A simple screening test of alternative pozzolanic materials

Ingemar Löfgren
M.Sc., Ph.D., adjunct professor
Thomas Concrete Group & Chalmers University of Technology

e-mail: Ingemar.lofgren@C-lab.se

ABSTRACT

In this paper, results from an experimental investigation of alternative supplementary materials are presented. In the experiments, the compressive strength as well as consumption of calcium hydroxide and chemically bound water was determined on mortar prisms which provided useful information about the pozzolanic reactivity.

Key words: Binders, Supplementary Cementitious Materials, Pozzolanic, Sustainability, Strength Activity Index, Testing.

1. INTRODUCTION

1.1 General

With an increasing focus on sustainability there is an urgent need for alternative supplementary cementitious materials (SCMs) to complement the traditional ones already used, e.g. coal fly ash (FA) and ground granulated blast furnace slag (GGBS). Currently there is no EN standard available for natural or calcined pozzolans, but in UK two standards were published in 2019 [1] & [2]. In the cement standards EN 197-1 [3], however, pozzolanic materials (natural and natural calcined) are accepted as a main constituent but there is no requirement on the chemical composition other than that they should have “pozzolanic properties” and consist essentially of reactive silicon dioxide (SiO_2) and aluminium oxide (Al_2O_3), together with iron oxide (Fe_2O_3) and other remaining oxides. The only “strict” requirement is that the reactive silica content should not be less than 25.0 % by mass, but it is not stated how this should be tested. In EN 196-5 [4] a method for assessing pozzolanicity is described and this is based on comparing the concentration of calcium ions in a solution in contact with the pozzolanic material. This test is also known as the Frattini test. More recently there has been development of test methods for SCMs and the Rilem R^3 method was implemented as an ASTM standard, ASTM C1897 [5]. The different test methods have their advantages and disadvantages, se e.g. [6]. For example, the strength activity index test is used to assess the reactivity of fly ash and slag but it provides no indication whether the material is pozzolanic, latent hydraulic or only acts as a filler.

In this paper results for three different alternative SCMs are presented and compared with a coal fly ash and a limestone filler. The strength activity tests were in this study complemented with a procedure to measure the amount of calcium hydroxide (portlandite) content and bound water in the mortar mixes which makes it possible to differentiate between materials that only provide a filler effect and those that have a pozzolanic reaction and consume portlandite.

2. EXPERIMENTAL PROGRAMME

Six different binder systems were tested, which included: a reference cement (CEM I 52.5 N); coal fly ash; ground pumice; a chemically treated sewage sludge ash, referred to as “silica sand”; a non-quenched steel slag filler, referred to as “ReSiCa”; and an “inert” limestone filler. For the SCMs and non-reactive fillers the cement replacement were 25 % by mass.

2.1 Materials

The materials tested in this study are presented in a ternary diagram, see figure 1 (GGBS added for comparison purpose). It should be noted that for the silica sand the content of Fe_2O_3 was 26 % and the pumice had 17 %. The MgO content of the non-quenched slag (ReSiCa) was 8.5 %.

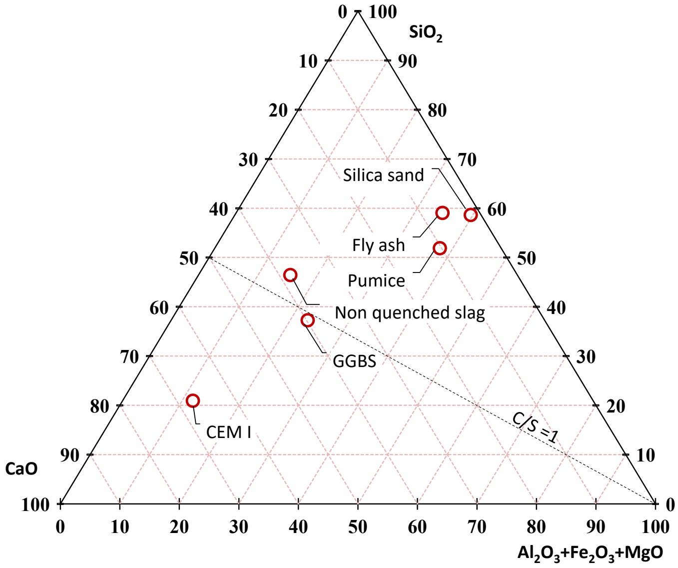


Figure 1 – Chemical composition of materials (main oxides) in a ternary diagram.

