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Effects of Curing Conditions on Shrinkage of Alkali-Activated High-MgO Swedish Slag Concrete

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This study aimed to determine the effects of curing regime on shrinkage of alkali-activated concretes produced from a Swedish high-MgO blast furnace slag. Sodium carbonate (SC), sodium silicate (SS), and their combination were used as alkali activators. The studied curing procedure included heat-treatment, no heat-treatment, sealed and non-sealed conditions. The heat curing increased the compressive strengths of the concretes activated with SS and with the combination of SS and SC. Sealed-curing applied for a period of 1 month reduced the measured drying shrinkage by up to 50% for all studied heat-treated samples. Conversely, the same curing procedure significantly increased the development of the drying shrinkage once the seal was removed after 28 days of curing in the case of the SC-activated concretes non-heat treated. Higher degree of reaction/hydration reached by the binders in these concretes was indicated as the main factor. All of the concretes studied had showed a significant microcracking of the binder matrix, with the most extensive cracking observed in the sealed lab-cured mixes. The heat-cured mixes activated with SS and combination of SC and SS showed the most homogenous microstructure and low extensive micro cracking comparing with lab-cured ones.

Keywords: Alkali activated slag concrete, microstructure, drying shrinkage, curing regime, high MgO granulated blast furnace slag

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INTRODUCTION

Granulated blast furnace slag (GBFS) is a by-product derived from the iron-making process, and is quenched from its molten state to form an amorphous material consisting mainly of a calcium-magnesium aluminosilicate glass. Ground GBFS is also widely used as a supplementary cementitious material in Portland cement—based systems (PC), to lower the hydration heat and to improve the chemical durability of concrete, especially in acid exposures (Chidiac and Panesar, 2008). The use of the alkali-activated slag (AAS) binders to produce concretes enabled up to 75% reduction of the carbon emissions footprint in comparison with plain Portland cement (Yang et al., 2013).

The mechanical properties of AAS concretes are controlled by physical properties and chemical composition of the used slag precursor, the activator type and its dose, curing temperature but also a number of other parameters (Atí et al., 2009; Bernal et al., 2014a; Myers et al., 2017; Criado et al., 2018). The most commonly used alkali activators include: sodium hydroxide, sodium silicate, sodium sulfate, and sodium carbonate (Provis and Van Deventer, 2014; Criado et al., 2018).
The setting times of AAS pastes activated by sodium silicate and sodium carbonate were found to be significantly longer than that of the OPC. On the other hand, slag paste activated with sodium carbonate had similar setting behavior as the PC-based concretes (Atiš et al., 2009).

AAS concretes showed good resistance to acid attack, fire exposure and corrosion (Jiang et al., 1997; Chidiac and Panesar, 2008; Karahan and Yakupoğlu, 2011; Mundra et al., 2017). Most AAS concretes activated by sodium silicate or sodium hydroxide showed higher early and flexural strength (Bakharev et al., 1999), but they are more sensitive to the curing temperature than PC. The apparent activation energy is higher but it develops lower hydration heat and faster reaction rates (Shi and Day, 1996; Shi et al., 2006; Aydin and Baradan, 2012; Provis et al., 2015).

