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Extending internal curing to concrete mixtures with W/C higher than 0.42

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1. Introduction

To obtain the required durability, strength and high performance during the life cycle of the structure, curing concrete is crucial from the first hours after its setting. Therefore, an effective in situ curing is required to maximize degree of hydration of cementitious material and to minimize cracking problems due to drying shrinkage.

1.1. Cementitious materials hydration

Hydration starts when mixing water contacts the cementitious materials, causing chemical reactions that produce calcium silicate hydrates, which make concrete stronger, and other hydration products such as calcium hydroxide and monosulphate. The formation of the hydration products is associated with a reduction of the original volume, what is called chemical shrinkage [1].

Some part of the mixing water becomes chemically bonded to the hydration products, some other adsorbed at the surface of the hydration products, and the rest remains in solution at the capillary pores formed during hydration. Cementitious materials get the water needed to promote hydration from the capillary pores, which generates surface tensions that result in volumetric reductions known as autogenous shrinkage when occurs in a closed isothermal system that is not subjected to external forces [1].

ABSTRACT

Internal curing (IC) is an effective method for improving performance of low W/C – low permeability concretes because they require additional water to hydrate the cementitious materials. Conventional concretes, on the other hand, contain enough water to hydrate the cementitious materials, but are frequently not properly cured, allowing drying and compromising strength gain and durability. The aim of this investigation is to assess the effect of IC as a complement to traditional curing in relatively high W/C concretes (W/C above 0.42) under drying conditions. Degree of hydration, compressive strength, and permeability were measured in concretes with IC and without IC. Results show that even under drying conditions, mixtures with IC exhibit 16% higher hydration, 19% higher compressive strength, and 30% lower permeability than their counterparts with no IC. This suggests that IC can be very useful for improving performance in concrete mixtures with relatively high W/C under poor curing conditions.

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Completing cementitious materials hydration process requires two conditions: (i) there must be enough space for the formation of hydration products and (ii) there must be enough water to keep internal relative humidity above 80% for chemical reactions to occur. In concretes with W/C lower than 0.36 condition (i) is not met while in concretes with W/C between 0.36 and 0.42 condition (ii) is not met requiring additional curing water to approach to complete hydration [2].

1.2. Curing of concrete

Curing of concrete plays an important role to minimize shrinkage and early cement paste desiccation; proper curing is vital to the quality of concrete. Concrete mixtures with W/C higher than 0.42 contain more water than required for the complete cement hydration; however, any water loss by evaporation will limit the maximum achievable degree of hydration; curing aims precisely to prevent such loss. An important aspect, often overlooked, is that curing should not only prevent water evaporation and keep concrete mixtures to appropriate internal relative humidity and temperature, but also should minimize shrinkage.

1.3. Application of internal curing to traditional concrete

Internal curing is a curing method studied and applied to low W/C concretes, because they have insufficient water to hydrate the entire cement they have and, due to their low permeability, they prevent access of external curing water [3]. This technique consists of adding curing water reserves within the concrete,

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which will be available to assist the hydration of cementitious materials when the initial water has been used and/or lost to the environment.

Nevertheless, water loss and poor curing conditions can be also present in concrete mixtures with W/C of 0.42 and above. Additionally, current practice shows a tendency to use finer cement or cements containing higher amounts of C_3S , leading an increase in hydration's rate and water use; and also a decrease in permeability at early ages. Both could make traditional curing insufficient to properly assist hydration, making internal curing an attractive alternative not only for low W/C mixtures but also for traditional mixtures.

1.4. Results of an effective curing on concrete properties

The increase in cement hydration, promoted by an effective curing, has been shown to increase mechanical properties such as strength and elastic modulus [4]. However, an effective curing can also reduce shrinkage, and specifically cracking caused by shrinkage, which can also increase strength and elastic modulus [3].

Another mechanical property that has shown improvements with effective curing is creep; studies on concretes with W/C of 0.23 with internal curing with expanded slate as curing agent showed less creep compared with normal aggregate concrete on long-term [5].

