Internally Cured High Performance Concrete with Magnesium based Expansive Agent using Coal Bottom Ash Particles as Water Reservoirs

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Abstract

Shrinkage is one of the main concerns related to high performance concrete (HPC) durability. Its high density paste, consequence of a low water to binder ratio, can be unprofitable if cracks appear due to excessive tension when volume changes are restrained. Therefore, volume stability is a priority. In this work, three different strategies have been studied with that purpose: integration of fly ash as a low reactive supplementary cementitious material, internal curing via coal bottom ash particles as water reservoirs and the use of an expansive agent based on magnesium oxide (MEA). Many research works address the three shrinkage reduction strategies individually. However, studies regarding their simultaneous use are not prevalent so this work proposes its combined application. Results indicate that internal curing and MEA have a synergistic effect in HPC. Internal curing enhances MEA expansion due to the lack of water in this kind of concrete, contributing to autogenous shrinkage compensation. When concrete is affected by air-drying conditions, the use of MEA, internal curing, or both together make shrinkage to increase. MEA effectively expands in wet cured HPC although internal curing is not effective in this condition due to the absence of self-desiccation and limited porous aggregate water desorption. Taking into account the effects of each shrinkage reduction strategy and curing condition, it has been concluded that the use of fly ash as supplementary cementitious material, internal curing and MEA is recommended together with prevention of water evaporation from HPC surface.

Keywords: Fly Ash; Magnesium based Expansive Agent; Coal Bottom Ash; Internal Curing; Autogenous shrinkage; Drying shrinkage; Swelling.

Highlights:

- Three shrinkage reduction strategies were used in high performance concrete (HPC)
- Internal curing water can contribute to magnesium expansive agent (MEA) hydration
- MEA makes shrinkage to increase in HPC affected by air-drying curing conditions
- MEA hydration product, magnesium hydroxide, carbonates on concrete surface

1. Introduction and objectives

Volume stability is a remarkable issue that must be taken into account when designing concrete for structural and non-structural applications. Different phenomena can make concrete to suffer deformations, generating internal stress if they are constrained. This tension can exceed tensile strength and produce cracks letting potentially harmful agents to ingress concrete. Therefore, concrete shrinkage can lead to important durability problems [1].

Concrete deformations can be positive (volume increase, i.e. swelling or expansion) or negative (volume reduction, i.e. shrinkage) and some of them can be absent depending on concrete composition and surrounding environmental conditions. Concrete external volume changes are known as autogenous deformations when they are intrinsic to material's composition, no matter surrounding conditions. These kind of deformations can increase (autogenous swelling) or reduce (autogenous shrinkage) concrete volume as a consequence of different physical and chemical processes.

Cement hydration provokes an external volume reduction before setting, and it is the driving force of autogenous shrinkage after setting. Concrete internal relative humidity decreases due to cement hydration and other supplementary cementitious materials reactions that consume water, such as pozzolanic reactions [2]. This progressive process is named as self-desiccation and, when intense enough, it is the cause of autogenous shrinkage [3–5]. Therefore, self-desiccation is a special concern in concrete with a low water to cement ratio, i.e. high performance concrete (HPC) [3,6,7]. Cement hydration rate becomes lower as time goes by. Therefore, autogenous shrinkage is more remarkable during the first days after concrete mixing. However, an opposite deformation can compensate self-desiccation shrinkage at early ages. That is autogenous swelling and can be caused by different phenomena such as absorption of bleeding water, water adsorption by fillers, CH growth and primary ettringite formation [8,9]. Also, some recent approaches to the effect of cement hydration on a porous-system scale state that C-S-H formation decisively contributes to swelling as long as a sufficient relative humidity is maintained inside concrete [10,11]. Sometimes, autogenous swelling cannot be observed as it coexists with autogenous shrinkage in a minor magnitude, resulting an overall negative deformation [12]. That is one of the possible reasons for

it to be less studied [13] although it plays an important role in shrinkage compensation and cracking prevention at early ages [14].

Cement hydration is also the direct cause of thermal deformations, as it is an exothermic reaction. Heat of hydration makes concrete temperature to rise to a peak and then decrease, causing associated expansion and shrinkage. The magnitude of this temperature change depends on the rate of heat dissipation into the surrounding environment. This is why thermal deformations should not be considered autogenous.

When interaction of concrete with the environment is allowed, the moisture gradient forces unbound water to evaporate. Then, water migration from the capillary pores to the outside develops pressure on them and a volume reduction occurs. This mechanism is analogous to the one that governs self-desiccation [15]. High water to cement ratios and low environmental humidity increase drying shrinkage [16,17]. Drying shrinkage magnitude is related to specimens shape and size, as water release is not homogeneous across specimen's cross section [14,18–20].

On the opposite direction, from environment to concrete, there is another element that migrates: CO_2. Carbon dioxide is naturally present on air and can react with calcium based concrete compounds, mainly calcium hydroxide formed as a product of Portland cement hydration. This carbonation implies a volume reduction known as carbonation shrinkage, which is not completely understood yet [21].

All previously mentioned deformations can coexist in laboratory samples and construction elements, depending on their size, shape and surrounding environment. Different strategies can be used in order to mitigate them [2]: low reactive supplementary cementitious materials [22–25], internal restraint by fibre reinforcement [26–28], internal curing [29–32], shrinkage reducing additives (SRA) [33–35] and expansive agents [36–38] are among the most popular [39].

Some of these shrinkage reduction strategies are gathered in **Fig. 1**.

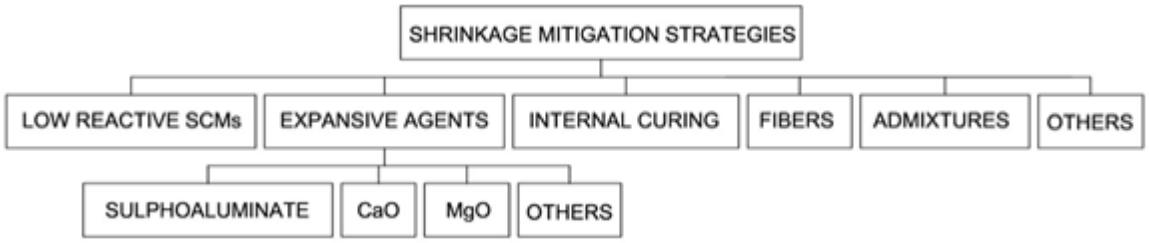


Fig. 1: Shrinkage reduction strategies.

Regarding expansive agents, the most known are based on calcium sulphoaluminate (C-$\overline{S}$-A) [38,40,41], calcium oxide (CaO) [42–49] and magnesium oxide (MgO). These expansive agents hydrate resulting in new compounds with a higher volume. The calcium sulphoaluminate can give rise to ettringite or monosulfate, with respective volumes of 9.3 and 4.1 times the initial C-$\overline{S}$-A particle [50]. Calcium oxide gives rise to calcium hydroxide, with a volume of 1.9 times CaO [50]. Finally, magnesium oxide gives rise to magnesium hydroxide, with a volume of 2.2 times MgO [51]. However, hydration is not the only important factor regarding the expansive process. Formation of surrounding hydrates subjected to the compressive forces produced by the expansive ingredients plays also an important role [50]. The effect of the use of expansive agents on compressive strength and tightness is strongly related to curing conditions and especially with restraining extent. Some studies conclude that restrained concrete maintains the values for these properties when including expansive agents into the mix, while some "loosening" can happen when no restrictions are applied, causing small decreases of compressive strength and tightness [50]. This must be taken into account when studying expansive agents' performance in small non-restrained laboratory specimens, as results can differ for reinforced structural elements.