In general AAS develop higher shrinkage in comparison with PC concretes (Allahverdi et al., 2017; Humad et al., 2018; Orosz et al., 2019). In the case of the SS activated GGBFS concretes, the high shrinkage was linked with the presence of more mesopores (Shi, 1996) which led to the increased tensile stresses and thus to a greater drying shrinkage (Häkkinen, 1993). SS-activated slag mortars developed up to six times higher drying shrinkage in comparison with mortars based on PC mortars (Atiš et al., 2009). Conversely, SC-activated slag showed lower or equal drying shrinkage values in comparison with PC based systems. The shrinkage kinetics appeared to be slower at a similar RH (Zheng, 2010; Ye and Radlinska, 2016). The hydration products of the AAS included C-S-H gel with a lower Ca/Si ratio than that in PC, with a Si-rich gel, containing higher amount of un-combined water content. This un-combined water would evaporate during the drying process, subsequently causing a significant shrinkage (Collins and Sanjayan, 2000; Ye and Radlinska, 2016; Ye et al., 2017). Shrinkage of the ASC is strongly influenced by the applied curing conditions. For example exposure to the relative humidity (RH) between 50 and 70% was indicated as especially unfavorable (Douglas et al., 1992; Bakharev et al., 1999; Krizan and Zivanovic, 2002; Provis and Van Deventer, 2014; Ye et al., 2017). A partial replacement of BFS with FA tended to lower the drying shrinkage (Wallah and Rangan, 2006; Singh et al., 2016; Humad et al., 2019). Decrease of the NaO to SiO₂ molar ratio of the SS alkali activation reduced the drying shrinkage values, but increased the autogenous shrinkage (Krizan and Zivanovic, 2002; Atiš et al., 2009; Humad et al., 2019).

Heat treatment can significantly accelerate the strength development of AAS concrete, and has been observed to reduce the ultimate shrinkage values in comparison with the PC concrete (Bakharev et al., 1999). The heat curing resulted in a coarser porosity and thus decreased drying shrinkage (Bakharev et al., 2000; Ismail et al., 2013). A similar effect was observed while using shrinkage reducing admixtures (SRA). In that case, the drying shrinkage was reduced by formation of a coarser pore structure which led to the reduction of the pore fluid surface tension (Palacios and Puertas, 2007).

The high MgO content GGBFS, which is the focus of the research results presented in this publication, can be found only in few locations around the world. Sweden is one example where all locally produced GGBFS has MgO content between 12 and 19-wt.%. Previous studies showed that the reaction chemistry and the developed microstructure are remarkably altered in those systems with a possible strong effect on shrinkage. The incorporation of Al in the C-S-H type gel which is typical for AAS systems was reduced at a high BFS MgO content. The available Al was strongly consumed through the formation of hydrotalcite-group minerals (Ben Haha et al., 2011). An increase of the MgO content in AAS systems activated by SS, enhanced the formation of hydrotalcite and formed a more disordered silicate gel microstructure (Ben Haha et al., 2011; Bernal et al., 2014b). Increasing the MgO content in alkali-activated slag system showed increased carbonation resistance (Bernal et al., 2014b; Park et al., 2018).

The problem of an excessive drying shrinkage in AAS concrete is known and has been studied well relatively for binder systems based on commonly available low MgO content blast furnace slags. On the contrary, the amount of the available data related to AAS based on high MgO content BFS is rather limited. The present research aimed to fill in this lack of knowledge and focused on effects of various curing conditions, mechanical properties, and microstructure on shrinkage of a typical high MgO blast furnace slag available in Sweden on the development of drying and autogenous shrinkage.

**MATERIALS AND METHODS**

A (GBFS), type Merit 5000 (MEROX, Sweden), was used as a main binder in this study. Its chemical composition determined using a PANalytical-Zetium XRF spectrometer is shown in **Table 1**. The physical properties were provided by the supplier, Sodium carbonate and sodium silicate were used as alkali activators. The powdered sodium carbonate (SC) was provided by CEICH SA from Poland while the liquid sodium silicate (SS) by the PQ Corporation. The SS as supplied had the alkali molar ratio \( M_s = \frac{\text{mass ratio SiO}_2/\text{Na}_2\text{O}}{2.2} \), with 34.37 wt.% \( \text{SiO}_2 \), 15.6 wt.% \( \text{Na}_2\text{O} \) and solids content of 49.97 wt.%. The \( M_s \) value was adjusted to 1.0 by addition of sodium hydroxide pellets (98% purity), with 76.31 wt.% \( \text{Na}_2\text{O} \). The alkali activator dosage varied between 5 and 10 wt.% of the total binder content, **Table 2**. The BFS content in all concrete mixes was 450 kg/m³, and the total water to binder (w/b) ratio was 0.45. Jehander group from Sweden provided coarse granite aggregates (size 4–8 mm) and fine aggregates which grading curves are shown in **Figure 1**. The fine aggregate content in the concrete mix was about 80 wt.% of the total aggregate amount. Liquid sodium silicate and powder sodium carbonate were dissolved in the mixing water 24 h before production of concrete. All dry ingredients including GBFS, coarse and fine aggregates were mixed for 1 min followed by an addition of the mixing water containing alkali activators and mixed for another 3 min. A rotating pan mixer type Zyklos-ZZ75HE was used to prepare all the mixes.

