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Factors controlling carbonation resistance of alkali-activated materials

Susan A. Bernal, Xinyuan Ke, Maria Criado, Shishir Mundra, John L. Provis

Synopsis: Although notable advances have been made in recent years in elucidating the relationship between the nature of the precursor and the activation conditions used for production of alkali-activated materials (AAMs), it remains largely unknown whether these materials can withstand various environmental threats during their service life. The interaction between a cementitious material and the CO₂ present in the air is referred to as carbonation, and while this is largely a well understood phenomenon for portland cement systems, its long-term effects in AAMs are unknown. This is a consequence of the large number of variables controlling microstructural development and therefore macro-scale properties of AAMs, and the lack of standardized methodologies for testing their carbonation resistance. This study reports an overview of recently identified factors inducing microstructural changes in AAMs upon exposure to CO₂, and the influence of carbonation on the corrosion resistance of some steel reinforced AAMs.

Keywords: Durability, Alkali-activated Materials, Carbonation, Corrosion

INTRODUCTION

Alkali-activated materials (AAMs) are produced via a chemical reaction of a powdered aluminosilicate source and an alkaline solution, and have been the object of study over the past century as technically sound and sustainable potential substitutes for portland cement in production of concretes [1]. Particular attention has been paid in recent years to the identification of raw materials derived from industrial, agricultural and urban processes, to be used as precursors for the production of AAMs. This has been investigated with the goal of utilizing alkali-activation technology as a means for the safe and sustainable valorization of wastes and by-products, through production of valuable products [2].

Although significant advances have been made in elucidating the links between the chemistry of AAMs, particularly those based on blast furnace slag, and their microstructural and physico-mechanical properties [3, 4], it remains to some extent unknown if these materials can withstand different in-service environments. This is largely due to the limited track record of large scale structural applications of AAMs; the incomplete understanding of the chemical and/or physical interactions between AAMs and different chemical threats, such as CO₂, sulfates and chlorides; and the lack of standardized methods for evaluating potential performance of these materials [5]. However, it is worth noting that the analysis of existing structures based on these materials, particularly in Europe, demonstrates that they are structurally sound after more than 40 years of service [6, 7].

The chemical reaction between the hydrated products formed in a cement and the CO₂ present in the atmosphere is referred to as carbonation, and leads to decalcification of the strength-giving phases through formation of carbonate-rich products [8-10]. In some cementitious systems this type of chemical attack can cause severe matrix degradation, and it is also one of the main processes modifying the alkalinity within cement-based products. This can lead to the destabilization of the protective oxide layer forming on the steel reinforcement of reinforced structural concrete, making them susceptible to undergo corrosion. Hence, carbonation of AAMs is considered one of the main durability degradation phenomena that needs to be well understood to generate the required confidence for the utilization of AAMs in infrastructure applications.

How carbonation occurs in AAMs differs from the existing understanding of portland-cement based systems, due to the chemical and microstructural differences between these types of cements. For instance, portlandite (Ca(OH)₂) is not commonly identified as a reaction product in AAMs, and their pore solution is much more alkaline than that of portland cement [11]. In the early studies of carbonation of AAMs, there was a general consensus that these materials appear less resistant than portland cements [12-14]. However, all these studies were carried out under various accelerated carbonation environments (e.g. CO₂ concentrations between 3 to 100%; saturated or dried specimens), evaluating AAMs produced with different precursors and activation conditions. This meant that it was challenging to draw any general trends to identify of the mechanism leading to carbonation of AAMs. More recently, a different approach to assessing this phenomenon has been adopted, so that the factors controlling carbonation of AAMs have been elucidated, and therefore strategies can be identified to enhance their performance.

Bernal et al. [15, 16] proposed that accelerated carbonation AAMs is both a diffusion and chemically controlled mechanism, and its extent is governed by the mix design parameters promoting specific microstructural features, including pore network and permeability. Also, the environmental conditions adopted to induce carbonation (e.g. CO₂ concentration, temperature and relative humidity) strongly influence the mechanism and extent of degradation of AAMs [17-19]. This is a consequence of the changes of the pore solution carbonation, modifying the type and amount of carbonation products forming. This explains why divergences are identified between results obtained in specimens carbonated under natural in-service conditions, or under accelerated conditions in simulated laboratory environments [18].

