated materials (AAMs) are emerging as future-proof technology of concrete production, as they combine utilisation of industrial by-products and omission of ordinary Portland cement (OPC), hence maximizing environmental benefits of concrete [1]. In some parts of the world, this technology has been utilised in the last century due to shortages of OPC, and in the western world it has been extensively researched over the last 20 years [2]. AAMs typically have rapid strength development, good fire resistance, good bond with OPC substrate, fast setting time, and good acid resistance which makes them attractive as repair mortars [2,3]. This combined with environmental benefits justifies recent increase in development of AAMs in concrete repair [4].

The purpose of this research is to develop alkali-activated repair mortar based on locally available industrial by-products and furthermore to check its potential for structural strengthening of concrete structures by means of textile reinforced mortar (TRM). A previously published repair mortar mix design based on the ground granulated blast furnace slag [3,5] was chosen as a starting point, due to its high flexural and compressive strength and relatively low shrinkage and compatibility of the materials. Concerns had been raised that proper adhesive bond between this repair mortar from substrate was reported in the original study [3,5]. Keeping in mind that the adhesive bond is sensitive to the pre-treatment of the concrete substrate and the application procedure [6,7], authors had decided in favour of replication and further investigation. This paper reports on a verification of the feasibility of BFS based AAM repair mortar according to EN 1504-3 [8], looking into different molar ratios of the silicate activator solution, as well as into curing conditions. Behaviour in terms of workability, strength and bond interaction with concrete substrates are considered.

## 2. EXPERIMENTAL PROGRAM

As a precursor for alkali-activated repair mortars, locally available BFS, provided by Ecocem Benelux is used. The chemical composition of the BFS is determined by X-ray fluorescence (XRF), and presented in Table 1. Particle size distribution (PSD) and specific surface area of BFS is obtained with laser diffraction. Results are shown in Table 2. Commercially available sodium silicate solution (type Crystal 112), with molar ratio of the silicate solution (MS) of about 2; sodium hydroxide flakes with purity higher than 99%; and demineralized water are used to create the aqueous alkali solution. Standard CEN sand compliant with EN 196-1 [9] was used as the aggregate. The mortars are prepared with an aggregate to precursor ratio of 2.42, water to precursor ratio of 0.45 and Na<sub>2</sub>O content (present in the alkali solution) of 5 % per weight of precursor. The MS of the alkali solution is tailored by blending the sodium silicate and sodium hydroxide, while the concentration of the Na<sub>2</sub>O per weight of the precursor was kept the same. Solutions with MS = 0.24 as in [3], MS = 0.46 analogue to [10] and MS = 0.7 and MS = 1 were used as activators.

	Na <sub>2</sub> O	SiO <sub>2</sub>	CaO	Al <sub>2</sub> O <sub>3</sub>	SO <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO
BFS [wt%]	-	31.1	40.9	13.7	2.3	0.4	9.2
Sodium silicate [wt%]	15	29.75	-	-	-	-	-

Activators were created by mixing water, sodium silicate and sodium hydroxide 24 hours in advance, while precursors and sand were stored in laboratory condition together with mixing equipment. Mortars were prepared in a 31 mixer (type Hobart) that complies with EN 196-1 [9]. The aggregate and precursors were added in the mixing bowl and mixed for at least 180 seconds at low speed (140 rpm), then the alkaline solution was added to the mixing bowl slowly over the period of 60 seconds, while mixing at low speed. For the next 30 seconds, the mixing speed is increased to 285 rpm, after which mixing is stopped for 90 seconds; 30 for scraping the material from bowl's bottom and edges with a plastic scoop and another 60 seconds for the mixture to stabilize and rest; than the mixture is again mixed at high speed (285 rpm) for 60 seconds more. Five minutes after mixing, a flow test is conducted to determine consistency of the mortars according to EN 13395-1 [11] (and repeated after 10 and 30 minutes for the determination of the flow retention). Mortars were casted into moulds, which are subdivided into three prisms with following dimensions: length - 160 mm, width and height of 40 mm. The casting process is done in two layers. Every layer is compacted with at least 10 strokes with a tamper after which the mould is vibrated for 15-30 seconds to avoid air cavities.

