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Peer-reviewed author version

GIJBELS, Katrijn; Pontikes, Yiannis; Ion Iacobescu, Remus; SCHREURS, Sonja & SCHROEYERS, Wouter (2017) Mechanical properties of alkali-activated materials based on blast furnace slag and calcium sulphate dihydrate. In: Ion Iacobescu, Remus; Malfiet, Annelies (Ed.). Proceedings of the 5th International Slag Valorisation Symposium: From fundamentals to applications, Department of Materials Engineering, KU Leuven, p. 353-356.

Handle: <http://hdl.handle.net/1942/23957>

MECHANICAL PROPERTIES OF ALKALI-ACTIVATED MATERIALS BASED ON BLAST FURNACE SLAG AND CALCIUM SULPHATE DIHYDRATE

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Introduction

Alkali-activated materials (AAMs) are currently under research for providing opportunities for re-use of industrial residue streams in the construction sector. The valorisation of industrial residues into sustainable building materials has become an important feature for reducing the need of disposal and avoid the scarcity of primary resources. The use of ordinary Portland cement (OPC) as primary construction material has also been questioned extensively due to its environmental impact, i.e. CO₂ emission during production. The aforementioned issues have prompted various researches in an attempt to develop AAMs as sustainable alternative binders. The most popular industrial solid residues used in making AAMs are: ground granulated blast furnace slag (GGBFS), steel slag, phosphorus slag, fly ash, bauxite residue, among others¹. In some cases (phospho-)gypsum and flue gas desulfurisation gypsum are also used^{6,7,8}. This wide range of solid residues provides a wealth of potential precursors for AAMs¹. GGBFS, produced during iron production, is an excellent raw material for making high-strength building materials for civil engineering applications². The slag's main components are CaO, SiO₂, and Al₂O₃, available in a glassy state. GGBFS is used in a wide range of applications³, in which some of these contain a significant amount of sulphate (e.g. flue gas desulfurisation gypsum)^{6,7,8}. For this reason, GGBFS-based AAMs serve here as a test matrix in order to allow the incorporation of sulphate-rich residues. Alkali silicate based activator is the most used activating solution for GGBFS based AAMs¹. The modulus (M_s) of the activator solution (molar ratio SiO₂/Na₂O) is an important parameter for mechanical strength and is usually optimal between a ratio of 1 to 2⁴. It is the aim of this paper to investigate the influence of curing regimes on the mechanical properties of AAMs, focussing solely on pastes based on GGBFS and calcium sulphate dihydrate.

Experimental procedure

The materials used in this study were GGBFS, delivered by a Belgian steel company, and calcium sulphate dihydrate ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ - CS). CS, analytical reagent, was provided by Sigma-Aldrich. The alkali activator was a combination of sodium silicate solution (molar ratio $\text{SiO}_2/\text{Na}_2\text{O}$ 3.3 and 65% water, supplied by ABCR GmbH), sodium hydroxide pellets (grade 99%, supplied by Chem-Lab), and water. The chemical composition of GGBFS was determined by X-ray fluorescence analysis and consists of (in wt%): 36.2 SiO_2 , 40.3 CaO , 11.4 Al_2O_3 , 8.2 MgO , 1.1 S and 0.8 TiO_2 . The density of the slag was measured according to the ASTM C204 method and found to be 2.9 g/cm^3 . Milling was performed to a blaine specific surface of $4050 \text{ cm}^2/\text{g}$, determined according to EN 196-6. The alkali activator ratio used of $\text{H}_2\text{O}/\text{Na}_2\text{O}$ and $\text{SiO}_2/\text{Na}_2\text{O}$ is given in Table 1, wherein the mix design as well the curing regimes are also presented. Precursors (90 wt% GGBFS and 10 wt% CS) were mixed with the alkali activator and were homogenised for one minute by hand mixing. One sample with 100 wt% GGBFS was made as reference. Samples A, B, and C were prepared to a L/S ratio of 0.6, whereas the reference sample had a ratio of 0.4 to keep a good paste consistency. No aggregates were used. The fresh paste mix was cast in polymer coated steel moulds (20mm x 20mm x 80mm). Three curing regimes were tested in a laboratory oven for 24h, having the samples always wrapped to prevent drying: (A) at $20 \text{ }^\circ\text{C}$, (B) at $60 \text{ }^\circ\text{C}$, and (C) at $80 \text{ }^\circ\text{C}$. Then, the samples were demoulded and transferred for further curing till 28 days into sealed storage vessels at $20 \text{ }^\circ\text{C}$.

Table 1: Mix design of samples

Sample	$\text{H}_2\text{O}/\text{Na}_2\text{O}$	$\text{SiO}_2/\text{Na}_2\text{O}$	GGBFS (g)	CS (g)	Curing for the first 24h	L/S
Reference	20	2	50	0	$20 \text{ }^\circ\text{C}$	0.4
A	20	2	45	5	$20 \text{ }^\circ\text{C}$	0.6
B	20	2	45	5	$60 \text{ }^\circ\text{C}$	0.6
C	20	2	45	5	$80 \text{ }^\circ\text{C}$	0.6

After 28 days, specimens were tested for their mechanical properties using the 250 kN load cell testing machine in accordance with EN 196-1. SEM was used for microscopical observation. Specimens were taken from the middle of the sample. They were embedded in epoxy resin, polished and coated with a 5 nm layer of Pt.