Initial and final setting times were determined using the Vicat apparatus, following the ASTM C191-13 standard. Flexural strength was determined on 28-day old beams of 100 × 100 × 500 mm³, in three point bending following the SS-EN 12390-5:2009 standard. The applied load rate was 160 N/sec. 7 and 28-days compressive strengths were determined as average values from

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**Table 1** Physical properties were provided by the supplier.

**Table 2** BFS content in all concrete mixes was 450 kg/m³, and the total water to binder (w/b) ratio was 0.45.
### TABLE 1 | Details of GGBFS used in this work.

| Component | CaO | SiO₂ | Al₂O₃ | Fe₂O₃ | MgO | Na₂O | K₂O | TiO₂ | MnO | SO₃ | L.O.I |
|-----------|-----|------|-------|-------|-----|------|-----|------|-----|-----|------|
| Composition represented as oxides wt.% | 30.4 | 35 | 14.3 | 0.3 | 16.1 | 0.6 | 0.7 | 2.8 | 0.5 | 0.7 | 0.9 |
| Physical data | Specific surface(cm²/g) | 5,000 | | | | | | | | | |
| | Bulk density (kg/m³) | 1,100 | | | | | | | | | |
| | Particle density (kg/m³) | 2,950 | | | | | | | | | |

### TABLE 2 | Mix proportions of concretes.

| Mix ID | Binder (GGBFS) content kg/m³ | Total w/b ratio | Aggregate content (0–8 mm) kg/m³ | Activator type, % dosage as solid materials | Curing condition | pH value of the activator solution | Slump results in mm |
|--------|-----------------------------|-----------------|----------------------------------|-------------------------------------------|-----------------|-----------------------------------|---------------------|
| SC10 L | 450                          | 0.45            | 1,663                            | 10% SC                                    | Lab curing      | 11.2                              | 80                  |
| SC10 H | 450                          | 0.45            | 1,663                            | 10% SC                                    | Heat curing     | 13.7                              | 250                 |
| SS10 L | 450                          | 0.45            | 1,663                            | 10% SS, Mn=Mₛ = 1                          | Lab curing      | 13.3                              | 180                 |
| SS10 H | 450                          | 0.45            | 1,663                            | 10% SS, Mn=Mₛ = 1                          | Heat curing     |                                    |                     |
| SC5 + SS5 L | 450               | 0.45          | 1,663                           | 5% SC+5% SS                               | Lab curing      | 11.2                              | 80                  |
| SC5 + SS5 H | 450               | 0.45          | 1,663                           | 5% SC+5% SS                               | Heat curing     |                                    |                     |

SS, sodium silicate; SC, sodium carbonate; L suffix after mix refers to laboratory-cured mix; H suffix after mix refers to heat-cured mix.

### FIGURE 1 | Grading curve of combined coarse and fine aggregates.

### FIGURE 2 | Initial and final setting times of AAS concrete mixes.

Shrinkage was determined using cylinders having a diameter of 100 mm and a height of 200 mm. The shrinkage measurements started 1.5 days after casting for the heat-cured sample and after 2–3 days for the laboratory-cured samples, depending on the measured final setting time, Figure 2. All samples were subsequently stored in laboratory conditions. Strain values were recorded using a strain gauge DEMEC type product by Mayes Instruments Limited. Two pairs of stainless steel measuring discs were glued with an epoxy resin onto each cylinder and were spaced at 100 mm apart.

