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# DURABILITY OF ALKALI-ACTIVATED FLY ASH AND SLAG CONCRETE

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**ABSTRACT:** Alkali-activated materials (AAM) have received increasing interest during the past few decades due to their environmental and technological advantages. However the durability of these binders is still considered as an unproven issue that needs to be addressed before their commercial adoption and large-scale production. The present work is focussed on the investigation of durability performances of concretes manufactured through the alkaline activation of fly ash and slag blends using a low-concentrated activating solution (formulated by blending commercial sodium silicate and sodium hydroxide). The aim of this study is to provide new insights on durability properties of AAM and to assess their resistance under severe conditions. For that purpose, concrete specimens have been exposed to accelerated carbonation (varying the curing and the exposure times from 7 to 28 days). After 28 days curing, their chloride resistance has been assessed using the non-steady-state chloride migration experiments following the NordTest method (NT Built 492).

Prior to durability test, the compressive strength of investigated mixtures was determined at early ages (1, 7 days) and after 28, 56, 90 days curing. The slag-rich mixtures have shown high compressive strength values (~45MPa after 1 day and ~80MPa after 28 days curing). Excellent mechanical properties have been also found for the fly ash-rich mixtures at early and late curing ages (15MPa after only 1 day and ~50MPa after 28 days curing). From carbonation test, it has been found that the carbonation depth increases as increasing fly ash/slag ratio or the exposure time. However when curing time is increased, a decrease on carbonation depth was observed. Slag-rich concretes were practically not carbonated after 7, 14 days exposure and showed 3-7mm carbonation depth after 28 days exposure. A higher carbonation depth (~18mm) was observed in fly ash-rich mixture. When the concrete specimens were exposed to chloride ingress, a high chloride permeability associated with higher concrete porosity was found for fly ash-rich mixtures with a chloride penetration depth of 18mm and chloride migration coefficient near  $15-17 \times 10^{-12} \text{m}^2 \text{s}^{-1}$ . These parameters decreased considerably as increasing slag content reaching values near 5mm and  $2 \times 10^{-12} \text{m}^2 \text{s}^{-1}$ , respectively.

**Keywords:** Geopolymer, Alkali-activated materials (AAM), Durability, Fly ash, slag.

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## INTRODUCTION

The growing demand for concretes with high performances, lower cost and reduced environmental impact when compared to those produced with conventional Portland cements has promoted the development of clinker-free or almost free alternative cementitious materials. Among them, alkali-activated materials (AAM) [1-6] and those classified as geopolymers [7, 8], are a high-profile example able to produce cement and concrete with the advantages of Portland cements but with a large reduction in CO<sub>2</sub> emissions and general aspects of good performances such as high mechanical strength at early ages of curing, high stability in aggressive environments and resistance to elevated temperatures, among others [9-13]. The bulk application of these new binders is still limited because there are still some uncertainties regarding their durability and degradation mechanisms under severe conditions [14-17]. The effect of chloride diffusion and CO<sub>2</sub> exposure on the concrete structure is probably the most extensively observed and assessed aspect of concrete durability as both attacks lead to the degradation of embedded steel reinforcing and account for over 50% percent of the deterioration of concrete structures [18]. However, there is limited existing knowledge about these phenomena in concretes based on AAM despite the increasing interest on durability issues of these new binders along the past few years. In most published works dealing with AAM resistance either to carbonation or to chloride ingress, there is no general agreement whether these materials exhibit better, similar or worse durability performances than conventional cements. There are also some contradictory conclusions regarding the evolution of carbonation rate, the effect of carbonation on mechanical properties or even the correlation between natural and accelerated carbonation of AAM [19-25]. From thermodynamic calculations related to pore-solution chemistry, Bernal et al. [21] stated that if an alkali-activated concrete and a Portland cement concrete show the same carbonation depth in an accelerated test, the AAM will suffer less carbonation under natural conditions, and thus the service life would be much longer. Low et al. [19] found that the carbonation of alkali activated slag was higher than that of blended slag-Portland cement, and Portland cement based concrete. Hakkinen [22] observed 40% higher compressive strength after 22 months of accelerated carbonation compared to the same materials after 28 days of curing; however Bernal et al. [23, 24] found that these materials decrease notably in compressive strength with accelerated carbonation. Bakharev et al. [20] reported higher strength reduction and higher susceptibility to carbonation in alkali-activated slag concrete than in ordinary Portland concretes. Conversely, Deja [25] reported that alkali-activated slag mortars and concretes showed carbonation depths comparable to those obtained for reference samples of Portland cement, along with increased compressive strengths as increasing exposure time to CO<sub>2</sub>. These results were associated with a refinement of the pore structure, as carbonates precipitated during the carbonation reaction. It has been reported [26] that AAM demonstrate better performance against chloride ingress, according to both accelerated (NordTest NT Build 492) and ponding (ASTM C1543) methods; but Law et al. [19] concluded that alkali-activated slag concretes exhibited lower durability properties (water sorptivity, chloride and carbonation resistance) than Portland cement and blended concretes.

