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Curing effect in the shrinkage of a lower strength self-compacting concrete

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HIGHLIGHTS

- Laboratory tests results on the shrinkage of a lower strength SCC are presented.
- The curing effect on different mixtures was evaluated.
- The compositions have a higher long-term shrinkage when subject to a curing period.
- The higher total shrinkage was related to the refinement of structural porosity.
- The results show that the curing time is essential to minimise the shrinkage at early ages.

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ABSTRACT

Self-compacting concrete possesses special properties that recommend its application in many repair jobs. However, in some practical cases, inappropriate performance of the repair material has been observed in the early stages of hydration, including cracks or delamination due to shrinkage. As the use of self-compacting concrete becomes more prevalent, some novel techniques to combat this phenomenon have been developed, but control of curing conditions continues to be an important prerequisite. In this study, experimental work on the curing effect on the total shrinkage of a lower strength self-compacting concrete, is presented. The curing effect was evaluated on compositions made with different commercial shrinkage-compensating products.

The results obtained indicate that all the compositions (with or without shrinkage reducing admixtures and expansive product) have a higher long-term total shrinkage when subject to cure. It was found that the specimens subject to a longer curing period have lower median pore diameter. The higher total shrinkage was related to the refinement of structural porosity.

The results also confirm that the curing time is essential to minimise the shrinkage at early ages. It causes a lower drying mass loss causing a delaying effect in the development of shrinkage allowing the natural strength development.

1. Introduction

Self-compacting concrete is presented as an innovative material, which has shown to have a high potential in the areas of productivity, working conditions and even in matters arising from their inherent characteristics. This type of concrete possesses special properties that justify its application in many repair jobs. Nevertheless, in some practical cases, inappropriate behaviour of the repair material was observed in the early stages of hydration, including cracks or delamination due to shrinkage [1]. As the use of self-compacting concrete becomes more prevalent, some novel techniques to combat this singularity have been developed. The means and methods for mitigating shrinkage include cement modification, mineral additives, chemical admixtures, fibres, control of curing conditions and advanced methods of internal curing.

In the last two decades, Shrinkage Reducing Admixtures (SRA) has been developed and used with success in reducing shrinkage [2–5].

Ribeiro et al. [6], aiming at reaching a higher shrinkage reduction, tested the synergy of two different SRA, and the results obtained indicate a cumulative effect in shrinkage reduction.

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However, in another work [7], limitations in decreasing shrinkage with the use of increasing dosages of SRA have been detected.

Before the development of shrinkage reducing admixtures, expansive cements were used for over four decades to minimise the effects of drying shrinkage [8–11]. This method has shown good results by improving joint space and limit curing, but its application requires special precautions [12].

To overcome the limitations resulting from the use of individual products to minimise shrinkage, in the last years the effect of combined use of expansive and SRA has been tested. In all the literature reviewed, a synergistic effect resulting from the combined use of expansive and SRA is reported, justifying its use in shrinkage control [13–18].

In order to optimise the shrinkage reducing effect, a proper curing is usually recommended, since curing has influence on shrinkage and cracking [19,20]. In this study, experimental work on the curing effect on the total shrinkage of a lower strength self-compacting concrete, is presented. The curing effect was evaluated on compositions made with different commercial shrinkage-compensating products. The objective of the experimental work carried out is to identify proper conditions to achieve the best shrinkage reduction, using available materials and standard construction practise, but the present paper is focused only in the analyses of the effect of the curing procedure.

2. Materials and methods

In this study, 6 different concrete mixtures were studied. The preparation of specimens (40 mm × 40 mm × 160 mm) was performed according to EN 196-1, in a room with a temperature of 20 ± 2 °C and relative humidity of 55 ± 5%. However, the aggregates and mixture proportions used in the study are different from those established in EN 196-1. Due to the low viscosity of the mixture (SCC), the test specimens were not compacted mechanically.