Magnesium oxide is present in Portland cement as dead burnt periclase, coming mostly from impurities of magnesite in calcite used as raw material for its manufacturing. In this case, magnesite is submitted to a temperature over 1400 ºC, producing low reactive a kind of magnesia that is only able to expand after long periods of time. This can cause cracks and associated durability problems [51]. However, magnesium oxide can be obtained from less intense burning (lightly burnt MgO), which is able to expand at relatively early ages. **Eq. 4** represents the involved chemical reaction in MgO manufacturing process:

$$MgCO_3 \rightarrow MgO + CO_2 \quad \textbf{(1)}$$

112 Lightly burnt MgO has been proved to be convenient for compensating autogenous shrinkage [52–54],
113 thermal shrinkage in laboratory [55,56] and field conditions [57], and it even shows self-healing capacity
114 [52,58,59]. The expansive mechanism involves hydration of MgO, producing magnesium hydroxide (also
115 known as brucite), with a much higher molar solid volume (117 % bigger than MgO) [51]. **Eq. 5**
116 represents this chemical reaction. Kinetics of MgO dissolution are extendedly explained in [60].

$$MgO + H_2O \rightarrow Mg(OH)_2 \quad \textbf{(2)}$$

117 Research about lightly burnt MgO started when it was reported that no cracks were formed due to thermal
118 shrinkage in Baishan concrete arch gravity dam in China. This fact was attributed to the use of a Portland
119 Cement with high content of magnesia burnt at a lower than usual temperature [61]. Since then,
120 intentionally lightly burnt MgO has been used to fabricate expansive agents known as magnesium based
121 expansive agents (MEA). Most common MEAs have a high content of magnesium oxide, as they are
122 obtained from pure magnesite extracted in quarries. Less effective but more sustainable and cheap MEA,
123 with less content of MgO, can be obtained from impure magnesite or industrial wastes too [62–64].

124 When comparing MEA with calcium and ettringite based expansive agents, the main advantages are its
125 relatively low need of water to react and the stability of the reaction product, magnesium hydroxide [53].
126 Positive effects of MEA on other durability properties more than shrinkage have been reported [65,66]
127 and also on mechanical properties under restrain condition [67].

128 Supplementary cementitious materials have a remarkable influence on autogenous and thermal stress,
129 tending to reduce those parameters when they are low reactive. For instance, class F fly ash is frequently
130 included in concrete with MEA [37,53,55–57,59,65,66,68,69], working both elements in favour of
131 volume stability. Blended cements containing MEA, especially with class F fly ash, have been widely
132 studied and used for construction of massive dams in China [70].

133 An alternative strategy that can make autogenous shrinkage to decrease is internal curing. This consists in
134 the supply of water to a cementitious mixture using pre-wetted lightweight aggregate, or other materials
135 that readily release water from within particles, thereby mitigating self-desiccation and sustaining
136 hydration [71]. These pre-wetted particles are also known as water reservoirs. Lightweight aggregates and

superabsorbent polymers are the most common elements used as water reservoirs [30]. Internal curing is especially useful in high performance concrete, with a very low water to binder ratio. Water in HPC is hardly available for cement hydration, and additional amounts cannot be effectively provided by external curing due to paste low permeability [31]. The use of MEA does not seem a good option for this kind of concrete, as it needs a certain amount of water to react. However, the simultaneous utilization of internal curing and MEA could be a promising combination, as the former could provide the latter with the necessary water to expand. Only a few studies have explored this option [36].

2. Research significance

Worries about HPC early age volume stability have motivated the publication of many papers about shrinkage reduction strategies. However, the most of these research works address each strategy individually. Studies about their combined effect are not prevalent, although this perspective is also interesting because of potential synergistic effects. In addition, the proposal of more than one strategy in order to avoid any concrete dysfunction is usually convenient as they all can present some drawbacks (not only from a technical perspective, but also economical, ecological…). This is the case of the three shrinkage reduction strategies proposed in this study: the use of fly ash as low reactive supplementary cementitious material, internal curing via saturated coal bottom ash as water reservoirs and a magnesium based expansive agent. Synergistic effects have been found and negative consequences of the use of each of them can be minimized when reducing the amount of each of them in favour of a multi-constituent approach. Moreover, three laboratory curing conditions have been stablished in this study with the aim of getting information about the performance of the materials under study on a real field situation. The authors conclude that HPC with high volume of fly ash, MEA and internal curing is volumetrically more stable than conventional HPC and shows an acceptable strength.

3. Materials characterization and mix design

All materials have a particle size below 4 mm. Therefore, the object of the experimental programme is the mortar phase of a hypothetical HPC. Designed mixes do not contain coarse aggregates but only fine aggregates and paste constituents.

Coal bottom ash particles (CBA) have been used as water reservoirs for internal curing. CBA is a porous granular waste generated in coal power stations. As internal curing affects a wider volume of paste when using small size water reservoirs [72], CBA particles were sieved to reach a 4 mm maximum size. Some photographs of different size CBA fractions are shown in **Fig. 2**.

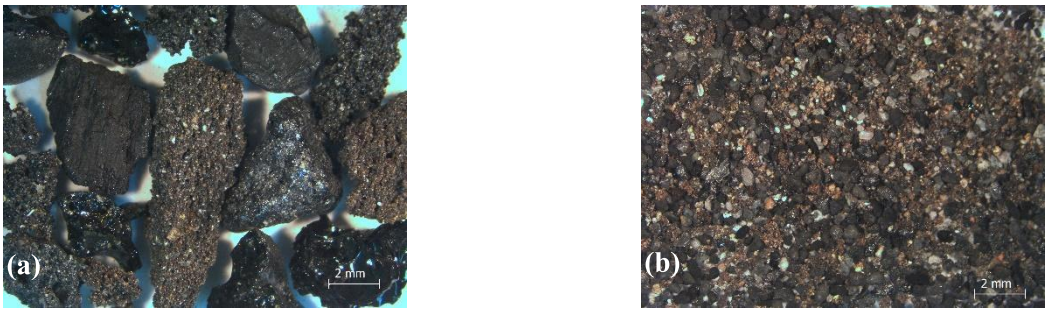


Fig. 2: CBA with a particle size between 2 and 4 mm **(a)**. CBA with a particle size between 0.125 and 0.5 mm **(b)**

Sieved CBA has a dry-state density of 1.19 g/cm^3 and a 24 h water absorption of 36.66 %. This element partially replaces conventional sand (S) in designed mortars, which is crushed sand with a maximum size of 4 mm, a dry-state density of 2.47 g/cm^3 and a water absorption of 2.43 % after 24 h. Both granular materials present a similar particle size distribution and fineness modulus, being 2.33 for CBA and 2.78 for conventional sand.