EFFECT OF AAM MIX DESIGN

The type of precursor used in producing AAMs is the main factor controlling phase assemblage and pore structure, and consequently resistance to chemical threats. The most widely used precursors are granulated blast furnace slag (GBFS) from the iron making industry, coal combustion fly ashes (FA), and calcined clays such as metakaolin (MK) [20]. Different contents of Al₂O₃ and SiO₂ can be present in these precursors, but their main difference lies in their CaO content, which modifies the chemistry of AAMs, so that completely different microstructures are favored depending on the content of CaO present, and its solubility under the activation conditions adopted.

In alkali-activated GBFS or high-calcium FA, aluminum substituted calcium silicate hydrate (C-A-S-H) type products are typically identified as the main reaction products [21], while in low calcium systems the formation of an alkali aluminosilicate hydrate (N,K-A-S-H) type gel prevails [22]. For high-Ca AAMs, carbonation mainly leads to decalcification of the C-A-S-H type phases through formation of different calcium carbonate polymorphs and a highly crosslinked aluminosilicate type gel [13]; while in low-Ca AAMs notable differences in their microstructure are not detected (Fig. 1) [16], formation of sodium-rich carbonates has been reported [16, 23].

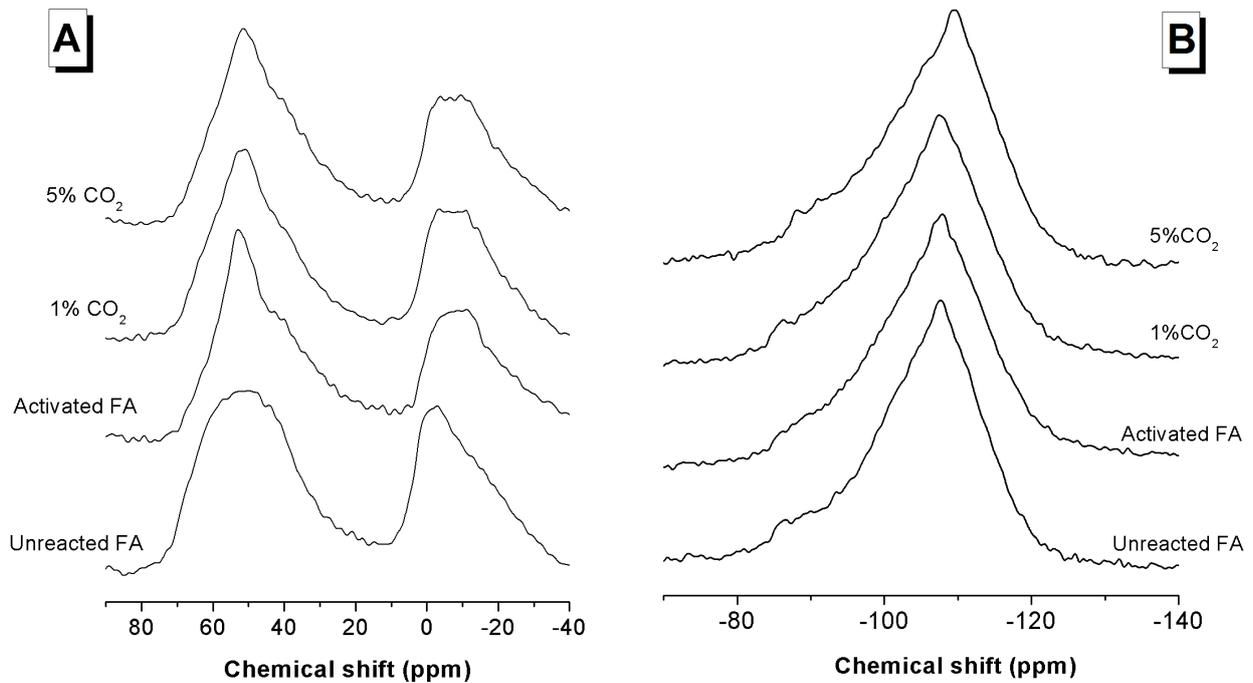


Fig. 1 - Solid state (A) ^{27}Al MAS and (B) ^{29}Si MAS NMR spectra of alkali-activated FA cements exposed to different CO_2 concentrations. Adapted from [16]

