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Impact of the water-curing time on the carbonation initiation period of high-volume limestone powder concrete

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ABSTRACT

The high-volume limestone powder concrete (HVLPC) is one of the potential CO_2 mitigation strategies within ready-mixed concrete sector. As with other low-clinker concretes, its durability related properties, carbonation resistance especially, could present a problem in the structural applications. This work deals with the influence of the water-curing time on the carbonation initiation period of HVLPC. An experimental campaign was designed which included various curing times, from 1 day to 28 days. Beside the reference mix (ordinary Portland cement concrete - OPC), HVLPC mixes with three different percentages of limestone powder (LP) in the powder phase (47%, 58% and 65%), and with two different LP particle size distributions within each group, were designed to have similar compressive strength. All mixes were tested under accelerated and natural carbonation conditions. Test results showed significant impact of the curing time on the HVLPC carbonation resistance, but different from OPC. The Model Code 2020 prediction model was tested against test results and it was found that it largely overestimated the impact of curing duration for short curing times (1 and 3 days), while underestimated this impact for 14 and 28 days of curing. New expression for the execution transfer parameter kc was proposed based on the own experimental results.

1 Introduction

In a pure mass-flow sense, concrete is by far the largest human-made product. Some research showed that human made mass, with concrete and aggregates making its large majority already exceeded all living biomass on Earth [1]. According to Global Cement and Concrete Association GCCA [2], 14 billion m^3 of concrete was produced in 2020. Global average of CO_2 emissions per tonne of cement varied between 0.6-0.7 tonnes in 2020 depending on the source, including energy related emissions [3-4]. If global yearly cement production is about 4.1 Gt, cement production emits approximately 2.7 Gt of CO_2 per year. This made about 7% of total CO_2 anthropogenic emissions in 2022, which were about 40 Gt according to [5].

About two-third of CO_2 emissions [6] in cement production originates from the decomposition of limestone (calcination) and the rest is associated with energy use. Because of that, cement industry is considered one of the most difficult to decarbonize as decarbonisation of the energy supply will not eliminate the unavoidable CO_2 emissions from calcination [7].

There are multiple ways of reducing CO_2 emissions from cement and concrete production. Savings in cement content, including the clinker substitution with so-called supplementary cementitious materials (SCM), is one of them. However, it is expected that global availability of the most widely used SCMs, fly ash (FA) and blast furnace slag (BFS), will decrease in near and medium term. FA comes from coal power plants and BFS from the steel industry's blast furnaces and these industries are also transitioning [8]. Therefore, other suitable materials must be looked for. Limestone is currently also used as SCM but in small quantities. Since it is abundantly available all over the world, the attempt was made in previous research to increase the participation of the limestone powder in the binder content and therefore to reduce CO_2 emissions from cement and concrete production.

Concrete with high amount of the limestone powder (over 35% in the powder phase) is called high-volume limestone powder concrete – HVLPC. Previous research showed that it was possible to achieve the HVLPC mechanical properties required for the structural concrete, if optimisation of the paste composition, improved particle packing and lowering the water content were applied [9-12]. However, the carbonation resistance remained significantly lower compared with reference clinker-based concrete due to lower alkalinity and higher porosity of HVLPC [13-17].

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Prediction models developed for carbonation initiation period of clinker-based concrete are not directly applicable to HVLPC, as shown by [17, 18].

Radović et al. [17] proposed modification of the Model Code 2010 [19] prediction model for the carbonation depth calculation in the initiation period. The proposed relationship between inverse effective carbonation resistance under natural and accelerated conditions was based on the statistical analysis of experimental results, own and available in literature. However, the authors assumed that curing impact (k_c) proposed in the Model Code 2010 equation was valid for the HVLPC as well. This assumption has to be experimentally verified since curing type and time can differently affect the carbonation initiation period in HVLPC [20]. The problem was rarely investigated in the previous research and the only experimental results found in literature were those reported in [21]. However, the authors tested concretes with up to 30% cement replacement with limestone powder (LP), which cannot be considered as HVLPC.

