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Alkali-aggregate behaviour of alkali-activated slag mortars: Effect of aggregate type

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ABSTRACT

The alkali–silica reaction in waterglass-alkali-activated slag (waterglass-AAS) and ordinary Portland cement (OPC) mortars was evaluated using three types of (siliceous and calcareous) aggregates. The tests were conducted to the ASTM C1260-94 standard test method. The mortars were studied by volume stability, mechanical strength and Hg intrusion porosity. The ASR products were studied with XRD, FTIR and SEM/EDX techniques.

According to the results obtained, under the test conditions applied in this study, waterglass-AAS mortars are stronger and more resistant to alkali-aggregate reactions than OPC mortars. When the mortars were made with a reactive siliceous aggregate, expansion was four times greater in the OPC than in the AAS material. When a reactive calcareous (dolomite) aggregate was used, no expansion was detected in any of the mortars after 14 days, although the characterization results showed that the dolomite had reacted and calcareous-alkali products (brucite) had in fact formed in both mortars. These reactive processes were more intense in OPC than in AAS mortars, probably due to the absence of portlandite in the latter. When the calcareous aggregate was non-reactive, no expansions were observed in any of the mortars, although a substantial rise was recorded in the mechanical strength of AAS mortars exposed to the most aggressive conditions (1 M NaOH and 80 °C).

1. Introduction

The world-wide need to reduce the energy used and the greenhouse gases emitted during cement manufacture has led to the pursuit of more eco-efficient materials, such as alkali-activated slag cements (AAS). These cements are obtained by mixing of blast furnace slags (by-products from the iron industry) with alkaline solutions. The behaviour and durability of AAS mortars and concretes have been extensively studied [1–15]. These researches have shown that such materials can develop comparable, and in some cases greater, mechanical strength than mortars made with ordinary Portland cement (OPC) [3,4]. They are also highly sulphate and seawater resistant [7,8] and perform well when exposed to acid media [9,10], chloride ions [11] and freeze-thaw cycles [12,13]. All these characteristics and properties are essentially associated with the special nature of the main hydration product in these systems (a less basic and more highly polymerized CSH gel than formed in OPC systems) [16–19] and the lower porosity and permeability of the respective mortars and concretes [20].

Recent studies [21,22] have shown that while AAS mortars do not generate $Ca(OH)_2$ during hydration, they are more readily and intensely carbonated than OPC mortars. In AAS systems the CSH gel itself is carbonated directly, prompting decalcification and the formation of silica gels of varying composition and structure. These same studies have shown, however, that the mechanical strength of AAS mortars is not adversely affected by such more intense carbonation.

The high concentration of alkalis in alkaline cements (over 3%) suggests that alkali-activated cements may be more vulnerable to alkali-aggregate reaction (AAR)-induced expansion than OPC mortars, although it is likewise true that in these alkaline systems the Ca^{2+} ion concentration in the pore solution is lower, a determining factor in the alkali-aggregate reaction [23]. The studies conducted to date have yielded varying and at times clearly contradictory results. Metso [24] reported differences in the AAR-mediated expansive behaviour of two slags activated with the same amount of alkali. Gifford and Gillot [25] found behavioural differences depending on the nature of the aggregate: in the presence of reactive calcareous aggregate, AAS concretes were more vulnerable to the reaction and more prone to form expansive products than OPC concretes, whereas the findings were reversed when reactive siliceous aggregate was used. The results obtained by Bakharev et al. [26], in turn, showed that alkali-aggregate reaction-induced expansion was greater in waterglass-AAS than in OPC concretes, particularly in the first 5 months of the experiment, when an alkali–siliceous gel was observed to form around the aggregate. By contrast, Fernandez-Jiménez and Puertas [27] found that NaOH-AAS mortars made with reactive opaline aggregate exhibited a lower expansion rate than OPC mortars prepared and

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Table 1
Chemical analysis of cement and blast furnace slag (% by weight).

Binder	CaO	SiO_2	Al_2O_3	MgO	Fe_2O_3	SO_3^-	S^{2-}	Na_2O	Free CaO	L.I.O.	I.R.	Blaine (m^2/Kg)
Cement	65.03	18.61	4.46	2.20	4.39	2.94	–	0.12	0.64	0.43	0.18	527
Slag	41.00	35.34	13.65	4.11	0.39	0.06	1.91	0.01	–	2.72	0.64	325
Mineralogical composition of the cement												
	C_3S	C_2S	C_3A	C_4AF	K_2SO_4	$CaSO_4 \cdot 2H_2O$	$CaSO_4 \cdot 1/2H_2O$	$CaSO_4$				
Cement	70.8(4)	10.3(4)	2.7(1)	13.6(2)	0.6(1)	–	1.1(1)	0.9(1)				

stored under the same conditions. Similar results have been reported by other authors [28] for waterglass-AAS concretes. According to the results of Krivenko [29], the Al_2O_3 content in aluminosilicate constituent is a determining factor in the alkali-aggregate reaction in alkaline cementitious systems.

These discrepancies may be due to the use of different materials (nature of the slag, type and concentration of the alkaline activator, nature and percentage of the reactive aggregate) and the alkali-aggregate reaction test conditions and standards applied.