Durability of concrete depends on several factors and properties, including permeability, degree of hydration, shrinkage, strength of the cement paste, presence of microcracks, among others. All of them can be improved through an effective curing, whether internal or external. Studies have shown that an effective internal curing decreases permeability of concrete, making more difficult that harmful agents penetrate concrete, increasing its durability. A study on chloride ion permeability in concrete of a low W/C [6] showed that permeability of those mixtures with pre-wetted lightweight aggregate (internal curing agent) had a 39% and 29% of the permeability of the same mixture without internal curing at age 1 and 3 years, respectively.

A more effective curing tends to produce less shrinkage and cracking [1,7]. Studies in concrete with W/C of 0.23, with internal curing using expanded slate as curing agent, showed less shrinkage compared to normal aggregate concrete with long-term [5].

2. Research significance

Nowadays, projects need to be open for service in shorter times each time; this undermines the implementation of a careful and effective traditional curing methods. There are many articles showing the benefits of using internal curing in concretes of low permeability and low W/C, but it is not the same in concretes with W/C higher than 0.42, which remain not studied yet.

The motivation of this research is the applicability of internal curing to complement traditional external curing, in concrete with W/C higher than 0.4 to assess improvement in its properties when subjected to insufficient traditional curing. It is expected that internal curing could maintain high internal relative humidity by delivering water to cementitious materials promoting hydration.

3. Experimental procedure

A total of 10 concrete mixtures were fabricated during the experimental program. Five different W/C were considered (from 0.400, 0.425, 0.450, 0.475, and 0.500); for each W/C, concrete mixtures were prepares prepared with internal curing or without internal curing.

Cement paste and aggregate volume remained constant for all concrete mixtures.

Table 1

Properties internal curing agent used.

Aggregate	Expanded clay sand
Size range (mm)	0.08-5
Absorption at 1 day	21.2%
Absorption at 3 days	28.5%
Absorption at 21 days	30.6%
Absorption at 32 days	30.8%
Dry specific gravity (kg/m ³)	1653
SSD specific gravity (kg/m ³)	1920
Porosity ^a	41%
Desorption ^b (%/days \times 10 ⁵)	10.1

^a Porosity was measured through the Mercury Intrusion Porosimetry (MIP) test. ^b Desorption was measured by monitoring the amount of water loss by the lightweight aggregate – initially immersed for 3 days – when kept at 97% relative humidity. The reported value is the slope of water release versus time in%/ days $\times 10^5$.

Six cylinders (100 \times 200 mm) were cast for each concrete mixture. All specimens were demoulded 24 h after casting, and then placed in a chamber at 23 °C and 50% of relative humidity until age of testing in order to produce drying conditions instead of curing conditions. All test were performed at the age of 90 days.

3.1. Internal curing agent used

Although there are several potential curing agents to implement internal curing, this investigation used expanded clay sand (an artificial lightweight aggregate) given that this has been applied successfully in the past with low W/C mixtures.

This curing agent was been chosen because of its absorption (water uptake), desorption (water release) and its particle size which allows better and more uniform distribution of the curing agent within the mixture; and thus, optimize the efficiency of internal curing minimizing the water transport distance which is limited [8].

The properties of the expanded clay lightweight aggregate are shown in Table 1.

3.2. Mixtures designs

The mixture designs of the concretes used in the study and their fresh properties are shown in Table 2.

The investigation started with W/C of 0.4 in order to produce data below the limit for fully hydration, which is 0.42; and ended with W/C of 0.5 in order to assess the effect of internal curing at relatively high W/C.

Mixtures were produced using 38% and 31% by volume of fine and coarse aggregate, respectively in all mixtures. Entrapped air was assumed to be 2% by volume. No water-reducing admixtures were considered in order to avoid possible effects in the properties of interest. Since mixtures contained different mixing water dosages, workability, measured by slump, varied between 1 and 4 cm; however, all mixtures could be well consolidated by an internal vibrator.

