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Experimental carbonation study for durability assessment of novel cementitious materials

EXPERIMENTAL CARBONATION STUDY FOR DURABILITY ASSESSMENT OF NOVEL CEMENTITIOUS MATERIALS

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SUMMARY: The design process of concrete structures is carried out using standards and guidelines, while the durability predictions of concrete structures is supported only with exposure classes and experience-based requirements. To improve durability predictions of the carbonation resistance of concrete, a numerical model is being developed within the Horizon 2020 project EnDurCrete, coupling the rate of carbonation, and the drying rate. To verify the numerical model, an accelerated carbonation study was carried out. Experiments were conducted on mortars incorporating a novel CEM II/C (S-LL) cement, developed within the EnDurCrete project, and a commercially available reference cement CEM II/A-S. EnDurCrete mortars (EnM) and reference mortars (RefM) were prepared with water-cement ratios of 0.6 and 0.5 (denoted with label extensions -06 and -05). Visual assessments and thermogravimetric analysis (TGA) were used to measure the carbonation rates, which were found to be $\sim 1.0 \text{ mm day}^{-0.5}$ in EnM-06 and $\sim 0.6 \text{ mm day}^{-0.5}$ in RefM-06, while in EnM-05 and RefM-05 the values were ~ 0.7 and $\sim 0.2 \text{ mm day}^{-0.5}$ respectively. Additionally, TGA shows that the initial portlandite (CH) content is $\sim 1.5 \text{ wt\%}$ in EnM-06 as opposed to $\sim 3.0 \text{ wt\%}$ in RefM-06. The difference in the initial CH content in the two hydrated binders might explain the difference in their carbonation rate. During the moisture transport experiments a gravimetric method was used to determine mass changes as specimens underwent drying and resaturation with and without CO₂ present. The drying led to a decrease in mass, but in the presence of CO₂ this mass loss was compensated by the mass gain due to uptake of CO₂ during carbonation. The resaturation experiments indicate an increase in the suction porosity in the carbonated samples compared to the non-carbonated samples.

KEYWORDS: Concrete, absorption of water, carbonation, durability assessment, model verification.

1 INTRODUCTION

The design process of concrete structures relies on standards like Eurocode 2 [1], [2] and guidelines like fib Model Code 2010 [3]. The main focus of the existing codes and guidelines is on mechanical performance, which is analysed with advanced structural design models. In contrast, durability related phenomena are addressed mainly through the selection of the exposure classes and experience-based requirements. As a result, concrete durability forecasts are only crude approximations not supported by the sophisticated modelling of concrete degradation over time. The deterioration and maintenance of concrete are having a significant impact on the public sector budget. Maintenance costs and shutdowns of the infrastructure like tunnels, bridges or power plants are particularly important, due to their impact on the wider community.

When concrete is exposed to an aggressive environment with an increased concentration of carbon dioxide in combination with moisture, concrete undergoes a carbonation process. The main process in carbonation of concrete is the reaction of portlandite ($\text{Ca}(\text{OH})_2$, commonly denoted CH) with carbon dioxide (CO_2). The reaction products are calcium carbonate (CaCO_3) and water (H_2O). This reaction leads to a net mass gain, as the uptake of CO_2 (44 g/mol) leads to a larger mass increase compared to the release of water (12 g/mol). While the products of this chemical reaction are not detrimental to concrete durability per se, the consumption of CH reduces the pH value of the pore solution, which may lead to the corrosion of reinforcement steel. The rate of carbonation is influenced by the curing conditions of concrete and by the climatic and local conditions to which the concrete is exposed. To accurately predict concrete deterioration, a numerical model for concrete carbonation is being developed within the Horizon 2020 project EnDurCrete.

The European Commission and European Construction Industry push to improve and develop technologies that reduce CO_2 emissions as well as energy and material usage in the cement production processes [4]. Many studies attempt to develop environmentally-friendly concrete by reducing Portland clinker content in cement and substituting it with supplementary cementitious materials [5]–[7]. In this manner, the above-mentioned model development is also linked to another aim of the EnDurCrete project namely, the development of low carbon-footprint binders aligned with the new edition of prEN 197-1 [5]. One such novel cement is CEM II/C (S-LL), a low-clinker cement containing high-value industrial by-products. The use of this cement is aiming to create cost-effective sustainable concrete on one hand and high durability concrete, which can withstand exposure to aggressive environments on the other hand. Details of the novel cement CEM II/C (S-LL) developed by *HeidelbergCement* are summarized by Bolte et al. [6].