The objective of this work was to experimentally determine the impact of the curing time on the HVLPC carbonation resistance, and to propose the empirical equation for the execution transfer parameter k_c within the Model Code 2020 (MC 2020) [22] carbonation initiation model. For that reason, an experimental campaign was designed which included various water-curing times, from 1 day to 28 days. Beside the reference mix (ordinary Portland cement concrete - OPC), HVLPC mixes with three different percentages of LP in the powder phase (47%, 58% and 65%), and with two different LP particle size distributions within each group, were designed to have similar compressive strength. All mixes were tested under accelerated and natural (420 days, indoor) carbonation conditions.

2 Experimental procedures

2.1 Component materials and concrete mixture proportions

For this experimental campaign, Ordinary Portland cement CEM I 42.5R [23] with corresponding mean particle size of d_{50} = 11.1 μm (Figure 1) was used. The density of cement was approximately 3100 kg/m^3. The effect of limestone fineness was evaluated using two high-purity LPs, with the same chemical composition (98% $CaCO_3$ content, meeting the EN 197-1 [23] requirements), but different particle size distributions. Determination of particle size distribution was done by laser diffraction method. Limestone powder L_{12} had a particle size distribution (d_{50} = 11.7 μm) similar to OPC, while limestone powder L_3 contained much finer particles (d_{50} = 2.9 μm), Figure 1. The densities of L_3 and L_{12} were about 2720 kg/m^3 and 2690 kg/m^3, respectively.

Locally available river aggregate, divided into three fractions I (0-4 mm), II (4-8 mm) and III (8-16 mm) was applied. The participation of each fraction in the total aggregate quantity (Table 1) was 52%, 21%, and 27% respectively. The workability of fresh concrete was controlled by adjusting the dosage of a polycarboxylate-based superplasticizer.

The target workability class for all mixtures was S4 or S5, with a required slump of ≥ 200 mm, as specified in EN 206-1 [24]. To achieve a meaningful comparison of the results, the target compressive strength was set at $f_{cm,cube}$ = 50±5 MPa, determined using 100 mm cube specimens. This strength range aligns with widely used concrete classes C25/30 and C30/37 according to Eurocode 2 [25]. By selecting this compressive strength, the study ensures that the tested mixtures reflect typical structural concrete applications, allowing for a realistic evaluation of performance. The chosen strength classes also facilitate direct comparisons with existing standards recommendations for commonly used concrete mixtures in construction practice.

Mixtures proportions were determined by the absolute volume method. In the reference mixture, the cement content was 334 kg/m³, and it was partially replaced by 30%, 45%, and 55% using each of L_3 and L_{12} limestone powder respectively. To ensure that the compressive strength remained within the desired target range, it was necessary to reduce the water-to-cement (w/c) ratio in mixtures incorporating limestone powder. This reduction resulted in a lower volume of cement paste, requiring an adjustment in the mixture composition. Consequently, a greater amount of limestone powder was added compared to the quantity of cement replaced, in order to compensate for the volumetric changes and maintain the overall performance of the concrete. The mixture proportions of the tested concretes are presented in Table 1. The mixture designation consists of the type of limestone powder (L_3 or L_{12}) and its mass percentage in the total powder phase (cement + limestone) of the concrete, which were 47%, 58%, and 65%.

