

DURABILITY OF GEOPOLYMER CONCRETE



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Summary

The durability of concrete in general is one of the most significant issues within the civil engineering community. This concern is even more important for geopolymer-based concrete as it is still considered as one of the unsolved problems towards their large-scale production and commercialization. The present report discusses test results with regard to the durability of three concretes produced and supplied by SQAPE (named MI, MII and MIII) derived from the alkaline activation of industrial by-products. The results are focused on their resistance to sulfate attack, chloride ingress and carbon dioxide penetration. A comparison of the investigated concretes shows that MI has an excellent durability performance with regard to the aggressive agents used in this study. In comparison with OPC concrete or other mixtures prepared with alkali-activated aluminosilicates, MI has demonstrated a similar or even better performance and could be considered a suitable candidate as resistant concrete to be used in civil structures. Mixture MIII showed a relatively high vulnerability mainly to chloride and CO₂ ingress. MII presents acceptable performances with a good resistance to carbonation and chloride ingress.

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I. Introduction

Among the family of alternative cements also referred as non-Portland cements, alkali activated materials (AAM) have emerged as new engineering composites with relatively high performances such as good mechanical properties in the hardened state, good durability during service, a reduced environmental footprint, and acceptable early-age rheology [1,2], if adequately composed and proper curing conditions are adopted. AAM are basically formed by a reaction between an alkali source, often (but not necessarily) supplied as a liquid, and a solid aluminosilicate either found in nature (metakaolin, pozzolan, etc.) or derived from industrial by-products/wastes such as powder coal fly ash (PCFA), ground granulated blast furnace slag (GGBS), silica fume (SF), rice husk ash (RHA). PCFA and GGBS have been reported as the most promising precursors for large-scale industrial production of geopolymer concretes due to the more favorable rheological properties and lower water demand achievable when compared to mixes based on other precursors such as calcined clays. Chemical principles, reaction phenomena and engineering properties of AAM have been studied extensively [2, 3-9]; however durability is still regarded as the key issue and unsolved question in the development and application of alkali activation technology [10].

This report presents results on the performance of three different (PCFA/GGBS) varying alkali activated concrete mixtures which are not optimized to any specific practical application. The aim of this study is to determine their durability properties in term of resistance mainly to sulfate attack, chloride ingress and carbonation for a better understanding of their behavior in these aggressive media. A comparison between obtained results and others reported elsewhere either on OPC concrete or dealing with other alkali activated materials as well as some concluding remarks and recommendations are also provided in this report.

II. Test methods

Specimens subjected to durability testing have been casted in steel cubic moulds with side length of 150 mm by SQAPE. In total 3 mixtures with varying binder compositions were tested:

- MI - high GGBS, low PCFA
- MII - moderate GGBS, moderate PCFA
- MIII - low GGBS, high PCFA

After 24 hours curing in ambient/laboratory conditions, specimens have been demoulded and transferred to the controlled-humidity chamber (20°C, 99%RH) in the Macrolab (TU Delft) where they have been cured for additional 27 days before starting durability tests (Figure 1). The tested mixtures were defined to have an average composition/workability:

- River aggregates sand 0-4mm and gravel 4-16mm
- Binder content 400 kg/m³
- L/B ratio 0.4-0.45
- Slump ≥ 170 mm (S4 classification)
- Slump flow ≥ 300 mm
- Compressive strength class C40/50

Additional information on concrete compositions, production method and related fresh and hardened properties are reported elsewhere [11].

To execute the durability tests of 3 investigated mixtures, cylinder-shaped-specimens of different sizes (Table 1) have been drilled from cubic samples with side length of 150 mm. The standards adopted for these tests as well as the parameters to be determined are summarized in Table 1.

