

EFFECTIVENESS OF ALKALI-ACTIVATED SLAG BASED BINDERS AND FLY-ASH BASED GEOPOLYMERS AGAINST ALKALI SILICA REACTION (ASR)

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Abstract

Cement-free alkali-activated binders and geopolymers are activated by concentrated alkaline solution, thus raising questions in the presence of reactive aggregates. Alkali-activated slag-based binders and fly ash-based geopolymers were subjected to accelerated mortar bar and concrete prism testing, respectively, using three different reactive aggregates and a 8M NaOH alkaline activator solution.

Alkali-activated slag-based mortars behaved differently depending on the reactive aggregate. Mortars incorporating a moderately reactive aggregate suffered 14-day expansion below 0.10%, while those made with a highly reactive siliceous limestone showed a rapid expansion ($\approx 0.4\%$ after 3 days) with slight increase afterwards. All fly ash-based geopolymer concretes suffered low expansion until about 40 weeks and then the expansion rate increased to reach values up to 3 times those measured on control (cement) concretes at 78 weeks. More fundamental testing is necessary to better understand the behavior of alkali-activated binders and geopolymers in the presence of reactive aggregates.

Keywords: Alkali silica reactivity, alkali-activated slag cements, fly-ash based geopolymers, expansion.

1 INTRODUCTION

Portland cement has historically been the most commonly used binder material for concrete making. In fact, the World cement production was estimated to 4 billion tonnes in 2013 [1]. The production of portland cement generates large amounts of CO₂ from decarbonation of limestone, well-known for its global warming effect. The production of one ton of portland cement generates approximately one ton of CO₂ [2]. It is estimated that the cement industry generates approximately 7% of global man made CO₂ emissions [3]. From the point of view of sustainable development, only systems where the clinker content in the binder is reduced or completely replaced will be able to meet the anticipated growth of concrete volumes. The alkaline activation of aluminosilicate waste materials can produce a binder often called *geopolymer* or *alkali-activated binders* with similar properties as portland cement but with fewer drawbacks linked to greenhouse gas emissions [4,5].

Several researchers have raised the importance of evaluating the durability of alkali-activated concretes, especially the alkali silica reactivity (ASR) [6-9]. Indeed, geopolymers and alkali-activated binders are generally characterized by very high alkali content, but also by a low calcium content for class F fly ash-based geopolymers or a high calcium content for both alkali-activated slag-based binders and class C fly ash-based geopolymers. By introducing a large amount of alkalis, mainly from the alkaline activator solution, it is likely that some ASR can occur in these alkalis rich systems [7].

To date, there are no evaluation methods that have been designed exclusively to address the alkali silica potential of geopolymers or alkali-activated binders. In the case of conventional cementitious systems, CSA and ASTM standards offer two tests that are normally used to evaluate the potential alkali reactivity of aggregates. These tests are the accelerated mortar-bar test (CSA A23.2-25A [10], ASTM C1260 [11] and ASTM C1567 [12]) and the standard test method for determination of length change of concrete due to alkali silica reaction also called concrete prism expansion test (CSA A23.2-14A [13], ASTM C1293 [14]). These testing methods were therefore used by some authors to assess the alkali silica potential of geopolymers and alkali-activated binders.

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In general, geopolymers and alkali-activated binders seem to perform better than portland cement against ASR. According to Garcia-Lodeiro & al. [7], this behavior is due to the formation of a dense matrix close to aggregate surfaces and to some competition for the availability of alkali ions. On one side, alkalis are consumed by the geopolymerization reaction and on the other they participate in the development of the ASR. Results obtained by Kupwade-Patil & Allouche [9] show a good behavior of geopolymers against ASR for samples tested according to the accelerated mortar bar test (ASTM C1260). Using different fly ashes (two class F and one class C) and three aggregates of different reactivity levels, these authors compared the behavior of these 36 mixtures to that of portland cement mixtures. None of the geopolymer mixtures exceeded the threshold expansion limit of 0.1% after 34 days of testing, while the portland cement mixes presented higher values and exceeded the 0.1% limit. However, the presence of silica gel, the ASR reaction product, was observed by scanning electron microscope analysis (SEM) in class C fly ash-based geopolymers.