Considering the current state-of-the-art, there are still some uncertainties and lack of consensus regarding the durability performances of AAM mostly when compared to Portland cement under carbon dioxide or chloride ingress. The present study reports an investigation on durability performances of alkali-activated fly ash and slag concretes. The aim of this work is to provide new insights for a better understanding of durability properties of AAM and to assess their resistance under accelerated carbonation and chloride ingress.

## MATERIALS AND METHODS

### Materials

Solid precursors used for the preparation of concretes are a class F fly ash (according to ASTM C618-12a [27] from VLIEGASUNIE BV and a blast furnace slag (BFS) supplied by ORCEM (The Netherlands). FA has a specific gravity of  $2440 \text{ kg/m}^3$  and 82% of particles with size less than  $45 \mu\text{m}$ , compared to  $2890 \text{ kg/m}^3$  and 95% of particles with an average size of  $45 \mu\text{m}$  for BFS. The chemical compositions of the FA and BFS determined by X-ray fluorescence (XRF) spectroscopy are shown in Table 1. Gravel is crushed granite with nominal sizes of 4-16 mm and a specific gravity of  $2640 \text{ kg/m}^3$ . Fine aggregate is natural sand having a specific gravity of  $2620 \text{ kg/m}^3$  and particle size of 0-4 mm. All mixtures were activated using a multi-compound activator solutions (low concentration) formulated by blending a commercial sodium silicate solution (27 wt%  $\text{SiO}_2$ , 8 wt%  $\text{Na}_2\text{O}$  and 65 wt%  $\text{H}_2\text{O}$ ), and NaOH solution. The  $\text{Na}_2\text{SiO}_3$  solution supplied by BRENTAG (Netherlands) has a specific gravity of  $1350 \text{ kg/m}^3$  and a modulus ratio  $M_s=3.37$ .

Table 1 Chemical composition of FA and BFS as deduced from XRF data

Oxide	$\text{SiO}_2$	$\text{Al}_2\text{O}_3$	$\text{CaO}$	$\text{Mg}$	$\text{Fe}_2\text{O}_3$	$\text{SO}_3$	$\text{Na}_2$	$\text{K}_2$	$\text{TiO}$	$\text{P}_2\text{O}$	L.O.
BFS	34.4	11.53	39.1	7.81	1.42	1.6	0.23	0.58	-	-	1.15
FA	54.2	23.32	4.23	1.62	8.01	0.6	0.85	1.97	1.23	0.54	3.37

### Concrete preparation

Concrete specimens were produced in a 40 L laboratory pan mixture by mixing first the coarse and fine aggregates for 2 min followed by the precursors for additional 3 min to get a homogenous mixture and the premixed alkaline activator solution (prepared one day before casting) was then added gradually and the mixing continued further for about 3-5 min. The concretes were produced in 150 mm cubic moulds with binder content of  $400 \text{ kg/m}^3$ , activator solution to binder ratio of 0.50 and a binder to aggregates ratio of 1:4.3 by weight. After casting, The specimens were sealed and cured in laboratory conditions for 24 h, then demoulded and stored in a curing chamber (99%RH,  $20 \pm 2 \text{ C}$ ) until testing. Three mixtures with the following FA/BFS ratios 70:30, 50:50, 30:70 named respectively S30, S50 and S70 have been prepared.

### Testing methods

The concrete carbonation resistance was assessed according to a slightly modified form of the standard EN 13295:2004 [28]. After the desired curing is achieved (7, 14, 21 and 28 days) the specimens were removed from the humidity chamber, and then the cylinders needed for carbonation test ( $\phi=75 \pm 1 \text{ mm}$ ,  $h=150 \pm 1 \text{ mm}$ ) are drilled from cubic specimens. Drilled samples are dried for 24h in laboratory condition ( $55 \pm 5\% \text{ RH}$ ,  $20 \pm 2^\circ\text{C}$ ) and immediately transferred to the carbonation chamber for carbonation testing. This procedure was applied to all samples regardless their curing period.