The formation of sodium carbonates in carbonated AAMs indicates that their pore solution plays a key role in how these materials react in the presence of CO_2 . It has been identified that depending on the environmental conditions used to induce accelerated carbonation of AAMs [19] or carbonation of their simulated pore solutions [24], formation of sodium carbonate products with different compositions and densities (e.g. natron, nahcolite or trona) is favoured. This has a negative impact on the potential pore-blocking ability through deposition of carbonation products in open pores, and therefore the pore solution carbonation alone is less likely to hinder CO_2 diffusivity within these specimens when more dense carbonate products are formed, which is particularly the case when carbonation is induced under accelerated conditions with CO_2 concentrations higher than 1% CO_2 .

In the case of GBFS-based AAMs, it has been identified that the content of MgO within the slag also influences its carbonation resistance [25]. Using high MgO -containing slags (> 5 wt. % MgO) favors the formation of layered double hydroxides with a hydrotalcite type structure, with the ability to chemically bind CO_2 under highly alkaline conditions [26, 27]. In Fig. 2A it can be seen that using a slag with high MgO gave a reduced compressive strength and higher permeable voids content in mortars, compared to that of a low- MgO content slag, when activated under similar activation conditions. However, the carbonation depths of the activated high- MgO content slag mortars were almost half of those of low- MgO content slag despite the higher permeable voids content of the high- MgO slag based mortars. These results highlight the notable effect of the formation of hydrotalcite in increasing the carbonation resistance of AAMs.

Consistently, in GBFS-activated systems, accelerated carbonation induced densification of NaOH -activated GBFS mortars compared to specimens activated with a sodium silicate solution, although NaOH -activated GBFS binders are typically more porous than those produced with sodium silicate. Puertas et al. [13] attributed this behavior to the precipitation of a larger amount of carbonation products when using NaOH , refining the pore structure of the specimens. However, as each activator will promote the formation of a different phase assemblage, further investigation is required to elucidate the chemical factors that might be leading to the differences in carbonation rates, as it is unlikely that the differences are solely attributable to changes in diffusivity of CO_2 in these materials.

