



Relation between carbonation resistance, mix design and exposure of mortar and concrete



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ABSTRACT

When cement with mineral additions is employed, the carbonation resistance of mortar and concrete may be decreased. In this study, mortars containing mineral additions are exposed both to accelerated carbonation (1% and 4% CO₂) and to natural carbonation. Additionally, concrete mixtures produced with different cements, water-to-cement ratios and paste volumes are exposed to natural carbonation. The comparison of the carbonation coefficients determined in the different exposure conditions indicates that mortar and concrete containing slag and microsilica underperform in the accelerated carbonation test compared to field conditions. The carbonation resistance in mortar and concrete is mainly governed by the CO₂ buffer capacity per volume of cement paste. It can be expressed by the ratio between water added during production and the amount of reactive CaO present in the binder (w/CaO_{reactive}) resulting in a novel parameter to assess carbonation resistance of mortar and concrete containing mineral additions.

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1. Introduction

The increasing use of mineral additions, e.g., ground granulated blast-furnace slag (GGBS), fly ash or limestone powder, to lessen the CO₂ emission of cementitious binders affects the durability of mortar and concrete. It can both improve and worsen their performance depending on the type of interaction [e.g. 1–5]]. In regard to carbonation resistance, a general decrease is observed when cement clinker is replaced by mineral additions [6–11]. An increased risk for rebar corrosion in reinforced concrete structures due to carbonation of the concrete cover may result.

Accelerated tests can be used to assess the carbonation resistance of mortar or concrete produced with cements containing mineral additions. However, a wide range of CO₂ concentrations, different relative humidity conditions and temperatures are used in such accelerated tests [8,10,12–14]. Therefore it is not clear, whether the results obtained with one specific accelerated test are transferable to another one and, even more important, to natural carbonation [15–20]. Another important point is the curing of the samples before they are exposed to carbonation. Carbonation resistance increases with increased time of curing [6,15,21,22], as longer curing allows a higher degree of hydration of the binder. However, the reactivity of cement clinker and mineral additions like GGBS or fly ash can be quite different, leading to

binder-specific effects of curing on carbonation. Additionally, it has to be taken into account that the effect of curing is dependent on the water-to-cement-ratio (w/c), with a stronger effect on mortar and concrete produced with high w/c [23,24]. Such effects may be the reason why no material parameter governing the carbonation resistance of systems with different mineral additions has been identified yet.

This study aims at empirically identifying a material parameter (or a number of parameters) that defines the carbonation resistance of mortar and concrete produced with cements containing mineral additions. To ensure optimal curing, all samples were moist cured for 28 days before starting exposure to CO₂. In a first series of experiments, mortars were produced. Their buffer capacity and diffusivity were systematically changed by addition of limestone powder, microsilica, portlandite and the use of GGBS cement. The carbonation resistance was determined under accelerated (1% and 4% CO₂) and natural conditions (sheltered and unsheltered). Additionally, compressive strength and oxygen diffusion were determined. Selected samples were further investigated with mercury intrusion porosimetry (MIP) and scanning electron microscopy (SEM), combined with energy dispersive X-ray spectroscopy (EDX). In a second series of experiments, concrete mixtures produced with different w/c, cement type and paste volume were exposed to natural carbonation for 2.5 years. Furthermore, compressive strength and oxygen diffusion were measured.

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2. Materials and methods

2.1. Materials

2.1.1. Mortars

The composition of the ordinary Portland cement (OPC/CEM I 52.5 R), the GGBS cement (CEM III/B 42.5, GGBS content of 66–80 mass-%) and the microsilica used for the mortars are given in Table 1. The limestone powder used contains 92 mass-% of calcite, 5.5 mass-% of dolomite and minor amounts of quartz and mica. A laboratory grade portlandite was used. The aggregate was standard quartz sand with a grain size of 0–2 mm.

The mix design of the mortars (Table 2) was systematically varied to study the effect of changing diffusivity and buffer capacity on the carbonation resistance. By increasing the w/c of the mixtures produced solely with OPC and by replacing cement with limestone powder, the diffusivity was increased and the buffer capacity decreased. Cement was replaced with microsilica to decrease both diffusivity and buffer capacity. The replacement of cement with portlandite was expected to slightly increase the buffer capacity and the diffusivity. The volume of paste was kept constant for all mixtures and the water-to-binder ratio by mass (w/b) was the same for all mortars containing mineral additions. The names of the mortars (Table 2) and the concrete mixtures (Tables 3 and 4) refer to the mineral additions used and their approximate content in mass-%.

The mortars were produced according to EN 197-1 using a Hobart mixer. From each mixture 15 mortar prisms ($40 \times 40 \times 160 \text{ mm}^3$) and one cube ($150 \times 150 \times 150 \text{ mm}^3$) were produced to measure compressive strength (3 prisms), carbonation resistance (12 prisms) and oxygen diffusion (cube). All samples were demolded after 24 h and subsequently moist cured (>95% relative humidity) until the age of 28 days.