All concrete mixtures with internal curing had the same amount of internal curing water (81 kg/m³ of concrete). This fixed value corresponds to the amount of water lost by a concrete mixture with W/C of 0.6 during 1 year at 50% relative humidity [9]. The internal curing water is contained within the expanded clay lightweight aggregate, which replaces a fixed amount of the normal fine aggregate. The expanded clay used was pre-wetted by immersing the lightweight aggregate in water for 3 days starting from a dry condition. Before mixing, lightweight aggregate was removed from water, let drain and then dried with paper towel to obtain a "surface dry" condition. This was possible because of the relatively low volumes used in the batches. In industrial applications, moisture of the lightweight aggreg yate needs to be measure in order to adjust the water in the mix accordingly.

3.3. Testing

Degree of hydration (LOI, loss on ignition), compressive strength, and chloride ion permeability were measured on all concrete mixtures. The age of testing was 90 days in order to describe mixtures with an advanced time of hardening.

Four samples obtained from four different concrete specimens were tested for degree of hydration for each mixture. Samples were obtained by grinding down concrete using a mortar. Samples were obtained from the surface and center of the specimens in order to obtain a representative result. Approximately, 30 g of crushed concrete were placed in ceramic crucibles. First, the samples were placed at 105 °C for 24 h, in order to remove the evaporable water (water not chemically bonded). Then, the samples were placed for a period of 3 h at 1100 °C. It should be pointed out that at above 1000 °C chemical bonds are broken and the water that has reacted with cement to form the hydration products (known as chemically bonded water) is removed. By knowing the mass loss of the sample between 105 and 1100 °C and the loss on ignition of the unhydrated cement and aggregates (both

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Table 2

Mixtures designs in SSD condition (quantities in kg/m³ of concrete).

Constituents	IC ^a	NIC ^b	IC	NIC	IC	NIC	IC	NIC	IC	NIC
W/C	0.400		0.425		0.450		0.475		0.500	
Water	161		166		170		173		177	
Cement ^c	403		389		377		364		354	
Coarse aggregate ^d	841		841		841		841		841	
Fine aggregate ^e	563	1028	563	1028	563	1028	563	1028	563	1028
Expanded clay	331	-	331	-	331	-	331	-	331	-
Fresh unit weight (kg/m ³)	2263	2464	2237	2434	2278	2449	2287	2463	2288	2432
Slump (cm)	1.0	1.5	1.0	2.0	1.5	1.0	2.5	2.5	2.5	4.0

^a IC: Internal curing applied.

^b NIC: No internal curing applied.

^c Ordinary Portland cement.

^d Siliceous aggregate 10-mm maximum size aggregate.

^e Siliceous aggregate 5-mm maximum size aggregate.

Table 3

Test results and coefficient of variation.

W/C	Curing condition	Degree of hydration		Compressive strength		Chloride ion permeability	
		%	CV ^a (%)	MPa	CV (%)	Coulombs	CV (%)
0.400	IC	62	7.3	74.0	5.6	1671	5.9
	NIC	47	18.4	67.5	8.7	1768	23.8
0.425	IC	70	16.6	61.8	6.6	2846	2.1
	NIC	48	11.1	50.8	7.9	3290	1.8
0.450	IC	66	16.2	66.8	1.0	1587	0.6
	NIC	51	15.9	49.1	7.3	3237	9.3
0.475	IC	69	2.5	55.4	7.5	2649	5.5
	NIC	59	12.0	47.5	11.0	3131	0.8
0.500	IC	67	7.4	61.9	3.2	1625	9.5
	NIC	50	13.7	55.0	3.2	3342	7.3
Average	IC	67	10.0	64.0	4.8	2076	4.7
	NIC	51	14.2	54.0	7.6	2954	8.6

^a Coefficient of variation (standard deviation divided by average value).

normalweight and lightweight), the degree of hydration reached by the concrete mixtures was calculated. In these calculations the non-evaporable water content for fully hydrated cement is considered to be 0.23 g of water by 1 g of cement [10]. This value might vary with cement type, but for comparison purposes required in this investigation the assumed value is adequate.