Bakharev & al. [6] obtained an expansion higher than the limit of 0.04% at 52 weeks for an alkali-activated slag-based binder tested with the concrete prism method (A23.2-14A CSA, ASTM C1293). The expansion was significantly higher than that measured for portland cement concretes. These authors concluded that alkali-activated slag-based concretes had lower resistance to ASR than that of portland cement concrete of similar grade.

All these contradictory findings show the importance of evaluating the behavior of geopolymers and alkali-activated binders against ASR.

2 MATERIALS AND METHODS

2.1 Aluminosilicate materials (precursor)

One class F fly ash (FA) and two ground granulated blast furnace slag (GBFS) were selected as cementitious materials in this study. Table 1 presents the chemical composition of these materials. Major oxides were measured by X-ray fluorescence spectrometry.

2.2 Alkaline activators

Alkali activators allow the dissolution of the aluminosilicate source (precursor) and the formation of a low solubility gel. The activator solutions generally used are hydroxides or alkali metal salts. In this study, sodium hydroxide (NaOH) solution was used as activator at concentration of 8 M prepared from solid pellets reagent.

2.3 Aggregates

To evaluate the performance of fly ash-based geopolymers and alkali-activated slag-based binders against ASR, three aggregates of different reactivity levels were selected (Sudbury (Su), Spratt (Sp), and New Mexico (Nm)). Table 2 shows the results of the petrographic examination and presents the various potentially reactive phases associated with these aggregates. A non-reactive granitic sand was used in the manufacture of geopolymer concrete.

2.4 Methods for assessment and analysis

General

The evaluation of the durability of fly ash-based geopolymers and alkali-activated slag-based binders against ASR was carried out using the accelerated mortar bar test (CSA A23.2-25 or ASTM C 1260) and the concrete expansion test (CSA A23.2-14A or ASTM C 1293). For each test, the behavior of the three aggregates (Sp, Su and Nm) was evaluated and compared to that obtained by conventional portland cement mortars and concretes.

Accelerated Mortar Bar Method (CSA A23.2-25A or ASTM C 1260)

Alkali-activated slag-based mortars were made using ground granulated blast furnace slag (GBFS1 and GBFS2) as precursor and 8M NaOH solution as the alkaline activator. Three mortar bars, 25 x 25 x 28.5 mm in size, were manufactured for each mixture. Control mixtures (100% portland cement) were also made with a water: binder ratio of 0.50. In the case of alkali-activated slag-based mortar, a ratio of solution to binder of 0.57 was used to allow good workability of the mixtures. Aggregates have been previously crushed and sieved to obtain the required particle sizes of sand. The different size fractions were recombined according to the requirements of CSA A23.2-25A.

The mortar bars were prepared by following the various steps described in CSA A23.2-25A test procedure. After casting of the mortar bars, the moulds were covered with a damp cloth and a plastic sheet and allowed to stand for 24 hours at 23°C. Following this initial treatment, the bars were removed from their moulds, immersed in an airtight container containing water at 23°C and stored in

an oven at 80°C for 24 hours. A measurement of initial length was then performed, whereupon the bars were transferred to an airtight container containing a 1M sodium hydroxide (NaOH) solution and then stored in an oven at 80°C for 150 days.

Measurements were made periodically to evaluate the longitudinal expansion of mortar bars. According to CSA A23.2-25A, the monitoring of the expansion is carried out over a period of 14 days for conventional cementitious systems. In this study, the test has been extended to 150 days to evaluate the long-term behavior of these new binders, but also to check whether this testing method should be regarded as a representative assessment tool of the performance of alkali-activated slag-based binders against ASR.

Concrete Prism Expansion Test (CSA A23.2-14A or ASTM C1293)

Fly ash-based geopolymer concretes were manufactured with the three reactive aggregates Su, Sp and Nm and a non-reactive granitic sand. A 8M NaOH solution was used as alkaline activator. Three concrete prisms, 75 x 75 x 300 mm in size, were made for each mixture. The coarse aggregates ranged from 5 to 20 mm in size. Control mixtures (100% portland cement) were also made for comparison. Table 3 presents the mix design of the concrete prisms, including a cement content of $420 \pm 2 \text{ kg/m}^3$ and w/b of 0.42 ± 0.01 for the control concretes, and SCM content of 409 kg/m^3 and w/c of 0.35 for the fly ash-based geopolymer concretes.