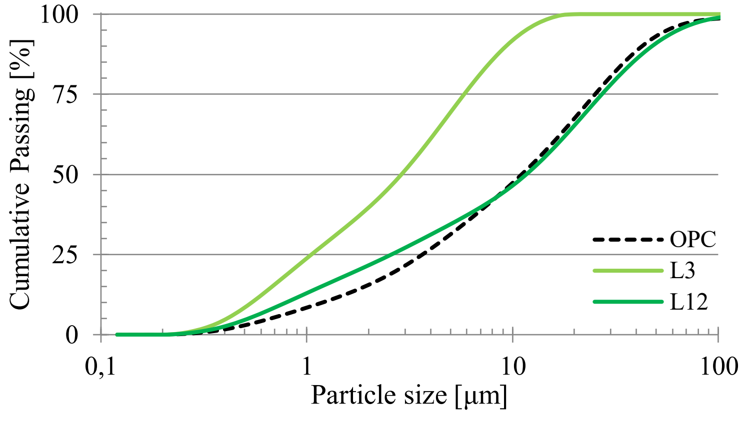

Figure 1. Particle size distribution of cement and limestone powders

Table 1. Proportions of concrete mixtures

Concrete mix	m_c [kg/m^3]	m_{LS} [kg/m^3]	m_w [kg/m^3]	w/c [–]	Aggregate [kg/m^3]	SP [kg/m^3]
OPC	334	0	171	0.51	1874	3.30
L_3-47	230	200	143	0.62	1847	6.45
L_{12}-47	230	200	143	0.62	1846	6.45
L_3-58	182	252	127	0.70	1868	8.60
L_{12}-58	182	252	127	0.70	1866	8.60
L_3-65	153	285	114	0.75	1883	8.60
L_{12}-65	153	285	114	0.75	1881	8.60

2.2 Preparation, casting, curing, and testing of concrete specimens

The mixing process for each concrete mixture lasted a total of five minutes. After verification of the workability with a standard slump test [26], the concrete was cast into moulds and compacted immediately after placing. The specimens were then covered with a plastic sheet to prevent moisture loss and stored in the laboratory environment. After 24 hours, the specimens were demoulded and subjected to five different curing conditions for a period of 28 days. The curing conditions are identified by numbers corresponding to the duration of standard curing (1, 3, 7, 14, and 28 days), as defined in [26]. A detailed description of these curing conditions is provided in Table 2.

Compressive strength at 28 days was tested on 100 mm cubic samples, following the EN 12390-3 [27], using three cubes for each mixture and each curing condition.

The carbonation resistance was assessed using 120×120×360 mm prismatic specimens, with two prisms prepared for each mixture. After completing the 28-day initial curing (as described in Table 2), the specimens underwent an additional 14-day conditioning in a climate chamber at 20±2°C and 65±5% relative humidity, to ensure uniform moisture distribution before carbonation exposure. Following this period, the prisms were halved, with one half exposed to accelerated carbonation and the other half used for a natural carbonation test. The accelerated carbonation test was conducted in a carbonation chamber under controlled conditions of 2% CO_2, 20±2°C, and 65±5% relative humidity for 28 days. In contrast, specimens designated for natural carbonation were stored under laboratory conditions (Figure 2) for 420 days to simulate long-term environmental exposure. The mean values and standard deviations of the laboratory environment parameters (relative humidity, temperature, and CO_2 concentration) are detailed in Table 3. The final carbonation depth, for both accelerated and natural carbonation, was determined as the average of eight measurements taken on each side of each prism, in accordance with EN 14630 [28].

Table 2. Curing conditions

Curing period	Curing (1d)	Curing (3d)	Curing (7d)	Curing (14d)	Curing (28d)
1 day	Mould	Mould	Mould	Mould	Mould
1-3 days	Air*	Water	Water	Water	Water
3-7 days	Air	Air	Water	Water	Water
7-14 days	Air	Air	Air	Water	Water
14-28 days	Air	Air	Air	Air	Water

*Air – laboratory-controlled environment (20°C, 65% RH); Water (20°C)

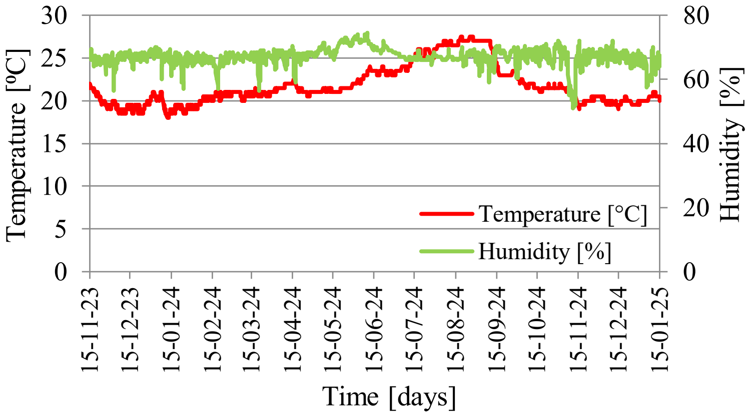

Figure 2. Natural environmental conditions during testing

Table 3. Mean values and standard deviations of environmental conditions

	Temperature [°C]	Relative humidity [%]	CO_2 [ppm]
Mean	21.8	67.1	440
St. deviation	2.3	2.8	70.5