Testing of compressive strength was performed at 90 days of age, according to ASTM C39. For each W/C, four 100×200 mm cylindrical specimens were tested.

Chloride ion permeability was measured according to ASTM C1202 in two 100×50 mm disc-shaped specimens saw-cut at the age of 90 days. The test consists on placing the flat ends of the specimens on two cells containing different solutions, one cell contains 3% NaCl solution (which will be connected to the negative pole) and the other cell contains 0.3 N NaOH solution (which will be connected to the positive pole). The two poles are then connected a 60 V power supply and the resulting current intensity through the specimen is measured every 5 min for 6 h. The chloride ion permeability is expressed as the charged passed during the 6 h through the specimen.

4. Test results and analysis

Table 3 presents the results of degree of hydration, compressive strength, and chloride ion permeability as W/C varies from 0.4 to 0.5 in the mixtures with and without internal curing.

4.1. Degree of hydration

Fig. 1 presents the results of degree of hydration for the 10 mixtures under investigation.

Theoretically, 100% hydration of cement can be reached in all mixtures with W/C higher than 0.36. This is only if optimum and prolonged curing is considered [11].

As shown in Fig. 1, none of the mixtures reached a value near to a degree of hydration of 100% in despite of some of them having W/C 0.42 and above. This was expected since curing conditions were not optimum and the time of curing was only 90 days.

From Fig. 1, it can be concluded that all mixtures with internal curing improved the degree of hydration compared with those mixtures without internal curing. The largest increase in the degree of hydration was obtained by the mixture with W/C of 0.425 while the lowest one for the mixture with W/C of 0.475; these values correspond to 22% and 10% respectively.

It is interesting to note that under poor curing conditions, such as those used in this investigation; the obtained degree of hydration does not increase as the W/C increases. This is valid for both cases: with internal curing and without it. This might be explained based on the fact that as W/C increases, the pore structure of the cement paste becomes more interconnected, as shown by the chloride ion permeability results below. This increase in permeability suggests an increase in the water lost by drying with the subsequent reduction on the degree of hydration.

A previous study [12] measured a degree of hydration of 70.2% at 90 days of age in mixtures with Type I Portland cement at W/C of 0.4 when cured at 20 °C and 100% relative humidity with seal curing. The values in Table 3 show that mixtures with W/C of 0.4 without internal curing reached a degree of hydration of only 47% when stored at 50% relative humidity and without sealing. This suggests that the poor curing conditions used in this investigation might have decrease degree of hydration from 70% to 47% (23% drop in degree of hydration) when no internal curing is applied. However, mixtures with internal curing reached a degree of hydration of 62% suggesting that internal curing can reduce the negative effects of the external drying (only 8% drop in degree of hydration).

This implies that internal curing allows for protecting/assisting hydration at 90 days even under drying conditions. Internal curing proves to be a powerful complement to traditional curing, especially when the later is not well applied.

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Fig. 2. Effective W/C based on the degree of hydration versus initial W/C of the mixture.

Since some of the initial water content is lost to the environment during the 90 days drying of the specimens, the effective W/C on each of the samples can be estimated in order to quantify the impact of drying on the mixtures with internal curing and those without internal curing.

In order to estimate the actual water used for hydration on each of the concrete mixtures, the initial porosity and the effective W/C was calculated based on the model developed by Powers [13].

$$p = \frac{W/C}{W/C + \rho_w/\rho_c} \tag{1}$$

$$\alpha_{\max} = \frac{p}{1.1(1-p)} \tag{2}$$

where *p* is the initial porosity, W/C is water/binder ratio, ρ_w is water density, ρ_c is cement density, and α_{max} is the maximum degree of hydration

By assuming that the maximum attainable degree of hydration has been reached after 90 days of age when stored at 50% of relative humidity, the model above can be used with the experimental degree of hydration shown in Table 3. An "experimental initial porosity" can be estimated using the experimental degree of hydration and Eq. (2). Then, the effective W/C can be calculated by using the experimental initial porosity and Eq. (1).