After casting of the concrete prisms, the moulds were covered with a damp cloth and a plastic sheet and allowed to stand for 24 hours at 23°C. Following this initial treatment, the prisms were removed from their moulds. In the case of the control mixtures, an initial measurement was carried out before placing the concrete prism samples in sealed plastic pails containing a moistened geotextile and water at the bottom of the pail to keep relative humidity $\geq 95\%$. Then, the pails were stored in a temperature-controlled room (38°C) for 78 weeks. In the case of the fly ash-based geopolymer concretes, once demolded, prisms were placed in an airtight container with a moisture source. These containers were subsequently placed for 48 hours in an oven at 80°C. Then, the initial measurement was carried out after the specimens were cooled down to 23°C and geopolymer prisms were stored at 38°C as for the control concretes. Measurements were made periodically to evaluate the longitudinal expansion of concrete prisms over a period of 78 weeks.

3 RESULTS

3.1 Accelerated Mortar Bar Method (CSA A23.2-25A or ASTM C 1260)

Figures 1 to 3 show the expansion of the mortar bars as a function of time for aggregates of different reactivity levels. The expansion limit of 0.10% at 14 days for the control systems with portland cement is shown for comparison. In presence of the moderately reactive aggregate Su, the control mixture has an expansion of 0.276% at 14 days (Figure 1). The alkali-activated slag-based mortars present expansion below the limit of 0.10% at 14 days. The expansion measured at 14 days was 0.041% and 0.057% for Su-GBFS-1 and Su-GBFS-2, respectively.

However, a completely different behavior is observed for mortar bars made with the highly reactive siliceous limestone Spratt aggregate (Sp) (Figure 2). The control sample shows an expansion of 0.272% at 14 days. However, Sp-GBFS-1 and Sp-GBFS-2 mixtures show a very significant early increase of the expansion, only 3 days after the beginning of testing. This initial expansion reached values of 0.391 and 0.502% at 3 days for Sp-GBFS-1 and Sp-GBFS-2 samples respectively. A slight increase of the expansion can be seen between 3 and 10 days followed by a complete stabilization of the expansion. At 28 days, expansion of 0.505% (Sp-GBFS-1) and 0.606% (Sp-GBFS-2) was measured.

Figure 3 shows the expansion, as a function of time, for the highly reactive Nm aggregate. The control sample made with portland cement shows an expansion of 0.894% at 14 days. Alkali-activated slag-based mortars show a much lower expansion than that obtained for the control mixture. However, expansion measured at 14 days for these two mixtures is greater than the limit of 0.10%. Values of 0.139% and 0.124% were obtained at 14 days for the Nm-GBFS-1 and Nm-GBFS-2, respectively. In addition, the expansion of these mixtures gradually increases for the whole duration of the test.

Figures 1 to 3 show that the behavior of alkali-activated slag-based mortars varies depending on the type of aggregate used. Figure 4 compares the expansion data obtained at 14, 28, 77 and 150 days. For the moderately reactive aggregate Su, alkali-activated slag-based mortars show lower expansion than the control sample at 14 and 28 days. Although the expansion progressively continues to increase over time, the values achieved at 150 days for the alkali-activated slag-based mortars are still lower than that of the control reached at 28 days.

The samples made with the highly reactive aggregate Spratt show little expansion difference in the longer term between the control sample and the alkali-activated slag-based mortars. The latter showed a very significant expansion at the beginning of the test and only a slight increase afterwards.

In the case of the very highly reactive Nm aggregate, the expansion of all mixtures, control or alkali-activated slag-based mortars, is higher than the 0.10% expansion limit. This expansion continues to increase with time to reach a very high value of 1.181% at 28 days for the control mixture. Although the expansion of alkali-activated slag-based mortars is reduced relative to the control, expansion obtained remains well above the limit.

3.2 Concrete Prism Expansion Test (CSA A23.2-14A or ASTM C1293)

Figure 5 shows the concrete prism expansion results obtained on geopolymer concretes produced by alkali activation of a class F fly ash (FA) with 8N NaOH as alkaline activator.