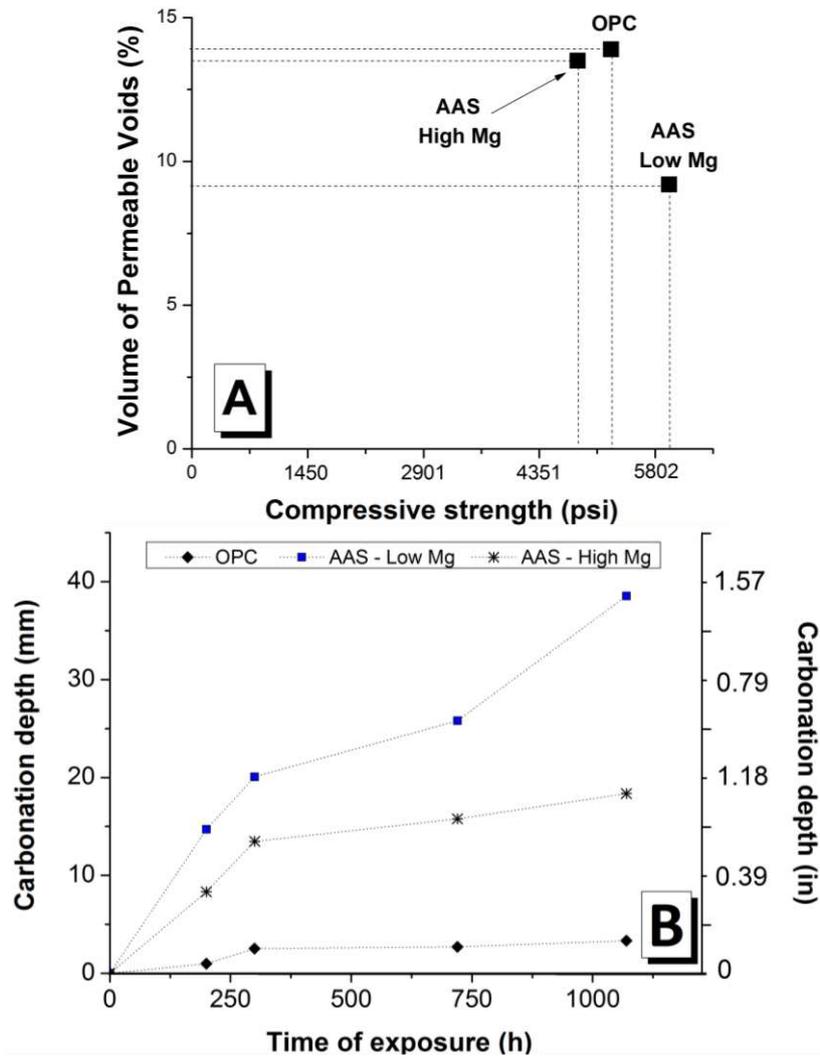


Fig. 2 - (A) 28-day compressive strength (1000 psi = 6.895 MPa) and volume of permeable voids (ASTM C642), and (B) accelerated carbonation (1% CO₂, 65% RH, 25°C (77°F)) depths of silicate-activated GBFS mortars, compared to a CEM I Portland cement mortar. All the specimens were formulated with a similar water/binder ratio (0.40)

The partial replacement of GBFS by MK in AAMs also has a notable influence on their resistance to carbonation. The chemical reaction between MK and the alkalis present in the pore solution of these binary systems modifies the mechanism of carbonation of AAMs, so that formation of sodium carbonates is not identified with MK additions of at least 20 wt.% of the total binder [28]. This means that any CO₂ diffusing into the material will react with the hydrated binding phases in these binders rather than the pore solution, which induces notable microporosity (Fig. 3) [28].

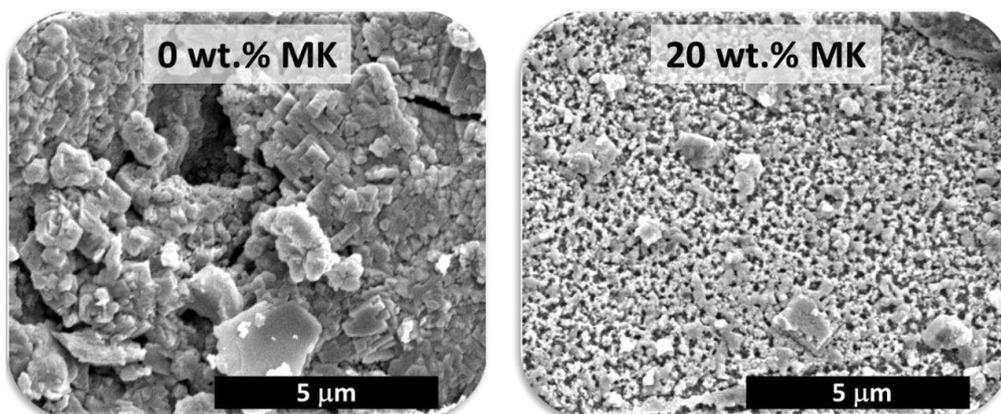


Fig. 3 - Secondary electron micrographs of fractured alkali-activated slag/metakaolin binders after 540 h of exposure to 1% CO₂, 65% RH and 25°C (77°F). Adapted from [28]

Carbonated mortar samples with 20 wt.% MK showed negligible changes in compressive strength, although reductions in porosity were observed [29]. Conversely, when carbonating concrete produced with similar mix designs to that of the carbonated mortars assessed in [29], exposure to high CO₂ concentrations led to a reduction in compressive strength, and an increased porosity [30]. Considering that the main difference between mortars and concretes based on a similar cementitious matrix is their permeability, which is influenced by the presence of the coarse aggregate, it can be inferred that in this particular case the CO₂ diffusivity into the material is the leading factor controlling the rate of carbonation and consequently the extent of microstructural changes induced.

In the case of carbonated alkali-activated FA cements, sodium carbonates are the predominant carbonation products [16, 23], most likely derived from the carbonation of the pore solution, although alkalis could also potentially be removed from the N-A-S-H type gel upon CO₂ exposure. When using a FA with low content of CaO (5 wt.%), formation of calcium carbonates is identified [31]. The addition of CaO to these binders, for instance by adding GBFS, has a notable impact in how carbonation of these materials proceeds, as high fractions of GBFS favor formation of C-(N)-A-S-H type phases, with varying incorporation of Ca [32]. These phases will carbonate similarly to the process identified in cements produced solely with activated GBFS. Consequently, the main carbonation products identified in carbonated FA/GBFS activated blends are the apparently unaltered N-A-S-H type product resulted from the activation of FA, the Si-rich aluminosilicate type phase derived from the carbonation of the C-(N)-A-S-H, along with alkali carbonate precipitates [16].