This study aims to present experimental results of an accelerated carbonation programme conducted on mortar samples incorporating novel CEM II/C (S-LL) binder developed within the EnDurCrete project and to compare it with the performance of mortars prepared with the commercially available cement CEM II/A-S. The specimens underwent accelerated carbonation in the conditioning chamber with controlled CO_2 concentration, temperature and relative humidity. The carbonation rate was assessed by measuring the depth of carbonation over time visually using a pH indicator, and by thermogravimetric analysis (TGA). The impact of carbonation on moisture transport was evaluated by the gravimetric method on specimens exposed to a specific protocol of drying and wetting.

The results of the accelerated carbonation tests will serve for the validation of the numerical model dealing with carbonation processes in concrete namely, the coupling between saturation degree, water transport and carbonation extent. The numerical model simplifies carbonation to the dissolution of one combined Ca phase, which embodies all Ca-based hydrates in the hydrated cement, and the precipitation of the calcite phase [7]. In addition, the results aim to further the understanding of concrete deterioration in a CO_2 -rich environment. Consequently, the design of concrete structures in terms of durability will be improved resulting in reduced maintenance costs and shutdowns of the infrastructure caused by the deterioration of concrete due to carbonation.

2 MATERIALS AND METHODS

Concrete used in tunnels is normally designed with a relatively low water/cement (w/c) ratio and therefore measurable carbonation only occurs during long-term exposure. To accelerate the process, mortar mixes used in this study have a higher w/c ratio than usually applied for such structures, namely 0.50 or 0.60 so that measurable carbonation takes place in a reasonable timeframe. Mortar mix designs developed by *HeidelbergCement* are shown in Table 1. Specimens were labelled EnM for EnDurCrete mortar using novel CEM II/C (S-LL) cement and RefM for reference mortar using CEM II/A-S cement. The label extension -05 or -06 was used to denote w/c ratio. The two cements are composite cements blends containing the same type of components namely clinker (K), ground granulated blast furnace slag (S) and limestone filler (LL), however, in different ratios [6] as shown in Table 1.

Mortar prisms measuring 10 cm × 10 cm × 40 cm were cast for this study. The prisms were cured in a humidity chamber with relative humidity (RH) > 95 % at 20 ± 2 °C for 21 days as per EN 12390-2 [8]. Next, the prisms were wrapped in plastic foil and transported from the casting to the testing facilities where they were cured at 20 °C up to 76 days. The moisture state of the specimens was checked at this stage using the method described in [9], [10] to confirm that the specimens did not dry out. After curing, the prisms were cut laterally either into thick blocks with dimensions 10 cm × 10 cm × 7 cm or into thin plates with dimensions 10 cm × 10 cm × 2 cm. The thick blocks were used for the carbonation rate measurements, while the thin plates were used for moisture transport experiments.

Table 1: Mortar mix design, stating the mass of components needed for 1 m³ of mortar. EnM and RefM stand for mortar made with EnDurCrete and reference cement respectively, while extensions -06 and -05 denote w/c ratios. The composition of cement is given as wt% of clinker (K), ground granulated blast-furnace slag (S) and limestone (LL). Mortar mix designs and types of cement were provided by HeidelbergCement.

Components		Mass of component per 1 m ³ of mortar [kg]						
		K (%)	S (%)	LL (%)	EnM-06	RefM-06	EnM-05	RefM-05
Cement	CEM II/C (S-LL)	50	40	10	487	/	552	/
	CEM II/A-S	83	13	4	/	487	/	552
Aggregate		Sand 0/4			1524	1524	1498	1496
Admixture		Superplasticizer			1.5	1.8	3.2	3.8
Water					292	292	264	263

2.1 Carbonation rate

Eight thick blocks of each mix were placed into the carbonation chamber with a temperature of 21 ± 2 °C, RH 60 ± 10 % and 1 % CO₂ according to EN 13295 [11]. Four blocks were used for the pH-indicator method while the other four were used for the TGA method.