3 Results and discussion

The compressive strengths measured at 28 days for cube samples ($f_{cm,cube}$) and calculated for cylinder samples ($f_{cm,cyl}$), for concrete mixes cured under various conditions, are presented in Table 4.

Table 4. Measured properties of tested concrete mixtures

Mixtures	Curing period [days]	$f_{cm,cube}$ [MPa]	$f_{cm,cyl}$ [MPa]
OPC	1	49.8	37.4
	3	52.4	39.3
	7	52.9	39.7
	14	51.1	38.3
	28	50.1	37.6
L_3-47	1	52.3	39.2
	3	51.3	38.5
	7	55.4	41.6
	14	55.9	41.9
	28	50.2	37.7
L_{12}-47	1	52.9	39.7
	3	53.6	40.2
	7	55.8	41.9
	14	55.7	41.8
	28	50.2	37.7
L_3-58	1	50.7	38.0
	3	54.1	40.6
	7	54.6	41.0
	14	53.4	40.1
	28	44	33.0
L_{12}-58	1	48.3	36.2
	3	50.3	37.7
	7	54.3	40.7
	14	52.4	39.3
	28	47.8	35.9
L_3-65	1	56.1	42.1
	3	57.4	43.1
	7	57.9	43.4
	14	61.6	46.2
	28	51.8	38.9
L_{12}-65	1	46.0	34.5
	3	47.5	35.6
	7	51.2	38.4
	14	49.4	37.1
	28	45.4	34.1

The results showed that there was practically no difference in compressive strength of HVLPC mixes with different fineness of LP (the differences were within 0.6%) for L-47 and L-58 concretes. In mixes with the highest LP content (L-65), limestone powder with higher fineness provided higher compressive strength. The increase of compressive strength was 20% (mixes with L_3 compared to mixes with L_{12} powder which is similar to cement). Except for the L_{12}-65 mix, the compressive strengths of the HVLPC mixes were similar to those of the reference OPC concrete. For all samples cured in water for 28 days, the measured compressive strengths varied between 45.4 and 51.8 MPa, aligning with the initial hypothesis of producing concrete mixes with consistent strength of 50±5 MPa. The only concrete that had strength lower than 45 MPa was L_3-58, whose strength was 44 MPa for samples cured for 28 days in water. However, all samples of this concrete cured for less than 28 days had strength greater than 50 MPa.

The 7 days curing period had a beneficial impact on the achieved compressive strength values, with longer curing period leading to lower compressive strengths. Concrete mixes cured in water for 28 days exhibited the lowest compressive strength compared to all other curing regimes. This outcome may be attributed to the increased amount of water absorbed by the concrete, which remained within the material at the time of testing under compressive load. The retained water, when subjected to external pressure, generates internal stresses and water vapor pressure, which ultimately reduces the fracture toughness of the concrete. Similar conclusions were reached in the study [29]. It is assumed that complete drying of samples cured in water for 28 days would result in greater compressive strength compared to alternative curing methods. As for curing time of 14 days, all concrete mixes showed compressive strengths approximately equal to those of concrete mixes cured for 7 days in water, with the exception of the L_3-65 mix.

As observed, curing had a more pronounced influence on the development of compressive strength in the HVLPC than in the OPC concrete. Among the HVLPC mixes, the curing regime's contribution to strength development appeared independent of the fineness of the LP. Additionally, variations in curing conditions did not significantly affect the rate of strength gain of the tested concretes.