Ordinary Portland cement (OPC) with a density of 3.12 g/cm^3 and Class F fly ash (FA) with a density of 2.21 g/cm^3 have been used as the main powder paste constituents. A magnesium based expansive agent (MEA) has been used in this study. It was obtained by calcination of pure magnesite (see composition in **Table 1**) with a maximum particle size of 100 µm at 900 ºC during 2 h (light burning) as it was done in previous works by Sherir et al. [59]. Other authors have also deeply studied the influence of different parameters of the calcining process over MEA performance [69,73]. Small samples of 2.7 kg, divided into three different containers, were burnt at a time to guarantee a homogeneous calcination (up to an average lost on ignition of 34 %). The resulting powder compound is basically magnesium oxide (MgO), with a purity of 90 % and a density of 3.58 g/cm^3.

Table 1: Magnesium carbonate chemical composition

CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	LOI
0.46 %	4.17 %	0.52 %	0.49 %	46.72 %	47.21 %”

After burning, containers were carefully taken out of the kiln and MEA was spread on aluminium trays to ensure a quick temperature drop of the material. After a cooling time of 15 min, MEA was kept in glass jars inside a sealed box containing soda lime and silica gel, which are granular materials with a high water vapour absorption capacity. The cooling procedure is very important as magnesium oxide is an unstable compound that can easily react with environmental humidity. Some stages of this process can be seen in **Fig. 3**.

Fig. 3: MEA obtaining process: taking out from the kiln, pouring and spreading on trays and packing.

A high range water reducing admixture (HRWRA) with a 1.05±0,02 g/cm^3 density and solid residue of 20.3±1 % has been used in order to enhance fluidity of HPC mortar phase.

Taking into account the shown characteristics of each material involved in the study, twelve different HPC mortar phase mixes were designed. The next criteria were considered to stablish the mix design:

- One half of the mixes contain CBA as internal curing water reservoirs. This material is introduced in the mortar substituting the 30 % of conventional sand (S), by volume.
- Two different binders are used: Ordinary Portland cement (OPC) and blended cement (BC). BC is obtained by mixing a 40 % of OPC with a 60 % of FA, proportioned on a volume basis.
- Two different MEA contents are stablished: 3 % and 5 %. MEA replaces OPC when no FA is in binder, while it substitutes FA in BC mixes. This substitution is carried out by volume in both cases.

Mix proportions in relation with mortars nomenclature are described in **Table 2**.

Table 2: Mortar mixes

NOTATION		GRANULAR MATERIALS			POWDER MATERIALS				
		Ø / IC	S	CBA	CM/BCM	C	FA	Ø / MEA3 / MEA5	MEA
CM SERIES	CM-MEA0		100 %	0 %	CM	100 %	0 %	MEA0	0 %
	CM-MEA3				CM	97 %		MEA3	3 %
	CM-MEA5				CM	95 %		MEA5	5 %
	ICCM-MEA0	IC	70 %	30 %	CM	100 %		MEA0	0 %
	ICCM-MEA3	IC			CM	97 %		MEA3	3 %
	ICCM-MEA5	IC			CM	95 %		MEA5	5 %
BCM SERIES	BCM-MEA0		100 %	0 %	BCM	40 %	60 %	MEA0	0 %
	BCM-MEA3				BCM		57 %	MEA3	3 %
	BCM-MEA5				BCM		55 %	MEA5	5 %
	ICBCM-MEA0	IC	70 %	30 %	BCM		60 %	MEA0	0 %
	ICBCM-MEA3	IC			BCM		57 %	MEA3	3 %
	ICBCM-MEA5	IC			BCM		55 %	MEA5	5 %

Conventional sand and coal bottom ash were soaked in water during 24 h prior to mixing, with an amount of water equivalent to their 24 h water absorption capacity. Water inside these particles performs as an internal curing agent, so it is referred as internal curing water in conventional sand (ICW-S) and internal curing water in coal bottom ash (ICW-CBA). It must be noted that the amount of ICW-S is much lower than ICW-CBA, as coal bottom ash particles have a much higher water absorption. Therefore, only mortars with CBA are referred as internally cured mortars (ICCM).

Mixing water (W) to powder materials ratio in terms of volume remains constant for all mixes. Note that mixing water does not include internal curing water referred in previous paragraph. HRWRA dosage is 0.80 % in terms of solid residue/OPC, by volume. Note this dosage is not referred to the sum of all powder materials but only to OPC. HRWRA is dosed as to get a fluid mortar with self-compacting behaviour. Mix proportions of resulting mixtures are described in Table 3.

Table 3: Mix proportions [kg/m^3].

		OPC	FA	MEA	S	ICW-S	CBA	ICW-CBA	W	HRWRA
CM SERIES	CM-MEA0	923	0	0	1112	27	0	0	252	15
	CM-MEA3	896	0	32	1112	27	0	0	252	15
	CM-MEA5	878	0	53	1112	27	0	0	252	15
	ICCM-MEA0	923	0	0	778	19	161	59	252	15
	ICCM-MEA3	896	0	32	778	19	161	59	252	15
	ICCM-MEA5	878	0	53	778	19	161	59	252	15
BCM SERIES	BCM-MEA0	370	393	0	1112	27	0	0	252	6
	BCM-MEA3	370	373	32	1112	27	0	0	252	6
	BCM-MEA5	370	360	53	1112	27	0	0	252	6
	ICBCM-MEA0	370	393	0	1112	19	161	59	252	6
	ICBCM-MEA3	370	373	32	1112	19	161	59	252	6
	ICBCM-MEA5	370	360	53	1112	19	161	59	252	6

4. Test methods

Including MEA in concrete as a separate powder (not previously mixed with other elements in other processes like, for example, cement grinding) demands a longer than usual mixing time [53,61]. Concrete homogeneity when using an expansive agent is vital as irregular expansions may cause differential tensions in paste and consequent damage [68]. Therefore, a 5.5 minutes mixing procedure was chosen, which is considered to be enough for MEA proper dispersion. A planetary 20 litres capacity pan mixer was used.

Length and mass change were measured using specimens with dimensions 25x25x285 mm^3 (gauge length of 250 mm), according to UNE 80112 [74]. Specimens were exposed to three different curing conditions: sealed with aluminium foil (**Fig. 4**), air-drying in an 50 % RH environment (**Fig. 5**) and water immersion (**Fig. 6**). Temperature was kept constant in all curing environments at 24±2 ºC. Length and mass changes were measured from 18 h after casting, when specimens were demoulded. An earlier than conventional demoulding age (specimens for shrinkage test are commonly demoulded 24 h after casting) was chosen to start measurements at a time as close as possible to final setting. Length change at different ages was divided by initial length, obtaining strain (με), while mass changes were divided by specimens' surface in order to get a unitary mass change (g/m^2). Mass results normalization by area is chosen as a criterion based on EN 16322 [75].

Fig. 4: Sealed specimens

Fig. 5: Specimens in air-drying conditions

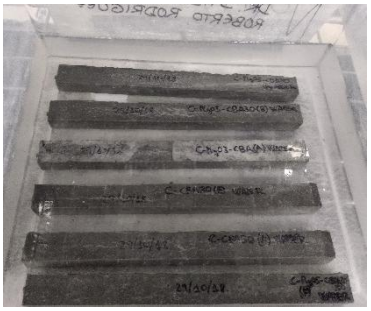


Fig. 6: Specimens immersed in water

Compressive strength was measured on sealed cubic samples with 50 mm side at an early age, 7 days, and long term, 244 days. This way, information about strength development over time can be obtained.

5. Results and discussion

5.1 Sealed condition: compressive strength and length change

Long term compressive strength of all mixes with OPC as the only binder reach 65 MPa, and mixes with ended cement surpass 40 MPa (**Fig. 7**). These results are considered to be acceptable for a mortar phase HPC.