The removal of moulds took place about 26 h after mixing. This period of time was defined as the minimum necessary to ensure concrete strength between 2 and 5 MPa, thus avoiding damage of the specimens due to moulds removal. Subsequently, the specimens were weighed and their length was registered.

After mould removal, 3 levels of curing were specified:

– Uncured (air curing with the temperature of 20 ± 2 °C and relative humidity of 55 ± 5%);
– Curing until the concrete reaches approximately 50% of the average strength at 28 days.
– Curing until the concrete reaches about 70% of the average strength at 28 days.

For these three levels of cure, the correspondent specimens were protected with a plastic film for 0, 3 or 7 days.

For comparison purposes, autogenous shrinkage and immersion expansion was also measured in additional specimens.

Shrinkage deformations of each specimen were measured using a length comparator, sensitivity of 1 μm, and gage studs on the end sections of the concrete prisms. Stability of the length comparator was checked by a reference invar bar.

Samples were weighed and measured at 1, 2, 3, 5, 7, 14 and 28 days, and 2, 3, 4, 5, 6, 7, 8 and 9 months.

The specimens were prepared with Portland cement CEM II/B-L 32.5N (Tables 1 and 2), according to EN 197-1, siliceous fly ash from Compostilla in Spain (Tables 3 and 4), natural siliceous river sand and crushed limestone coarse aggregate from Algarve in Portugal (Table 5), potable tap water, two SRA, one superplasticizer and one expansive mineral admixture (Table 6).

Table 7 shows the concrete mixtures. The reference mixture (Ref) does not contain shrinkage reducing products. In the other five mixtures, chemical admixtures to control shrinkage were used. The two SRA products were used individually (SRA1, SRA2) or together (SRA 1 + 2). Two of these mixtures also incorporate the expansive agent (+20E, +40E). The superplasticizer amount was adjusted (2.29–3.50 l/m^3) to keep constant the water/powder and the spread flow (68–70 cm).

Small variations on the binder and water contents are required to keep constant the concrete workability, but they are low enough to be despised in the analysis, since all mixtures were made with $W/(C+F) = 0.457 \pm 0.001$, and with a cement content of $C = 260 \pm 5$ kg/m^3.

Taking into account that the expansive powder is a cementitious material, we have considered the hypothesis of using this product as a partial replacement of the cement. However, as the basis for the work is to define a reference mixture and to analyse the reduction of shrinkage provided by the use of different products, the addition of the expansive product was the option chosen. This option has also the advantage of avoiding the problem of uncertainty of the clinker contents of the cement and expansive product.

Table 8 shows some of the properties measured in fresh and hardened states of the reference mixture.

The flow of all the remaining mixtures was in the range 680–700 mm.

The use of SRAs usually leads to a mechanical strength decrease, especially at early ages. However, the introduction of expansive product has the opposite effect. Fig. 1 shows the typical variation obtained with the admixtures.

Table 1
Chemical properties of cement.

Property	Standard	Un.	CEM II B-L 32.5N
Loss on ignition	EN 196-2	%	11.17
Insoluble residue		%	1.60
SiO_2		%	16.05
Al_2O_3		%	4.46
Fe_2O_3		%	2.53
CaO		%	60.29
MgO		%	1.10
SO_3		%	3.02
Cl–		%	0.02
Free lime	ASTM C 114	%	1.00

Table 2
Physical properties of cements.

Property	Standard	Un.	CEM II B-L 32.5N
Density	LNEC E64	kg/m^3	3020
Fineness (Blaine)	EN 196-6	m^2/kg	428
Water for standard consistence	EN 196-3	%	26.1
Initial setting time		min.	120
Final setting time		min.	175
Soundness		mm	1.2
Compressive strength at 2 days	EN 196–1	MPa	19.0
Compressive strength at 7 days		MPa	30.9
Compressive strength at 28 days		MPa	38.8

Table 3
Chemical properties of the fly ash.