Fig. 2 shows the initial W/C versus the effective W/C required to reach the degree of hydration obtained from LOI if there were no water exchanges with the environment (sealed curing). It can be noted that for every W/C, mixtures with internal curing had a higher effective W/C than its counterpart without internal curing. That is, mixtures with internal curing in drying conditions for 90 days performed similarly to sealed mixtures of W/C between 0.22 and 0.25 while mixtures without internal curing in drying conditions for 90 days performed similarly to sealed mixtures of W/C between 0.17 and 0.21.

Fig. 3 shows the amount of water effectively lost by each mixture calculated as the difference between the initial amount of free water (see Table 2) and the effective water (calculated as the effective W/C multiplied by the amount of cement).

Fig. 3 suggests that regardless of the initial W/C, mixtures with internal curing showed less water effectively lost than those without internal curing; on average, mixtures with internal curing had

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19.8 kg less of water effectively lost than their counterparts. This suggests that even though both types of mixtures were stored at 50% relative humidity, the cement paste of those with internal curing was less affected by drying than the cement paste of the mixtures without internal curing. This lower effect of drying in mixtures with internal curing is believed to be caused by the presence of the internally stored water within the lightweight aggregate. As cement paste lost water to the environment, "new" water migrated from the lightweight aggregate to the paste to assist hydration.

Fig. 3 also suggests that the amount of water effectively lost increases as the initial W/C increases. That is, mixtures with higher initial porosity (higher initial W/C) show higher amount of water effectively lost suggesting that they are more severely affected by drying.

4.2. Compressive strength

Fig. 4 presents the average compressive strength at 90 days of age for the 10 mixtures under investigation and its variability.

As shown in Fig. 4, mixtures with internal curing exhibit higher compressive strength compared with those without internal curing, for the same W/C.

The largest increase in compressive strength – 18 MPa – was obtained for mixtures with W/C of 0.45 while the lowest one – 6 MPa – was obtained for mixtures with W/C of 0.40. On average mixtures with internal curing showed a compressive strength 19% higher than those without it.

Since lightweight aggregate intrinsic strength is lower than that of normal weight aggregate, a decrease in concrete strength when using lightweight aggregate can be expected (this is known as ceiling strength [14]). Nevertheless, mixtures with internal curing (with lightweight aggregate) not only did not show a decrease in strength but also an increase. It can be concluded that the strength gained due to a higher degree of hydration is more important than the eventual decrease in strength caused by the use of a lower intrinsic strength lightweight aggregate as curing agent. It should be noted that all mixtures had the same amount of lightweight aggregate (amount of internal curing water remained constant for all W/C), then reduction in compressive strength due to the use of lightweight aggregate should be similar in all of them.

It is interesting to note that compressive strength of mixtures without internal curing did not vary importantly for W/C of 0.425 and above. This might be related to the drying conditions that produced important water losses and limited the attainable degree of hydration. This suggests that under poor curing conditions a reduction in W/C does not necessarily provide higher strength. This was also true for mixtures with internal curing meaning that this curing method should not replace external/traditional curing, but it should be a complement of it.

4.3. Chloride ion permeability

Fig. 5 presents the average chloride ion permeability at 90 days of age for the 10 mixtures under investigation and its variability.

As shown in Fig. 5, mixtures with internal curing exhibit lower permeability compared with those without internal curing, for the same W/C.

The largest decrease in chloride ion permeability – 1717 C – was obtained for mixtures with W/C of 0.50 while the smallest decrease in chloride ion permeability – 97 C – was obtained for mixtures with W/C of 0.40. On average mixtures with internal curing showed chloride ion permeability 30% lower than those without it. These results were expected since the mixtures with internal

curing showed a higher degree of hydration meaning that they

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Table 4Statistical comparison of concrete properties.