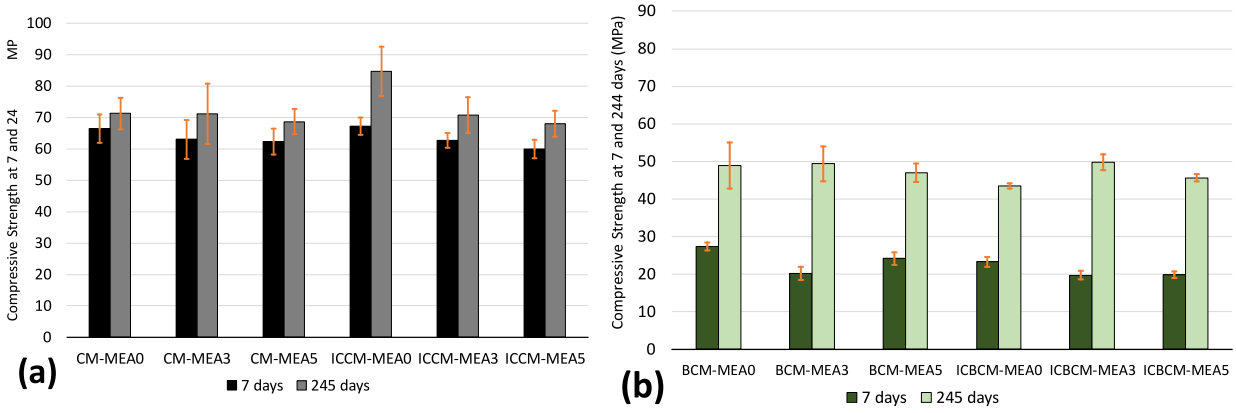


Fig. 7: Compressive Strength of CM **(a)** and BCM **(b)** series at 7 and 244 days [MPa]

The use of MEA slightly reduces strength of CM mixes because, in these mixes, MEA replaces part of Portland cement. When blended cement is used, the effect of MEA on strength is not so clear as cement content remains constant and not all fly ash is expected to have been reacted even at 245 days. Strength index of mixes with CBA, considering mixes without CBA as reference, slightly varies around 1, being

246 0.89 the minimum (ICBCM-MEA0) and 1.19 the maximum (ICCM-MEA0). Therefore, the effect of the
247 alternative aggregate on compressive strength can be neglected. As expected, mixes with fly ash gain
248 strength slowly, reaching about a 50 % of their maximum at 7 days. On the other hand, CM mortars
249 strengthen faster, achieving the 90 % of their maximum at the same early age.

250 In sealed condition, control mixes without internal curing or MEA show the highest deformation of their
251 respective series at all times, as can be seen in **Fig. 8**.

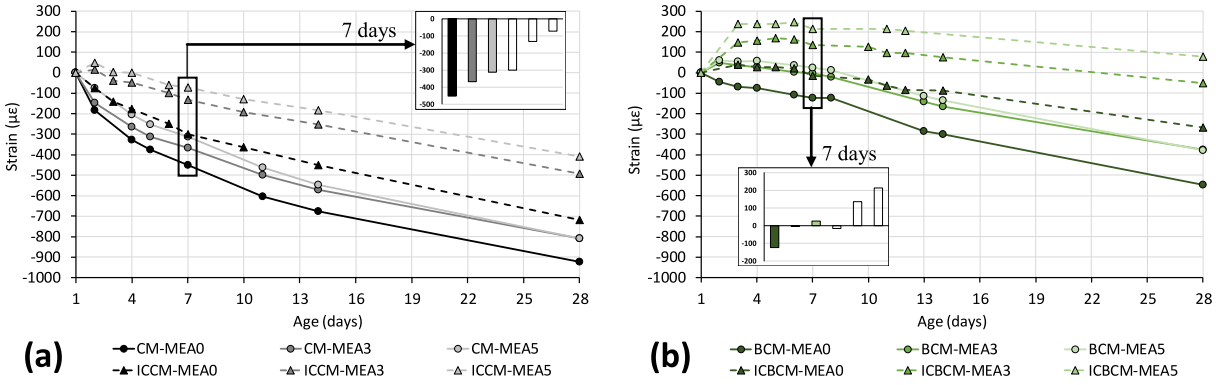


Fig. 8: CM **(a)** and BCM **(b)** strain in sealed conditions [με].

252 For some of the mixes, it has been recorded an initial positive deformation which is possibly caused by
253 swelling due to internal bleeding, i.e. aggregates water desorption, and MEA expansion. Blended cement
254 internally cured mixes with MEA show the greatest positive deformation (**Table 4**). The lower hydration
255 activity in BCM mixes consumes less water at early ages, reducing paste self-desiccation and leaving
256 more water available for MEA hydration. Saturated CBA desorb water (internal bleeding) causing
257 swelling and enhancing MEA expansion.

Table 4: Swelling peak time (days) and strain (με).

	CM-MEA0	CM-MEA3	CM-MEA5	ICCM-MEA0	ICCM-MEA3	ICCM-MEA5
Time	-	-	-	-	2	2
Value	-	-	-	-	16	18
	BCM-MEA0	BCM-MEA3	BCM-MEA5	ICBCM-MEA0	ICBCM-MEA3	ICBCM-MEA5
Time	-	2	2	3	5	6
Value	-	48	62	40	168	246

258

After the swelling peak, shrinkage starts. In sealed curing condition, with no moisture exchange with the environment, recorded shrinkage is autogenous shrinkage. In **Fig. 9**, Swelling has been removed from the total recorded deformations in order to only analyse autogenous shrinkage. Over time, all mixes maintain their relative position from the one with the highest shrinkage to the one with the lowest.

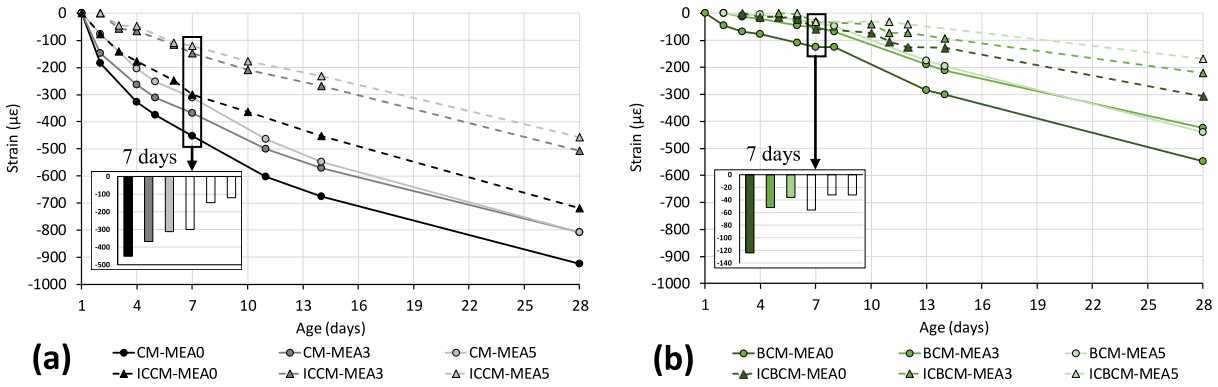


Fig. 9: CM **(a)** and BCM **(b)** autogenous shrinkage [με].

