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## Review Article

# Review of Carbonation Resistance in Hydrated Cement Based Materials

Joseph Mwiti Marangu <sup>1</sup>, Joseph Karanja Thiong'o,<sup>1</sup> and Jackson Muthengia Wachira <sup>2</sup>

<sup>1</sup>Department of Chemistry, Kenyatta University, Nairobi, Kenya

<sup>2</sup>Department of Physical Sciences, University of Embu, Embu, Kenya

Correspondence should be addressed to Joseph Mwiti Marangu; [jmarangu2011@gmail.com](mailto:jmarangu2011@gmail.com)

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Blended cements are preferred to Ordinary Portland Cement (OPC) in construction industry due to costs and technological and environmental benefits associated with them. Prevalence of significant quantities of carbon dioxide (CO<sub>2</sub>) in the atmosphere due to increased industrial emission is deleterious to hydrated cement materials due to carbonation. Recent research has shown that blended cements are more susceptible to degradation due to carbonation than OPC. The ingress of CO<sub>2</sub> within the porous mortar matrix is a diffusion controlled process. Subsequent chemical reaction between CO<sub>2</sub> and cement hydration products (mostly calcium hydroxide [CH] and calcium silicate hydrate [CSH]) results in degradation of cement based materials. CH offers the buffering capacity against carbonation in hydrated cements. Partial substitution of OPC with pozzolanic materials however decreases the amount of CH in hydrated blended cements. Therefore, low amounts of CH in hydrated blended cements make them more susceptible to degradation as a result of carbonation compared to OPC. The magnitude of carbonation affects the service life of cement based structures significantly. It is therefore apparent that sufficient attention is given to carbonation process in order to ensure resilient cementitious structures. In this paper, an indepth review of the recent advances on carbonation process, factors affecting carbonation resistance, and the effects of carbonation on hardened cement materials have been discussed. In conclusion, carbonation process is influenced by internal and external factors, and it has also been found to have both beneficial and deleterious effects on hardened cement matrix.

## 1. Introduction

Cement is the main building binder used in housing and general construction throughout the world [1]. Ordinary Portland Cement (OPC) is the most common type of cement used in construction [1, 2]. However, OPC production process involves significant environmental damage with respect to carbon dioxide (CO<sub>2</sub>) production and raw material requisition [3]. Cement manufacturing releases CO<sub>2</sub> in the atmosphere when calcium carbonate (CaCO<sub>3</sub>) is heated [4]. The gas is also emitted when petroleum based fuel is burnt in the kiln during clinkerisation process [4]. The cement industry is the second largest CO<sub>2</sub> emitting industry behind power generation[5]. CO<sub>2</sub> is considered as the main greenhouse gas causing global warming and climate change in the world. There is an increasing demand for eco- friendly cement in the world in order to meet the sustainable development goals.

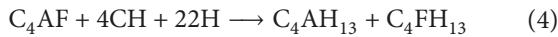
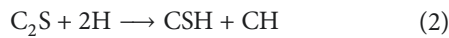
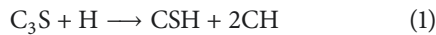
The increased emission of CO<sub>2</sub> subsequently increases the atmospheric CO<sub>2</sub> reaction with cement based materials. Ingress of CO<sub>2</sub> in hydrated cement materials is mainly a diffusion controlled process [5]. Increased atmospheric CO<sub>2</sub> results in serious durability concerns for field-exposed reinforced concretes due to carbonation. The effects carbonation-induced corrosion accounts for 78% of reduction in service life of cement based materials and increased maintenance related costs[5].

The durability of reinforced cement based structures is a global pervasive problem[6–9]. Poor understanding of deterioration processes and inadequate ingress resistance criteria for aggressive substances such as CO<sub>2</sub>, chlorides and sulphates, among others on site concrete changes in cement properties and construction practices are some of the reasons that account for the deterioration of cement based structures[6, 10–17]. As a result, many cement based

structures deteriorate prematurely resulting in short service life [17–19]. Repair and maintenance costs are greatly increased in the attempts to mitigate the adverse effects of aggressive substances [6, 18–23]. In this paper, an indepth review on current advances on carbonation process, factors affecting carbonation, and effects of carbonation on hardened cement materials have been discussed in detail.