The fly ash-based geopolymer concretes show expansion values lower than that obtained with the control mixtures in the first half of the test. Indeed, the expansion values of 0.024%, 0.129% and 0.100% were obtained at 44 weeks for mixtures of geopolymers with aggregates Nm, Su and Sp, respectively. These values are lower than the expansions obtained for the control mixtures (100% portland cement), i.e. 0.209%, 0.125% and 0.180% for mixtures Nm, Su and Sp.

In the second half of the test (> 44 weeks), a very significant expansion is observed for mixtures of fly ash-based geopolymers compared to that measured on the control mixes. At 78 weeks, the expansion of geopolymer concretes is approximately three times greater than that of the control mixtures. Expansion values of 0.694%, 0.625% and 0.550% were obtained for geopolymer samples made with Nm, Su and Sp aggregates, respectively, while the control mixtures show expansion of 0.224%, 0.164% and 0.199% for the same aggregate materials. It is interesting to note that the expansion of the control concretes had generally levelled off after 52 weeks of testing.

4 DISCUSSION

The results obtained on alkali-activated slag-based mortars showed a very different behavior depending on the reactivity level of the aggregate tested. For a moderately reactive aggregate (Su - 0.276% at 14 days), alkali-activated slag-based mortars show a slight expansion at the beginning of the test (\approx 0.05%, <3 days) and the expansion remained fairly limited (<0.10%) over the rest of the 28-day period. In the case of a highly reactive siliceous aggregate (Sp - 0.272% at 14 days), the behavior was similar, except that a very rapid onset of expansion of about 0.4% is measured after only 3 days of testing. Although the expansion remained constant up to 28 days, expansion of alkali-activated slag-based mortars was similar or slightly greater than that of the control mortar in the case of Spratt aggregate.

The behavior of the alkali-activated slag-based mortars made with the very highly reactive Nm aggregate differs considerably from that observed with the Spratt and Sudbury aggregates. First, the Nm mortar shows an expansion (0.894% at 14 days) three times greater than that obtained with Sp aggregate (0.272%) for control mixtures with portland cement. However, for the alkali-activated slag-based mortars, expansions obtained with the Nm aggregate are about three times lower than the expansion obtained with the control Spratt mortar at 14 days. Not only the expansion values are well below the control mixture, but no rapid expansion was observed early in the test (<5 days). However, a constant expansion rate was observed throughout the testing period in presence of the Nm aggregate.

The type of aggregate has a significant impact on the results obtained at the accelerated mortar bar test (ASTM C1260). This influence is not limited to the reactivity level of the aggregate tested. The nature of the aggregate seems to have an importance on the expansion rates achieved.

The accelerated mortar bar test (ASTM C1260) is often described as very severe due to the harsh environment of the immersion solution for the evaluation of the potential reactivity of aggregates in conventional systems based on portland cement. Is this test severe enough or simply a realistic environment to evaluate the behavior of geopolymers or alkali-activated binders? In this test, the mortar bars are soaked in a 1M NaOH alkaline solution and then stored in an oven at 80°C for 14 days. The temperature, the alkaline solution and the testing period represent parameters that can influence the results and are all favorable to the development of a strong geopolymer or alkali-activated binders. Indeed, a solution of at least 8N NaOH and a temperature of 80°C are generally used in the synthesis of geopolymers or alkali-activated binders. By immersing the mortar bars in a 1N NaOH solution, the soaking solution is less concentrated in NaOH than the geopolymer or alkali-activated mortars themselves. In addition, there is some competition for the availability of alkalis. On the one hand, geopolymers and alkali-activated binders use these alkalis paired with the high

temperature (80°C) for producing a densification of the matrix and, on the other side, alkalis diffuse into the matrix to attack the reactive siliceous aggregates and then participate in the formation of the expansive gel. The first 2-3 days of testing are equivalent to the activation conditions used for the geopolymer or alkali-activated binder curing to optimize its strength. Thereafter, the availability and diffusion of alkali ions through the matrix would be more difficult. Fernandez-Jimenez & Puertas [15] suggest that the alkali silica reactivity will develop in geopolymers but the expansion is delayed due to the competition for the alkali ions at the beginning of the test. A 28-day testing period may not be sufficient to assess the expansion induced by this reaction. By extending the test for a period of 150 days, the results suggested that the reaction proceeds. For all mixtures of alkali-activated slag-based binders, expansion measured at 77 and 150 days is greater than the limit of 0.10%.