In light of the need, for a more rigorous procedure for establishing the behaviour of AAS mortars and concretes made from aggregate with varying degrees of reactivity, the present study was undertaken to ascertain the behaviour of waterglass-AAS mortars prepared with calcareous and siliceous aggregate under the conditions laid down in ASTM standard C1260-94.

2. Experimental

2.1. Materials

The chemical composition of CEM I 52.5 N cement and the vitreous blast furnace slag used in the study is given in Table 1. The table also shows the quantitative mineralogical composition, pursuant to Rietveld refinement of XRD patterns [30]. The blast furnace slag had a vitreous content of 99% [31].

The slag was alkali-activated with a waterglass solution ($Na_2O \cdot nSiO_2 \cdot mH_2O + NaOH$) containing 4% Na_2O by slag mass and a SiO_2/Na_2O ratio of 1.08.

The physical and chemical characteristics of the three types of aggregate used to prepare the mortars, two calcareous and one siliceous, are summarized in Table 2. Aggregate water absorption and density were determined as specified in Spanish standard UNE 1097-6. The potential reactivity of the siliceous aggregate was evaluated to Spanish standard UNE 146507 EX. XRD, FTIR and thin section microscopic techniques were used to determine the mineralogy of the three types of aggregate.

2.2. Mortar preparation

Table 3 lists all the mortars, which were prepared as specified in European standard EN-UNE 196-1. The alkali-aggregate reaction was evaluated according to the method described in ASTM standard C1260-94. Further to this standard, the aggregate must have a specific particle size distribution (see Table 4). While all the mortars prepared had the particle size distribution called for in the standard, the liquid/cement ratio in the AAS mortars had to be raised to 0.52, for the mortars were unworkable with less. A water/cement ratio of 0.47 was maintained for the OPC mortars, however.

Mortar specimens measuring 2.5 × 2.5 × 28.7 cm were prepared for each binder/aggregate combination, with a binder/aggregate ratio of 1/2.25.The specimens were cured in a moist cabinet (99% R.H., 21 ± 2 °C) for the first 24 h and then de-moulded. The de-moulded samples were submerged in water and kept in an oven at 80 °C for 24 h, after which the first shrinkage measurement was taken. They were subsequently submerged in deionized water at room temperature (control mortars) or in 1 M NaOH at 80 °C and stored under these conditions for 14 days, according to the ASTM standard (see storage conditions on Table 3). Previous studies [27] concluded that for determining the potential expansion due to alkali-aggregate reaction in AAS mortars, 14 days is not a suitable period and a longer period is required. Consequently in the present study the AAS mortars were additionally stored in the different media during 4 months.

Table 2
Aggregate characteristics and composition.

Aggregate	Density (g/cm^3)	Absorp. coefficient (A_ρ)	Potential reactivity	Mineralogical composition
Siliceous (S)	2.64	0.7	Negative	Quartz, calcite and albite-type feldspars
Non-reactive (C) calcareous	2.65	0.5	–	Calcite and quartz traces
Reactive calcareous (CR)	2.67	0.4	–	Calcite, dolomite and quartz traces

Table 3
Mortars prepared.

Mortar	Binder	Aggregate	Mixing liquid	Liquid/solid	Storage medium
OPC-S_1	OPC	S	Water	0.47	In water: 99% R.H., T 21 ± °C
OPC-S_2	OPC	S	Water	0.47	1 M NaOH, T 80 °C
OPC-C_1	OPC	C	Water	0.47	In water: 99% R.H., T 21 ± °C
OPC-C_2	OPC	C	Water	0.47	1 M NaOH, T 80 °C
OPC-CR_1	OPC	CR	Water	0.47	In water: 99% R.H., T 21 ± °C
OPC-CR_2	OPC	CR	Water	0.47	1 M NaOH, T 80 °C
AAS-S_1	AAS	S	Wg.4% Na_2O	0.52	In water: 99% R.H., T 21 ± °C
AAS-S_2	AAS	S	Wg.4% Na_2O	0.52	1 M NaOH, T 80 °C
AAS-C_1	AAS	C	Wg.4% Na_2O	0.52	In water: 99% R.H., T 21 ± °C
AAS-C_2	AAS	C	Wg.4% Na_2O	0.52	1 M NaOH, T 80 °C
AAS-CR_1	AAS	CR	Wg.4% Na_2O	0.52	In water: 99% R.H., T 21 ± °C
AAS-CR_2	AAS	CR	Wg.4% Na_2O	0.52	1 M NaOH, T 80 °C

Table 4
Grading criteria.

Passing	Retained on	Mass %
4.75 mm	2.36 mm	10
2.36 mm	1.18 mm	25
1.18 mm	600 μm	25
600 μm	300 μm	25
600 μm	150 mm	15

At the same time, 3 × 3 × 3-cm OPC and AAS mortar specimens were prepared and subjected to the alkali-aggregate test described above. Compressive strength was determined on these mortars.

2.3. Tests conducted

The tests conducted were:

2.3.1. Volume stability

Volume stability measurements were taken throughout the 14 days and 4 months-day trial according to UNE 80-112-89 standard.

2.3.2. Compressive strength

Specimen compressive strength according to EN 196-1 standard was found after 14 days and 4 months of storage in the media described.