## 2. Hydration of Cement

The main clinker phases present in cements are tricalcium silicate ( $C_3S$ ), dicalcium silicate ( $C_2S$ ), tricalcium aluminate ( $C_3A$ ), and tetra calcium aluminoferrate ( $C_4AF$ ). The hydration of these phases in OPC is given in [24]



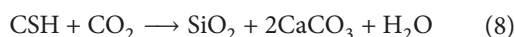
Hydration of  $C_3S$  and  $C_2S$  results in the formation of CH and CSH. CSH is a cementitious material that imparts strength to cement based materials [24]. The hydration products of  $C_3A$  and  $C_4AF$  are not of significance to the strength of hardened cement [24]. At the early ages of mortar/concrete curing, CH is useful since it provides the necessary alkalinity to the hardened cement [24]. In blended cements, CH produced during the hydration of OPC reacts with active silica and alumina in presence of water to form more CSH and calcium aluminate hydrate (CAH) during the pozzolanic reaction as shown in [24]



The consumption of CH during the pozzolanic reaction in blended cements results in lower amount of CH and increased CSH [25–29]. The CH and CSH are the most susceptible phases to carbonation [30]. The amount CH and severity of  $CO_2$  attack on CSH has been found to greatly affect the rate of carbonation process [30].

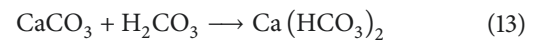
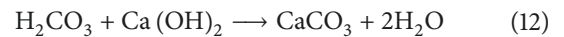
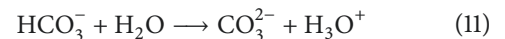
## 3. Carbonation Process

Carbonation refers to the reaction of hydration products dissolved in pore water (mostly CH and CSH) with  $CO_2$  in the atmosphere [31]. In hydrated cement based materials, carbonation occurs when they are exposed to atmospheric  $CO_2$  or dissolved  $CO_2$  in water in form of carbonic acid [32]. The CH and CSH are the major phases attacked by  $CO_2$  during carbonation as shown in (7) and (8), respectively [33];



Equation (7) represents moderate carbonation occurring on the surface layer of cement based materials leading

to the formation of  $CaCO_3$  [33]. The  $CaCO_3$  formed is deposited on the pore network in the hydrated cement matrix resulting in pore refinement of the carbonated layer. This is beneficial since pore refinement subsequently results in increased strength, reduced porosity and improved durability of hydrated cement [34]. Conversely, according to (8), excess ingress of  $CO_2$  attacks CSH resulting in its disintegration and subsequent loss of strength in cement based structures [35]. Additionally, during carbonation,  $OH^-$  present in CH is consumed resulting in the decrease of pore solution pH [36]. In case of reinforced concrete, reduction in the pH value leads to the destruction of protective passive surface layer around steel reinforcement resulting in rebar corrosion [36]. In areas with high concentration of dissolved  $CO_2$  for example in estuaries, the effects of carbonation are more deleterious since carbonic water is more acidic in seawater than fresh water [37]. This can be worsened by the decaying organic matter releasing  $CO_2$ . The carbonation related chemical reactions that result in the degradation of cement based materials due to dissolved  $CO_2$  are presented in [13, 38]



Equation (9) represents the formation of carbonic acid ( $H_2CO_3$ ) from dissolved  $CO_2$  present either in decaying organic matter or polluted water bodies [13, 38]. Based on (10) and (11),  $H_3O^+$  produced results in significant reduction in the pore solution pH in hydrated cement matrix [13]. Reduction in the alkalinity of the pore solution is deleterious since it results in carbonation-induced corrosion of rebars through dissolution of its passive surface layer [39]. In addition, (13) shows that significant penetration of  $CO_2$  results in the conversion of insoluble  $CaCO_3$  to soluble  $Ca(HCO_3)_2$  which is easily leached out thus decreasing the porosity of hydrated cement [13].

In conclusion the mechanism of carbonation in hydrated cements has been summarized as [17]

Step I: Diffusion of  $CO_2(g)$  in the gaseous layer surrounding the solid

Step II: Diffusion of  $CO_2(g)$  through the solid

Step III: Solvation of  $CO_2(g)$  to  $CO_2(aq)$  in the pore water

Step IV: Hydration of  $CO_2(aq)$  to  $H_2CO_3(aq)$

Step V: Ionization of  $H_2CO_3$  to  $H^+$ ,  $HCO_3^-$  and  $CO_3^{2-}$

Step VI: Dissolution of calcium containing phases to  $Ca^{2+}$

Step VII: Nucleation of  $CaCO_3(s)$

Step VIII: Precipitation of solid phases.

#### 4. Factors Affecting Carbonation Resistance

**4.1. Relative Humidity.** The extent of carbonation largely depends on the existing Relative Humidity (RH) upon which the hydrated cement exists [40]. RH determines the long term availability of moisture/water in the hardened cement. Water influences the reactivity and diffusion of carbon dioxide in the pore network of hydrated cement. According to [17], water is essential for the dissolution of  $\text{Ca}^{2+}$  from calcium containing phases such as (CH), hydration of  $\text{CO}_2$  to form  $\text{H}_2\text{CO}_3$  and besides solvation of gaseous  $\text{CO}_2$  to form aqueous  $\text{CO}_2$ . In presence of insufficient water,  $\text{CO}_2$  and CH do not ionised fully. Conversely, in presence of sufficient water, carbonation process takes place leading to the formation of pore filling solid  $\text{CaCO}_3$  in the hydrated cement pore network as the diffusivity of  $\text{CO}_2$  is lower in water than in air.