The concrete prism test seems more appropriate for assessing the performance of geopolymers or alkali-activated binders against ASR. The concrete prism test is more representative of the actual behavior of concrete when used as construction material. However, the testing conditions (ie 38°C and 100% RH) can influence the performance of geopolymer or alkali-activated concretes. Indeed, the temperature and relative humidity are important parameters in the synthesis of geopolymers and alkali-activated binders and their presence helps to promote the geopolymerization reaction. In this way, the quality of the matrix would be improved, resulting in a reduction of the porosity and therefore a decrease in the diffusion of alkaline ionic species to the reactive silica aggregates. The expansion results obtained at 78 weeks in concrete prism test show that the fly ash-based geopolymer samples outperform the control mixtures in the first half of the test, but the rate of geopolymer expansion is much higher than the control mixtures afterwards. This observation can possibly be explained by the conditions at the beginning of the testing period (38°C and 100% RH) that contribute positively to the geopolymerization reaction, so there would be only a slight increase of expansion. Once the matrix formed and densified, the testing conditions would possibly allow ASR to take place. However, the sudden increase in the expansion observed after 44 weeks of testing remains somewhat “unusual” and further testing is currently in progress to confirm such behaviour.

5 CONCLUSIONS

The goal of this research was to evaluate the behaviour of geopolymers and alkali-activated binders against alkali silica reactivity. The behavior of alkali-activated slag-based mortars in accelerated test conditions (80°C, immersion in 1N NaOH) varies depending on the reactive aggregate. In the presence of a moderately reactive aggregate, expansions measured at 14 days are below the conventional limit of 0.10%. However, in the presence of a highly reactive siliceous limestone, a very rapid onset of expansion, of about 0.4%, is measured after only 3 days of testing. On the other hand, the very-highly reactive Nm gravel did not produce such a rapid onset of expansion but rather a very progressive expansion rate over the 28-day testing period that reached about 0.20% expansion. It is clear from these results that the type of reactive aggregate has a strong influence in the development of the expansion in alkali-activated slag-based mortars.

Very low concrete prism expansion was measured for fly ash-based geopolymers until about 40 weeks of testing and then the expansion rate is suddenly increased, reaching values up to 3 times those measured on a control mixture (portland cement only) at 78 weeks.

Expansion results obtained so far are not conclusive and more fundamental testing is necessary to better assess the behavior of geopolymers and alkali-activated binders in the presence of reactive aggregates. The test parameters used to accelerate the alkali silica reactivity (high temperature, high alkalinity and high relative humidity) are favorable to the geopolymerization reaction but it is not clear if the long term behavior measured according to ASTM C1260 and ASTM C1293 testing procedures are representative of the real behavior of geopolymers or alkali-activated binders.

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TABLE 1: Chemical composition of the aluminosilicate materials.

Composition (%)	SiO ₂	Fe ₂ O ₃	Al ₂ O ₃	CaO	MgO	TiO ₂	Na ₂ O	K ₂ O
FA	47.5	15.3	24.4	4.36	0.92	1.21	0.94	1.74
GBFS-1	33.5	1.41	13.9	43.7	4.42	0.49	0.28	0.41
GBFS-2	33.2	0.28	13.5	40.6	5.83	0.45	0.31	0.33
Composition (%)	P ₂ O ₅	MnO	Cr ₂ O ₃	LOI	Sum	Na ₂ O _{eq}	Specific gravity	
FA	0.39	0.03	0.02	3.27	96.8	2.08	2.63	
GBFS-1	0.03	0.20	0.01	0.90	98.4	0.55	2.97	
GBFS-2	0.01	0.46	0.01	3.21	95.0	0.53	3.01	

TABLE 2: Origin, composition and physical properties of the reactive aggregates studied (adapted from Villeneuve 2011 [16]).

Aggregate	Location	Lithological composition	Specific gravity	Absorption (%)	Potentially reactive phases	Reactivity
Spratt (Sp)	Ottawa (Canada)	Siliceous limestone	2.67	0.61	Micro to cryptocrystalline quartz	High
Sudbury (Su)	Sudbury (Canada)	Gravel (greywacke, sandstone, granite)	2.66	0.52	Micro-crystalline and strained quartz	Moderate to high
New Mexico (Nm)	New Mexico (USA)	Polymictic gravel (mixed volcanic, quartzite, chert)	2.50	0.65	Volcanic glass, micro-crystalline and strained quartz.	Very high

TABLE 3: Mix design of concrete samples.