For the pH-indicator method, one block was taken out of the carbonation chamber after 14, 28 and 90 days and, depending on the w/c ratio, either after 146 (w/c 0.60) or 167 days (w/c 0.50) of exposure. It was split perpendicularly to the sawn surface. The carbonation front was visually determined with a ruler on a freshly split surface with dimensions 7 × 10 cm², which was sprayed with a thymolphthalein pH-indicator solution. Where the mortar is unaffected by the carbonation (pH above 9.3 to 10.5) indicator turns blue while the carbonated surface remains uncoloured.

For the TGA method, the remaining four blocks were used, which were taken out of the carbonation chamber the same day as the blocks for the pH indicator test. The blocks were profile-ground inwards from the sawn surface. The powder extracted from the consecutive layers was used for the TGA measurements using a *Mettler Toledo TGA/DSC3+* device. The amount of CH relative to the ignited mass of the powder (CH_{ignited}) at 900 °C was calculated as

$$CH_{\text{ignited}} = \frac{w_{400} - w_{550}}{w_{900}} \times \frac{M_{\text{CH}}}{M_{\text{H}_2\text{O}}}$$

where w_{400} , w_{550} and w_{900} refer to the mass of powder at 400, 550 and 900 °C respectively and M is the molar mass of either CH or water [12]. Since carbonation consumes CH, its content is a measure of the degree of carbonation. The CH content was plotted as a function of the depth from the exposed surface. A detailed description of the experimental set-up can be found in [10].

2.2 Moisture transport

The moisture transport experiment was carried out on the thin plates. It consisted of several stages of conditioning as shown in Figure 1, where stage 1 corresponds to curing described at the beginning of Ch. 2. After 76 days of curing, thin plates were sawn and the plates were organized into two groups, namely group “0 %” and group “3 %” indicating the CO₂ concentration to which groups were exposed in stage 2. Throughout the experiment, the mass of the plates was measured at regular intervals using a balance with accuracy 0.01 g.

In stage 2, the thin plates of the “3 %” group were placed in the carbonation chamber with a temperature of 21 ± 2 °C, RH 60 ± 10 % and 3.1 % CO₂ according to EN 13295 [11]. The higher CO₂ concentration, compared to 1 % used on the thick blocks in Ch. 2.1, was assumed to have no impact on the type of phases that form during the carbonation process [13]. The thin plates in the “0 %” group were stored in the conditioning chamber with a temperature of 18 – 25 °C and RH 50 – 65 % at a reduced CO₂ concentration. Soda lime as a CO₂ trap was put under the specimens to capture carbon dioxide and reduce its concentration in the chamber’s atmosphere. Stage 2 lasted for 146 days. At the end of stage 2, one plate from each group was split in half and the carbonation depth was measured using a phenolphthalein pH-indicator solution. The rest of the plates proceeded to stage 3.

In stage 3, the remaining plates from both groups were re-saturated. The plates were first saturated by capillary absorption. The water level was gradually increased over several days until the specimens were completely immersed.

Again, the mass of the plates was measured at regular intervals. When they all reached a near to constant mass, they were further saturated by vacuum saturation according to EN 12390-11 [14]. Stage 3 lasted for 63 days.

In stage 4, the specimens were kept in normal atmospheric conditions at 20 °C and RH 60 %. This stage lasted 33 days. Once the plates reached a constant mass, they were further conditioned at 20 °C and RH 30 % in stage 5. Upon completion of stage 5 the total porosity of specimens was determined according to SIA 262-1 [15].

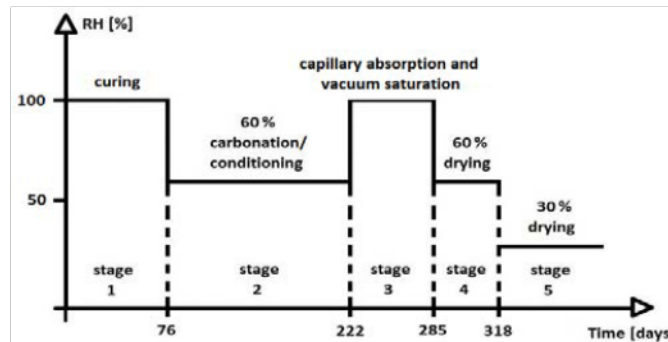


Figure 1: Stages of the moisture transport experiment carried out on the thin plates.