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Data	IC ^a	NIC ^b	IC	NIC	IC	NIC	
Test	Compressive	strength	Permeability		Degree of hydration		
Average	63 MPa	54 MPa	2076 C	2953 C	67%	51%	
Standard deviation	3.426		301.38		0.025		
Degrees of freedom	28		18		36		
t-Value	2.70		2.91		6.25		
Significance (a)	1.20%		1.00%		0.00010%		
$T_{(1-\alpha)}$	2.69		2.88		5.88		
Hypothesis (H_0)	$IC_{strenght} = NIC_{strength}$		$IC_{permeability} = NIC_{permeability}$		$IC_{hydration} = NIC_{hydration}$		
Rejection interval	t -Value $\geq t_{(1-1)}$	-α)	t -Value $\geq t_{(1-\alpha)}$:)	t -Value $\ge t_{(1)}$	t -Value $\geq t_{(1-\alpha)}$	

^a IC: Internal curing applied.

^b NIC: No internal curing applied.

have achieved a more compact and less permeable cement paste microstructure.

It should be considered that the decrease in chloride ion permeability was obtained in despite of the fact that lightweight aggregate was used as internal curing agent. The use of a more porous aggregate could have increased permeability. However, as concluded in previous studies [6,15], in this case concrete permeability is controlled by cement paste permeability rather than the aggregate permeability.

4.4. Statistical comparison of concrete properties and discussion of results

It is well known that a higher degree of hydration in cementitious materials increases strength and decreases permeability, which are probably the two most commonly sought properties in a high performance concrete.

The results of tests conducted in the different mixtures with internal curing showed precisely an increase in strength and a decrease in permeability in comparison with those mixtures without it.

The experimental design allowed performing statistical comparisons to know whether internal curing brings significant benefits in increasing the degree of hydration. Experimental design was analyzed through *t*-student test to evaluate the performance of internal curing on: hydration, compressive strength and chloride ion permeability as shown in Table 4. The results of *t*-student test showed that internal curing:

- Significantly increased cement degree of hydration with 99.9% of confidence level.
- Significantly increased compressive strength with 98.8% of confidence level.
- Significantly decreased chloride ion permeability of concrete with 99.0% of confidence level.

Mixtures with internal curing did not only present higher degree of hydration in comparison with those without it, but also presented lower variability as shown by the lower coefficient of variation (see Table 3). Reduction in variability in mixtures with internal curing was also observed in compressive strength and chloride ion permeability. That is, coefficients of variation of mixtures with internal curing were 63% and 55% of that of mixtures without internal curing for compressive strength and chloride ion permeability, respectively.

5. Conclusions

The increased competitiveness in the construction industry requires the production of a higher performance concrete, with higher strength and durability. These characteristics do not depend only on the constituents and proportioning of the mixture but also on curing. However, current construction speed requirements are generally against the implementation of a proper concrete curing.

This research aims to apply internal curing, a technology widely investigated in the last decade with low W/C concrete mixtures, as

a complement to traditional curing on site for concrete mixtures with W/C higher than 0.42.

Mixtures with and without internal curing were kept at 50% of relative humidity in order to represent poor curing conditions. From the results obtained herein, it can be concluded that the mixtures with internal curing showed less water effectively lost than the mixtures without it, That is, internal curing was able to maintain better curing conditions for the cement paste even under drying conditions.

Note that properties of concrete are strongly affected by a poor curing, e.g., the degree of hydration in mixtures without internal curing was generally below 55% and none of them were above 60%. This had a direct impact on compressive strength, chloride ion permeability, and other properties.

The use of internal curing allowed a 15% increase in degree of hydration under the drying conditions considered herein. This increase in hydration is believed to be the cause of the 19% increase in compressive strength observed in mixtures with internal curing with respect to those without it. Also a 30% decrease in chloride ion permeability was observed when using internal curing.

It is concluded that the application of internal curing brings benefits when it is applied on tradition concrete as a complement to traditional curing methods.

Internal curing reported benefits regardless of the W/C considered. That is, mixtures with high W/C under poor curing conditions, as those considered herein, tend to lose water and reach a limited degree of hydration. Thus, internal curing is helpful regardless the initial water content in the mixture.

This investigation considered the same amount on internal curing water regardless the W/C of the mixture. Future investigations should increase internal curing water as W/C increases, so the water loss in somehow counteracted.

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