Measured carbonation depths under accelerated (ACC) conditions after 28 days of exposure and natural (NAC) conditions after 420 days of exposure, along with the relationships between carbonation depth and water-curing period for all concrete mixtures, are shown in Figures 3 and 4. It can be seen that with increasing the water-curing period, the carbonation depth decreases. However, the effect of curing was smallest with the reference OPC concrete (17%). As for HVLPC mixes, the impact of water-curing duration increased with the LP content increase in both accelerated and natural conditions. The trend was similar for both types of LP. What however is interesting is that the mixes with 47%

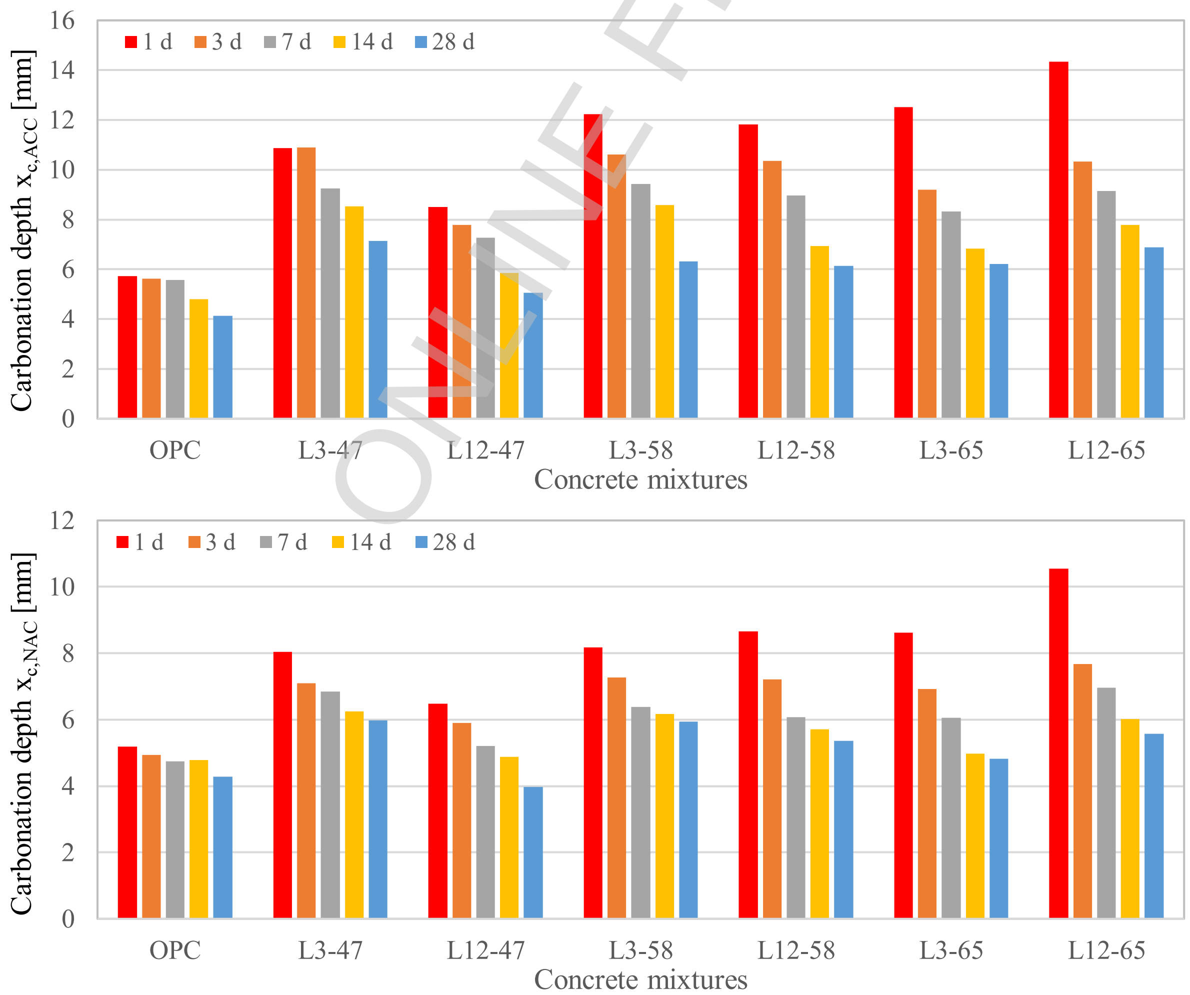

Figure 3. Measured carbonation depths under accelerated (ACC) and natural (NAC) conditions for different curing periods