Concretes	W/B	Constituents, kg/m ³					
		Binder	Sand (dry)	Stone (dry)	NaOH	SP	Additional water
Control	0.43±0.01	420±2	710 ± 18	1053 ± 26	2 (solid)	--	--
Geopolymer	0.35	409 (FA)	554	1294	143 (8M soln)	8	50

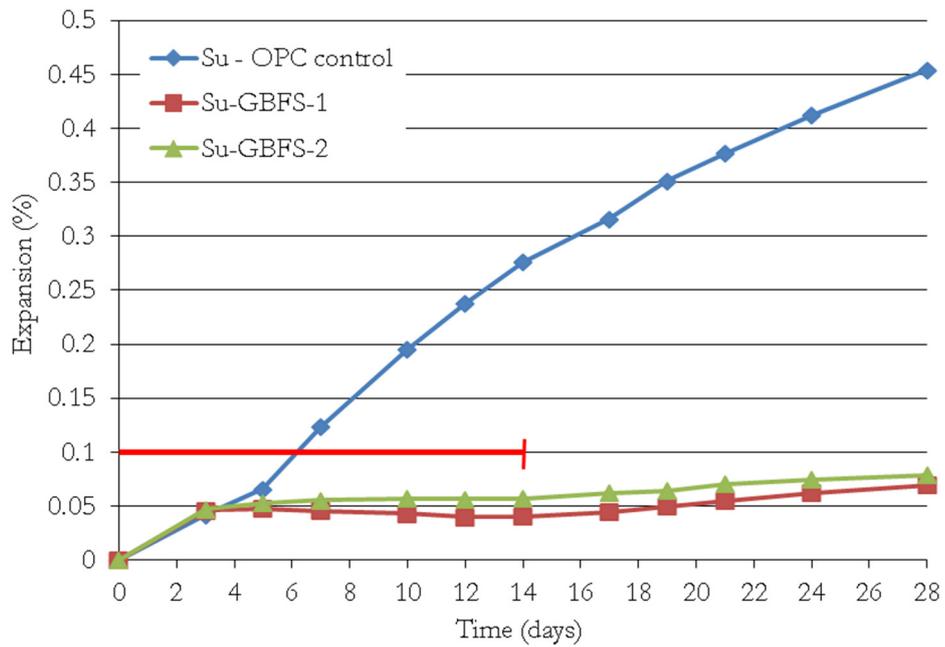


FIGURE 1: Expansion of mortar bars tested according to ASTM C1260 for control mixture (100% portland cement) and alkali-activated slag-based mortars (GBFS-1 and GBFS-2) in the presence of the moderately reactive aggregate Su. Each curve in this figure corresponds to the average values obtained from a set of 3 mortar bars.

