

ALKALI ACTIVATION OF SYNTHETIC $\text{SiO}_2\text{-CaO-FeO}_x\text{-Al}_2\text{O}_3\text{-MgO}$ GLASS

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Introduction

Towards a low carbon circular economy, intense research has led to alternative binders, such as geopolymers, coming from aluminosilicate sources.¹ A multitude of work in this area demonstrates a number of possible secondary resources that can be used as precursors of these novel binders. Some examples are bauxite residue², fayalitic slag³ and ferronickel slag⁴. Apart from the aforementioned resources, research has also been carried out on municipal solid waste incinerator (MSWI) ashes for their potential to be used as precursors for the synthesis of inorganic polymers (IP).⁵ The current work aspires to contribute to the field of using MSWI bottom ash (BA) as a standalone precursor for IP synthesis. This will eventually enable the upcycling of the ashes coming from MSWI plants, with a view to their utilisation in novel materials.

Continuing on this line, there are many factors affecting the performance of the IPs in terms of compressive strength, such as the activating solution and its water content. Increase in compressive strength of the IP can be achieved by increased concentrations of both alkalis and silicates. However, there is an optimal value for the alkaline concentration, while surpassing will lead to lower compressive strength.^{4,6} Similarly, diluting the activating solution also leads eventually to decreased mechanical strength.⁷

To gain a better understanding on using MSWI BA as a precursor for the IPs, the effect of different $\text{SiO}_2/\text{Na}_2\text{O}$ (0.8 and 1.6) and $\text{H}_2\text{O}/\text{Na}_2\text{O}$ (16 and 25) molar ratios of activating solutions were investigated, regarding the kinetics and compressive strength. Since MSWI BA from incineration plants have variable and unpredictable composition, the current work is based on using as a precursor a $\text{SiO}_2\text{-CaO-FeO}_x\text{-Al}_2\text{O}_3\text{-MgO}$ synthetic glass, which replicates a certain MSWI BA chemistry. Additional data points will eventually create the envelope of possible chemistries one could encounter for MSWI BA.

Materials and methods

In the present work, the precursor that was used was a synthetic glass (SG) made by mixing quartz, metallic iron, iron (III) oxide and calcium oxide, and subsequent melting. The composition of the glass was designed to represent the glass coming from the vitrification process of BA, as previous work did for representing non-ferrous metallurgy slag⁸. Melting of the starting mixture took place in an induction furnace (Indutherm TF4000), using an Al₂O₃ crucible. The heating temperature was chosen to be 100°C above the one calculated by Factsage 7.0 software, using the databases FactPS and FToxid. Melting was performed under air atmosphere and subsequently the melt was quenched in water. The produced glass was afterwards milled in a Fritsch pulverisette 13 disc mill, until a Blaine specific surface of 4000 ± 200 cm²/g was achieved. The chemical composition was determined by X-ray Fluorescence (XRF) spectrometer (Philips PW2400). The mineralogy was evaluated by quantitative X-ray powder diffraction analysis (QXRPD, D2 Phaser, Bruker AXS). For the quantification, 10 wt% of zincite (ZnO) was added to the powder glass as a standard material. The blend was mixed for 7.5 min (McCrone micronising) in ethanol. The quantitative analysis was performed using Topas Academic software.

Inorganic polymer pastes were prepared by mixing different activating solutions SiO₂/Na₂O (0.8 and 1.6) and H₂O/Na₂O (16 and 25) molar ratios with the powder glass, under the liquid/solid ratio of 0.4. Curing was performed in sealed plastic boxes at 20°C. Calorimetry measurements were performed in a TAM Air Isothermal Calorimeter at 20°C. The compressive strength of the paste samples was tested for 2 and 7 days (Instron 5985 testing machine) using a 250 kN load cell.

Results and discussion

The chemical composition of the synthetic glass (denoted by SG) as well as the amorphous content are presented in Table 1. The liquidus temperature was calculated using FactSage and found to be 1265°C.