2.3.3. Microstructural and mineralogical characterization

Total porosity and pore size distribution in tested 14 days specimens were found by Hg intrusion porosimetry (MIP) using a Micromeritics 9320 porosimeter. Also mortar samples were studied under SEM/EDX using a JOEL 5400 microscope attached to an Oxford-Link ISIS EDX microanalysis unit. The mineralogical study was conducted on binder-enriched samples, tested 14 days samples. These samples were studied by XRD using a Philips PW-1730 diffractometer and by FTIR using a ATIMATTSON, Genesis FTIR-TM spectrometer.

3. Results and discussion

3.1. Volume stability

According to ASTM standard C1260, mortars that expand by more than 0.1% when stored for 14 days in 1 M NaOH at 80 °C are regarded to have undergone an alkali-aggregate reaction. Fig. 1 shows the variation in volume recorded for the mortar specimens stored in water and in the 1 M NaOH solution at 80 °C up to 4 months. An analysis of the data in these figures shows that both the OPC and AAS mortars made with siliceous aggregate expanded, while no change in volume was observed when calcareous aggregate was used.

In mortars containing siliceous aggregate, the nature of the binder (OPC and AAS) also appeared to affect expansion intensity. In OPC mortars, volume changes were recorded in the first 72 h of storage in the alkaline medium, with 14 days expansion values on the order of 0.36%. In mortars in which the binder was AAS, expansion also began after around 72 h of storage, but total 14 days expansion was just 0.1%. Similar results had been obtained in previous studies [27], which showed that the alkali-aggregate reaction was greater in OPC than NaOH-AAS siliceous aggregate mortars when both were subjected to identical test conditions. The primary difference between that research and the present study was the nature of the alkaline activator, which was waterglass in this case.

The mortar properties were studied after 4 months of storage conditions. Only with siliceous aggregate and accelerated conditions the expansion of the mortars was observed, although an slight increase of the volume were observed in AAS mortars with

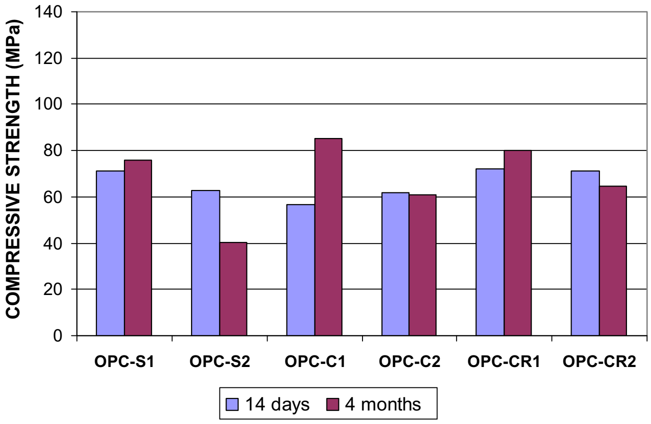


Fig. 2. Mechanical strength in OPC mortars.

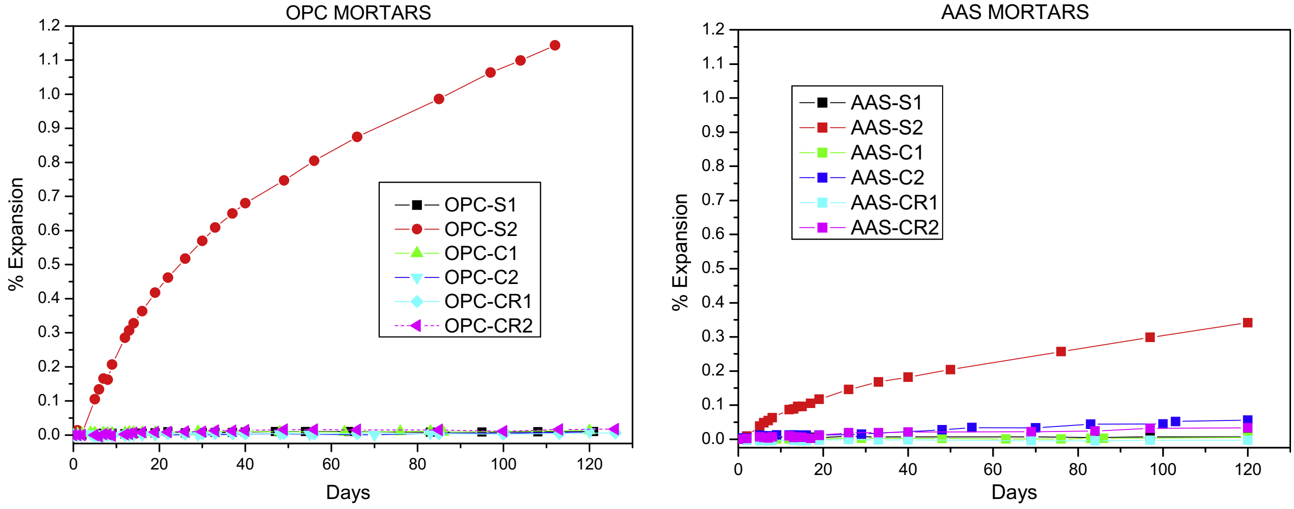


Fig. 1. Expansion curves.

both calcareous submerged to alkaline medium; but in any case those expansion values were lower than 0.1%.