RH determines the quantity of atmospheric water upon which cement based material is exposed during curing [31, 36, 40–42]. The existing water in the hydrated cementitious system especially during curing process primarily depends on the pore network as well as external RH. Moisture exchanges usually occur at any given RH between the pores in hydrated cement based materials and the atmosphere until an equilibrium is established [43]. The pore sizes in hydrated cement that remains saturated can be related to the RH by the Kelvin equation given in [44]

$$r = \frac{-2\gamma M \cos \theta}{\rho_w RT \ln H} \quad (14)$$

where  $r$  is the radius of the pores (m),  $\gamma$  is the surface tension of water ( $0.076 \text{ N m}^{-1}$ ),  $M$  is the molar mass of water ( $0.018 \text{ kg mol}^{-1}$ ),  $\theta$  is the contact angle,  $\rho_w$  is the density of water ( $1000 \text{ kg m}^{-3}$ ), and  $H$  is the external RH.

Assuming that the contact angle between water and hardened cement is  $0^\circ$  and (14) can be reduced to the form given in [44]

$$r = \frac{10^{-9}}{\ln H} \quad (15)$$

According to [44, 45] (15) is strictly applicable to capillaries of uniform dimension solely to provide qualitative approximate values due to the rather complex pore geometries in cementitious systems. Based on (15), when the external RH is zero, the water content in the hydrated cement material is also zero and therefore carbonation reactions do not take place [46, 47]. On the other hand, assuming that the RH is 100%, the cementitious system is therefore fully saturated, since all pores are filled with water which inhibits the diffusion pathway of  $\text{CO}_2$  [20]. It is apparent that the RH has a considerable influence on the magnitude of carbonation [44].

Several researchers have conducted experimental work to determine the optimum RH for carbonation process [20, 40–42, 48]. In most of the cementitious materials, carbonation is more rapid at an external relative humidity of 50–60% [10, 18, 49]. At 50–60 % RH, the equilibrium water content in the hydrated cementitious materials tends to reach optimum for the carbonation reaction. Generally, the ideal RH for optimum carbonation is at 50 % (range 40–70 %). This is

because when the RH is less than 40 %, then  $\text{CO}_2$  cannot dissolve. Conversely, when the RH is greater than 70%, the diffusion of  $\text{CO}_2$  is inhibited by the water that has filled the pores and hence  $\text{CO}_2$  cannot ingress in the cement based structure [31, 50].

**4.2. Concentration of Carbon Dioxide ( $\text{CO}_2$ ).** The rate of carbonation in the hydrated cementitious materials depends on the concentration of environmental  $\text{CO}_2$  [51]. Generally, a high concentration of  $\text{CO}_2$  increases the diffusion rate of  $\text{CO}_2$  into the cementitious matrix and hence increases the carbonation rate [52]. However, the use of a high concentration of  $\text{CO}_2$  also increases the water content in the pores because of the high rate of reaction and evolution of water within the specimen [50]. Therefore, if thin specimens are considered at the optimum RH, carbonation in low concentrations of  $\text{CO}_2$  proceeds at a faster rate than in high concentrations [30]. When there are minimal increases atmospheric  $\text{CO}_2$  concentrations, the rates of carbonation reactions are increased and this has great positive influence on strength development of hydrated cementitious materials.

**4.3. Type of Cement.** Blended cements have higher carbonation rate than OPC [52]. In blended cements, pozzolanic reactions also consume CH [53–55]. As a result, less CH is available and the rate carbonation is higher than in OPC which contains higher amount of CH [30]. Moreover, the lower CH availability results in more CSH carbonation. In contrast with CH carbonation, CSH carbonation does not densify the pore structure [52].

**4.4. Porosity.** Highly porous cement materials allow greater penetration of  $\text{CO}_2$  in the bulk of hydrated cementitious materials. Porosity in combination with factors such RH has a significant impact on  $\text{CO}_2$  diffusion, i.e., the pore sizes. At a specific RH, the finer the pore size is, the easier the capillary condensation occurs according to the Kelvin equation. For instance, when mortars are exposed to a 57% RH environment, a calculated Kelvin radius would be 4 nm. When the cement matrix has a larger fraction of pores below this pore size,  $\text{CO}_2$  diffusion will be very slow since pores with sizes below 4 nm will be filled by condensed water.

Reference [11] studied the effect of crack opening on carbon dioxide penetration in cracked mortar samples. The authors observed that the presence of cracks in hydrated cement matrix promote the ingress of  $\text{CO}_2$  due to increased porosity.