Pasupathy et al. [33] evaluated naturally carbonated alkali-activated FA/GBFS concretes, and identified calcium carbonates in the colorless regions of phenolphthalein sprayed samples. Differences in mix design (e.g. FA/GBFS ratio and type of activator used) of the aged concretes had a notable impact on their permeability, and consequently on their carbonation rates, consistent with a higher diffusivity of CO₂ in the most porous materials. Bijen and Waltje [34] also identified an acceleration in carbonation of NaOH-activated FA/GBFS binders due to intensive microcracking of their specimens during drying prior to CO₂ exposure, compared with portland cement samples. Consistent with the observations made when adding MK to GBFS-based AAMs, the diffusivity of CO₂ strongly influences the resistance to carbonation of these materials, independent of the chemistry of the cementitious matrix.

IMPACT OF CARBONATION OF AAMs IN CORROSION OF STEEL REINFORCEMENT

Undamaged concrete provides the pH conditions necessary for the passivation of mild steel reinforcement, which protects them against the effects of aggressive species such as chlorides. In portland cement-based concretes the pH is mainly controlled by the hydration product portlandite (Ca(OH)₂); however, in the case of AAMs this phase is not typically identified, and therefore, the pH of the system is controlled solely by the pore solution.

As carbonation of AAMs will induce variations in the pore solution pH, the effect of carbonation on the long-term performance of these materials is of great concern. If the pore solution pH of carbonated AAMs falls below the highly alkaline range in which the oxide passivation layer on the steel is stable, this will lead to formation of anodic and cathodic regions in the steel rebar, promoting localized corrosion. Bernal et al. [19] demonstrated the significant differences in the reaction products forming during natural and accelerated testing of AAMs, particularly related to the carbonate/bicarbonate equilibria and in the nature of the carbonation products. Bicarbonate forms at high CO₂ partial pressures, typical of accelerated carbonation conditions ([CO₂] > 1%), promoting a greater decrease in pH upon carbonation that would be identified in natural CO₂ environments, which means that the carbonated pore solution environment in an accelerated test becomes far more damaging to embedded steel reinforcement than is the case in real service conditions. Thermodynamic simulations of the carbonate/bicarbonate equilibria showed that natural carbonation is unlikely to reduce the pore solution pH below 10, even when considering alkali leaching effects, and therefore the likelihood of initiation of a corrosion process due to natural carbonation is considered on this basis to be small.

The observations derived from thermodynamic simulations [19] are in good agreement with recently reported experimental data by Pouhet et al. [24] evaluating the pH evolution of simulated pore solution of AAMs exposed to atmospheric CO₂ concentrations for 1 year, where the pH values of the pore solution was higher than the steel depassivation limit of 9. Similar observations have also been identified in 8-year old naturally carbonated activated FA/GBFS concretes, where in the carbonated region the pH did not drop below 10 (Fig. 4) [33], independent of the concrete mix design. These results demonstrate that, considering the chemistry of carbonated pore solutions in AAMs, it is unlikely that corrosion initiation, will take place in these materials as a direct consequence of carbonation of the matrix.

The corrosion potential of steel reinforcement embedded in AAMs remains largely unknown at present, and electrochemical studies in carbonated AAMs concretes, in the absence of chlorides, are limited [31, 35]. Long-term assessment (200 days) of naturally carbonation of reinforced low-Ca FA alkali-activated concretes showed that the system has the ability to passivate the steel reinforcement although carbonation is occurring, despite the observation of reductions

in the corrosion potential at extended exposure times (>150 days), remaining within the range of minimal corrosion risk. Similar results were observed in high-Ca FA alkali-activated concretes, however, the corrosion potentials identified for these specimens were within the range of high corrosion risk [31]. This demonstrates how small variations in mix design of AAMs, in this case the composition of the precursor used, leads to notable changes in the electrochemical determination of durability performance of these materials.