3.2. Compressive strengths

The mechanical behaviour of the 14 days and 4 months specimens stored in water and the alkaline medium are shown in Figs. 2 and 3.

According to these findings, after 14 days of storage, the strength of both OPC and AAS mortars made with siliceous and calcareous aggregate increased after the accelerated test, except in OPC mortars with siliceous aggregate. The results obtained after 4 months of storage show a decrease (of about 35%) in the compressive strength in these same mortars with siliceous aggregate. Also at this time (4 months) a slight decrease on mechanical strengths was observed in the OPC mortar with reactive calcareous aggregate in aggressive conditions.

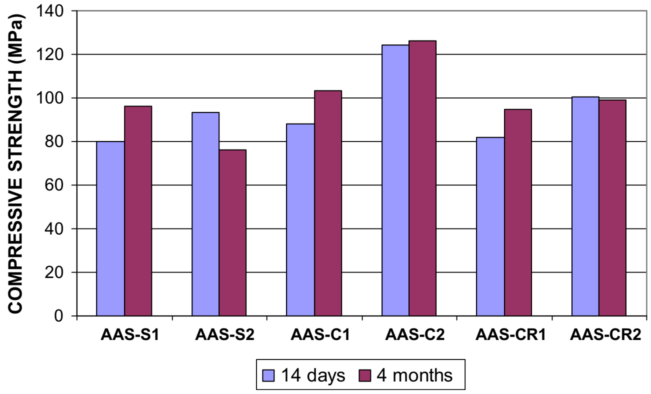


Fig. 3. Mechanical strength in AAS mortars.

All the AAS mortars (see Fig. 3) stored during 14 days in the alkaline medium exhibited much higher strength values than the specimens stored in water, with non-dolomite aggregate AAS mortars (AAS-C2) reaching values of 124 MPa, i.e., 29% higher than the water-stored samples. Only the AAS mortar with siliceous aggregate and stored in alkaline medium during 4 months shows a decrease on the compressive strength. In the rest of the mortars an increase of the strengths were observed.

3.3. Microstructural and mineralogical characterization

This mechanical behaviour is explained, in part, by the porosity and pore size distribution results obtained for the mortars. Fig. 4 shows the MIP findings for total porosity and pore size distribution in OPC mortars after 14 days of accelerated test, while Fig. 5 gives the results for the AAS mortars.

The OPC mortars stored in an alkaline medium had from 10% to 24% greater total porosity than the mortars stored in water, with the highest values found for the materials made with siliceous aggregate. As a rule, the pore size in the mortars stored under the most aggressive conditions was from 0.1 to 0.01 μm greater than in the controls. These mortars also exhibited an increase in the proportion of larger pores (10–0.1 μm). Such variations in porosity did not appear to suffice to induce a decline in strength values.

Total porosity was lower and meso-porosity greater in the AAS than the OPC mortars, a finding that concurs with reports by other authors [32]. A sharp decline in total porosity was observed in this study in all the mortars submerged in the alkaline solution, irre-

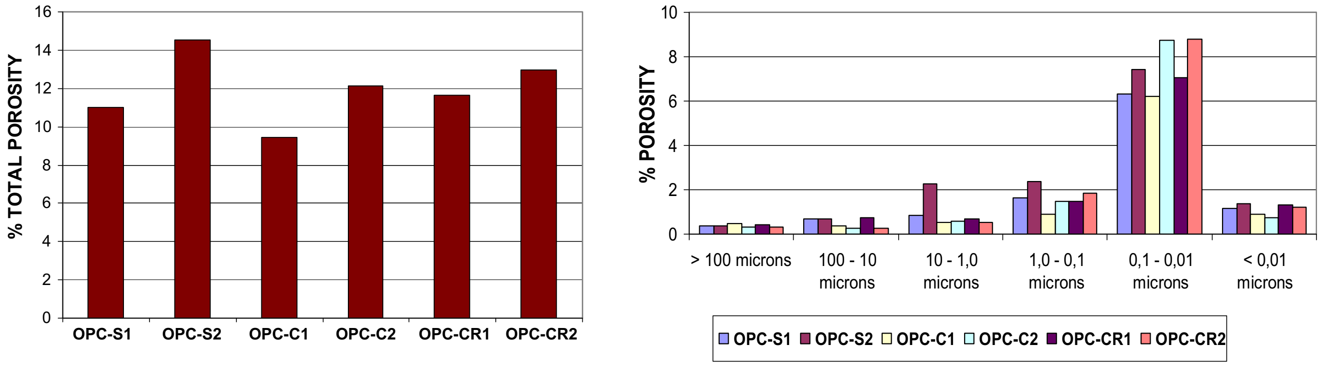


Fig. 4. Total porosity and pore size distribution in OPC mortars after 14 days of accelerated test.