Concrete specimens of different mixtures were exposed during 7, 14, 21 and 28 days to  $1 \pm 0.1\% \text{ CO}_2$ , at a temperature of  $21 \pm 2^\circ\text{C}$  and RH of  $60 \pm 5\%$ . Specimens were removed from the chamber after achieving the desired exposure time, and the corresponding carbonation depth was measured by treating the surface of a freshly cleaved specimen with a 1 % solution of phenolphthalein in alcohol. Depending on the alkalinity of concrete contact area with the

phenolphthalein, purple-red coloration (noncarbonated) and colourless region (carbonated) can be distinguished. The carbonation depth was measured in 6 different points per sample using four replicate samples for each mixture.

The rapid chloride migration (RCM) test was conducted following the NordTest method NT Build 492 [29] using cylindrical specimens with a diameter of 100 mm and a thickness of 50 mm, sliced from drilled cores of  $\phi 100 \times 150 \text{ mm}^2$  after 28 days of curing. A 0.1 M silver nitrate ( $\text{AgNO}_3$ ) solution was applied to freshly split samples to determine chloride penetration depth.  $\text{AgNO}_3$  reacts with both chloride and hydroxyl ions to form white silver chloride and dark brown silver oxide, respectively. Chloride penetration depth is measured as the visible boundary between white precipitation of silver chloride when chloride ion is present, and precipitation of brown silver oxide.

## RESULTS AND DISCUSSIONS

### Compressive strength

The dependence of compressive strength on curing ages of investigated concrete mixtures is shown in Figure 1. It can be observed that the compressive strength increases as increasing slag content reaching values close to 100 MPa for S70 after 90 days of curing. It can be also deduced that all mixtures exhibit high early age strength even for fly ash-rich mixture (S30) which has about 15 MPa after 1 day and near 40 MPa after one week of curing. It is important to highlight that the compressive strength of this mixture after 56 and 90 days of curing are practically the same. Contrarily, concrete mixtures with slag amount  $\geq 50\%$  are still developing additional strength during the same curing period but with strength gain compared to early age strength development.

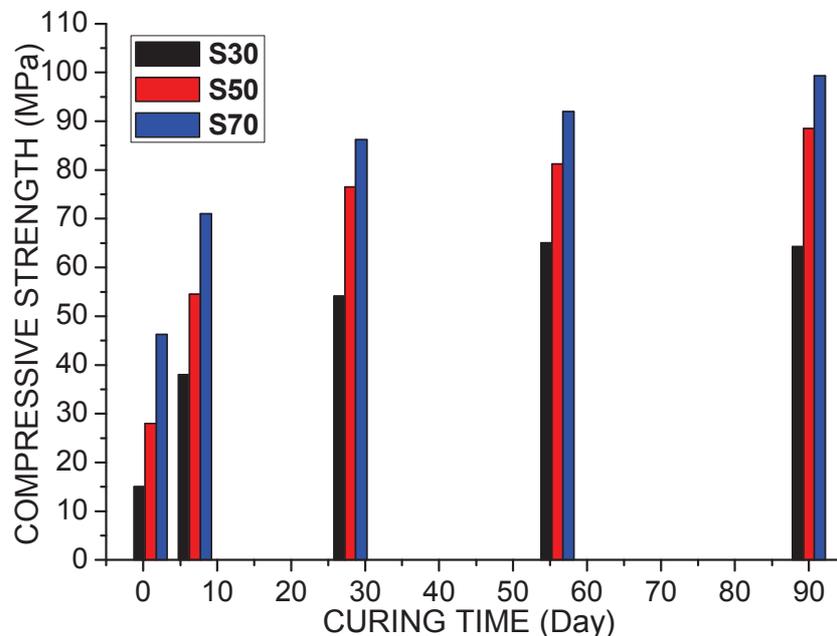


Figure 1 Compressive strength of the indicated concrete mixtures after 1, 7, 28, 56 and 90 days of curing

## Carbonation resistance

The investigated concretes have been cured at different ages and then exposed to 1% CO<sub>2</sub>. Their carbonation resistance has been assessed through the evaluation of the carbonation depth measured by mean of the phenolphthalein test. The effects of the exposure time, the fly ash to slag ratio as well as the curing time on carbonation depth are shown in figure 2 and 3.