Property	Un.	Fly ash*
SiO_2	%	41.65
$SiO_2 + Al_2O_3 + Fe_2O_3$	%	92.71
CaO (free)	%	0.02
CaO (reactive)	%	2.80
MgO (%)	%	2.10
SO_3 (%)	%	0.27
Cl– (%)	%	0.00

* From the autocontrol testing provided by the manufacturer (EN 450-2).

Table 4
Physical properties of the fly ash.

Property	Un.	Fly ash*
Density	kg/m^3	2330
Fineness (Blaine)	m^2/kg	428
Particle size >0.045 mm	%	11.1
Activity index$_{28\ days}$	%	85.5
Activity index$_{90\ days}$	%	104.3

* From the autocontrol testing provided by the manufacturer (EN 450-2).

Table 5
Properties of the aggregates.

Sand		Gravel	
Particles dimensions (mm)	0.125–1	Particles dimensions (mm)	8–12.5
Density (SSD) (kg/m^3)	2660	Density (SSD) kg/m^3	2620

Table 6
Properties of the admixtures.

Admixture	Type	Delivery condition	Density (kg/m^3)	Main Component	Recommended dosage
SP1	Superplasticizer	Liquid	1070	Polycarboxylate ether	0.6–1.2 kg/100 kg of binder
SRA1	SRA	Liquid	980	Butoxyethanol	7.5 l/m^3
SRA2	SRA	Liquid	1010	Alkyl-ether	0.5–2% of weight of cement
EXP	Expansive	Powder	3090	Clinker with high free lime content	20 kg/m^3

Table 7
Mix proportions of tested concrete.

Mixture		Ref	SRA1	SRA2	SRA (1 + 2)	SRA (1 + 2) + 20E	SRA (1 + 2) + 40E
Water/powder		0.458					
kg/m^3	CEM II/B-L 32.5N	265	263	263	261	260	258
	Fly ash	173	172	172	171	170	168
	Sand	780	774	776	770	765	760
	Gravel	768	763	765	759	754	749
	Expansive	–	–	–	–	19.6	39.0
l/m^3	SP1	3.50	2.65	2.66	2.31	2.29	2.67
	SRA1	–	7.45	–	7.41	7.37	7.31
	SRA2	–	–	5.31	5.27	5.24	5.20
	Mixing water	200	199	199	198	197	195

Table 8
Properties of the self-compacting reference mixture.

Self-compacting ability		Compressive strength (MPa)		
Method	Ref	Time (days)	Average (3 samples)	SD
Flow (EN 12350-8)	680 mm SF2	1	9	1.5
V funnel (EN 12350-9)	4 sVF1	7	20	2.7
L Box (3 bars) (EN 12350-10)	0.84 PA2	28	27	1.8

In the absence of the expansive agent, the simultaneous use of the two SRA products caused a reduction in compressive strength at 28 days of 31.2%. In the tests conducted at 9 months the strength reduction was 30.0%. A strength reduction of 5–10% is usual due to the use of a SRA, but decreases up to 20% have been reported [6,7,21]. The higher percentage obtained in this work should be related with the lower strength level of the concrete, being 8 MPa the difference in absolute value. It should also be mentioned that the small size of the specimens ($4 \times 4 \times 16$ cm^3), and the high value of $W/C = 0.76$, may contribute to portlandite leaching of the specimens submerged in water, which decreases the pozzolanic contribution of the fly ash. This may explain the abnormal high decrease of compressive strength.

The simultaneous use of the SRAs combined with 20 kg/m^3 dosage of the expansive agent lead to a strength reduction at 28 days of 13.3%. In tests carried out at 9 months of age, only 1.6% reduction was observed. This better behaviour of the mixtures with expansive product should be related with the increase of the clinker content, and the consequent decrease of water/binder. This is supposed to have not only a direct effect on strength but an indirect benefit related with portlandite leaching.