**4.5. Concentration of  $\text{Ca}(\text{OH})_2$ .** The amount of calcium hydroxide present in cementitious materials determines the durability of cement based structures against carbonation since it maintains a high pH of 12.6 [38]. It is accepted that the calcium hydroxide content has a significant impact on the carbonation rate, since higher total calcium hydroxide has higher  $\text{CO}_2$  binding capacity [56].

## 5. Effects of Carbonation on Hydrated Cement

Apart from the effect of carbon dioxide on  $\text{Ca}(\text{OH})_2$ , CSH, and other  $\text{CO}_2$  reactive compounds, carbonation has been noted to influence other material properties and effects. Some of the major effects are introduced in this section.

**5.1. Compressive Strength.** Higher compressive strengths are obtained in mortars cured at 90 percent than their corresponding mortars cured at 60 percent relative humidity [57]. This can be attributed to the different curing humidity conditions that the mortars were subjected to. Low humidity (60 percent) allows more ingress of  $\text{CO}_2$  than mortars cured at high humidity (90 percent) [12]. CH, is one of the products formed during hydration of cement that readily reacts with  $\text{CO}_2$  resulting in precipitation of sparingly soluble calcium carbonate ( $\text{CaCO}_3$  as shown in (6) and (8)[13, 58–60].

Moderate carbonation process occurs on the surface layer of cement based materials leading to the formation of  $\text{CaCO}_3$ . The  $\text{CaCO}_3$  formed is deposited on the pore network in the hydrated cement matrix resulting in pore refinement of the carbonated layer [6, 21, 22, 61, 62]. This is beneficial since pore refinement subsequently results in increased strength, reduced porosity and improved durability of hydrated cement. This explains the increase in compressive strength of mortars/concrete with continued curing when exposed to  $\text{CO}_2$  laden environment. Conversely, as shown in (8), continued ingress of  $\text{CO}_2$  attacks CSH resulting in its disintegration and subsequent loss of strength in cement based structures [6, 21, 22, 61, 62]. This enhanced at low RH. This explains why mortars cured at low RH approximately 60% exhibit lower compressive strength than those cured at 90 % RH.

**5.2.  $\text{p}^{\text{H}}$  of Pore Solution.** Carbonation process occurring in cementitious materials results in the change in the pH of pore water [63]. The pore water has a solvent action on cementitious materials. The dissolution of the  $\text{Ca}(\text{OH})_2$  and CSH buffer the pH of pore solution in equilibrium with the cementitious materials to values between 10.5 and 12.5 [64]. The consumption of  $\text{Ca}(\text{OH})_2$  during carbonation results in relatively lower concentration of  $\text{Ca}(\text{OH})_2$  in the pore water resulting in lower pH [64]. The ingress and reaction of  $\text{CO}_2$  with  $\text{Ca}^{2+}$  in the pore solution and subsequent dissolution of  $\text{Ca}(\text{OH})_2$  and CSH. The consumption of  $\text{Ca}(\text{OH})_2$  and CSH leads to a drop of pH values of pore solution[17]. A fully carbonated surface shows a pH of approximately 8. In reinforced concrete, decreased pH in pore water is deleterious since it promotes corrosion resulting in reduced service life of cement based structures [65].

**5.3. Porosity.** The precipitation of the calcium carbonate produced by carbonation can lead step by step to a clogging of the pores [35, 66]. A reduction of 5-12% of the open porosity of concrete degraded by carbonation [67]. Another study showed a decrease of up to 26% in the pore volume of a cement-solidified waste system [68].

## 6. Conclusion

In conclusion, carbonation process is influenced by internal and external factors, and it has also been found to have both beneficial and deleterious effects on hardened cement matrix. With increased  $\text{CO}_2$  emission globally, there is need to closely conduct carbonation tests on various cement based structures/materials. The emerging infrastructural development in estuaries calls for enhanced carbonation studies on cement based materials used.

## Conflicts of Interest

The authors declare no conflicts of interest.

## Acknowledgments

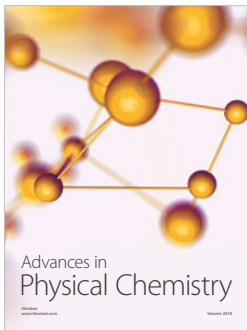
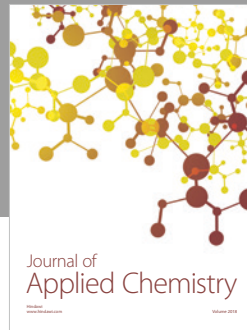
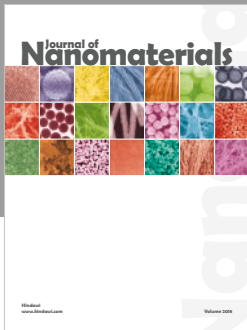
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