Table 1: Chemical composition of the synthetic glass, based on XRF data (relative error of semi quantitative analysis: 10%), the calculated liquidus temperature TL, and the amorphous content by XRD (estimated relative error is 10%)

	SiO ₂	CaO	Fe ₂ O ₃	Al ₂ O ₃	MgO	T _L (°C)	Amorphous
SG	48.9	22.3	11.6	10.5	2.1	1265	96

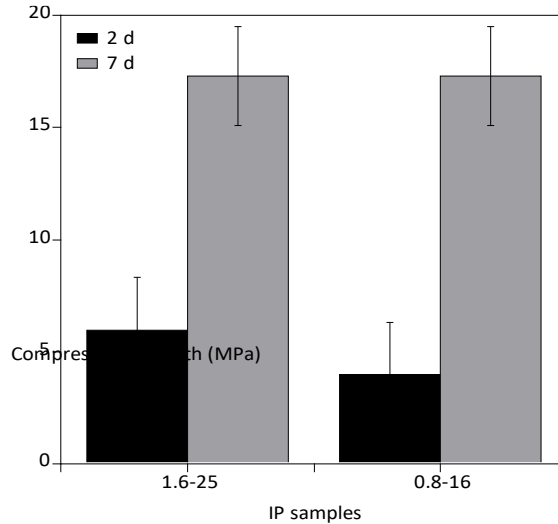


Figure 1: Compressive strength of the inorganic polymers for 2 days and 7 days; The error bars represent the standard deviation on the data

The compressive strength for the IPs is presented in Figure 1. While the early strength of the 1.6-25 IP is higher (7 MPa) than the one for the 0.8-16 IP (4 MPa), the 7 days strength increases significantly for both IPs.

Isothermal calorimetry measurements, presented in Figure 2, were performed to assess the reactivity with respect to heat release. The peaks observed in less than one hour for both IPs, are related to the dissolution and, because the onset of setting was observed within this timeframe, the formation of the polymeric network. The heat flow abruptly decreases after the initial reaction peak. Nevertheless, it should be noted that the reaction kinetics could not be directly correlated to the mechanical properties of the produced IPs.

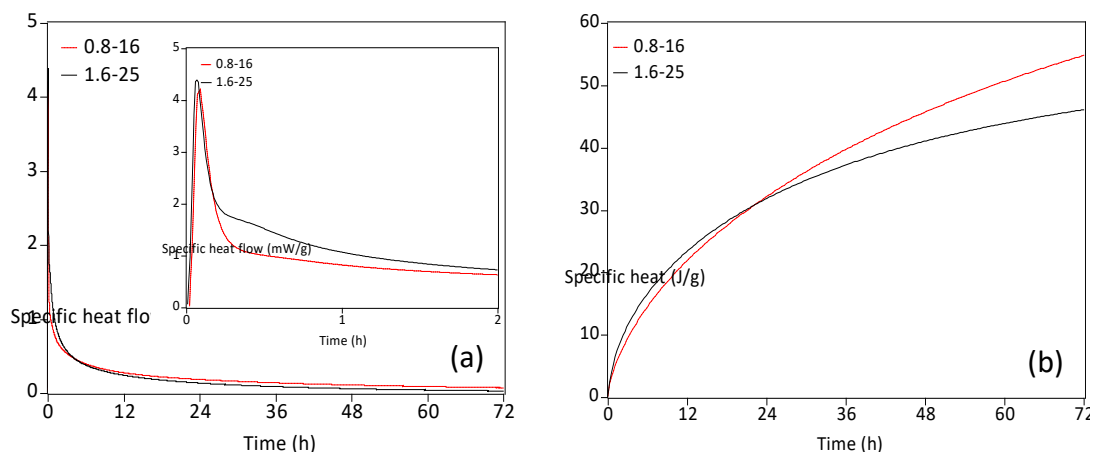


Figure 2: Specific heat flow (a) and (b) specific heat in isothermal calorimetry at 20°C during the first 70 h after mixing, normalised by the weight of the slag

Conclusions

The influence of the activating solution on a synthetic glass, replicating the chemistry of the MSWI BA, was studied by varying the $\text{SiO}_2/\text{Na}_2\text{O}$ and $\text{H}_2\text{O}/\text{Na}_2\text{O}$ molar ratios. The early age compressive strength (2 days) appears to be influenced by the ratio $\text{SiO}_2/\text{Na}_2\text{O}$, while no effect is observed on the 7 days strength. Independently from the activating solution employed, calorimetry measurements showed fast dissolution and polymerisation. Ongoing work will shed more light on the evolution of the compressive strength in 28 days and reveal the main reactions taking place.

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