However, some authors also refer the reduction in the degree of hydration of the cement due to the presence of SRA [22–27]. In the early ages, significant reductions in mechanical strength were reported [28–30,13].

3. Results and discussion

The test results obtained on the six series of specimens allowed comparing their relative performance regarding mass change, total shrinkage and porosity.

3.1. Mass change

The charts in the following figures (Figs. 2–7) show the mass variation for the mixtures: Ref; SRA1, SRA2, SRA (1 + 2), SRA (1 + 2) + 20E and SRA (1 + 2) + 40E, recorded up to 9 months (each value presented is the average of 7 specimens).

The mass variation presented was calculated as a percentage of the initial mass, recorded immediately after mould removal. The individual results deviation were very small (SD < 0.15%).

All figures clearly show the beginning of mass loss at the end of cure, as expected. The results also put in evidence that the cured specimens (3 or 7 days) have a lower mass loss than the not cured specimens.

Regarding the curing effect, one may observe that the test pieces subjected to curing (3 or 7 days) present a lower mass loss at 9 months. In fact, a longer curing period promotes better cement

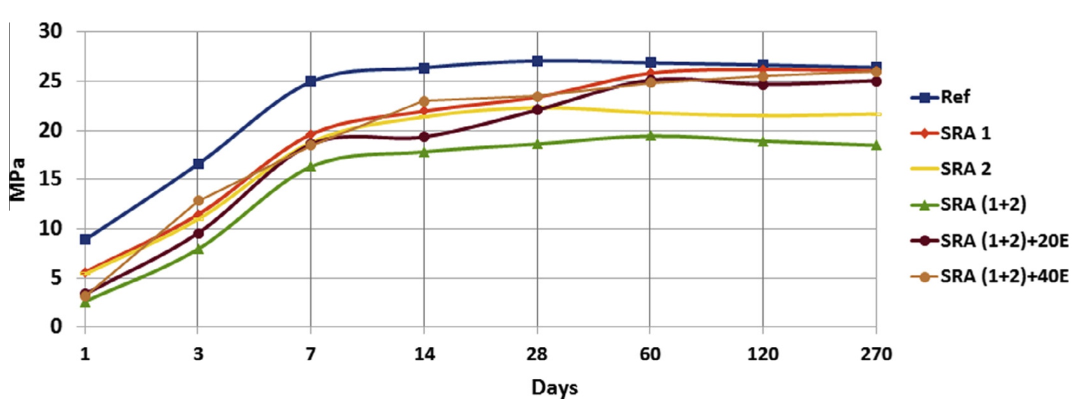

Fig. 1. Compressive strength.