Autogenous shrinkage is higher for CM specimens than for their BCM counterparts, as less cement hydration leads to less self-desiccation. Autogenous shrinkage is lower when internal curing is applied, as it mitigates paste self-desiccation. The positive effect of internal curing is more remarkable during the first days and remains over time.

MEA expansion partially compensates autogenous shrinkage for both CM and BCM series. This effect, however, is only visible during the first days after demoulding if internal curing is not applied. For that case, there is no proportional improvement in late autogenous shrinkage (28 days) when increasing the amount of MEA from 3 to 5 %. CM-MEA3 shows barely the same autogenous shrinkage at 28 days than CM-MEA5, and there is no significant difference between BCM-MEA3 and BCM-MEA5 either. HPC, produced with a small amount of mixing water, seems not to be able to provide enough water for MEA to expand. Some extra water is needed and internal curing can provide it. Proof of this are the improvements of ICCM-MEA5 over ICCM-MEA3 and of ICBCM-MEA5 over ICBCM-MEA3 (84 and 130 με at 28 days, respectively). Available water in non-internally cured mixes is not enough even for hydrating all MEA in mixes with a 3 % content. This is stated after having registered a 228 με compensation at 28 days of ICCM-MEA3 over ICCM-MEA0, and a quite inferior, 116 με, of CM-MEA3 over CM. Similar

behaviour is found for mortars with fly ash, with a 216 με compensation of ICBCM-MEA3 over ICBCM-MEA0, and an inferior one, 171 με, of BCM-MEA3 over BCM. However, this lack of water for proper MEA hydration in HPC is not detected during the first days after demoulding. At that time, the higher the MEA content the lower the shrinkage in case of CM series, and the higher the swelling in case of BCM series.

Internal curing is a shrinkage reducing strategy playing a double role in HPC with MEA. It contributes not only to autogenous shrinkage reduction but also to compensate it by providing extra water for MEA hydration. This beneficial synergistic effect has been also reported in other works [65].

5.2 Air-drying conditions: mass change and shrinkage

Mortars lose water by evaporation when submitted to air-drying conditions, which can be recorded as mass loss. Mass loss is especially remarkable during the first days after demoulding and tends to stabilize at early ages due to high surface to volume ratio of shrinkage specimens, and it even turns into mass gain after 14 days for BCM series due to carbonation. The described tendency can be observed in **Fig. 10**.

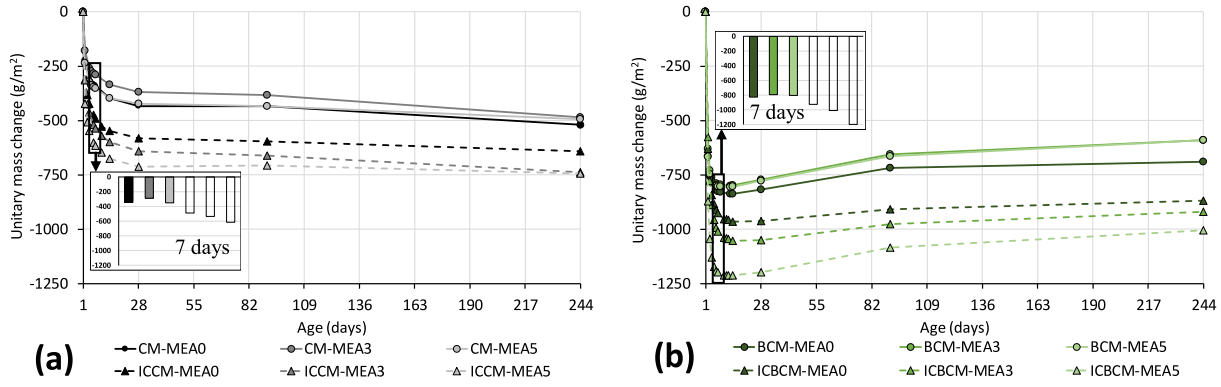


Fig. 10: CM **(a)** and BCM **(b)** mass loss in air-drying conditions (g/m^2).

The initial drastic drop is lower for CM series because they consume more water during setting, when mortar is still inside the sealed mould. Internally cured mixes have a higher mass loss as they contain a higher amount of water (ICW-CBA > ICW-S). Presence of MEA increases mass loss in internally cured specimens in a slight magnitude.

As said before, after 14 days, mass gain due to carbonation can be observed in BCM series. Carbon dioxide fixing capacity is typically lower when pozzolanic materials as fly ash are used due to portlandite consumption [76]. However, specimens under air-drying conditions have been poorly cured so pozzolanic reaction might not have taken place properly. Moreover, poorly cured BCM may develop a porous network that enhances carbon dioxide diffusion, in contrast with CM exposed to the same curing conditions. This long term tendency is not typically measurable when large specimens are used, but other studies have registered similar mass gain [5,77,78]. Total mass gain from 14 days onwards can be found in **Table 5**.

Table 5: Mass gain from 14 to 244 days age (g/m^2).

BCM-MEA0	BCM-MEA3	BCM-MEA5	ICBCM-MEA0	ICBCM-MEA3	ICBCM-MEA5
148	208	214	98	131	207

This registered mass gain is the result of the combination of CO_2 collection and water evaporation. That is the reason for internally cured mixes to show a lower mass gain. They still lose a considerable amount of water after 14 days, higher than their non-internally cured counterparts, because of their higher initial water content. This effect is also noticeable at the period between 7 and 14 days, when non-internally cured specimens already show a lower mass loss than the internally cured counterparts. For instance: BCM-MEA0 loses 11.59 g/m^2 during this time period, while ICBCM-MEA0 loses 39.84 g/m^2. The mass gain rhythm due to carbonation is constant when no significant mass loss is simultaneously occurring and drastically decreases when the carbonation depth reaches the whole specimen (with a small cross section), i.e. almost all carbonatable compounds have been carbonated. This tendency can be seen in non-internally cured specimens after 14 days, being the carbonation ending at around 91 days (**Table 6**).

Table 6: Mass gain rhythm ($g/dm^3 \cdot day$).

Time period	BCM-MEA0	BCM-MEA3	BCM-MEA5
14 to 28 days	1.59	1.77	1.85
28 to 91 days	1.56	1.88	1.82
91 to 244 days	0.18	0.43	0.48

This tendency cannot be clearly seen in internally cured mixes for the same period because they lose water for a longer time. Although mass gain is not clearly observable in CM series, a little amount of carbonation might coexist with water evaporation. Associated mass changes, positive and negative respectively, may balance each other resulting a negligible mass loss for this period. After that age, almost all carbonatable compounds are believed to be carbonated so a small mass loss due to water evaporation is registered.

The presence of MEA means an increase in carbonation mass gain (**Table 5**), as magnesium hydroxide can react with environmental carbon dioxide to form magnesium carbonate. This mass gain is also faster when MEA is used, what means it reacts with environmental CO_2 at the same time other compounds contained in fly ash and Portland cement do. For example, ICBCM-MEA0, ICBCM-MEA3 and ICBCM-MEA5 gain mass at a ratio of 0.87, 1.16 y 1.78 g/m^2·day, respectively, for the time period between 28 and 91 days.

In air-drying curing condition, the interactions of mortar with the surrounding environment cause continuous shrinkage (**Fig. 11**). Along time, all mixes maintain their relative position from the one with the highest shrinkage to the one with the lowest. Expansions due to MEA are not registered in air-drying curing conditions. What is more, mortars with MEA show a higher shrinkage.