3 RESULTS AND DISCUSSION

The results of the accelerated carbonation study, where carbonation rate was measured by a pH-indicator and by TGA, are presented in Ch. 3.1. Results of both methods are compared and discussed and they are found to have good correlation. Moisture transport results, given in Ch. 3.2, indicate an increase in porosity due to carbonation in specimens with w/c ratio 0.6.

3.1 Carbonation rate

Carbonation depth results assessed visually after spraying the pH-indicator solution on the freshly split surfaces are given in Figure 2 where carbonation depth is plotted against the square root of time. In general, there is a linear relationship between the carbonation depth and the square root of time, therefore a straight line was fitted to the results. It can be seen, that the carbonation rate of $\sim 1.0 \text{ mm day}^{-0.5}$ found in EnM-06 is the highest, while RefM-05 has the lowest carbonation rate of $\sim 0.2 \text{ mm day}^{-0.5}$. The other two mortars, namely EnM-05 and Ref-06, have approximately the same carbonation rate of $\sim 0.6 \text{ mm day}^{-0.5}$. Consequently, after 146 days of exposure, EnM-06 shows a carbonation depth of 13 mm as opposed to the 9 mm measured on RefM-06. Similarly, EnM-05 shows a carbonation depth of 8 mm while only 4 mm was measured on RefM-05. The results thus indicate that a higher w/c ratio leads to a higher carbonation depth. This is expected since higher w/c ratio generates higher porosity of the cement matrix, allowing a faster penetration of CO_2 into the sample [16].

The CH content determined by TGA as a function of depth for the samples prepared with a w/c ratio of 0.6 are shown in Figure 4. In general, the CH content decreases towards the surface where no CH can be detected because all of it was consumed during carbonation reaction. The plateau level of the CH content determined on deeper sections represents the amount initially present in the uncarbonated mortars. The depth at which the CH content equals zero corresponds well to the carbonation depth measured with the visual method and therefore results already shown in Figure 2.

The lower carbonation resistance of the EnDurCrete mortars compared to the reference mortars might be related to the difference in the CH content in the uncarbonated mortar samples (Figure 3). Since less CH is available in the EnDurCrete mortars compared to the reference mortars, these samples have a lower buffer capacity during carbonation. Additionally, Revert et al. [17] have observed that low clinker binders get a coarser porosity upon carbonation enabling faster carbonation rates.

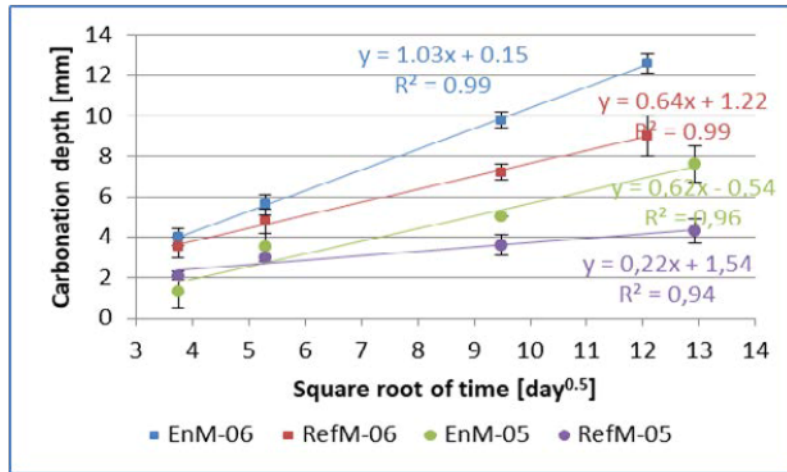


Figure 2: Carbonation depth measured visually after spraying the split samples with a pH indicator plotted against the square root of time. “EnM” and “RefM” stand for EnDurCrete and reference mortar respectively, while extensions “-06” and “-05” refer to water/cement ratios of 0.6 and 0.5. The error bars indicate the variation in the carbonation depth (10 measuring points) measured in one sample for each mortar and exposure time.