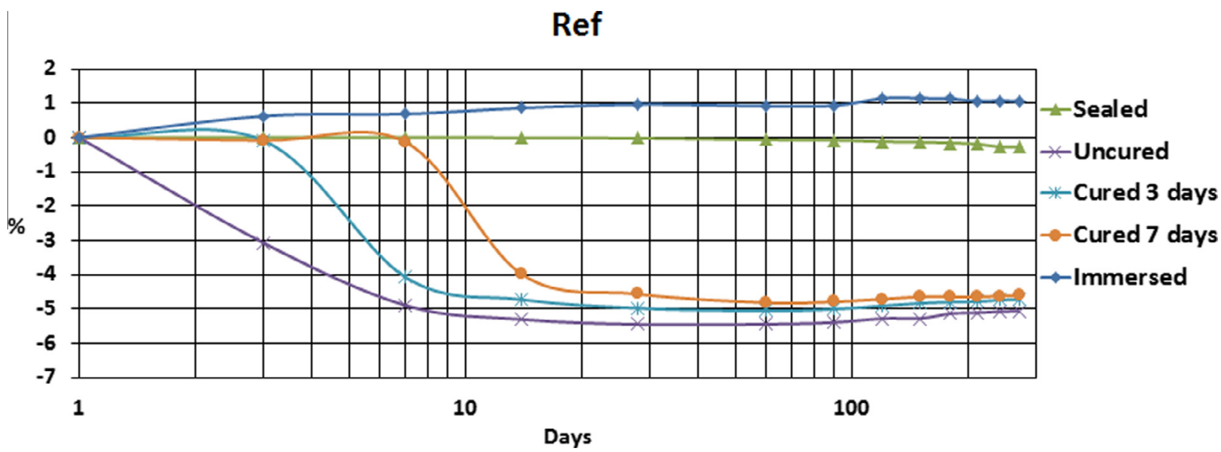

Fig. 2. Mass change of mixture Ref.

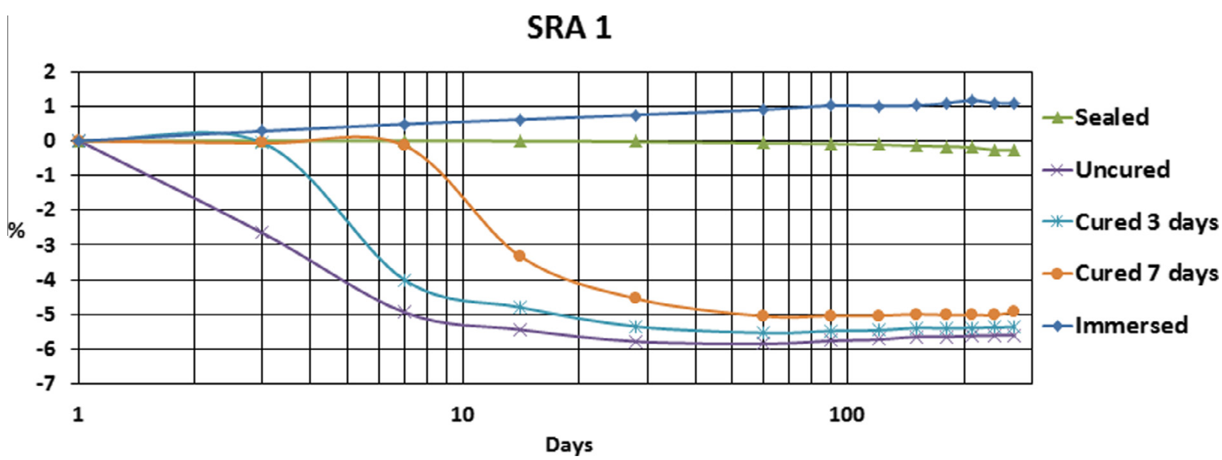

Fig. 3. Mass change of mixture SRA1.