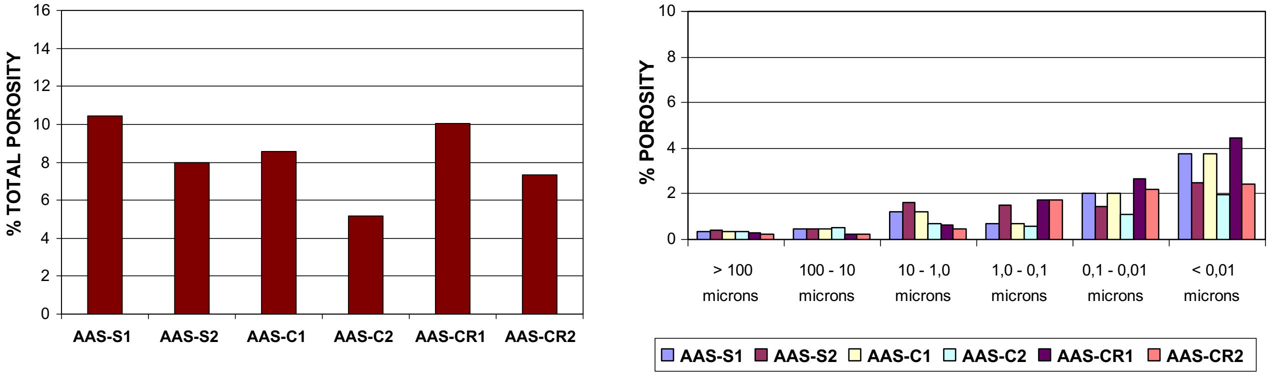


Fig. 5. Total porosity and pore size distribution in AAS mortars after 14 days of accelerated test.

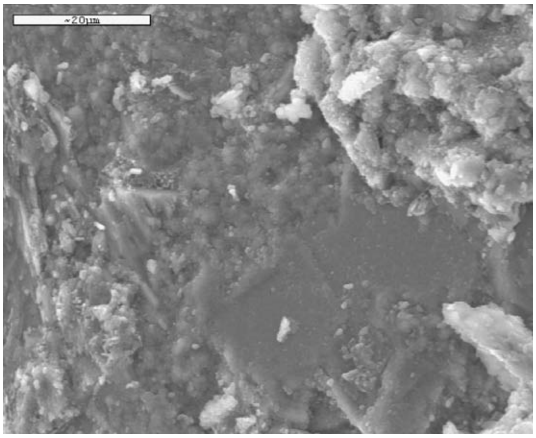


Fig. 6. Attack on calcareous aggregate (OPC-C_2).

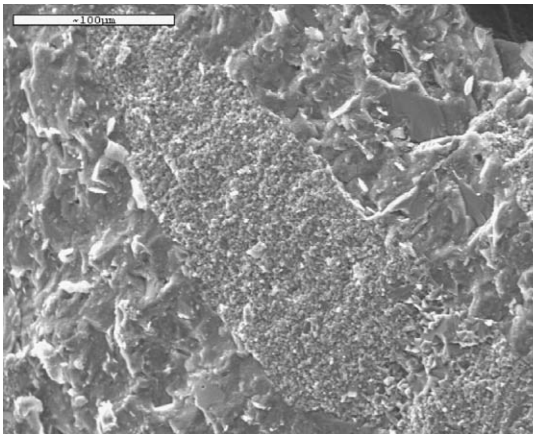


Fig. 7. Attack on calcareous aggregate (AAS-C_2).

spective of the type of aggregate, but particularly where non-dolomite or non-reactive calcareous aggregate was used, which would explain the high strength values obtained for these mortars. The most prominent effect on pore size distribution was the decline in the proportion of smaller pores in the mortar stored in an aggressive medium, although that decline was due, in part, to the lower total porosity in such mortars.

Porosity was consistently higher in OPC than AAS mortars, while this parameter declined more steeply in the AAS mortars stored in an alkaline solution. The explanation may be that the alkaline medium and the high storage temperature (80 °C) would continue to stimulate the reaction between any remaining anhydrous slag and the NaOH solution, generating new reaction products that would, in turn, reduce porosity and raise strength.

The mortar volume stability, mechanical strength and porosity results obtained showed that under equally aggressive conditions, strength and stability raised more in the AAS than in the OPC mortars.

The microstructural study conducted by SEM/EDX on the non-reactive calcareous aggregate OPC and AAS mortars showed that the aggregate surface was attacked in both cases when the mortars were stored in aggressive environments. The surface attack on the calcareous aggregate in mortars OPC-C_2 and AAS-C_2 is depicted in Figs. 6 and 7, respectively. When the latter mortar was studied with SEM/BSE techniques, a whitish layer was observed around the calcareous aggregate that appeared to be spreading into the matrix. The analysis of this layer showed a higher Ca content than in other inner-more areas of the matrix (see Fig. 8). These results are indicative of dissolution and partial attack on the aggregate; the subsequent diffusion of calcium ions towards the matrix would favour the formation of a more cohesive interface and explain the low total porosity and the high strength exhibited by these mortars.