After the moist curing, the mortar prisms used to test the carbonation resistance were conditioned at $20 \pm 1 \text{ }^\circ\text{C}$ and $57 \pm 2\%$ relative humidity until the age of 56 days. Then, the upper and lower sides of the prisms were coated with epoxy to allow CO_2 ingress only from the lateral cast surfaces. Subsequently, each of the four sets consisting of three prisms were moved to one of the following exposure conditions: carbonation chamber with 1% CO_2 ($20 \text{ }^\circ\text{C}$, 57% RH), carbonation chamber with 4% CO_2 ($20 \pm 1 \text{ }^\circ\text{C}$, $57 \pm 2\%$ RH), sheltered outdoor exposure and unsheltered outdoor exposure. The samples in the sheltered outdoor exposure were stored in a shelter having one side entirely open. The unsheltered exposure was on a roof where the samples were placed on steel profiles to prevent capillary suction of water accumulated on the roof top.

Cores (diameter of 100 mm, height of 30 mm) were taken from the cube after 28 days to measure oxygen diffusion. The cores were conditioned at $20 \pm 1 \text{ }^\circ\text{C}$ and $35 \pm 2\%$ RH for seven days and then dried in an oven at $50 \text{ }^\circ\text{C}$ for 24 h before the measurement started. The mass change during conditioning was recorded to obtain information on the drying behavior of the mortars.

The samples for the investigation with the SEM were taken from the mortar bars in the carbonation chamber after an exposure at 4% CO_2 for 35 days (mortars OPC-0.48, MS-15, P-15 and S-65). The samples were dried in an oven at $50 \text{ }^\circ\text{C}$ for three days, epoxy impregnated and polished.

The same mortars were used for the MIP analysis. Samples were extracted with pincers from carbonated and non-carbonated areas of mortar bars after an exposure in the carbonation chambers at 4% CO_2 for 35 or 91 days depending on the progress of carbonation. Afterwards, the pieces were stored in isopropanol for 7 days to remove the water and then dried at $50 \text{ }^\circ\text{C}$ for another 7 days.

The calculation of the reactive calcium oxide ($\text{CaO}_{\text{reactive}}$) and the reacted CaO ($\text{CaO}_{\text{reacted}}$) in the mortars displayed in Table 2 is explained in Section 4.

2.1.2. Concrete

Two ordinary Portland cements (CEM I 32.5 R, CEM I 42.5 N HS), a cement containing 15 mass-% limestone powder (CEM II/A-LL), a cement containing approximately 15 mass-% of limestone powder and 20 mass-% low-calcium fly ash (CEM II/B-M (V-LL) 32.5 R) and a GGBS cement (CEM III/B 32.5 N) were used to produce the concrete. The composition of the five cement types used is given in Table 3. The mix design of the concrete is given in Table 4. The cubes with a side length of 150 mm were demolded after 24 h and subsequently moist cured (>95% relative humidity) until the age of 28 days.

As the concrete mixtures were produced for another project dealing with sulfate resistance [25], only one cube per mixture was available for testing the carbonation resistance. At the age of 28 days, every cube was cut in the direction of casting in two halves of identical dimensions. All sides except the cut surface and the opposite cast surface were coated with epoxy. One half of the cube was stored unsheltered on a roof and the other half sheltered under a roof (identical storing conditions to the mortar prisms).

At 28 days, cores (diameter of 100 mm, height of 50 mm) were taken from one cube to determine oxygen diffusion. Conditioning was identical to the mortar samples. The mass change during conditioning was recorded to obtain information about the drying behavior of the different concrete mixtures.

The calculation of the reactive calcium oxide ($\text{CaO}_{\text{reactive}}$) and the reacted CaO ($\text{CaO}_{\text{reacted}}$) in the concrete mixtures shown in Table 4 is explained in Section 4.

2.2. Methods

Compressive strength of the mortar prisms and the concrete cubes were determined according to DIN EN 1015-11 [26] and EN 12390-3 [27] respectively.

The procedure for the determination of carbonation coefficient is based on SN 505 262/1 [28]. Before transferring the 12 mortar prisms per mixture to the four different exposure conditions, the initial carbonation depth was determined. An approximately 2.5 cm thick slice was split of the prisms. The freshly broken surface was sprayed with phenolphthalein and photographed. Carbonation depth was analyzed with image analysis on 10 points per lateral cast surface. As three prisms were used for each of the four different exposure conditions, the total number of measurements per exposure condition, age and mixture was 60. The measurements on the concrete were performed the same way. After the initial measurement, the carbonation depth of the samples in carbonation chambers was measured after an exposure of 7, 35

Table 1
Composition of the two cements and the microsilica used for the mortars.

Material	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	Cr ₂ O ₃	MnO	TiO ₂	P ₂ O ₅	MgO	K ₂ O	Na ₂ O	SO ₃	L.O.I.
CEM I 52.5 R	62.7	20.2	5.29	3.25	0.01	0.06	0.29	0.22	1.65	0.90	<0.03	3.82	1.34
CEM III/B 42.5 L-LH HS	48.8	30.6	9.73	1.18	n.a.	n.a.	n.a.	n.a.	5.19	0.59	0.29	1.43	n.a.
Microsilica	0.36	92.8	0.94	0.13	n.a.	0.03	0.01	0.05	0.74	1.09	0.39	0.54	2.66

L.O.I. = loss on ignition.

n.a. = not analyzed.

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