Results and discussion

Figure 1 shows the compressive strength mean values with a 95.4% confidence interval. For the reference sample, a high strength was obtained, compared to values in literature⁵. The incorporation of 10 wt% CS indicates a reduction in compressive strength when cured at $20 \text{ }^\circ\text{C}$; however, changing the

curing temperature results in an increase in strength. Still, even the best formulation has approximately 26 % lower strength compared to the reference one.

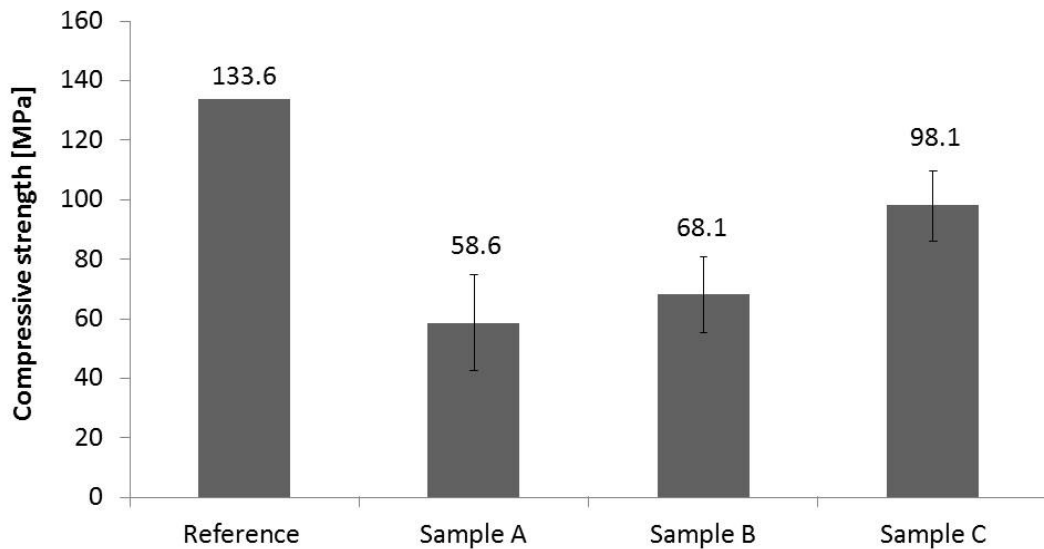


Figure 1: Mechanical properties of reference sample and samples A, B, and C cured at different temperatures

Backscattered images of samples A, B, and C are shown in Figure 2. It can be seen that higher curing temperature (i.e. 80 °C) results in a denser microstructure with less microcracks compared to curing at lower temperatures (i.e. 20 °C and 60 °C). This is attributed most likely to the faster kinetics taking place with respect to polymerisation and strength gain for the matrix, and is the main reason for the high compressive strength demonstrated.

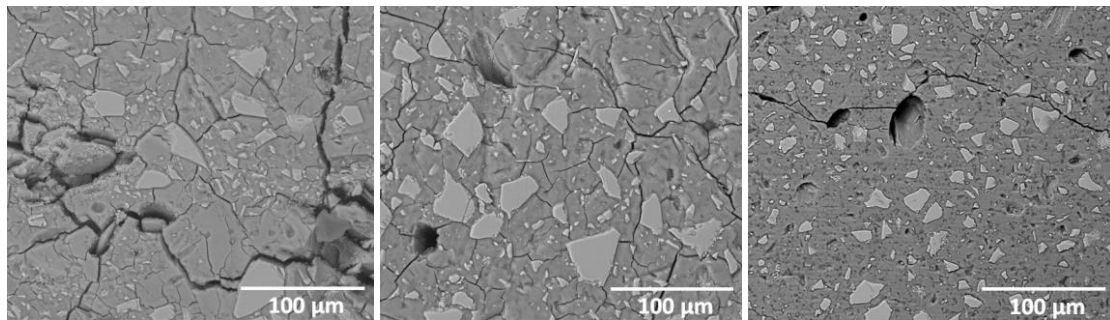


Figure 2: BSE at 100x magnification for different curing regimes (figure left 20 °C, figure middle 60 °C, figure right 80 °C)

Conclusion

This paper investigated the influence of curing regimes (i.e. 20 °C, 60 °C, and 80 °C) on the mechanical properties of AAM pastes made from 90 wt% GGBFS and 10 wt% CS as precursors. Addition of CS resulted in a decrease in strength; however, by

optimising the curing (i.e. 80 °C) the strength increased. SEM microscopical observation indicated a denser microstructure with less microcracks for curing at 80 °C compared to curing at lower temperatures (i.e. 20 °C and 60 °C). In further research, additional aspects with respect to processing (e.g. setting time) and microstructure and performance will be investigated.

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