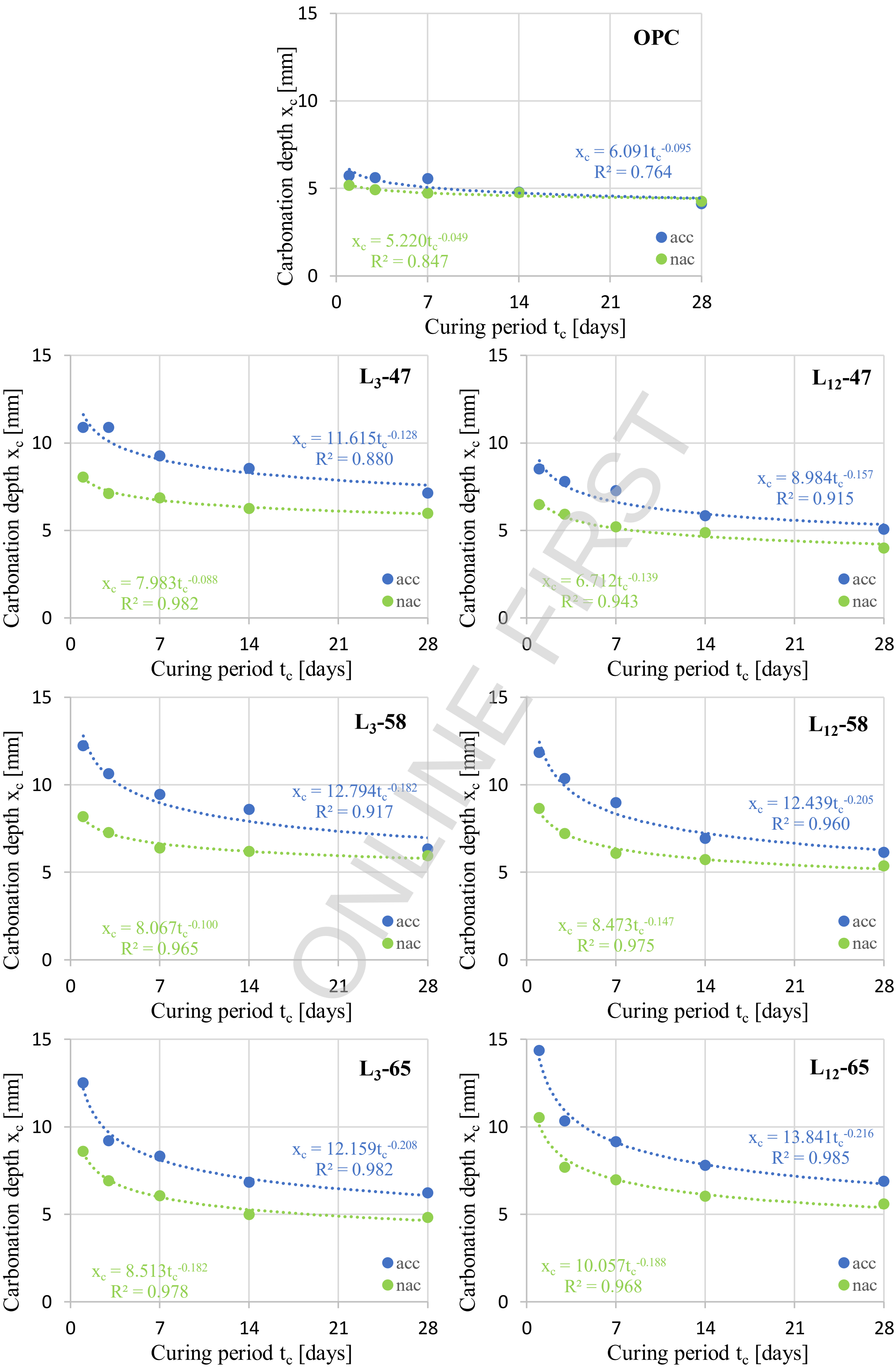

Figure 4. The relationship between the carbonation depth and water-curing period

and 58% of finer LP had generally higher carbonation depths compared with those mixes made with coarser LP for all curing periods. Only the L-65 mix with finer LP had lower carbonation depths under natural conditions (the carbonation depth of L_3-65 was 13%-19% lower compared to L_{12}-65, depending on the curing duration). This is in contrast to the results published in [29], where concrete mixes with finer LP had a lower carbonation depth.

In all cases, the HVLPC mixtures had lower carbonation resistance compared to the reference OPC concrete, although the compressive strength was generally similar or higher. The only mixture that had higher resistance was L_{12}-47, which had a 7% lower carbonation depth. Although the proposed mix design method – characterized by low water content and high plasticizer dosage – successfully maintained compressive strength, the issue of low alkaline reserve persisted, resulting in reduced carbonation resistance. Nevertheless, the test results demonstrated that it is feasible to design concrete mixes that meet both high workability and strength requirements for commonly used strength classes, using as little as 153 kg/m^3 of cement. However, HVLPC mixes had up to 40% greater carbonation depth in natural conditions compared to OPC, for standardly cured samples of 28 days. Generally, the measured carbonation depths after 420 days correspond to the expected values that can be found in the literature for these types of concrete [18].

In order to examine the influence of water-curing period on the HVLPC carbonation resistance, the MC 2020 [22] carbonation depth prediction model was applied. This model includes execution transfer parameter (k_c) that takes into account concrete curing period:

$$k_c = \left(\frac{t_c}{7}\right)^{b_c} \qquad (1)$$

where t_c is the period of moist curing (days), while b_c is exponent of regression with mean value equal to -0.567 and standard deviation equal to 0.024 (normal distribution). However, this exponent was calibrated on the ordinary and blended cement based concretes [30]. For concretes with up to 30% cement replacement with LP, this relationship was tested by Lollini & Redaelli [21], suggesting that this exponent should be -0.10. This relationship was evaluated as well for concretes tested in this research, i.e. concretes with more than about 50% in the powder phase.

In order to evaluate the relationship (1) for the tested HVLPC mixes, it was necessary to determine the coefficient k_c experimentally. This was done based on the measured values of natural carbonation depths of concretes cured for different curing periods $k_{c,t}$:

$$\frac{x_{c,t}}{x_{c,ref}} = \sqrt{\frac{k_{c,t}}{k_{c,ref}}} = \sqrt{k_{c,t}} \qquad (2)$$

where

$x_{c,t}$ carbonation depth of concrete water-cured for time t

$x_{c,ref}$ carbonation depth of concrete water-cured for 7 days

$k_{c,t}$ execution transfer parameter for concrete water-cured for time t

$k_{c,ref}$ execution transfer parameter for concrete water-cured for 7 days, $k_{c,ref} = k_{c,7} = 1$

Using the previously defined relationship (2), the coefficients k_c for OPC and HVLPC mixes were calculated based on the measured carbonation depths under natural conditions. Figure 5 shows the relationship between the execution transfer parameter (k_c) and duration of water-curing. In addition to the experimental results, the figure shows the MC 2020 relationship and Lollini & Redaelli [21] proposal as well.