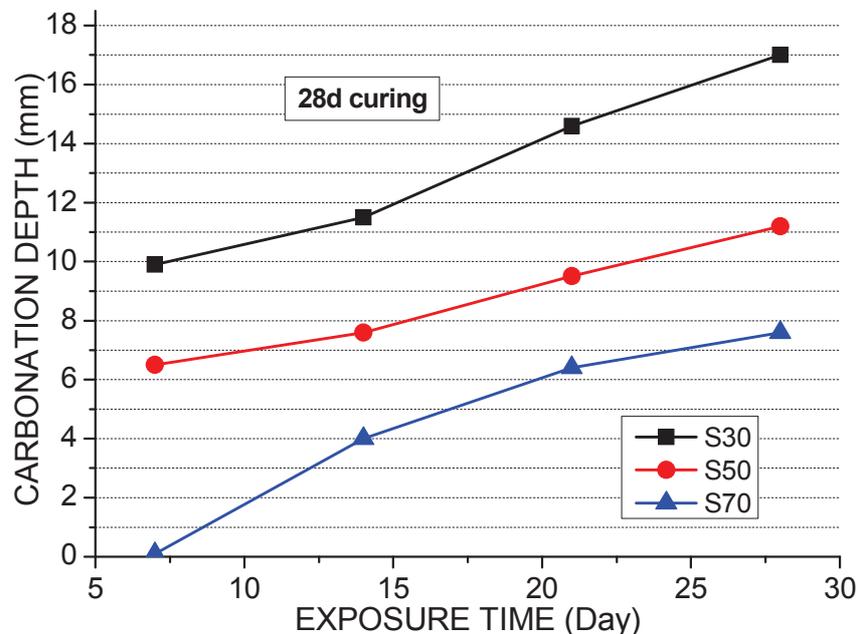


Figure 2 Effect of exposure time on carbonation depth of S30, S50 and S70 after 28d curing.

It can be observed from Figure 2 that, regardless the exposure time, the fly ash-rich mixture exhibits the highest carbonation depth and the slag-rich mixture the lowest one. This trend could be related to the higher porosity of concrete with high fly ash content [26]. An increase of the slag content produced a refinement of the concrete pore structure making difficult the penetration of CO<sub>2</sub>. After 7 days exposure, S70 was not carbonated whereas S50 and S30 show a carbonation depth of about 7 and 10 mm, respectively. As increasing the exposure time, all mixtures are carbonated but keeping the tendency already mentioned previously i.e. the slag-rich mixture was less carbonated than the porous concrete containing higher fly ash amount. After 28 days exposure, the carbonation depth of S30 reaches 17 mm which is about 1.5 times higher than for S50 and more than 2.5 times higher than that of S70. A comparison of the observed slopes shows that the mixture S30 shows a higher carbonation rate than S50 and S70. Based on these findings it could be expected that for reinforced fly ash/slag based concretes, the passivating layer will be damaged first in S30 then in S50 and finally in S70. Consequently S70 could exhibit the longest service life among the 3 mixtures.

The effect of curing time on carbonation resistance of the 3 investigate mixtures is shown in figure 2. At all curing ages, the increase of fly ash to slag ratio increases the carbonation depth. At early age of curing (7, 14 days), all samples are carbonated with a carbonation depth ranging from 6 to 18 mm. When the curing time is increased, a decrease on carbonation depth was observed. This decrease is induced by the refinement of the pore structure and the densification of concrete matrix as the geopolymerization process continues. After 28 days of curing, the slag-rich concrete exhibits a very low permeability. This mixture is not carbonated after 7 days of CO<sub>2</sub> exposure and its carbonation depth does not exceed 8 mm after 28 days

exposure. At 7 and 14 days of curing, the slopes of the exposure time dependence of the carbonation depth are practically similar indicating that the carbonation rate of the 3 mixtures is quite similar as the gel maturity (responsible for hardening and densification of the concrete) is not enough to prevent a high-rate CO<sub>2</sub> penetration. At increased curing ages (21 or 28 days), The slopes in S50 or S70 are lower than that of S30 due to the higher slag content which in turn improves the density and reduces the concrete chloride permeability.