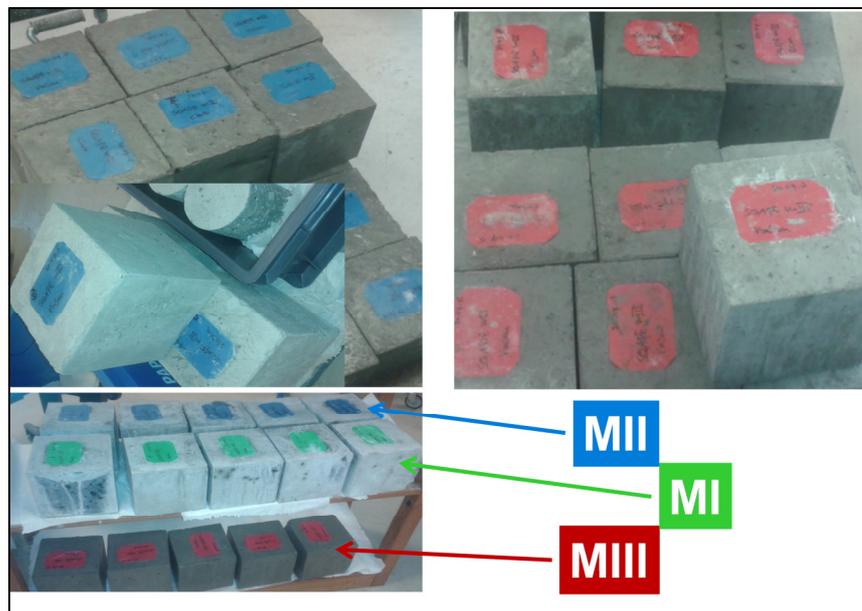


Figure 1. Specimens of 3 different mixtures provided by SQAPE for durability tests

Table 1. Standards, specimen size/shape and measured parameters for durability of SQAPE mixtures.

Durability test	Standards	Shape/size	Measured parameters
Sulfate	SIA 262/1 – App. D [12]	Cylinders ($\phi=34\text{mm}$, $h=150\text{mm}$)	<ul style="list-style-type: none"> • Length/volume • Mass loss • strength
Chloride	NT 492 (RCM) [13]	Cylinders ($\phi=100\text{mm}$, $h=50\text{mm}$)	<ul style="list-style-type: none"> • Penetration depths, x_d
Carbonation	DIN EN 13295-2004 [14]	Cylinders ($\phi=76\text{mm}$, $h=150\text{mm}$)	<ul style="list-style-type: none"> • Carbonation depth, d_k

III. Results

1. Sulfate resistance

To evaluate the length and mass changes after the sulfate tests, 6 cylinder specimens ($\phi=34\text{mm}$, $h=150\text{mm}$) have been drilled from 6 cubes of each mixture for the drying and immersion tests according to the adopted standard (Table 1). The strength evaluation has been conducted on cubic samples.

1.1. Compressive strength

Four cubic specimens of each mixture have been used to determine the compressive strength changes between 2 samples immersed in 5% Na_2SO_4 solution for 28 days and 2 samples kept in the laboratory conditions (reference samples) for the same period.

A decrease on compressive strength values in comparison with reference samples (kept in the laboratory) were observed after 28 days of sulfate immersion (Figure 2). The decrease was 10.84% for MI, 9.40% for MII and 17.44% for MIII. It can be observed that the reduction of mechanical strength is higher in MIII mixture (almost twice) indicating the lower resistance of this mixture for sulfate attack with respect to MI or MII which have a similar behavior. Taking into account that sodium sulfate could be considered as an activator, the compressive strength after sulfate immersion should increase. However, the opposite trend has been observed. The decrease of the compressive strength could be associated with the difference on relative humidity where reference samples were kept at $50\pm 5\% \text{RH}$ and immersed samples were fully saturated.

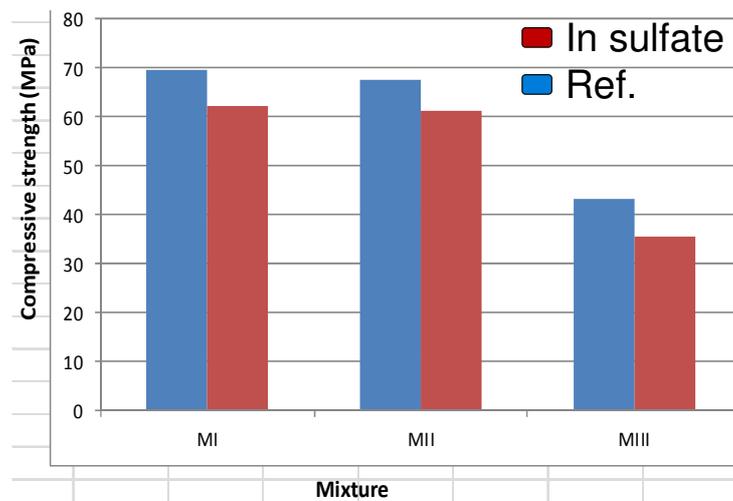


Figure 2. Compressive strength evolution of 3 mixtures after 28 days sulfate immersion (red) compared to reference samples without immersion (blue).