Table 2: Particle size distribution and specific surface area of the BFS obtained with laser diffraction

	Particl	le size distr	Specific surface area		
	$d_{10}$	d50 (µm)	d90 (µm)	$[m^2/g]$	
BFS	1.238	7.117	20.485	0.655	

After mixing, moulds are covered with plastic sheet and stored for 24 hours. During which specimens were demoulded, 3 of each was tested, and the rest is stored in the following way: part of the specimens was stored at so-called ambient curing (AC), meaning uncovered in a large climate room at 20°C and RH of 60%, and part of the specimens was stored in sealed conditions at the constant temperature of 20°C. The bulk density of mortars, flexural and compressive strengths were determined according to EN196-1 [9]. At least 3 specimens were tested for each time and curing condition. Specimens for testing bond strength of repair mortars are prepared and carried out according to EN 1542 [12]: all mix configurations were applied to a concrete substrate type MC(40) according to EN 1766 [13]. The substrates, 300x300x100 mm, were at least three months old, grit blasted to the roughness of 0.45 mm, thoroughly cleaned and every batch passed quality control in terms of compressive and tensile pull-off strength [14]. Wooden formwork was glued to the concrete to ensure thickness of the overlay of 20 mm. The surface was prewetted 30 minutes before the application (yet moistdry at the surface), and the mortar was hand-applied, with special attention to the creation of the interface layer between old and new material, and covered with plastic sheet. After 24 hours, wooden formwork is removed, specimens were sealed with plastic sheet for 6 more days, after which they were stored at ambient curing. 21 days after casting, 5 cores were drilled in every specimen and 28 days after casting every specimen was subjected to the tensile pull-off tests according to EN 1542 [12]. This bond test comprises direct pull-off of a steel dolly glued to the top of the core repair mortar previously cored through mortar and substrate until 15 mm in the substrate.

## 3. RESULTS AND DISCUSSION

The initial configuration MS 0.24 was designed [3] as a stiff, thixotropic trowel grade mortar suitable for application on vertical surfaces, but with increase of the MS ratio the initial flow increased to the value more characteristic to self-compacting mortars (Figure 1). However, the flow retention in terms of final setting was not influenced by the increase of MS ratio, all configurations were not workable after 30 mins, as previously reported in [3,5].



Figure 1: Flow values of different mortar configurations

The bulk densities of the mortars, are given in Figure 2, and range between 2220 and 2380 kg/m<sup>3</sup>. With the increase of MS ratio, the bulk density increases, which is probably due to increase in the microstructural density of the reaction products in the alkali-activation process [15]. Since bulk density was measured each time on a different set of specimens, no conclusions on weight loss could be drawn, but it is worth mentioning that AC specimens have lower density than their sealed counterparts. This difference is the most prominent on the 28 days old specimens of each configuration.



Figure 2: Bulk densities of tested mortar configurations

Compressive strength of tested configurations and curing regiments are presented in Figure 3. All configurations showed compressive strength above 45 MPa, which is the minimal strength for R4 repair mortars according to EN 1504-3 [8]. With the increase of the molar ratio of the silicate solution, compressive strength increases, from about 45 for MS 0.24 to around 94 MPa for MS 1. The curing condition does not affect the compressive strength of the mortars, with differences between sealed and AC specimens within margins of standard deviation. It does, however, affect flexural strength in all configurations.



Figure 3: Compressive strengths of tested mortar configurations

Ideally, for high-end structural repair mortars, flexural strength of 8 MPa is required [14]. For the reference mix MS 0.24 after 28 days sealed curing, this is approached with 7,5 MPa. This value was lower than 11.4 MPa reported in the original study [3] for the same age and same curing conditions.



Figure 4: Flexural strengths of tested mortar configurations

By increasing the MS, flexural strength of sealed specimens generally increased, yet not necessarily already after 1 day. A 28 days flexural strength of 11,5 MPa was obtained for MS1.0, for sealed curing.

When cured at unsealed ambient conditions, the flexural strength was significantly affected, sometimes with strength reductions of 50% with respect to their sealed counter parts. Only mortar with MS 1.0 achieved value above 8 MPa, while configurations MS 0.46 and MS 0.7 showed values above 6 MPa (Figure 4).