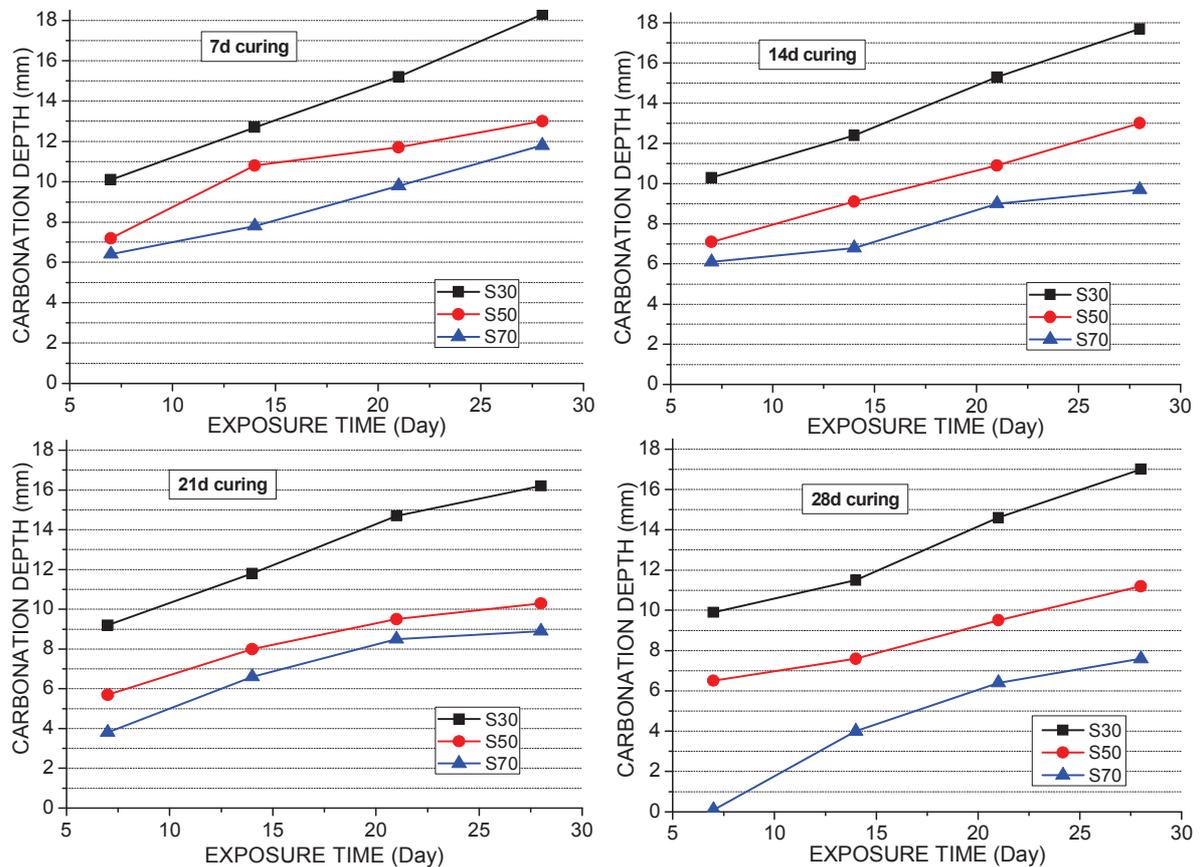


Figure 2 Effect of curing time on carbonation depth of S30, S50 and S70.

On the other hand it can be deduced that despite the lower carbonation depth mostly observed in slag rich concrete, these values are very high compared to those reported in aging structures (In Russia, Ukraine and Poland) where a carbonation rate of aged structures between 12 and 40 years does not exceed 1mm/year [5]. The divergence of these results open a new debate on the correlation between natural and accelerated carbonation resistance of alkali activated materials. The curing time before starting the accelerated carbonation test could be one of the parameters that affect the nature of the reaction products formed after carbonation as it is directly related to the microstructure development and gel maturity. According to Bernal et al. [21], the influence of gel maturity is essential when analysing the results of accelerated carbonation. During the exposure to natural carbonation the binder structure evolution lasts long term of period (years), while accelerated tests are applied on relatively young concretes for short time of period (weeks).

### Chloride resistance

The rapid chloride migration test (RCM) according to NT Build 492 standard has been used to evaluate the resistance of the investigated mixtures to the chloride ingress. For sake of comparison, the chloride resistance of Portland cement concrete (Cem I 42.5 R) used as

reference has been also assessed. From Figure 3, it can be deduced that the chloride resistance in alkali activated concretes is higher than that of Cem I which has a carbonation depth close to 25 mm. These results are in a good agreement with those reported by Ismail et al. [26]. Among the 3 investigated concretes, S30 exhibits the highest chloride penetration depth (~18 mm) and S70 the lowest one (~5mm) which indicates that the increase of slag content increases the concrete resistance to the chloride ingress. The non-steady-state migration coefficients deduced from the chloride penetration depth (Equation 4 in ref. 29) are 17.6, 4.4 and  $1.91 \times 10^{-12} \text{m}^2 \text{s}^{-1}$  for S30, S50 and S70, respectively.