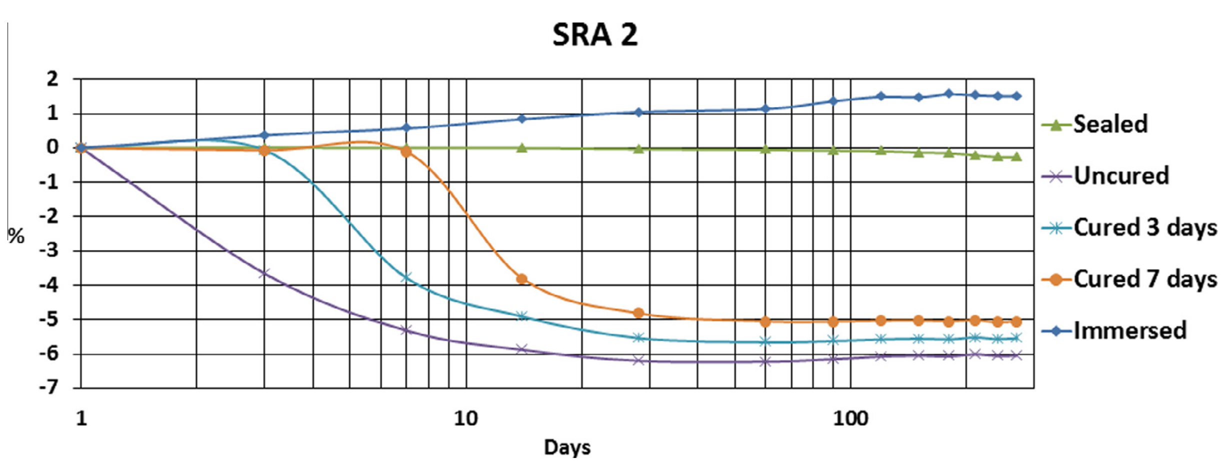

Fig. 4. Mass change of mixture SRA2.

hydration, leading to a more closed pore structure, thus inhibiting moisture exchange with the environment.

Sealed specimens show almost no mass change and immersed specimens show mass gain, as expected. The mass gain is higher in specimens with the expansive powder, which should be related with the higher paste content and the presence of expansive hydration products.

3.2. Total shrinkage

The following results, presented in the form of graphs, were obtained using the average measurements of seven specimens per mixture. For each mixture the solid curves present the average values and the dashed curves present the average plus or minus one standard deviation.

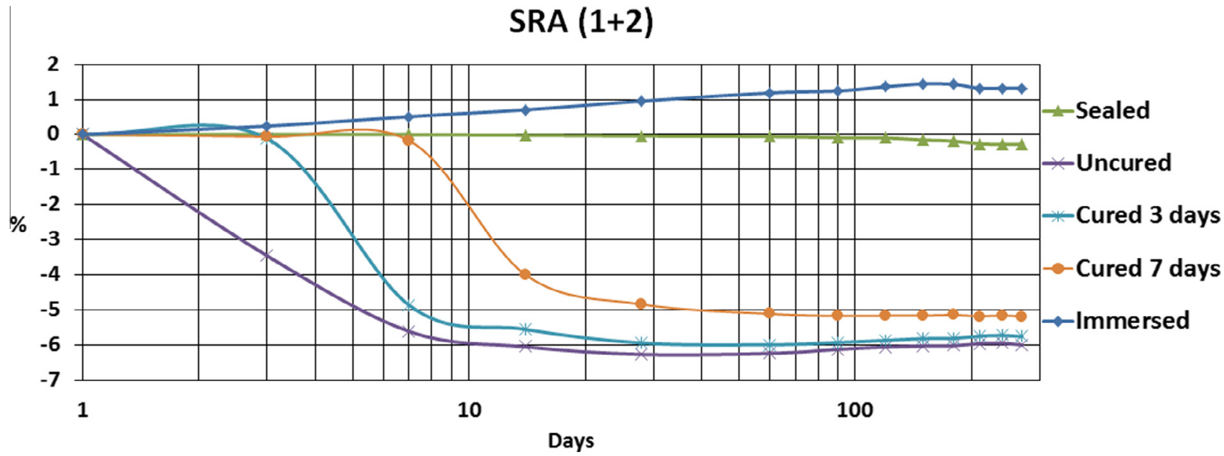

Fig. 5. Mass change of mixture SRA (1 + 2).