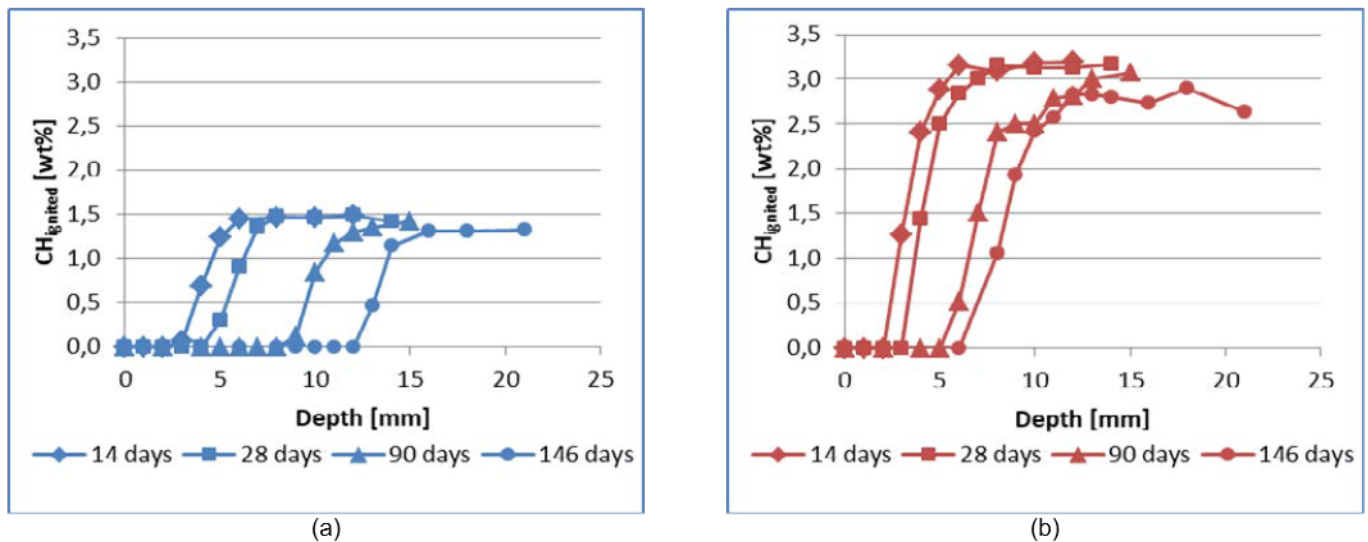


Figure 3: CH content measured with thermogravimetric analysis (TGA) after 14, 28, 90 and 146 days of exposure to accelerated carbonation for (a) EnDurCrete mortar (EnM-06) and (b) reference mortar (RefM-06) both made with w/c ratio of 0.6.

3.2 Moisture transport

The mass changes of the thin plates during the moisture transport experiments are shown in Figure 4 for w/c ratio 0.6 and in Figure 5 for w/c ratio 0.5. The results in both figures are normalised to the initial mass of each plate. The rapid drop of mass at the beginning of stage 2 is probably due to surface drying of the freshly cut samples. The plates in group “0 %” continue to lose mass until equilibrium is reached between 96 and 98 wt% of the initial mass. When looking at the drying profiles of the 0% series, we can see that the RefM-06 seems to dry faster and to a larger extent compared to the EnM-06. Plates in group “3 %”, on the other hand, first decrease in mass but start to increase in mass after ~3 days of exposure, at this point the net mass gain due to carbonation (see Ch. 1) compensates for the mass loss due to drying. Sanjuán et al. [18] also report an initial decrease in weight followed by mass gain despite their specimens having been stabilised at 60 % RH before carbonation. The only exception where the mass continues to decrease throughout stage 2 is EnM-06. The decrease or increase in mass in these experiments can give a hint on which of the two mechanisms (drying vs carbonation) has the strongest impact on the weight of the sample. When CO₂ is present, carbonation will cause a weight gain due to the binding of CO₂ in the reaction products. This weight gain will partially counteract the mass loss due to the simultaneous drying. According to Figure 2, the EnM-06 mortar carbonates faster than the RefM-06, however, due to the lower clinker content in the EnM-06 mortar, it has a lower CO₂ binding capacity compared to the RefM-06. This might explain why the EnM-06 mortar does not gain any mass during simultaneous drying and carbonation, whereas the RefM-06 mortar does (stage 2 in Figure 4 and Figure 5).