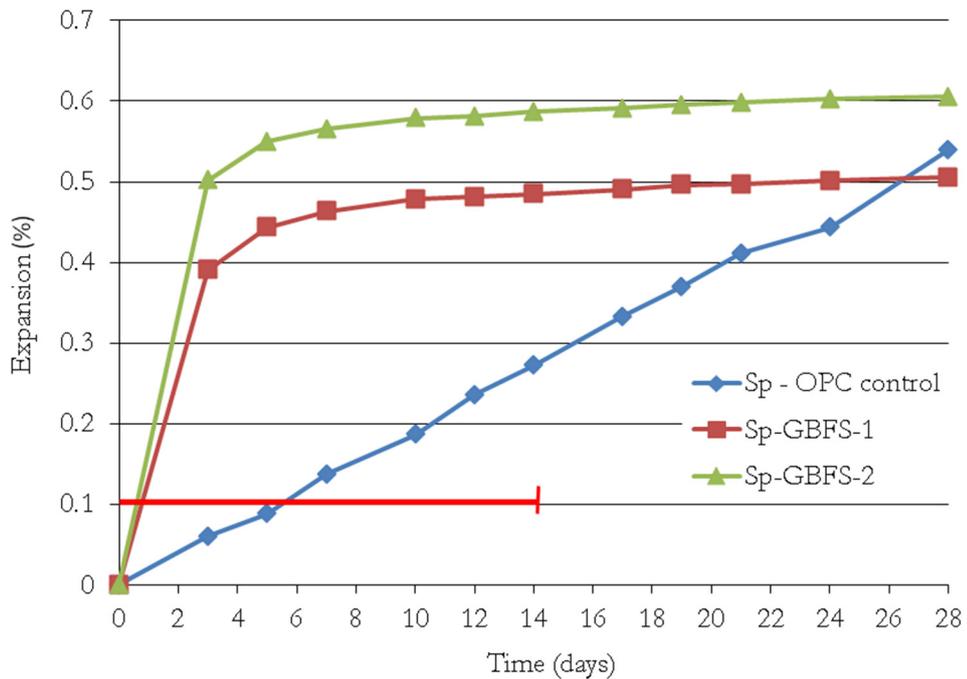


FIGURE 2: Expansion of mortar bars tested according to ASTM C1260 for control mixture (100% portland cement) and alkali-activated slag-based mortars (GBFS-1 and GBFS-2) in the presence of the highly reactive aggregate Sp. Each curve in this figure corresponds to the average values obtained from a set of 3 mortar bars.

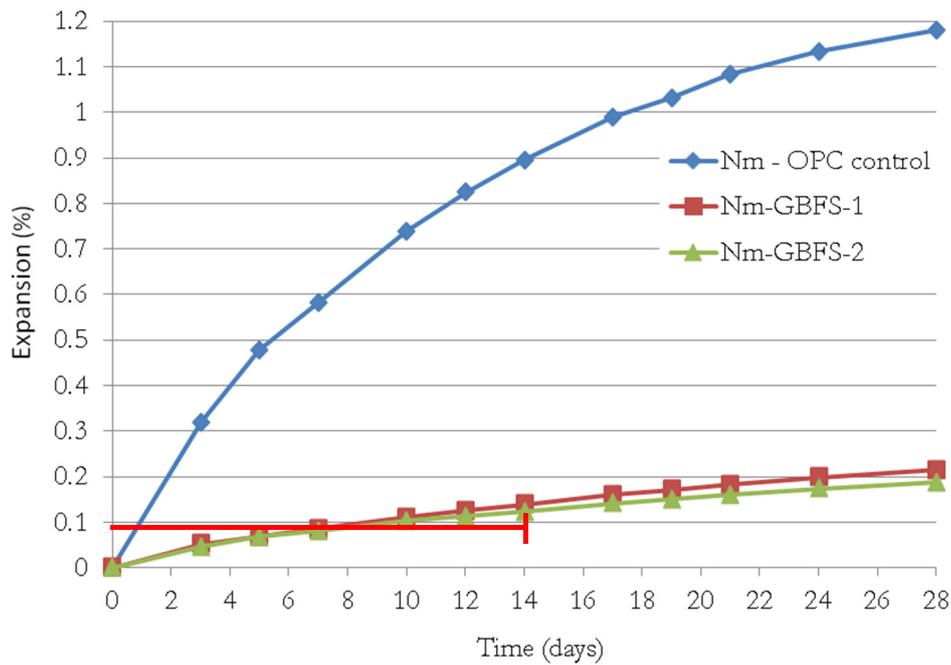


FIGURE 3: Expansion of mortar bars tested according to ASTM C1260 for control mixture (100% portland cement) and alkali-activated slag-based mortars (GBFS-1 and GBFS-2) in the presence of the very highly reactive aggregate Nm. Each curve in this figure corresponds to the average values obtained from a set of 3 mortar bars.