2.2 Compressive strength

Mortar prisms where mixed and casted according to EN 196-1 [7] using CEN standard sand. Subsequently the prisms were cured and tested for compressive strength according to EN 196-1 at 7, 28, 56 and 91 days.

2.3 Change of mass on heating

After testing the compressive strength at the age of 28, 56 and 91 days, the tested prisms were used to determine the bound water content and the portlandite content by measuring the mass loss after heating. The tested prism (half of it) were dried in an oven at 105 °C for four hours and then ground to a fine powder (< 0,125 mm) which was placed in a vacuum sealed plastic bag. The day after grinding, approximately 200 g powder was weighed in a container which was then placed in an oven at 105 °C for 60 minutes. After drying the powder, (4.00 ± 0.05) g powder was weighed into a crucible and the initial weight was recorded and for each binder and testing age four sub-samples were tested. After the initial weight was recorded the samples were heated for 60 minutes in cycles to the temperatures: (1) 400 °C; (2) 500; and finally (3) 600 °C. Before each weighing the samples were allowed to cool down for 10 minutes in a desiccator. The mass change between 105 and 600 °C was used as an indication of the bound water content while the mass change between 400 and 500 °C was used to calculate the amount of portlandite. The measured portlandite was calculated according to [8]:

$$Ca(OH)_{2,measured} = WL_{Ca(OH)_2} \times \frac{m_{Ca(OH)_2}}{m_{H_2O}} = WL_{Ca(OH)_2} \times \frac{74}{18} \qquad (1)$$

Where: $WL_{Ca(OH)_2}$is the weight change between 400 and 500 °C and $\frac{m_{Ca(OH)_2}}{m_{H_2O}}$ is the molar ratio of portlandite and water.

The amount of portlandite per gram of binder was determined as:

$$Ca(OH)_2 = \frac{Ca(OH)_{2,measured}}{Weight\ at\ 600℃ \times 0.25}\ \text{[g/(g binder)]} \qquad (2)$$

The bound water content per gram of binder was determined as:

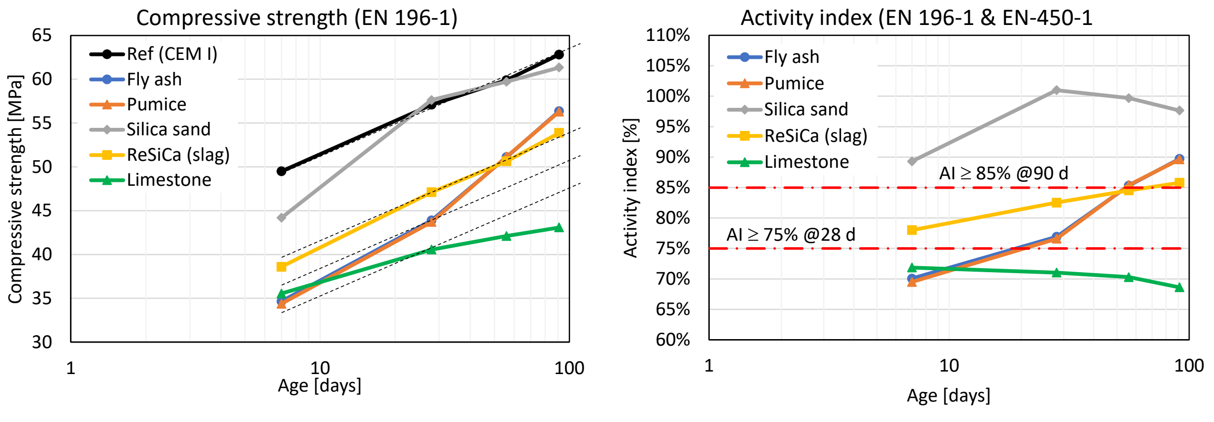
$$Bound\ water = \frac{Weight\ at\ 105℃ - Weight\ at\ 600℃}{Weight\ at\ 600℃ \times 0.25}\ \text{[g/(g binder)]} \qquad (2)$$

Where 0.25 is the amount of binder in the mortar in relation to the sand (0.25 = 450 g / 1350 g).