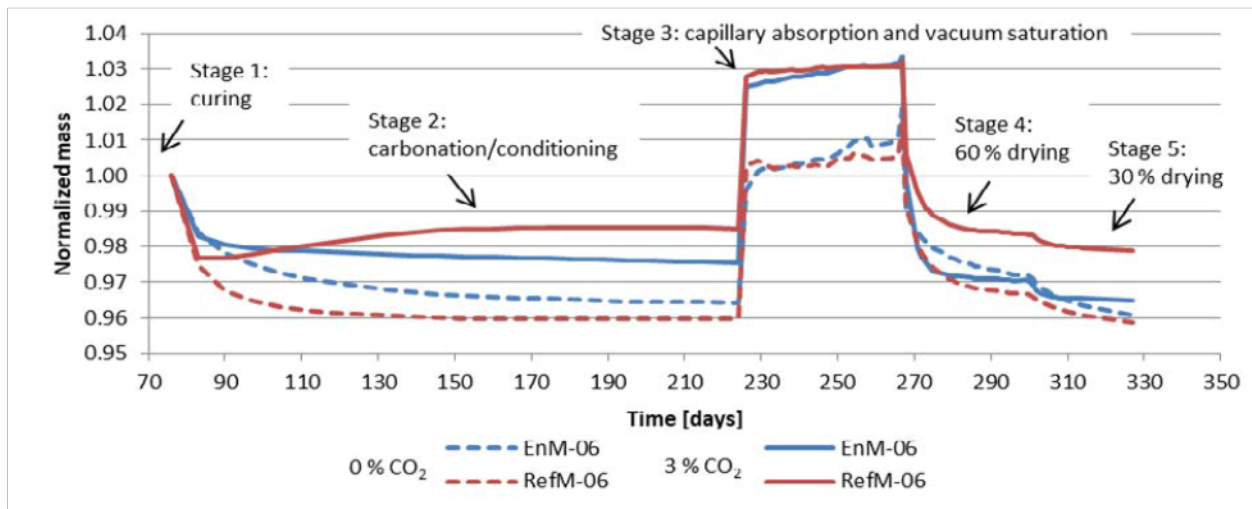


Figure 4: Mass changes during the moisture transport experiment for plates with water/cement ratio of 0.6. In stage 2 one group was exposed to carbonation in 3.1 % CO₂ environment and the other to conditioning in ~0 % CO₂ environment. “EnM” and “RefM” stand for EnDurCrete and reference mortar respectively.