To study the adhesive bond between the repair mortar and an OPC concrete substrate, the 4 mixes were tested further. The failure aspect of the specimens are shown in Figure 5 and bond strengths results are presented in Table 3.

Specimen MS 0.24 had no visible cracks or delaminations before testing. An average pulloff strength of 2.5 MPa was obtained fulfilling criteria for R4 class mortar from EN 1504-3 [8]. It was observed that failure is either on the interface between mortar and concrete or in the substrate itself. This indicates that the bond capacity was close to the tensile strength of the substrate. The clear difference between this and previously reported results, might come from various factors. Time and manner in which the wooden formwork removed might play a role, since it can damage material if removed too early, or constrain shrinkage/expansion and induce microcracks in the interface zone. Treatment of the substrate's surface can also play a role, as the grit-blasted surface facilitates mechanical interlocking between old concrete and repair mortar and prewetting minimises substrate's adsorption of the alkaline solution needed for the alkali-activation of the mortar. However, all of this is speculative and could be further investigated.

Configuration	MS 0.24	MS 0.46	MS 0.7	MS 1
Adhesive bond strength, mean value [MPa]	2.5	≥ 2.1	n.t.*	≥ 1.7

Table 3: Results of the pull-off tensile test

Standard deviation	0.27	0.31	n.t.*	0.30
Failure mode, cumulative for all tests	80% mortar- concrete interface 20% concrete	90% concrete, 10% mortar concrete interface	n.t.*	100% concrete

\*not tested

Specimen MS 0.46 had an average tensile bond strength of 2.1 MPa with no single value lower than 75% of the minimal requirement which fulfils requirement for R4 repair mortar [8]. There was no visible cracking of delamination observed on this specimen either.

Specimen MS 0.7 showed some minor surface cracks that appeared after 7 days, but it was mishandled during the preparation of cores and was excluded from the analysis. This, however, demonstrates the importance of proper preparation of specimens, and subsequently, the importance of handling concrete repairs with care, competence and skill.

Specimen MS1.0 showed surface cracks that appeared after 7 days, yet no delamination was observed. This specimen reached an average pull-off strength of 1.7 MPa, going hand in hand with tensile failure in the concrete substrate. Note that dolly #3 failed during drilling and could not be tested further. Given the fact that the substrate's tensile strength in this case was the weakest part of this system and lower than required by the standard (in spite of the batch passing quality control), no further conclusion can be made of the bond capacity of the repair mortar MS. Tentatively, in reference to a tensile bond strength of minimum 1.7 MPa, this would correspond with at least an R3 class repair mortar. More extensive testing is however needed to come to further conclusions.



Figure 5: Pull-off bond test specimens of the configuration MS 0.24 and MS 1

## 4. CONCLUSIONS AND FUTURE WORK

All mortar configurations showed mechanical properties that are putting them at least in class R3 repair mortars according to EN 1504-3 [8], even when cured in ambient conditions.

In terms of the flow and flow retention, all configurations performed as expected. With the increase of MS ratio, the initial flow of the mixtures increased, with no effect on final setting time. None of the mortars were sufficiently workable after 30 minutes. This remains as a challenge for future work.

Compressive strength was almost not affected by curing conditions, which might indicate that Na<sub>2</sub>O content of 5% per weight of precursor might be the right concentration for BFS based AAMs. In boundary conditions (MS 0.24 and MS 1), AC specimens showed slightly higher compressive strength, still within margins of standard deviations.

Flexural strength is the most affected by the curing condition. Restrained internal shrinkage effects, and associated microcracking in the material, might be a possible cause for this phenomena. This stresses the importance of establishing proper protocols when AAM repair mortars would be applied in practice, and the need for application robust AAM repair mortar mix designs in this respect.

By looking at the results of the pull-off tests, the good bond was observed, even in cracked specimen, indicating suitability of slag-based AAMs for concrete repair and the application in TRM systems. Shrinkage mitigation and evaluation of other durability properties according to EN 1504-3 [8] remains as a challenge for further work.

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