1. 2. Length and mass changes

For these measurements, 6 cylinder specimens ($\phi=34\text{mm}$, $h=150\text{mm}$) have been drilled from cubic samples of each mixture. Before starting the drying immersion cycles a metal plug has been glued on both ends of each cylinder using a sulfate resistant and volume stable glue for attaching the plugs to the specimens. After each cycle, the mass and length changes of all specimens were measured. The results obtained after cycle 1 and cycle 2 are given in Table 2. However, it was not possible to complete neither the rest of drying immersion cycles (cycle 3 and 4) nor the additional immersion cycles (without drying) due to a problem derived from sharing facilities (the oven in this case) with other users that happened in the beginning of drying process of cycle 3 after which the junction between metal plugs and specimen was broken making it difficult to measure correct values of mass and length. Measurements have been stopped after cycle 2 and it was not possible to repeat the test due to the lack of additional samples and limited time.

Table 2. Mass and length changes after 2 cycles of drying immersion in 5% Na₂SO₄ solution.

Mixture		Cycle 1		Cycle2	
		Δl_1 (%)	Δm_1 (Kg/m ³)	Δl_2 (%)	Δm_2 (Kg/m ³)
MI	1	6,69E-06	83,08084	1,34E-04	78,16785
	2	2,54E-04	81,00293	0,00123	77,5687
	3	1,34E-04	82,89654	4,28E-04	78,4295
	4	2,01E-05	82,96976	2,61E-04	80,07805
	5	3,34E-05	83,01867	8,88E-04	80,33101
	6	7,66E-05	82,24053	1,18E-04	80,9436
MII	1	2,67E-05	86,96572	6,01E-05	88,89664
	2	2,08E-05	83,36753	1,04E-04	84,29127
	3	6,02E-05	90,62166	1,40E-04	92,78465
	4	9,36E-05	84,91491	1,67E-04	85,87817
	5	2,08E-05	85,54414	1,04E-04	87,86032
	6	0,00244	85,14821	0,00318	84,77832
MIII	1	2,21E-04	114,0826	2,35E-04	114,1571
	2	-8,43E-05	113,3626	-9,13E-05	112,2695
	3	1,07E-04	121,9827	1,14E-04	122,6562
	4	7,01E-05	118,5432	4,91E-05	119,1688
	5	1,36E-04	114,3894	7,18E-05	113,9088
	6	6,60E-04	115,1353	2,52E-04	116,3724

From results obtained during cycle 1 and cycle 2, the variation of length for the specimens (Δl_i) is too small (10^{-3} - 10^{-6}) to be distinguished from the apparatus accuracy which makes it difficult to draw conclusions at this stage. Concerning changes on specimens weight, it could be concluded that after 2 cycles of drying-immersion, the mass increase is higher for MIII mixture (110-120 kg/m³) compared to that of MII (80-90 kg/m³) and MI (75-85 kg/m³). This result, which is in agreement with that deduced previously from compressive strength changes, indicates again the lower resistance of mixture MIII to sulfate attack.

2. Chloride resistance

For chloride resistance, the NT Build 492 standard (Rapid chloride migration, RCM) was adopted and cylinder specimens ($\phi=100$ mm, $h=50$ mm) have been used for this purpose.

The test starts with a preconditioning step in which specimens subjected to RCM are placed in a vacuum container for 3h and then a saturated Ca(OH)₂ solution was added. The specimens were kept in the solution for 18h. Immediately after saturation, the specimens are placed in the test

container as shown in Figure 3 and a voltage of 30V was applied between the anode/anolyte solution (0.3 M NaOH) and the cathode/catholyte solution (10% NaCl solution). Based on the initial measured current for each mixture, the duration of the RCM test is 24h and the applied voltage is 30 V for MI and MII mixtures but 15 V for mixture MIII.



Figure 3. RCM test of MI, MII and MIII mixtures at the beginning (left) and after 24h (right)

A visual inspection at the end of RCM test (right part of Figure 4) showed that the anolyte solution color of all mixtures has changed (more evident in the case of MII and MIII) and the electrode is slightly corroded.

After the RCM measurement, the specimens of each mixtures were split axially into 2 pieces (Figure 4) and a 0.1M AgNO_3 solution was sprayed onto the freshly split section. The chloride penetration depth was measured when the white AgCl_2 precipitates on the split surface is clear.