The SEM/EDX study of the siliceous aggregate OPC and AAS mortar microstructure confirmed that the aggregate was attacked under the aggressive test conditions. In the SEM image of mortar OPC-S_2 in Fig. 9, a characteristic morphology of ASR, in a pore, can be observed. The morphology and composition of an ASR

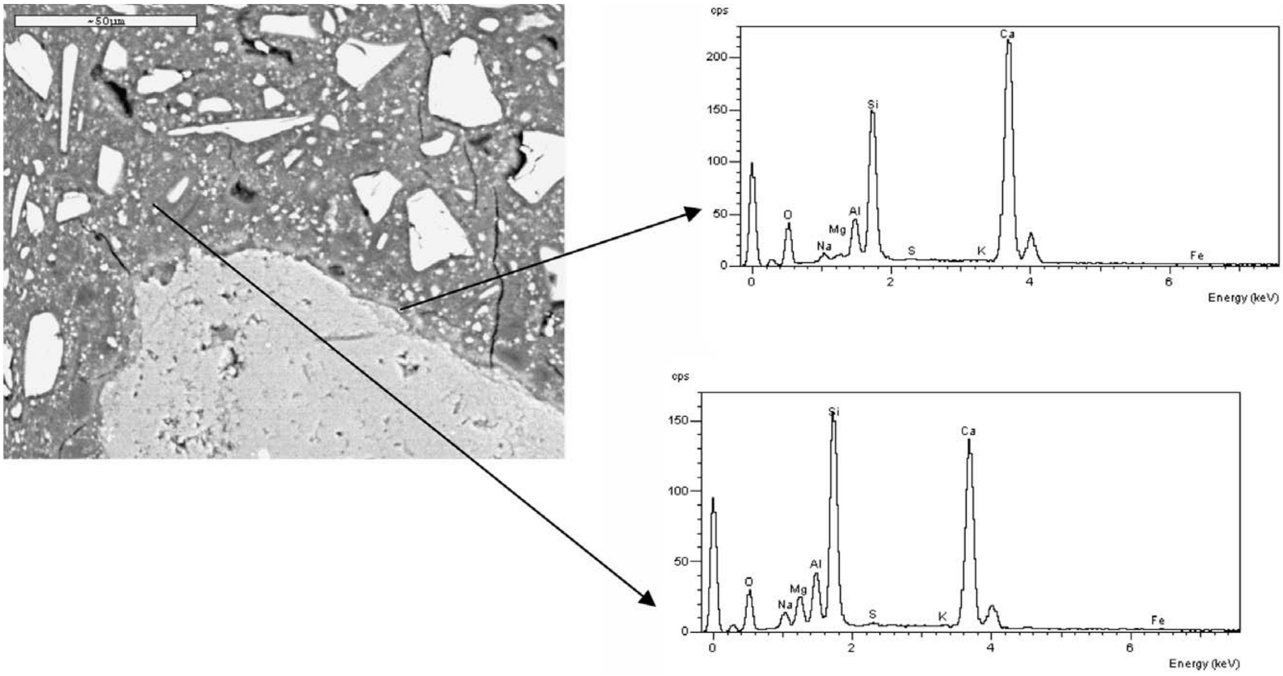


Fig. 8. SEM/BSE image and analysis of AAS-C_2 mortar after 14 days of accelerated test.

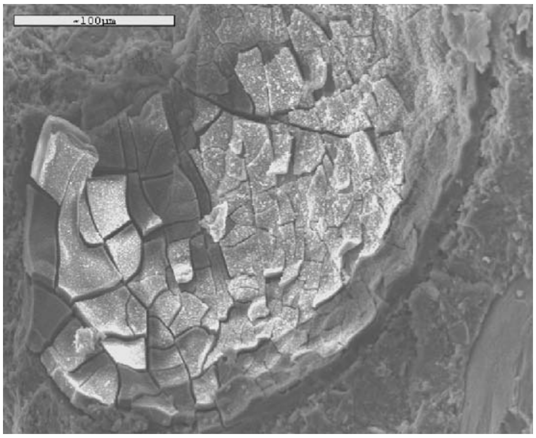


Fig. 9. Mortar OPC-S_2 after 14 days of accelerated test. Morphology of ASR in a pore.

products observed in the OPC-S_2 mortar are shown in Fig. 10. The formation of these expansive reaction products induces a decrease of the total porosity and the compressive strength as it has been discussed above. On the contrary, the morphology of the ASR products in the AAS mortars differed slightly from the findings described for the OPC mortars. Some of the morphologies and compositions of these ASR products observed in sample AAS-S_2, are given in Fig. 11. In this case, the formation of the expansive products does not have a negative effect on the compressive strength (see Fig. 3). In both mortars (OPC-S_2 and AAS-S_2) the reactivity of siliceous aggregate is due mainly to the presence of feldspars (see Table 2) in this aggregate. These feldspars suffer hydrolysis in the accelerated conditions due to an exchange between the metal ions of the feldspars and the ions present in the solution [33]. This hydrolysis process is increased by the high pH of the solution and the temperature.