The curing procedure applied to samples used for the determination of the compressive and flexural strength tests included sealing with plastic bags followed by storage of the specimens in the laboratory conditions at 20 ± 2°C and 40 ± 3% RH until testing. The second procedure included heat curing at 65°C for 24 h applied immediately after casting followed by a storage in the laboratory at 20 ± 2°C and 40 ± 3% RH. Samples used for shrinkage tests determination were subjected to two additional curing procedures, Table 3. The third procedure included sealing only for 2–3 days after hardening. After seal removal samples were stored at 20 ± 2°C and 40 ± 3% RH (L unsealed), Table 3. Heat curing of sealed samples at 65°C for 24 h was applied in the 4th variant and was followed by removal of the seal and storage of the specimens in the laboratory conditions at 20 ± 2°C and 40 ± 3% RH (H unsealed).

“Microstructure and microchemistry of hardened samples were studied using scanning electron microscope (SEM) model JSM-IT100 combined with a QUANTAX EDX (Energy-dispersive X-ray spectroscopy) analysis system from BRUKER and the ESPRIT 2 software. Concrete samples for the SEM analysis were cut from 28-days old specimens. Moisture was moved
from all specimens using an alcohol exchange techniques where sample were stored in alcohol for 48 h. In the following step, specimens were moved to the low vacuum impregnation chamber and impregnated with low viscosity resin. No heat drying of the samples was applied to maximally limit formation of microcracks. After curing for 24 h resin impregnated samples were grinded and polished in steps using polishing spray containing 9, 3, and 1 \( \mu \)m synthetic diamond particles. During polish a load of 35 N was applied in the polishing head. The SEM was operating at using 15.0 kV accelerating voltage and probe current of 61–72 mA."

**RESULTS AND DISCUSSION**

The shortest initial setting times were measured for mixes activated with 10 wt.% of SC and 10 wt.% of SS (SC10, SS10, respectively). The longest final setting times were observed for mixes activated with 10 wt.% SC and the combination of 5 wt.% of SC + 5 wt.% of SS (SC10 SC5 + SS5), Figure 2. These results can be explained by a high concentration of calcium carbonate and high MgO content which resulted in formation of gaylussite, hydrotalcite and calcite (Humad et al., 2018). Earlier studies showed that a presence of calcite CaCO\(_3\) tends to shorten the initial setting time rapidly and decreases the viscosity of the fresh mix. At the same time, it extended the final setting due to slower reaction rates and a longer induction period. The pH values of those systems were also observed to be significantly lower, (Sariibrahimoglu et al., 2012; Provis and Van Deventer, 2014; Bernal, 2016).

The flexural strength of the AAS concrete beams reached between 4.5 and 6.0 MPa with slightly lower values for the heat-cured samples, Figure 3. The highest 7- and 28-day compressive strength was measured for the heat-treated samples activated with 10 wt.% SS, while the lowest for the heat-cured sample activated with 10 wt.% SC (mix SC10 H), Figure 4. There is no evident correlation between compressive and flexural strength among tested samples: Few mixes had relatively high compressive strengths but showed lower flexural strengths, which could be related to microcracking of the binder matrix. Similar trends were also observed by others in the PC based systems (Wedatalla et al., 2019). The chemical composition and particles size distribution strongly affects the kinetics reaction and the evolution of solid structural phases forming in alkali-activated slag (AAS) system. At high MgO contents (MgO > 5%), the hydrotalcite phases and the C-(N)–A–S–H type gels were observed to be formed (Bernal et al., 2014b). The lowered Al uptake by the C-S-H resulted in an increased 28-days compressive strength. An increasing MgO content of SH- and SS-activated slag pastes showed more rapid strength development and higher compressive strength at early and late hydration times (Ben Haha et al., 2011). The highest increase of the compressive strength between 7 and 28 days was observed for the non-heat-treated samples activated with 10 wt.% SS (mix SS10 L) while the lowest for the heat-treated samples activated with 5 wt.% SC and 5 wt.% SS (mix SC5 + SS5 H). A lack of the moisture required to continue the reaction of GBFS could be indicated as one of the possible reasons (Collins and Sanjayan, 2001). Higher initial hydration/reaction rate observed in heat-cured mixes followed by its significant slowdown can be also related to the formation of a dense impermeable binder matrix surrounding the unreacted particles. The efficient amount of available GBFS will be limited. The formed binder matrix will
be also less homogenous with larger amount of pores (Helmuth and Verbeck, 1968; Shi et al., 2006).