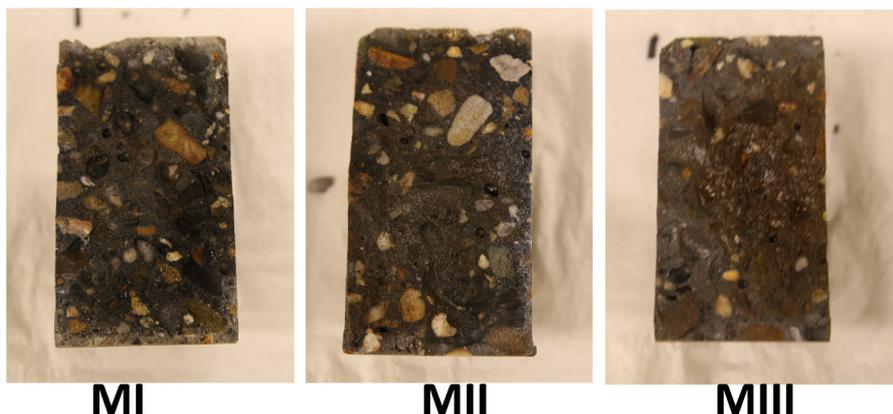


Figure 4. Aspect of split surfaces of the 3 mixtures after spraying the AgNO_3 solution

After the RCM test and the spraying of silver nitrate solution, the measured chloride penetration depth is about 5mm for MI, 6-9mm for MII and 18-23mm for MIII. From the

average value of chloride penetration depth, the deduced non-steady-state migration coefficients are $1.97 \times 10^{-12} \text{ m}^2/\text{s}$ for MI, $3.13 \times 10^{-12} \text{ m}^2/\text{s}$ for MII and $18.95 \times 10^{-12} \text{ m}^2/\text{s}$ for MIII.

3. Carbonation

For the 3 investigated mixtures, an accelerated carbonation test (1%CO₂, T≈22°C, RH≈60%,) in parallel with natural (indoor and outdoor) carbonation have been conducted on 6 cylinder specimens (ϕ 76mm, h=150 mm) drilled from 6 cubes of each mixture. For accelerated carbonation, specimens have been exposed during 28 and 56 days after which the carbonation depth (d_k) has been measured by applying phenolphthalein indicator on freshly broken specimens. All samples have been cured for 28 days before the start of the carbonation test.

4. 1. Carbonation depth after 28d exposure

As shown in Figures 5 and 6, specimens of 3 mixtures were carbonated after 28 days exposure to 1% CO₂. Through the phenolphthalein indicator, 2 different colors can be distinguished: i) the pinky area which corresponds to the non-carbonated region and ii) the uncolored area associated with the carbonated zone.

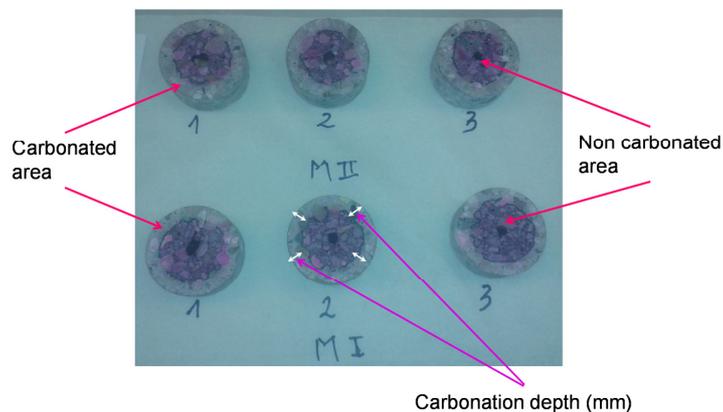


Figure 5. Aspect of carbonated specimens after spraying the phenolphthalein solution

The width of the carbonated area (Figure 6) which represents the carbonation depth (d_k) is higher for mixture MIII compared to MI and MII indicating a priori a higher carbonation rate and consequently a lower resistance to carbonation for MIII. The average carbonation depth value measured in 4 specimens of each mixtures is 11.1, 13.4 and 16.8 mm for MI, MII and MIII, respectively. The carbonation depths of each sample were determined by measuring 5

values in different points of the sample. The carbonation depth of each mixture was then determined by the average of carbonation depths of 4 samples from the same mixture.

After 28 days of natural carbonation (one sample stored in the laboratory and another kept in weathering conditions), the phenolphthalein test showed no color change for the three mixtures.