The OPC and AAS mortars with dolomite calcareous aggregate exhibited no variations in volume or adverse effects on their strength; nonetheless, the mineralogical characterization study conducted on the two mortars showed that alkali-calcareous aggregate reactions did take place after 14 days of test and that these reactions depended on the nature of the binder. The diffractograms for binder-enriched samples OPC-CR_1 and OPC-CR_2 obtained after the accelerated test are reproduced in Fig. 12a. Note that the diffraction lines assigned to dolomite at *d* (Å) 2.89 and 2.19 and the ettringite phase at *d* (Å) 9.66 and 5.60 disappeared in the sample stored in 1 N NaOH at 80 °C (OPC-CR_2). The low

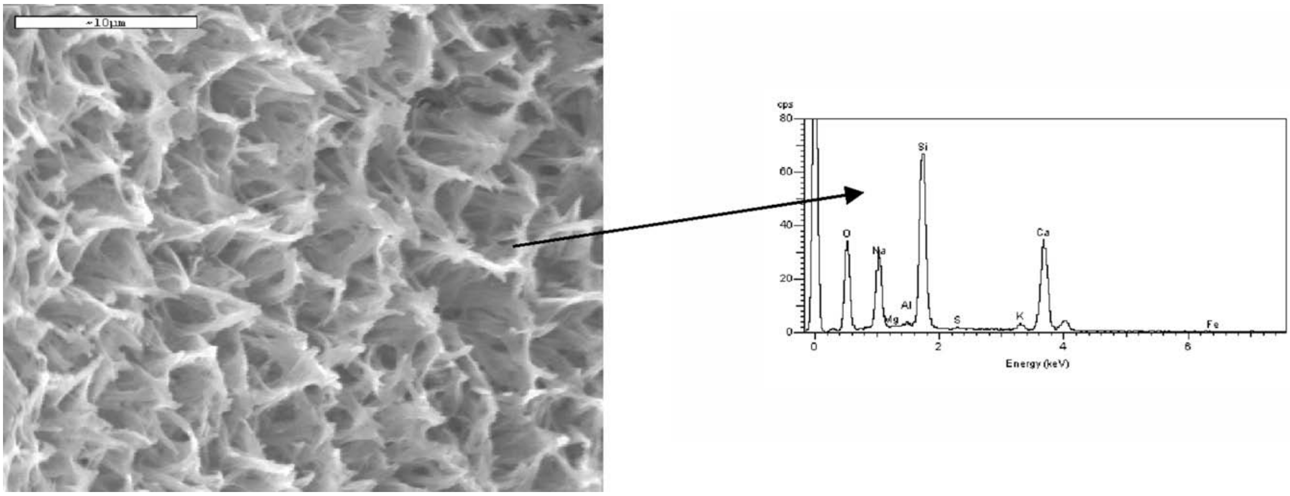


Fig. 10. Morphologies and compositions of ASR products in OPC-S_2 mortar after 14 days of accelerated test.

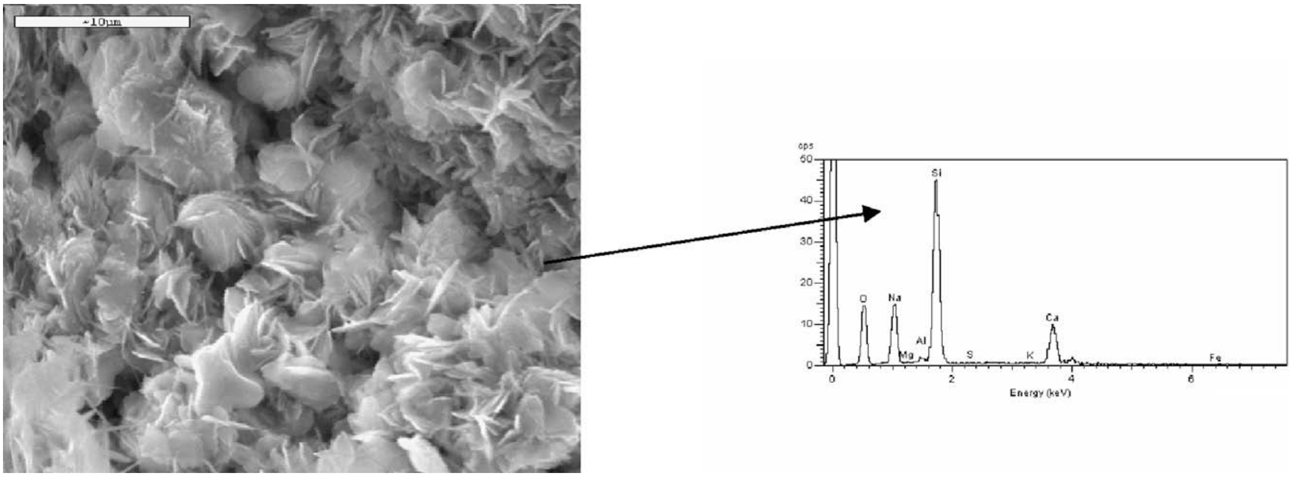
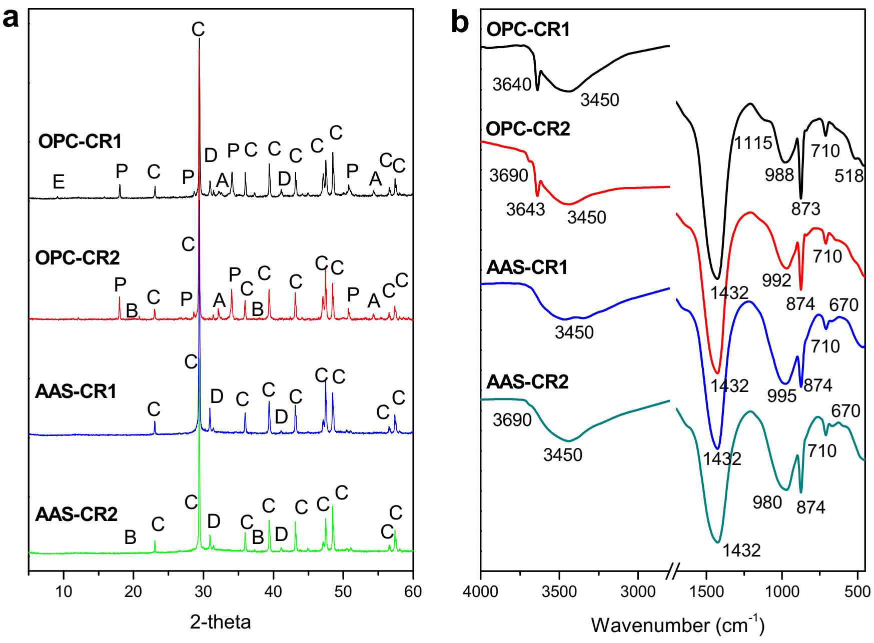