The used curing procedure aimed to simulate real life conditions where sufficient control is only possible for a limited amount of time. The main difference between the sealed and unsealed samples was the ultimate contribution of the autogenous and drying shrinkage. The sealed curing hindered the development of the drying shrinkage and produced a strong and dense binder matrix able to resist volumetric changes without extensive microcracking. Contrary, the used unsealed curing enabled a development of both types of shrinkage. The measured shrinkage values are shown in Figures 5–7. In general, concretes activated with the sodium carbonate, had a significantly lower shrinkage in comparison with mixes activated with the sodium silicate. The lowest shrinkage of 1.1 mm/m was measured on SC-activated slag concrete (mix SC10 H sealed) subjected to the heat-treatment, which was followed by one-month curing in sealed conditions, Figure 5. Mixes activated with 10 wt.% SS and 5 wt.% SC + 5 wt.% SS, subjected to the same curing procedure developed 2.4 and 1.5 mm/m of shrinkage, respectively. Those higher values can be related to more intensive chemical reactions
triggered by a high pH values. The formed microstructure was finer presumably also leading to intensive self-desiccation as observed by others (Zhang et al., 2013; Mosale Vijayakumar, 2014). The number of empty pores most probably increased due to more intensive reactions which eventually resulted in a higher shrinkage (Neto et al., 2008).

The applied heat treatment decreased the measured shrinkage. In the case of mixes activated with a combination of 5 wt.% SC + 5 wt.% SS the maximum reduction reached 41%. The heat treatment presumably produced coarser microstructure of the binder matrix which lead to lower tensile strains induced by the evaporation of the pore water and lower shrinkage values (Bakharev et al., 2000; Collins and Sanjayan, 2000; Ismail et al., 2013). As described earlier, a higher curing temperature most probably lead to a more intensive formation of reaction products around slag particles thus limiting or fully inhibiting its further dissolution which provides all required for the solidification reaction species (Bakharev et al., 1999). The heat-cured samples
showed only a moderate enhancement of the measure ultimate compressive strength, which further supports that mechanism. A similar effect of the high curing temperature was observed in Portland cement based concretes (Helmuth and Verbeck, 1968). A significant and unfortunately, unmeasured proportion of the shrinkage developed already during the heat treatment. The application of the 1-month sealed-curing reduced the measured shrinkage by up to 61% in the case of concretes activated with 10-wt.% SC, Figure 5. A combination of the heat treatment and the 1 month sealed curing reduced the measured shrinkage by up to 50% for the mix SC5 + SS5, Figure 7. The sealed curing presumably additionally increased the tensile strength of the binder matrix and decreased its carbonation (Ma and Dehn, 2017).

The SEM-BSE analysis showed an intensive microcracking of the binder matrix. In the analysis of the obtained images only cracks filled with resin were considered which excluded crack, which could form due to vacuum conditions present in the SEM specimen chamber. In general, the microcracking was more extensive in non-heat-treated samples, which can be related with the high early-age shrinkage, Figure 8. Conversely, the heat-cured samples showed less microcracking, especially the mixes SS10H and SC5 + SS5H due to stronger matrix and coarser microstructure.