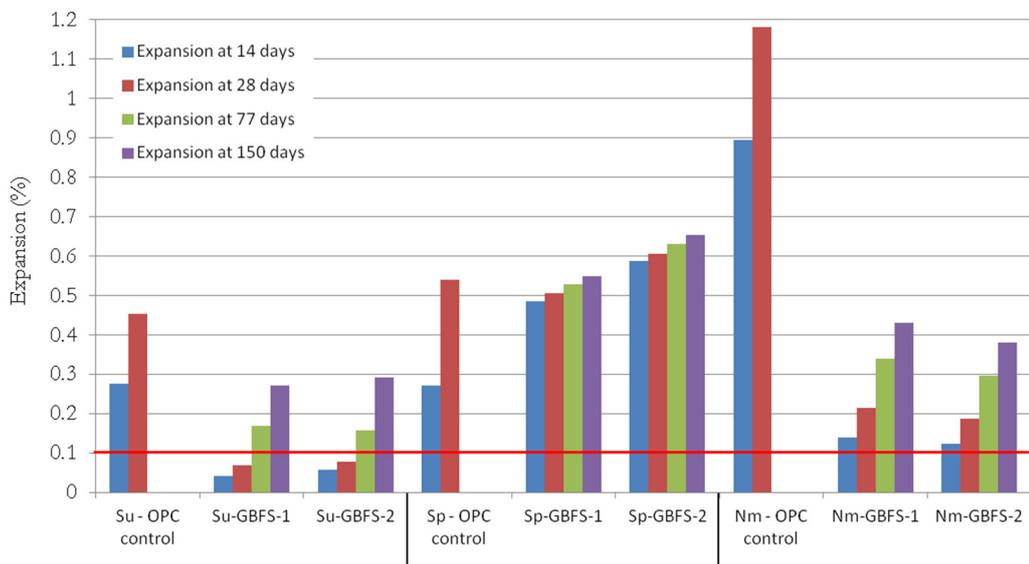


FIGURE 4: Expansion of mortar bars measured at 14, 28, 77 and 150 days (ASTM C1260) for control mixtures (100% portland cement) and alkali-activated slag-based mortars (GBFS-1 and GBFS-2) in presence of 3 aggregates of different reactivity levels. (Su – moderately reactive, Sp – highly reactive – Nm – very highly reactive).

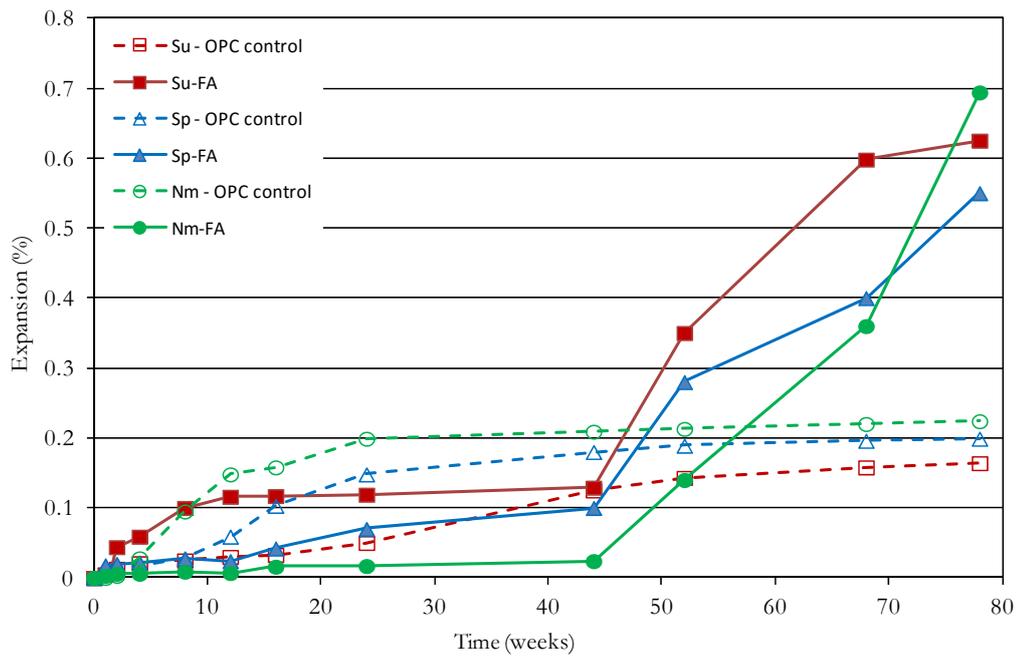


FIGURE 5: Expansion of concrete prisms measured according to ASTM for control mixtures (100% portland cement – empty symbols) and fly ash-based geopolymers (FA – full symbols) in the presence of 3 aggregates of different reactivity levels. (Su – moderately reactive, Sp – highly reactive – Nm – very highly reactive).