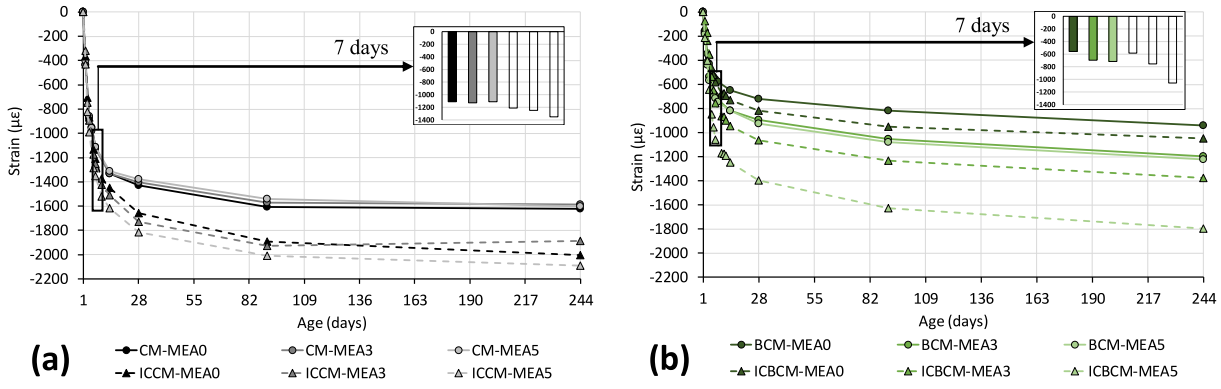
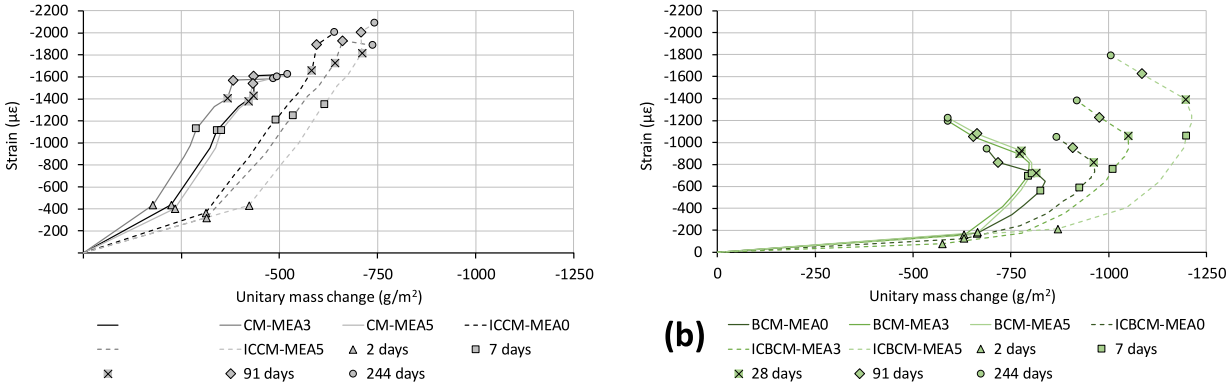


Fig. 11: CM **(a)** and BCM **(b)** shrinkage in air-drying conditions [με].

Shrinkage increase decelerates over time and CM series show higher values than BCM series. At this point, doing a quick comparison between **Fig. 10** and **Fig. 11**, it can be clearly observed that CM series lose less water but show a higher shrinkage than BCM series. This is not the usual correlation between

mass loss and shrinkage that can be found in studies about conventional concrete. In HPC and mortars with a low water to binder ratio, as it is the case of this study, paste self-desiccation coexists with drying and carbonation in air-drying curing condition. An approach about the importance of each of those phenomena on total shrinkage can be got by plotting shrinkage against unitary mass change ratio (**Fig. 12**).



and BCM **(b)** shrinkage (με) for corresponding unitary mass loss (g/m^2).

day after demoulding (from first record until the age of 2 days), for the same mass loss, er in non-internally than in the internally cured specimens for both CM and BCM series. -MEA0 has a shrinkage to mass loss ratio of 1.95 με/(g/m^2) at the age of 2 days while the internally cured counterpart has a shrinkage of 1.16 με/(g/m^2). This corroborates that self-desiccation shrinkage coexists with air-drying shrinkage at early ages, as internal curing is known to mitigate self-desiccation shrinkage.

For CM series, from 28 to 91 days, the possible coexistence of drying and carbonation makes a low mass loss to cause a relatively high shrinkage. Drying and carbonation produce mass loss and mass gain respectively, so they can compensate each other when occurring simultaneously. However, both phenomena cause negative strain, so their effects overlap in relation with length change. Self-desiccation is believed not to be causing shrinkage at this period because cement hydration rate after 28 days might be low in this air-drying curing condition. After 91 days, the tendency changes due to some residual drying in the absence of further carbonation. At this late period, drying shrinkage seems not to produce a strong shrinkage.

The overlapping of drying and carbonation shrinkage, while respective mass changes compensate each other, is a visible phenomenon in BCM series too, for the period between 7 and 28 days. Later, mass starts to increase due to predominance of carbonation over drying. Internally cured mixes show a slightly higher shrinkage to mass gain ratio after 28 days, as they suffer a higher drying.

The presence of MEA has a slight influence on shrinkage to mass ratio so the different substitution rates show a similar trend for both CM and BCM series.

5.3 Water immersion: unitary water absorption and positive deformation / swelling-expansion

When mortar specimens are immersed in water after demoulding, they continuously absorb water which can be recorded as mass gain. This mass gain is significant during the first days of immersion, following a lower rhythm at later ages (**Fig. 13**). Although HPC paste is too dense for an effective external wet curing, water can still penetrate to a certain depth in water immersion conditions. As specimens under study have a high surface to volume ratio, water is believed to access a substantial proportion of the mortar pores. Water gain over time is very similar for all mixes.

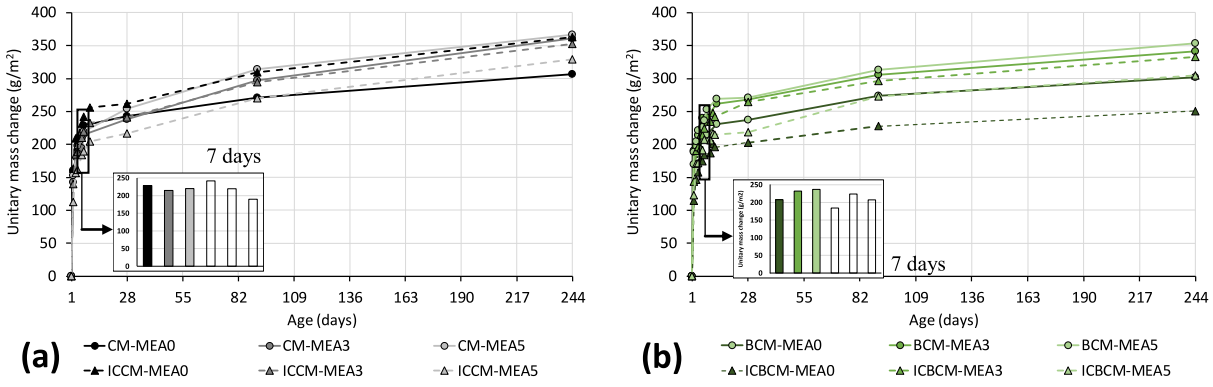


Fig. 13: CM **(a)** and BCM **(b)** mass gain (g/dm^3).