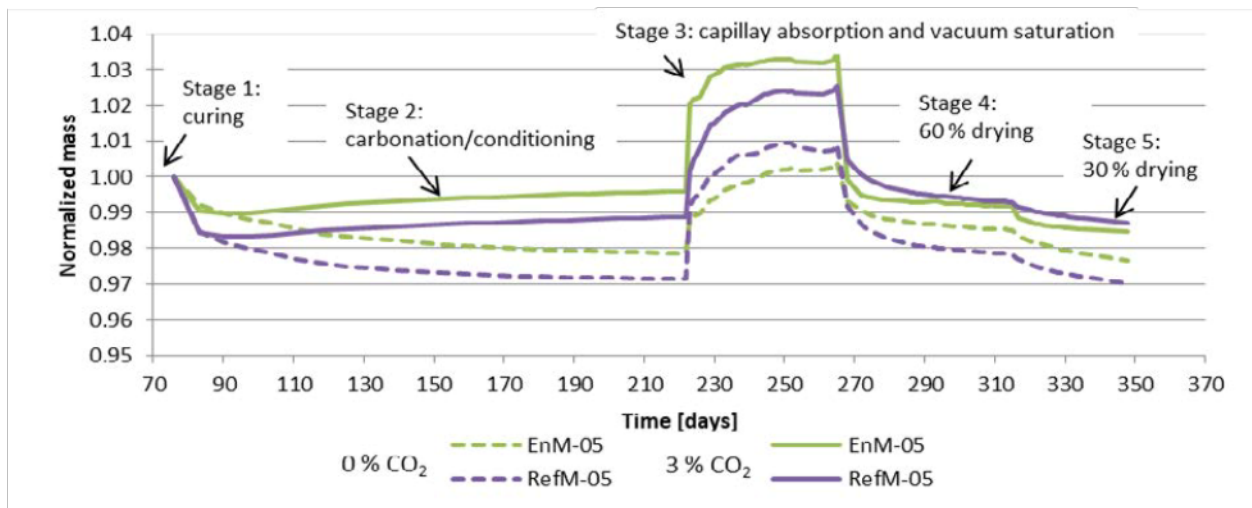


Figure 5: Mass changes during the moisture transport experiment for plates with water/cement ratio of 0.5. In stage 2 one group was exposed to carbonation in 3.1 % CO₂ environment and the other to conditioning in ~0 % CO₂ environment. “EnM” and “RefM” stand for EnDurCrete and reference mortar respectively.

In stage 3, significant differences between groups “0 %” and “3 %” are observed. The uncarbonated plates in the former group absorb significantly less water than carbonated plates in the latter group compared to their starting weight. This indicates an increase in capillary porosity during carbonation compared to the uncarbonated samples. This assumption is confirmed by porosity results shown in Figure 6b where one can see that in “-05” specimens porosity increased for ~2 % due to carbonation which isn’t the case for -06 samples

Stages 4 and 5 show gradual loss of mass. For specimens in “0 %” group, the mass at the end of stage 5 (30% RH) is approximately the same as mass at the end of stage 2 (60% RH). Normalised mass (to initial mass) of carbonated specimens is by ~2 % larger than the normalised mass of uncarbonated specimens.

The fact that the EnM-06 and RefM-06 were fully carbonated, corresponds to the plateauing of the carbonated samples in Figure 4 during stage 2, confirming that these samples have reached equilibrium at those conditions. The corresponding curves for the carbonated EnM-05 and RefM-05 samples in Figure 5, do not reach the plateau during stage 2, which agrees with the fact that they haven’t been fully carbonated yet as shown in Figure 6.

The degree of carbonation at the end of stage 2 was checked on one thin plate with the pH-indicator method. Plates were considered fully carbonated if the depth of carbonation was equal to half of the plate thickness, that is 10 mm. The

results for plates in groups “3 %” and “0 %” are shown in Figure 6a. Plates in the group “3 %” and w/c ratio 0.6 (EnM-06 and RefM-06) carbonated completely while those with w/c ratio 0.5 carbonated only half the depth in case of EnM-05 and quarter of the depth in the case of RefM-05. This is an undesired outcome since the numerical model calibration is planned to be performed on a fully carbonated material on one hand and completely uncarbonated on the other. The results show that the reduced CO₂ levels in the conditioning chamber were able to limit the carbonation depth of the 0% samples. No carbonation depth was measured on 0% EnM-05 and RefM-05 samples.