3. RESULTS AND DISCUSSION

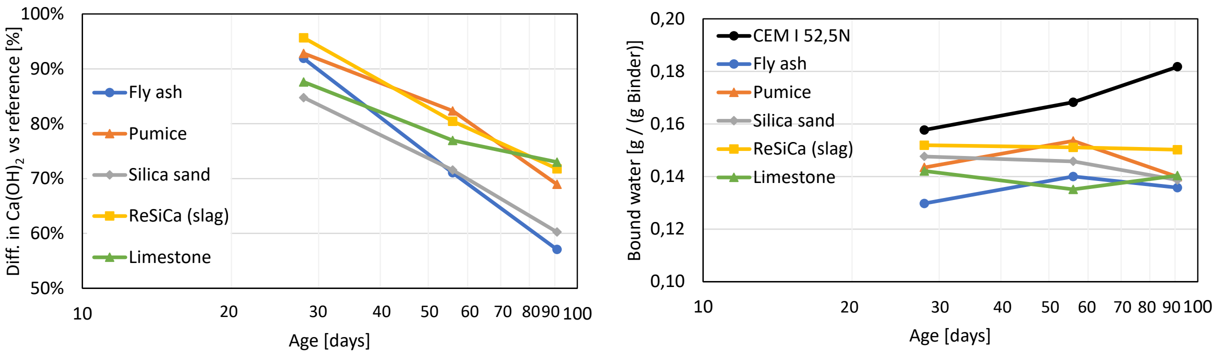
The compressive strength development and the activity index for the different SCMs is presented in figure 2 while in figure 3 the calcium hydroxide content (in % of the reference mix) and the bound water can be seen. For the strength development and activity index it can be seen that the silica sand (chemically treated sewage sludge ash) had the highest strength and at 28, 56 & 91 days the strength was equal to that of cement. The fly ash and the ground pumice had almost identical strength and the delayed pozzolanic reaction can be seen as the strength increases between 28 and 91 days. For the inert fillers, there was a significant difference between the slag and the limestone where the limestone resulted in the lowest strength. With respect to the portlandite content, the materials that "consumed" most were the fly ash and the silica sand while the pumice had a slightly lower content than the inert fillers. For the bound water content, the reference cement had the highest content while the other materials had a lower content where the fly ash had the lowest content of all tested materials.

The results from this investigation show that utilizing the tested prisms for determining the portlandite content can be useful as it show which materials have pozzolanic properties. When testing is done on ground mortars, compared to testing cement paste, there is some uncertainty about the sample preparation and the assumed binder content and in this experiment the coefficient of variation for the calcium hydroxide content and bound water was about 2 % which is deemed as satisfactory.



(a) *(b)*

Figure 2 – Development of compressive strength (a) and activity index (b).



(a) *(b)*

Figure 3 – Development of amount of calcium hydroxide compared to reference (CEM I) (a) and chemically bound water (105 to 600 °C) per gram of binder (b).

4. CONCLUSIONS

In this paper the strength development and activity index of alternative SCMs and inert fillers are presented and by utilizing the tested prisms for characterization of portlandite and bound water content complementary information about the pozzolanicity of the materials was obtained. The general conclusions are:

- The ground pumice had the same strength as the traditional fly ash but consumed less portlandite which could be due to less amount of SiO_2 (the content is 86% of the content in the fly ash).
- The chemically treated sewage sludge ash (silica sand) showed good reactivity and strength results. Moreover, the consumption of portlandite indicates pozzolanicity of the material.
- For the inert fillers, non-quenched slag (ReSiCa) and limestone filler, the portlandite content at 91 days was close to what is expected from the dilution effect (25 % replacement). However, also some of the inert materials like limestone does react and consumes some portlandite, e.g. in the formation of hemicarboaluminate.

REFERENCES

[1] BS 8615-1:2019: Specification for pozzolanic materials for use with Portland cement Natural pozzolana and natural calcined pozzolana.

[2] BS 8615-2:2019: Specification for pozzolanic materials for use with Portland cement High reactivity natural calcined pozzolana.

[3] EN 197-1:2011: Cement — Part 1: Composition, specifications and conformity criteria for common cements.

[4] EN 196-5:2011: Methods of testing cement Part 5: Pozzolanicity test for pozzolanic cement.

[5] ASTM C1897-20: Standard Test Methods for Measuring the Reactivity of Supplementary Cementitious Materials by Isothermal Calorimetry and Bound Water Measurements.

[6] Al-Shmaisani, S., Kalina, R. D., Ferron, R. D., Juenger, M. C.G.: Critical assessment of rapid methods to qualify supplementary cementitious materials for use in concrete, *Cement and Concrete Research* 153 (2022) 106709.

[7] EN 196-1:2016: Methods of testing cement Part 1: Determination of strength.

[8] Scrivener, K., Snellings, R., & Lotenbach, B. (ed): A Practical Guide to Microstructural Analysis of Cementitious Materials. CRC Press, 2016.