The relationship between k_c and t_c in the form:

$$k_c = \left(\frac{t_c}{7}\right)^{-0.281} \qquad (3)$$

fits well the test results for all HVLPC mixes, regardless of the content and fineness of LP ($R^2 = 0.88$). The MC 2020 expression for k_c largely overestimates the impact of water-curing duration for short curing times (1 and 3 days), while underestimates this impact for 14 and 28 days of curing. The

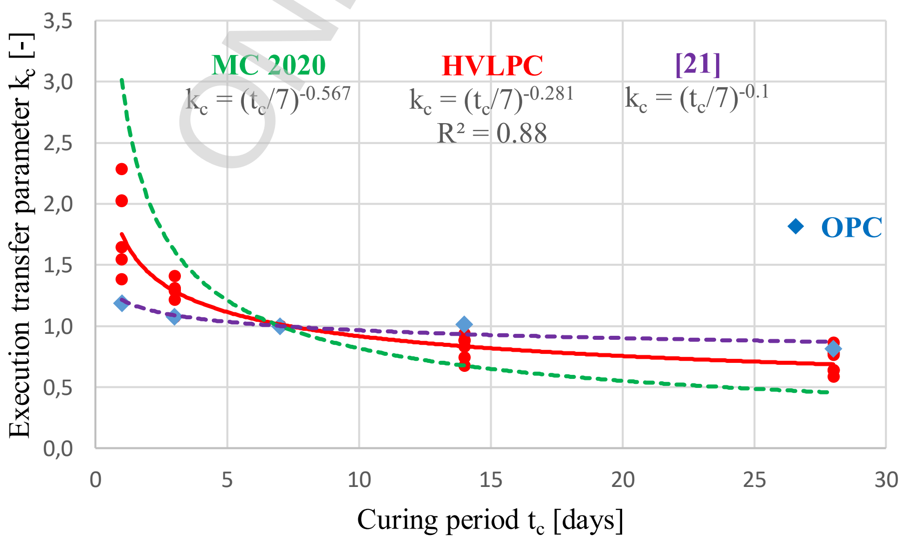

Figure 5. Relationship between execution transfer parameter (k_c) and water-curing period (t_c)

MC 2020 prediction behaves even worse for the tested OPC mix. This is probably the consequence of the calibration of this parameter on the mixes made with both OPC and commercial blended cements, which gave a large scatter of results, especially for curing periods of 1 and 3 days [31]. On the other hand, Lollini & Redaelli [21] proposal made originally for mixes with up to 30% of the LP fits well the OPC mix test results, suggesting that such mixes behaved similarly to OPC concrete. Having in mind that only one OPC mix was tested in this research, this is only a remark, not the conclusion.

4 Conclusions

The results of the experimental testing of the compressive strength and carbonation depth (under accelerated and natural conditions) of reference OPC mix and six different HVLPC mixes were presented. All mixes were water-cured for 1, 3, 7, 14, and 28 days, and for each curing period compressive strength and carbonation depth were determined. Following conclusions were drawn:

- the compressive strength at 28 days of all mixes varied between 44 MPa and 51.8 MPa (i.e. between 33 and 39 on cylindrical specimens);
- except for the L_{12}-65 mix, the compressive strengths of the HVLPC mixes were similar to those of the reference OPC concrete for all curing periods;
- among the HVLPC mixes, the impact of the curing regime on the strength development appeared independent of the fineness of the LP;
- with prolonging the water-curing period, the carbonation depths of all mixes decreased, i.e. carbonation resistance increased;
- impact of the water-curing period on the carbonation resistance is much more pronounced for the HVLPC mixes than for the reference OPC mix;
- impact of the water-curing duration slightly increased with the LP content increase in both accelerated and natural conditions regardless of the LP fineness;
- the MC 2020 expression for the execution transfer parameter k_c is hardly applicable to HVLPC for all tested LP contents and fineness.

The new expression for the execution transfer parameter k_c, which describes the effect of the water-curing time on the HVLPC carbonation resistance, was proposed. The conclusions and the proposal are limited to experimental results obtained within this research.

CRediT authorship contribution statement

A. Radović: Data curation, Investigation, Formal analysis, Resources, Writing - original draft.

V. Carević: Data curation, Investigation, Methodology, Formal analysis, Writing - original draft.

S. Marinković: Conceptualization, Methodology, Supervision, Writing - review & editing.

Declaration of competing interest

The authors declare no conflict of interest.

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