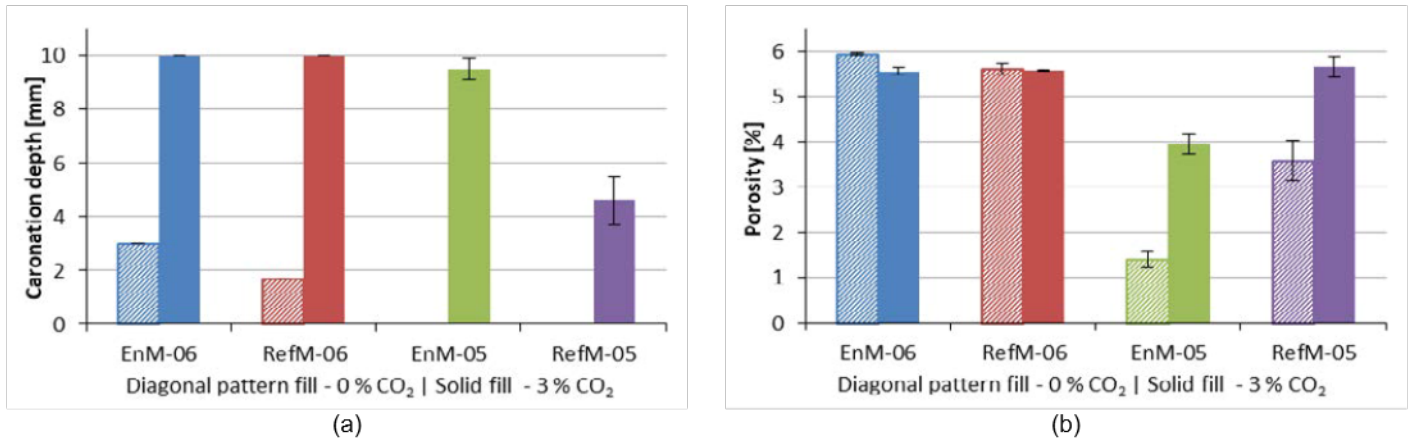


Figure 6: Carbonation depth (a) and total porosity (acc. SIA 262-1) (b) of conditioned specimens after stage 5. Specimens in group “3 %” were exposed to 3.1 % CO₂ atmosphere while specimens in group “0 %” were exposed to the atmosphere with reduced CO₂ content. “EnM” and “RefM” stand for EnDurCrete and reference mortar respectively, while extensions “-06” and “-05” refer to water/cement ratios of 0.6 and 0.5. The error bars indicate the variation in values measured on three sample.

4 CONCLUSIONS

An experimental programme was carried out to collect data for verification of numerical model studying (1) rate of carbonation, and (2) impact of carbonation on moisture transport. For the verification of the rate of carbonation model, thick mortar blocks were exposed to accelerated carbonation conditions and the depth of carbonation was measured visually on split samples sprayed with a pH-indicator and on profile-ground specimens using the TGA method. For the model verification, the impact of carbonation on moisture transport was monitored by measuring the mass of thin mortar plates subjected to a specific protocol of conditioning.

The results obtained in this study indicate that the carbonation depth is affected by the amount of clinker in the cement and w/c ratio. The lower the amount of clinker and/or higher the w/c ratio, the higher the carbonation rate is. Carbonation rates were found to be $\sim 1.0 \text{ mm day}^{-0.5}$ for EnM-06 and $\sim 0.6 \text{ mm day}^{-0.5}$ for Ref-06, while for EnM-05 and RefM-05 carbonation rate was ~ 0.7 and $\sim 0.2 \text{ mm day}^{-0.5}$ when exposed to accelerated carbonation with 1 % CO₂. Results of both methods, namely visual and TGA method, are in fairly good agreement. Additionally, TGA shows that initial CH content is $\sim 1.5 \text{ wt\%}$ in EnM-06 as opposed to $\sim 3.0 \text{ wt\%}$ in RefM-06. Based on the results of this study, the lower initial CH content of the EnDurCrete samples might be the reason for the larger carbonation depth.

The moisture transport experiments show that carbonation increases the total porosity, which can facilitate the transport of deleterious substances, and therefore can promote other deterioration processes in addition to corrosion of reinforcement. The results obtained on EnM-06 show that during carbonation at constant RH EnM-06 performs differently compared to other mortars. In contradiction to the results obtained in the carbonation rate experiment, during exposure to the CO₂-rich environment, its mass continued to drop while it exhibited the highest carbonation rate. The decrease or increase in mass shows which of the two mechanisms (drying vs carbonation) has the strongest impact. As an improvement to the experiment protocol authors recommend conditioning specimens at 60 % RH after stage 1 until a constant mass is achieved and then exposing them to carbonation.

The experimental results presented here will be used for verification of mechanistic/generic models developed within EnDurCrete project [7], which can be applied to novel cements with varying compositions. These models aim to improve prediction of concrete durability in a CO₂-rich environment and therefore reduce the maintenance-related costs, particularly in the public infrastructure sector.

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