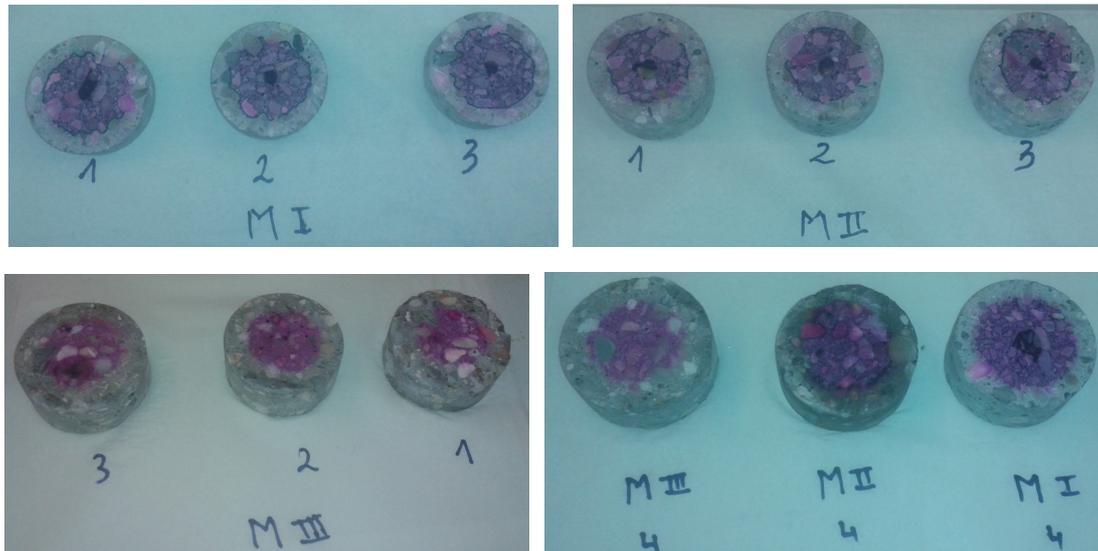


Figure 6. Visual comparison between carbonation depths of three investigated mixtures

4. 2. Carbonation depth after 56d exposure

From Figure 7, it can be observed that after 56 days exposure to 1% CO₂ all mixtures were carbonated and mixture MIII showed the highest carbonation depth followed by MII and then MI which showed less carbonated area after spraying phenolphthalein.

Applying the same procedure described previously, the measured carbonation depths are 16.1, 19.2 and 23.6 mm for MI, MII and MIII, respectively. These values follow the same tendency found after 28 days and showed again that the resistance to carbonation ingress is higher for mixture MI compared with mixture MII or mixture MIII which has the lowest resistance.

When specimens are exposed to natural carbonation (bottom of Figure 8), mixtures MI and MII were not carbonated. However, mixture MIII was slightly carbonated. The corresponding carbonation depth is about 4-5 mm.

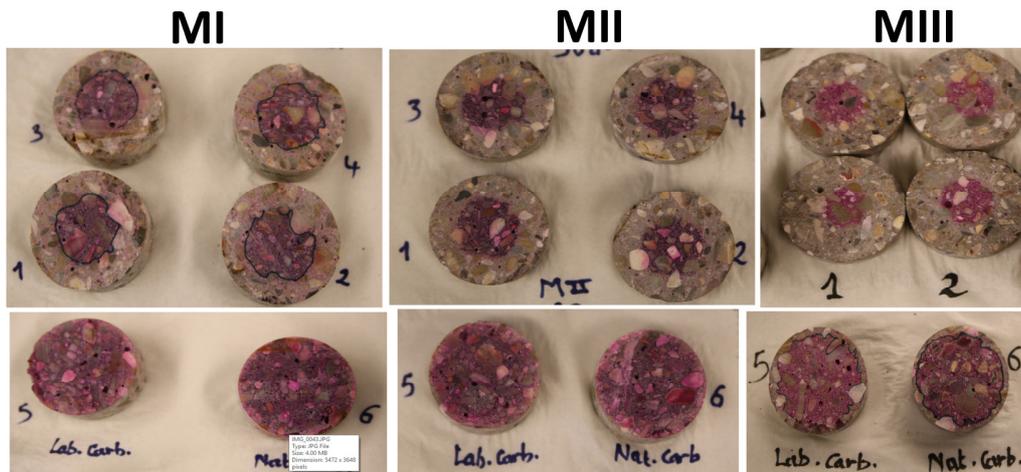


Figure 7. Aspect of the specimens after 56 days accelerated (top) and natural (bottom) carbonation of the 3 mixtures.