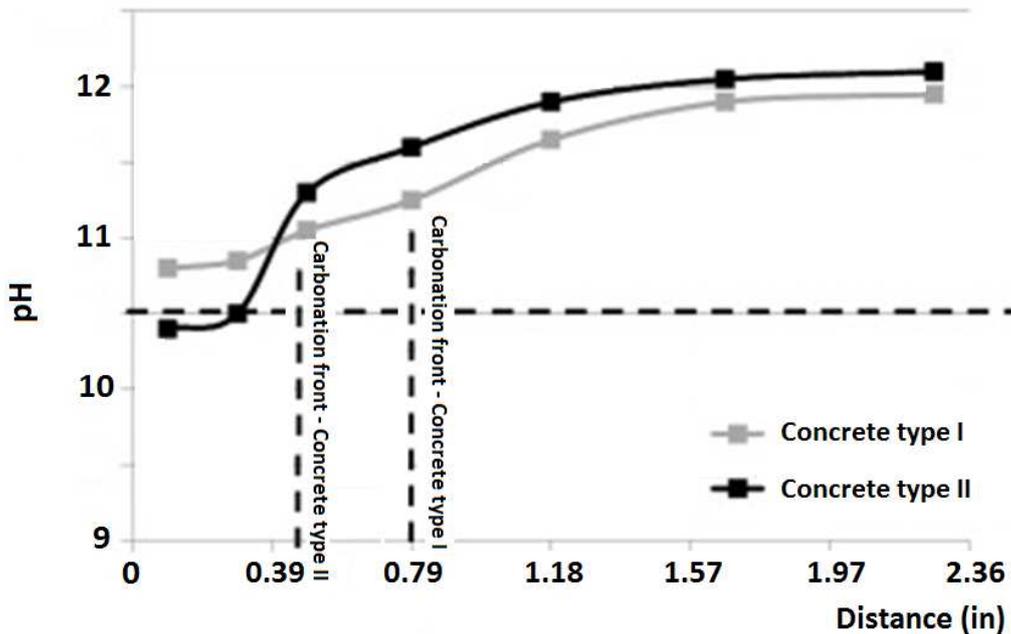


Fig. 4 - pH as a function of the distance from the surface in naturally carbonated 8 year old alkali-activated FA/GBFS concretes. Adapted from [33]

Most electrochemical studies have been focused on evaluating corrosion potential of steel reinforcement embedded in AAMs (mainly focused on alkali-activated FA mortars) exposed to chlorides rich environments. Although valuable information about the potential corrosion of steel reinforcement under highly alkaline conditions in the presence of chlorides has been obtained, it is important to recognise the limitations of those results in describing the mechanism leading to corrosion of all AAMs. This is a consequence of the complex interactions of all the different species present in the pore solution of AAMs when using different precursors and activation conditions for its production, and the significant changes taking place in pore solution chemistry as these materials age.

Hence, there is an imminent need to carry out corrosion studies in naturally carbonated AAMs to truly elucidate the passivation/depassivation taking place in carbonated AAMs during service life, and its effects on the stability of the steel reinforcement passivation layer, particularly in the absence of chlorides, as this threat is not always present in the intended infrastructure applications of AAMs.

GENERAL REMARKS

It cannot be generalized that AAMs are less resistant to carbonation than portland cements, as the accelerated carbonation rates identified in specimens tested under controlled laboratory conditions vary significantly when either the precursor or activator are modified, and direct correlations between these results and those observed in naturally carbonated concrete specimens have not been identified. Hence, the accelerated carbonation testing conditions adopted to date for assessing performance of AAMs do not replicate well with what is likely to occur during an extended period of time in service.

For a given AAM formulation the carbonation results identified in mortars are inconsistent with what is obtained in concrete specimens, mainly due to the permeability differences between samples. This is particularly critical for selecting the type of specimens to test, in aiming to collect meaningful and representative results about carbonation resistance of these materials. This requires particular attention as most studies of carbonation of AAMs have been, and currently are, carried out in mortar specimens.

The nature and amount of carbonation products derived from the interaction of the pore solution of AAMs and CO_2 are highly susceptible to the testing conditions (e.g. CO_2 concentration and temperature). Thermodynamic modeling, as well as measurements in simulated pore solutions and aged specimens, demonstrate that natural carbonation of AAMs will not

lead to reductions in the pH below 9.6, and therefore corrosion of steel reinforcement is unlikely to take place in these materials due solely to carbonation.

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