Absorbed water during immersion and interacts with the three different used binders and their reaction products. Both phenomena contribute to continuous volume increase in all specimens (**Fig. 14**). Along time, all mixes maintain their relative position from the one with the highest deformation to the one with the lowest.

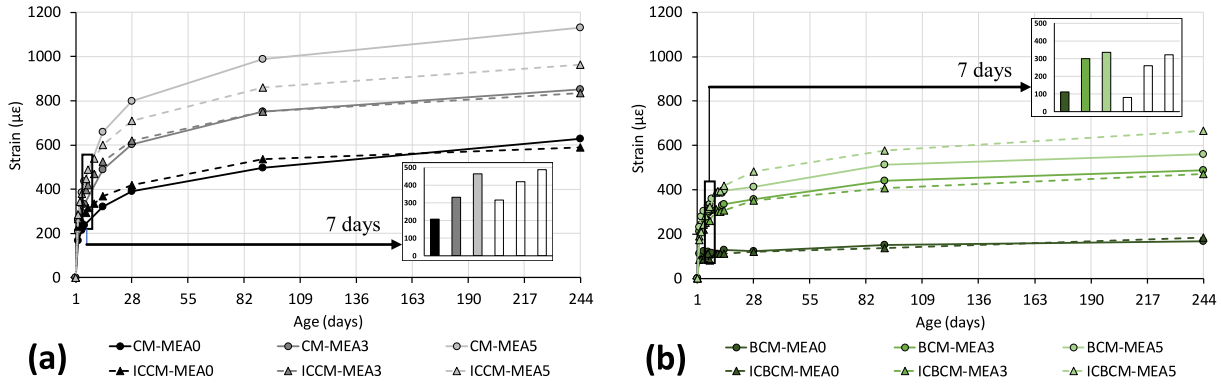


Fig. 14: CM **(a)** and BCM **(b)** swelling and expansion [με].

In general, the volume increase is more remarkable in CM than in BCM series, although their water gains are very similar. The principal reason might be the formation of C-S-H and a higher amount of primary ettringite, both cement hydration products. Ettringite is very porous (its structure is needle-like) and occupies more volume than involved reactants, so an expansion occurs when it forms [8]. Moreover, ettringite is able to adsorb big amounts of water causing associated expansion. The expansion due to C-S-H formation at a porous material scale is a recent approach proposed by [10,11]. Furthermore, negligible self-desiccation may occur in small specimens subjected to this curing condition, so no internal curing effect or MEA expansion enhancement is visible.

As expected, the more the MEA the more the expansion due to its effective hydration in this curing condition. This higher MEA expansion is observed in CM and BCM series, but it is proportionally higher in the second. For example, at 244 days, CM-MEA3 and CM-MEA5 show a deformation 1.35 and 1.80 times CM, while BCM-MEA3 and BCM-MEA5 show a deformation 2.65 and 3.04 times BCM. When MEA substitutes cement, it reduces the amount of C-S-H and ettringite and their commented associated expansion, so an expansion is replaced by another one and the difference between them is not drastic. On the other hand, when MEA substitutes fly ash, no OPC related expansion is avoided and they simply add up.

The relation between water absorption and specimens deformation follows a quadratic tendency (**Fig. 15**). The continuous expansion of MEA and OPC hydration products makes the pore network volume to decrease, letting less space for new products growth. This progressive lack of space for free expansions in

pores produce stronger tensions and associated external volume increase. This argument has been used by other authors to explain other expansive phenomena such as those related to delayed ettringite formation [79].

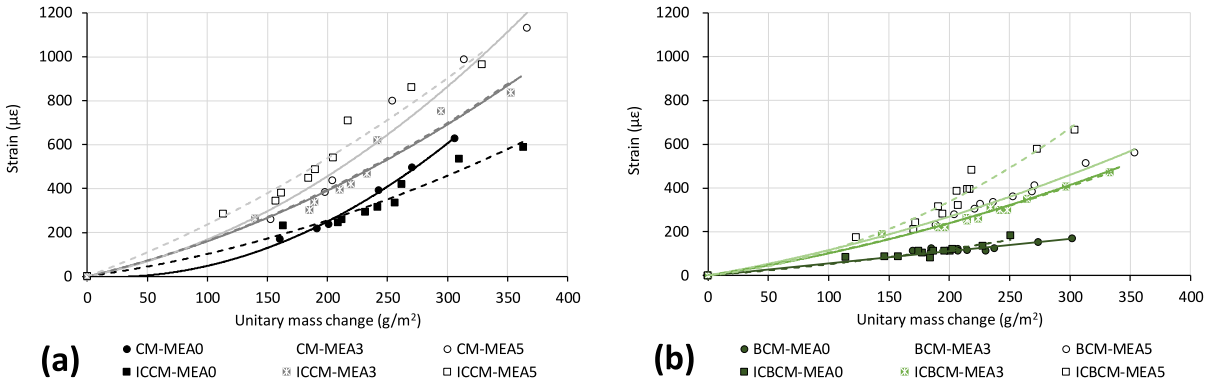


Fig. 15: CM **(a)** and BCM **(b)** deformation (με) for corresponding unitary mass gain (g/m^2).

CM series shows higher length increase for the same mass gain because the principal causes of expansion are related to cement hydration. Mixes with MEA register a higher expansion to water gain ratio. For instance: CM, CM-MEA3 and CM-MEA5 specimens increase their length by 2.05, 2.36 and 3.09 με/(g/m^2) after 244 days. As water penetrates the mortar, at the same rhythm independent on MEA content, it interacts with the different binders, hydrating MEA if present.

6. Effect of curing conditions on real applications

Three curing conditions have been used in this study: sealed, air-drying and water immersion. They have been chosen because of their correspondence with the standard UNE 80112 [74] and because of their similarity with some real field curing conditions. The correspondence between laboratory and real field curing conditions is stablished taking into account the fundamental differences between laboratory specimens and construction elements. These differences are related to size and geometry. Laboratory specimens used in this study are smaller and have a much higher surface to volume ratio than a construction element such as a standard building beam (**Table 7**).

Table 7: Comparison between the geometry of a laboratory specimen and a structural concrete element.

	Length (cm)	High (cm)	Width (cm)	Surface (cm^2)	Volume (cm^3)	Surface/Volume (cm^{-1})
Laboratory specimen*	30.0	2.5	2.5	312.5	187.5	1.67
Ordinary building beam**	360	30	20	37200	216000	0.17

*Laboratory standard specimen for length change measurement according to UNE 80112 [74].

**Ordinary building beam according to [80]

407

408 Environmental conditions affect any element from its surface. Therefore, elements with a high surface to
409 volume ratio, as it is the case of small laboratory specimens, get proportionally more affected by
410 surrounding conditions than a construction element for the same level of exposure (**Fig. 16**).