4. 3. Carbonation depth after 90d exposure

The accelerated carbonation of the 3 mixtures has also been investigated after 90 days exposure to 1%CO₂ and the obtained results are shown in Figure 8. It can be observed that the carbonation depth increased for all mixtures with respect to results obtained after 28 and 56 days and mixture MIII was almost completely carbonated (Figure 8, right) with a carbonation depth of about 29 mm. The same trend already observed for previous exposure conditions is kept after 90 days and the carbonation resistance is still the lowest for MIII in comparison with MI which exhibits a carbonation depth near 19 mm or MII whose carbonation depth was 22.3 mm.

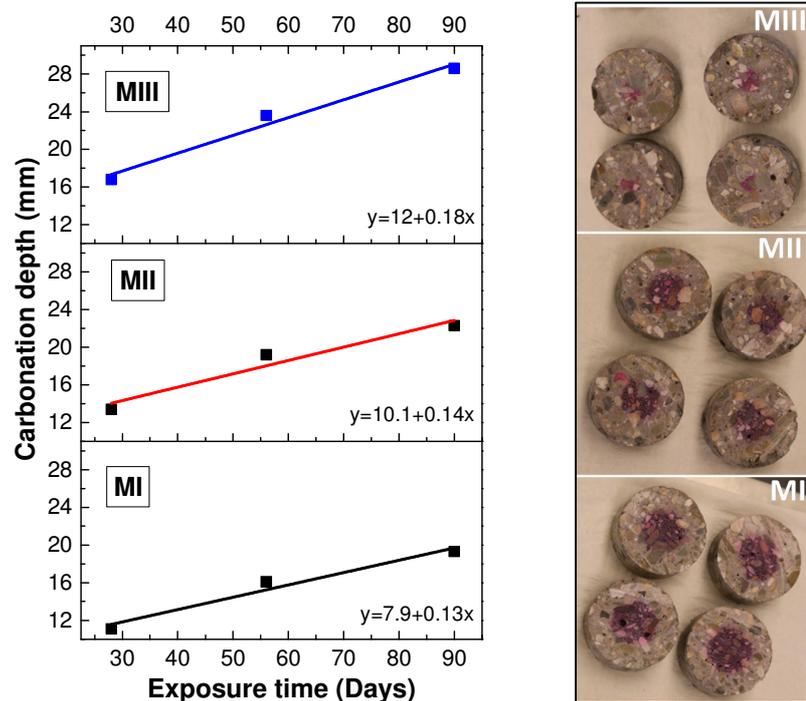


Figure 8. Evolution of carbonation depths in 3 mixtures after 90 days CO₂ exposure

IV. Comparison with OPC and other AAM

In this section we compare the results with regard to the durability of OPC-based concretes or other concretes derived from different alkali-activated materials taken from literature in order to check whether the investigated concretes in this report exhibit better, similar or worse durability performances.

Concerning sulfate attack results, only strength behavior will be considered for comparison with other AAM or OPC concretes.

In sulfate media, AAM have in general a better sulfate resistance than the OPC based concretes [15-17]. From the present study, the sulfate results have shown that compressive strength of the 3 mixtures was reduced after 28 days immersion in 5% sodium sulfate solution (~10% for MI and MII, ~17% for MIII). This trend has also been observed in other alkali-activated materials [15] or in OPC [15, 16]. A comparative study between alkali-activated slag (AAS) and OPC concrete exposed to 5% sodium sulfate solutions showed that the decrease of the compressive strength was 17% for AAS and about 25% for OPC based concrete [15]. However, in some others cases where the investigated concretes are mainly geopolymer, even an increase of compressive

strength was reported [18-19]. This behavior was attributed to the fact that geopolymer binder continues to stabilize and develop in the presence of the sodium sulfate (often used as an activator in alkali activated alumino-silicate systems), and the relatively high pH and high sodium content are helpful in maintaining the pore solution chemistry required for good stability of the geopolymer. Noting that in the present study, the reference samples used during sulfate test were stored in laboratory conditions ($50\pm 5\%$ RH, $20\pm 2^\circ\text{C}$). The big difference in relative humidity (50% in the laboratory and 100% in sulfate solution) could explain the reduction of compressive strength values of concretes MI, MII and MIII. Thus, the curing conditions of the reference samples (laboratory conditions, fog room, immersed in tap water) may have significant impact on their mechanical properties and could explain the divergent trends on compressive strength reported in the literature. Finally, it is important to highlight that in most reported results, the sulfate test was carried out over a long period (3 months to 2 years) whereas in the present study concrete specimens were immersed in sulfate solution for only 28 days. For a better comparison, an extended period of sulfate immersion is recommended.