Fig. 11. Morphologies and compositions of ASR products in AAS-S_2 mortar after 14 days of accelerated test.



P = portlandite, C = calcite, D= dolomite, A =alite, E=ettringite, B=Brucite

Fig. 12. (a) XRD and (b) FTIR of OPC and AAS mortars after 14 days of accelerated test.

intensity signals at *d* (Å) 4.76 and 2.36 on this diffractogram were attributed to $Mg(OH)_2$. The FTIR study also detected an absorption peak at $3690\ cm^{-1}$, attributed to the hydroxide O-H stretching vibrations in brucite ($Mg(OH)_2$) (see Fig. 12b). In addition, these spectra confirmed the absence of ettringite in the OPC-CR_2 mortars, for no present of S–O vibration bands of SO_4^{2-} groups at around $1115\ cm^{-2}$,that identify that hydrated phase.

Fig. 12a shows the diffractrograms for binder-enriched samples AAS-CR_1 and AAS-CR_2. In this case the dolomite signals did not disappear from sample AAS-CR_2 (stored in 1 M NaOH at 80 °C); nor did the XRD study clearly identify the formation of brucite ($Mg(OH)_2$). The shoulder at around $3690\ cm^{-1}$ on the FTIR spectrum (see Fig. 12b), however, may indicate the formation of this phase, albeit in a lower proportion than observed in the OPC samples.

The de-dolomitization of dolomite aggregate as a result of the reaction with alkaline solutions leads to the neo-formation of brucite ($Mg(OH)_2$) and the regeneration of the alkaline hydroxide, as shown below [34]:

$$CaMg(CO_3)_2 + 2NaOH \longrightarrow Mg(OH)_2 + CaCO_3 + Na_2CO_3$$
$$Na_2CO_3 + Ca(OH)_2 - \longrightarrow CaCO_3 + 2NaOH$$

The findings of the present study showed that this process was more intense in OPC than in AAS mortars, probably due to the presence of $Ca(OH)_2$ in the former and its absence in the latter.

Ettringite decomposed in both types of mortar after storage in 1 M NaOH at 80 °C. The explanation may be found in the instability of this mineral at temperatures of over 70 °C [35] in the presence of a high concentration of alkalis. According to Berger [36], ettringite decomposes in solutions with over 2.7% by weight of Me_2O (Me = Na, K, Li).

4. Conclusions

The OPC and waterglass-AAS mortars containing siliceous and calcareous aggregate behaved differently under the test conditions applied in this study (based on ASTM standard C1260-94):

(a) When a non-reactive calcareous aggregate was used, no variations in volume were observed in any of the mortars, although a substantial rise was recorded in the mechanical strength of AAS mortars exposed to the most aggressive conditions (1 M NaOH and 80 °C). Ca^{2+} ion diffusion from the aggregate toward the matrix was also observed, with the formation of highly cohesive aggregate-paste interfaces.

(b) Although both the OPC and the AAS specimens made with reactive siliceous aggregate expanded under the most aggressive conditions (submerged in 1 M NaOH at 80 °C), expansion was nearly four times larger in the OPC than the AAS mortars.

(c) When a calcareous (dolomite) reactive aggregate was used, none of the mortars expanded after 14 days; nonetheless, the results of the characterization study conducted showed that the dolomite reacted in both mortars and that calcareous-alkali products (brucite) did in fact form. These reactive processes were more intense in OPC than in AAS mortars, probably due to the absence of portlandite in the latter.

(d) Unlike the OPC mortars, under the most aggressive conditions for the aggregate the AAS mortars exhibited a decline in total porosity and a very significant increase in mechanical strength. These effects were probably due to the reaction between the remaining anhydrous slag and the alkaline solutions in the medium, with the concomitant formation of new hydration products.

In summary, under the test conditions applied in this study, waterglass-AAS mortars proved to be higher in strength and more resistant to the alkali-aggregate reaction than their OPC counterparts. Also, AAS mortars are more vulnerable to alkali-aggregate reaction and expansion with siliceous aggregate than calcareous aggregate.

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