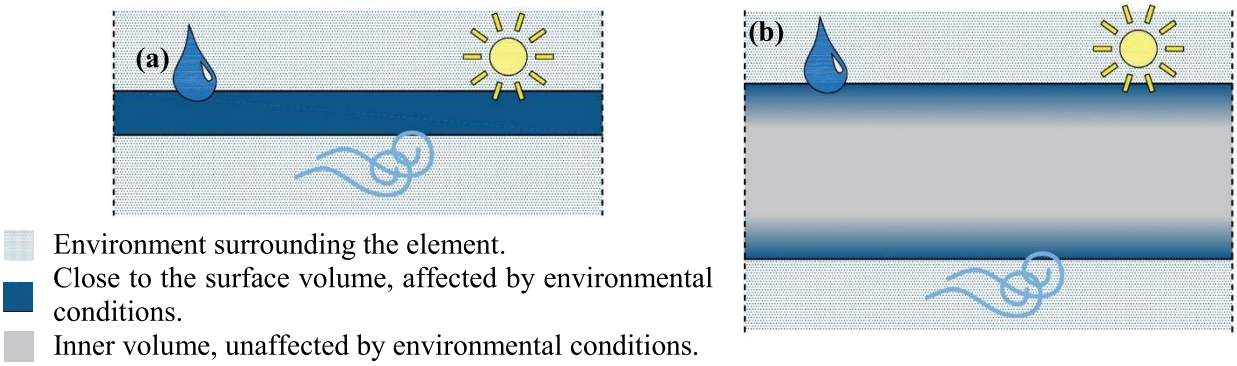


Fig. 16: Influence of environmental conditions on different concrete elements: (**a)** small laboratory specimen and (**b)** construction element.

411 A real construction element can be continuously wetted or isolated from environmental humidity changes
412 by covering it with burlaps, plastic sheets or hydrophobic substances (**Fig. 17**). If this is the case, it can be
413 considered to perform as a sealed laboratory specimen.

(a)

(b)

Fig. 17: External wet curing **(a)** and covering with burlaps and plastic sheet **(b).**

414 The absence of curing in field elements can have a similar effect on concrete close-to-surface volume
415 than air-drying conditions on laboratory specimens. Water immersion can be applied out of the laboratory
416 to small precast concrete elements. Finally, the non-affected zone of a HPC structural member by
417 environmental conditions (its inner part) can be considered to performance like laboratory specimens in
418 sealed conditions [39]. Relationships between analysed curing conditions and field curing conditions are
419 summarized in **Table 8**.

Table 8: Similarity between curing conditions in laboratory and field elements.

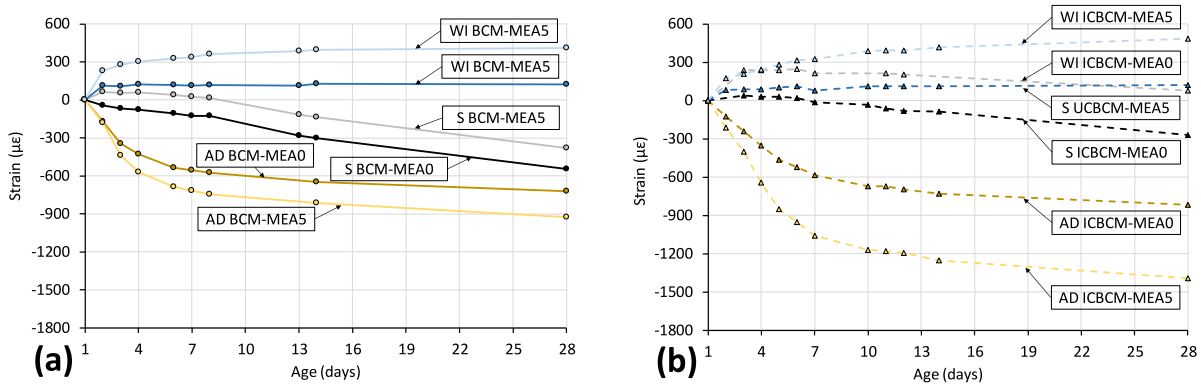
Analysed curing conditions	Sealed	Air-drying	Water immersion
Close-to-surface volume of a construction element	Wet external curing or covering	Absence of curing	Water immersion
Inner part of a HPC structural member	Any possible curing condition	-	-

420

421 In HPC, with a highly dense paste, the inner part can occupy the vast majority of a construction element.
422 Concrete in similar to sealed conditions is, therefore, the principal contributor to concrete strength, as it
423 constitutes the most of a hypothetical structural member. This is the reason why compressive strength was
424 tested on sealed specimens.

425 For each of the three chosen laboratory curing conditions, the effect of three shrinkage mitigation
426 strategies has been analysed: fly ash as a low reactive supplementary cementitious material, internal
427 curing and MEA. More than the results analysis carried out in previous paragraphs, results obtained with
428 different curing conditions can be compared between them (**Fig. 18**). BCM series is chosen for this

comparison as the use of blended cement is considered to be recommendable for economic and ecological reasons over OPC binder. Moreover, only the higher content of MEA (5 %) is presented in order to make graphs clearer.



WI = Water immersion | S = Sealed | AD = Air-drying

Fig. 18: Effect of the highest dosage of MEA (5 %) over blended cement mixes without (a) and with (b) internal curing, when the three curing conditions are applied.

Water immersion leads to expansions whereas air-drying condition lead to the highest values of shrinkage. Sealed condition lead to lower shrinkage than air-drying, and some mixes show initial swelling.

Following the correspondence between laboratory and real field curing conditions stablished in previous paragraphs (**Table 8**), the next can be stated. On the close to surface part of an HPC structural member, the combination of internal curing and MEA has a negative effect if water evaporation is not prevented somehow. On the other hand, if water curing is used, what can happen for small precast elements, undesired expansions on the close to the surface area could happen, being more probable if this kind of curing is applied for a long time. Finally, if HPC is covered with burlaps, plastic sheet or sealant, and in the inner part of a construction element whatever curing condition is used, the three shrinkage reduction strategies used in this study show a positive effect on shrinkage. What is more, internal curing and MEA show a synergistic effect, having a superior performance together than separately in HPC.

Therefore, for internally cured HPC incorporating MEA, it is recommended not to dispense with external curing and being careful with water immersion curing times. This way, it can be get better advantage of the combined effect of both shrinkage reduction strategies.

7. Conclusions

The combined effect on high performance concrete (HPC) of three shrinkage reduction strategies: the use of fly ash as a low reactive cementitious material, internal curing via saturated coal bottom ash and magnesium based expansive agent (MEA), has been studied. The following conclusions are drawn:

- The inclusion of small amounts of MEA (until 5 % of binder) and coal bottom ash as water reservoirs for internal curing (until 30 % of aggregate) in HPC has a slight effect on compressive strength.
- HPC with low content of water limits MEA expansion, however internal curing can effectively provide the extra water needed for this propose. The synergistic effect of both shrinkage reduction strategies is effective for minimising autogenous shrinkage.
- If water evaporation is not prevented, the use of both MEA and internal curing separately or together makes shrinkage to increase.
- MEA effectively expands in water immersed HPC and, in this case, internal curing has no effect over its expansion capacity.
- In comparison with a conventional HPC with no shrinkage reduction strategies, blended cement HPC with high volume of fly ash, MEA and internal curing presents a much better volume stability and acceptable compressive strength. Negative effects such as a possible higher shrinkage on concrete surface are avoidable by preventing water evaporation with conventional external curing methods.
- Other low reactive supplementary cementitious materials and other expansive agents could serve to the same propose, and other aggregates could be suitable to work as internal curing water reservoirs. Future research must study some alternative materials that could lead to comparable results.

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