The carbonation resistance of the 3 investigated mixtures will be compared with other results found in literature for similar mixtures and also in comparison with OPC concrete. Figure 9 shows the variation of carbonation depth as a function of time exposure to 1% CO_2 for the 3 investigated mixtures (right) and an alkali-activated mixture of slag/metakaolin taken from literature [20].

Similar behavior is observed after 28 days with a carbonation depth ranging between 12 and 18 mm (1% CO_2) for both mixtures (SQAPE and literature). After 42 days accelerated carbonation, the reference mixtures showed similar carbonation depth values compared to mixtures MI, MII and MIII but after 56 days exposure the results indicate that the SQAPE mixtures have a better resistance to carbonation than those made by blending slag and metakaolin. Even mixture MIII which showed the worst carbonation resistance of 3 mixtures, has similar performances to those reported by Bernal et al. [20].

Taken into consideration that carbonation depths for OPC concrete can reach 2-10 mm in only one year of natural carbonation [21], and one week of accelerated carbonation is equivalent to approximately 1-1.5 year of natural carbonation [22], we can confirm that mixture MI is better than OPC if we would like to design a carbonation-resistant composite.

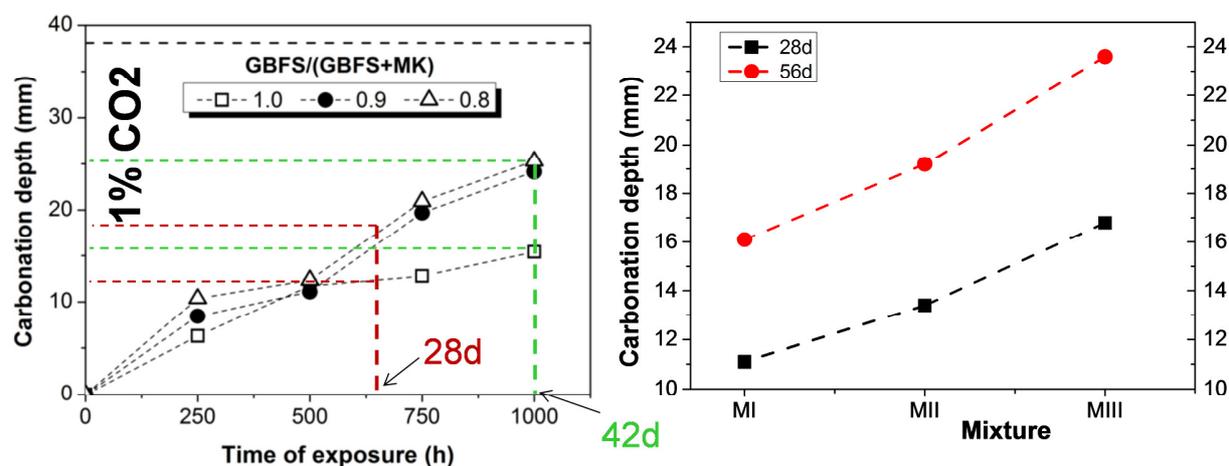


Figure 9. Time-exposure dependence of carbonation depth in (slag+metakaolin) mixtures (left) and SQAPE mixtures (right)

The chloride resistance results of the investigated concretes showed high chloride resistance (MI and MII having the lowest migration coefficients of $1.97 \times 10^{-12} \text{ m}^2/\text{s}$ and $3.13 \times 10^{-12} \text{ m}^2/\text{s}$, respectively). However, concrete MIII has a relatively high chloride penetration depth with the highest migration coefficient ($18.95 \times 10^{-12} \text{ m}^2/\text{s}$). In comparison with other concretes (see Figure 10), penetration depth values are in the same order of other alkali-activated concretes derived from slag and fly ash (FA); but all 3 concretes tested in this study exhibit better performances than OPC concrete. The lowest penetration depth corresponds to concrete MI indicating that this mixture has a low permeability to chloride migration which was confirmed by a lower migration coefficients. It is important to mention the good chloride resistance of concrete MII with a penetration depth 4 times lower than that reported in OPC concrete [23]. Even concrete MIII, which showed the worst chloride resistance of the 3 mixtures, has similar or better performances against chloride attack when compared to OPC based concrete. (Figure 10).