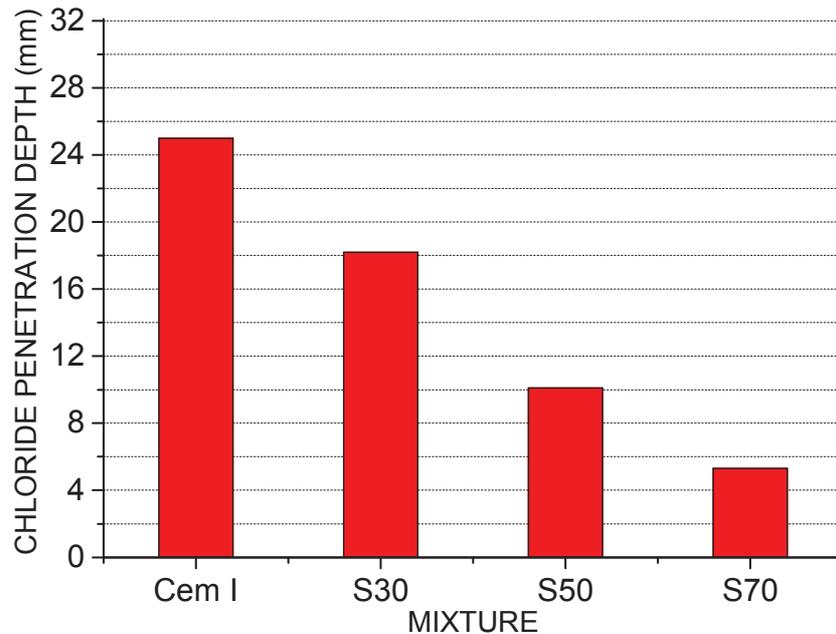


Figure 3 Evolution of the chloride penetration depth of Cem I, S30, S50 and S70 at 28 days of curing.

The initial current values obtained during the RCM test (applying a voltage of 30V conforming to NT Build 492 standard) are 85, 162.3, 52.3 and 33.8 mA for Cem I, S30, S50 and S70, respectively. This parameter could provide preliminary information about the concrete porosity. Taking into account that the measured current results from the migration of mobile species through the concrete pores, it can be deduced that S30 has an open pore structure (higher porosity) in comparison with S50 or S70 where the concrete matrix becomes denser as increasing the slag content. Therefore the chloride ingress will be easier in S30 than S50 or S70 which is in a good agreement with the measured chloride penetration depth values (see figure 3). However this trend is not observed when comparing Cem I with S30. Cem I has a lower initial current but higher chloride penetration depth. Several possible reasons could explain this behaviour: i) Despite the higher porosity of S30 in comparison to that of Cem I, the critical pore size may not be favourable for an optimum chloride mobility in the fly ash-rich concrete. The accommodation of mobile chloride species in the conduction paths connecting the pores is not suitable because they may spend more time between the wall-paths (zigzag movement) of bigger pores before crossing the specimen section in S30 than in Cem I. In accordance with this hypothesis, it has been reported [30] that the critical pore size in alkali silicate powder activated slag concretes is more influential than porosity in determining the chloride transport properties. ii) At a given initial voltage (30 V here), the measured initial current will define the applied voltage for each RCM test. As in the fly ash-rich mixture the measured initial current could involve chlorides and other mobile species,

this could underestimate/affect the value of the resulting applied voltage before starting the RCM test leading to a lower chloride penetration for S30 than Cem I. finally these results could rise some concerns regarding the suitability of the RCM test for the evaluation of chloride resistance in alkali activated materials and the need for new standards.

## CONCLUSIONS

The carbonation and chloride resistance of alkali activated fly ash and slag concretes have been examined in this study. It can be concluded that the carbonation depth is increased as increasing the fly ash to slag ratio or the CO<sub>2</sub> exposure time but decreases with increasing the curing age. Slag rich-concrete exhibits better durability performances than fly ash-rich mixture either against CO<sub>2</sub> or under chloride ingress. Cem I shows higher chloride penetration than alkali activated concretes even when compared with a high-volume fly ash concrete (S30). This behaviour was mainly related to the concrete porosity especially the pore distribution/connection and the critical pore size within the concrete matrix. The conventional testing protocols used to evaluate the durability of Portland cement are not necessary suitable for the assessment of durability performances in AAM.

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