Efflorescence was observed in non-heat-treated samples during de-molding, Figure 9. The phenomenon was caused by a migration of sodium hydroxide from the pore solution to the surface of samples during drying and its subsequent reaction with atmospheric carbon dioxide. As a result, the sodium carbonate was formed (Higgins, 1982). The hydroxide remains water-soluble only for a short period after the exposure to the atmosphere as it reacts with CO₂ to form carbonate salts (Higgins, 1982). The observed strong tendency of AAS binders toward the efflorescence can be related to the high alkali concentration in the pore solution. In certain alkali activated concretes, this can be exaggerated by an open microstructure caused by for example a lower reaction degree, and a weak exchangeable binding of Na (Wang et al., 1995; Lloyd et al., 2010; Allahverdi et al., 2017; Longhi et al., 2019). Earlier studies showed that in SC-activated binders, calcium and sodium-calcium carbonates tend to form at early stages rather than the strength-defining C-A-S-H phases (Fernández-Jiménez et al., 2003). After 28 days, a highly cross-linked C-A-S-H providing a higher strength phase was reported to form (Fernández-Jiménez et al., 2003; Xu et al., 2008).

The high MgO content of the slag tends to affect the early-age properties of the binder matrix. Earlier studies, also performed by the authors, indicated formation of hydrogarnet-like Mg-Al layered double hydroxides (Bernal et al., 2016; Humad et al., 2018; Abdollahnejad et al., 2019). The consumption of Al may also contribute to the strength development. The phase composition at each age can be controlled by the consumption of CO₃²⁻ species from the activator. For example, calcium carbonates are formed using Ca²⁺ released from the GBFS. Carbonates can be up taken by the Mg-Al layered double hydroxide phases. When the CO₃²⁻ species are consumed, the pH will increase and the reaction is believed to continue similarly as in the case of binders activated with sodium hydroxide (Xu et al., 2008; Bernal et al., 2015). In the present study, the applied heat curing (at 65°C) affected the dissolution process of the GBFS, and thus also the availability of Ca²⁺ and CO₃²⁻ at the early age. Although, not verified experimentally, it is very likely that the dissolution rate of GBFS was higher in the heat-cured samples thus leading to a more intensive formation of calcite and gaylussite. The formation of gaylussite was shown earlier to be predominant at an early age, in high-MgO AAS binders (Humad et al., 2018). The high sodium carbonate content in the mix SC10 provided an excess of Na ions, which supported the formation of gaylussite Na₂Ca(CO₃)₂.5H₂O (Allahverdi et al., 2017). In the next stage, the gaylussite started to dissolve and changed to more stable phases besides the formation of C-A-S-H was initiated (Allahverdi et al., 2017; Humad et al., 2018).

The results from the SEM-EDX analysis are shown in the form of ternary diagrams; CaO, Al₂O₃ and SiO₂, Figure 10.
CONCLUSIONS

High MgO content Swedish GGBFS was studied to determine effects of curing conditions on the development of shrinkage of alkali activated concretes. Additionally, setting times, microstructure, chemical composition and compressive strength were determined as well. Four different curing procedures were evaluated including heat-treatment and no-heat-treatment in combination with sealed and unsealed conditions. The obtained results confirmed that all measured parameters were affected by the applied curing procedure. The non-heat treated sealed-cured samples produced higher 28-day compressive strengths when activated with sodium carbonate. The application of the heat treatment combined with the sealed-curing for those concretes resulted in lower strengths. All non-heat-treated and unsealed samples showed higher shrinkage, while the application of the combined heat-treatment and sealed-curing reduced these values by 30–50%. The reduction was related to the development of more intensive hydration/geopolymerization reaction leading to a denser binder matrix having a higher tensile strength. Consequently, the drying and carbonation shrinkage were reduced by sealing. The heat-treated samples showed less micro-cracked and coarser microstructure additionally indicating higher tensile strength of the binder matrix.

All presented results are based only one high MgO BFS which limits to formulate generalized conclusions. Therefore, the future research should focus on testing slags with variable amounts of MgO.

DATA AVAILABILITY STATEMENT

All datasets generated for this study are included in the article/supplementary material.

AUTHOR CONTRIBUTIONS

AH: did all tests, organized the database, performed the statistical analysis, and wrote the first draft of the manuscript. JP and AC: revision read and approved the submitted version.

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Conflict of Interest: The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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