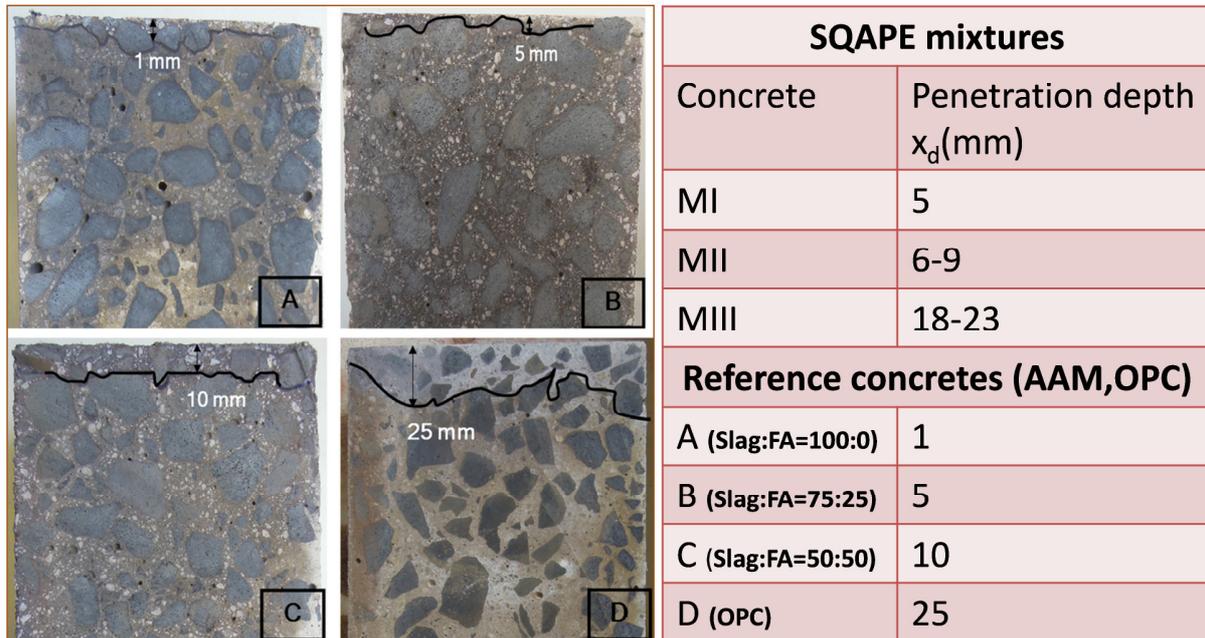


Figure 10. Chloride penetration depth values of investigated concretes in comparison with other concretes (A, B, C and D) taken from literature [23]

V. Concluding remarks & recommendations

A summary of all results deduced from the durability tests carried out on 3 different concrete mixtures is given in Table 3.

Table 3. Summary of durability tests on SQAPE mixtures including all measured parameters

Mixture	Measured Parameters						
	Mass change (kgm ⁻³)	Strength reduction (%)	Penetration depth (mm)	Diffusion coeff. (10 ⁻¹² m ² /s)	Carbonation depth (mm)		
					28d	56d	90d
MI	75-85	-10.84	3-5	1.97	11.1	16.1	19.3
MII	80-90	-9.40	6-9	3.13	13.4	19.2	22.3
MIII	110-120	-17.44	18-23	18.95	16.8	23.6	28.6
Test	Sulfate		Chloride		Carbonation		

From this study, the following conclusions can be drawn:

- The best durability performances have been found for concrete MI (high GGBS, low PCFA) which has demonstrated a higher resistance in all aggressive media used in this study.
- Concrete MII (moderate GGBS, moderate PCFA), showed also acceptable performance with a good resistance to carbonation and chloride ingress when compared with other concretes.
- In comparison with MI or MII, poor performances have been found for concrete MIII (low GGBS, high PCFA). The durability of this concrete could be enhanced by the tuning of some parameters such as the w/b ratio, the air content, the binder content, the activator, curing conditions, mix design, use of additives, etc. It should be kept in mind that the use of additives could positively or negatively affect the durability. On the other hand, it is well known that the use of some admixtures such as superplasticizers, retarders could improve the concrete performance. However, these chemical substances are usually combined with water and their stability and efficiency in the presence of strong alkaline solutions (such as waterglass) remain unclear and need to be investigated. Against some draw-backs of additives, the formulation of concrete with a low water content, a high strength and a low permeability is the key for the desired durability.

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