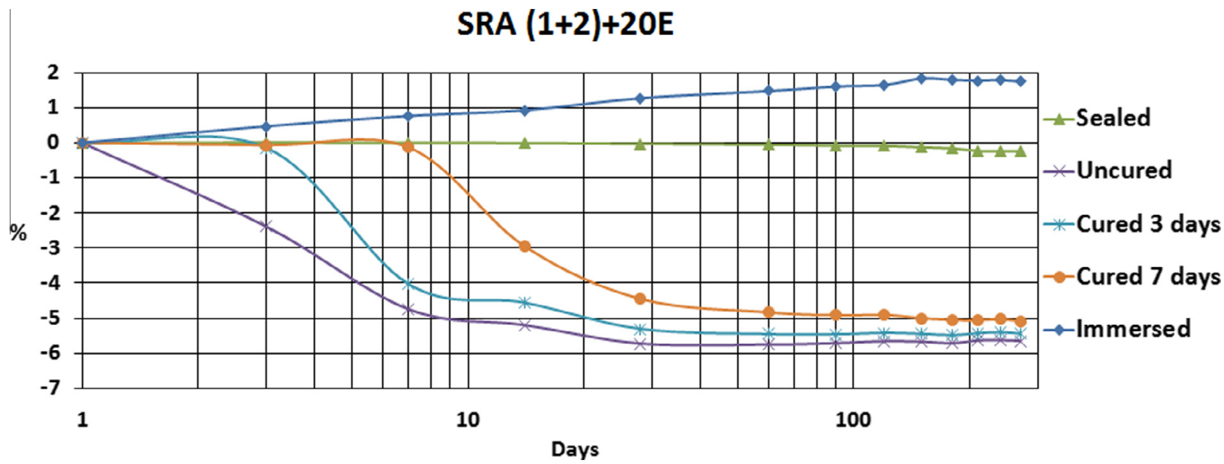

Fig. 6. Mass change of mixture SRA (1 + 2) + 20E.

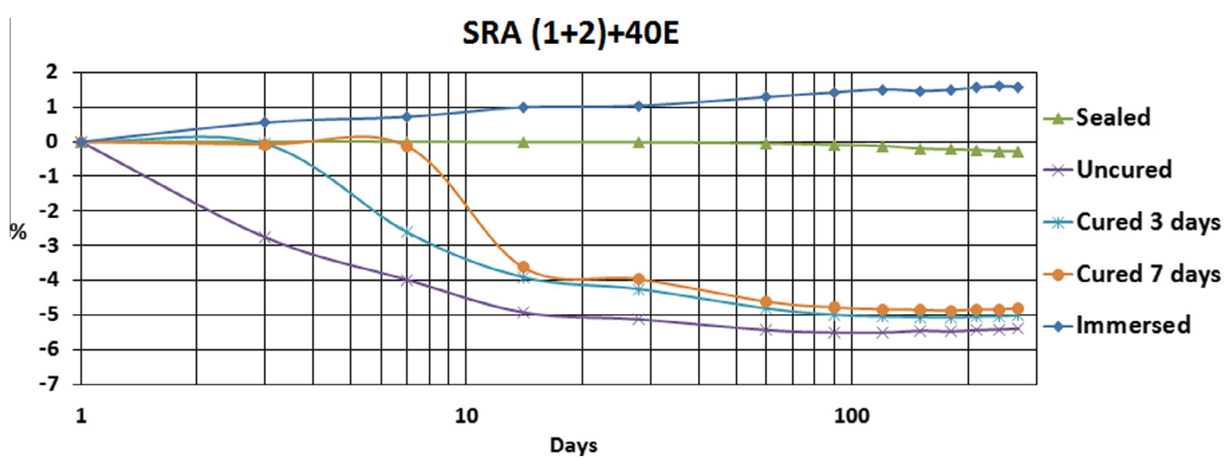

Fig. 7. Mass change of mixture SRA (1 + 2) + 40E.

Figs. 8–13 illustrate the total shrinkage evolution recorded up to 9 months of age for the different mixtures.

Fig. 8 shows the results obtained in the reference mixture, which contains no SRA or expansive powder. Immersed specimens show expansion due to the mass gain, and sealed specimens show low shrinkage values, due to the almost absence of drying.

Focusing now in the specimens subjected to drying, until 10 days the higher shrinkage is observed on the uncured specimens, followed by the specimens 3 days cured and the lowest shrinkage is observed on specimens 7 days cured. The behaviour observed is in accordance with the mass loss presented in Fig. 2, or, in other words, when there is more drying the result is larger shrinkage. This is in agreement with the shrinkage mechanisms more relevant for high humidity levels (capillary tension and disjoining pressure), assuming equal porous structures for the specimens subjected to the different curing procedures.

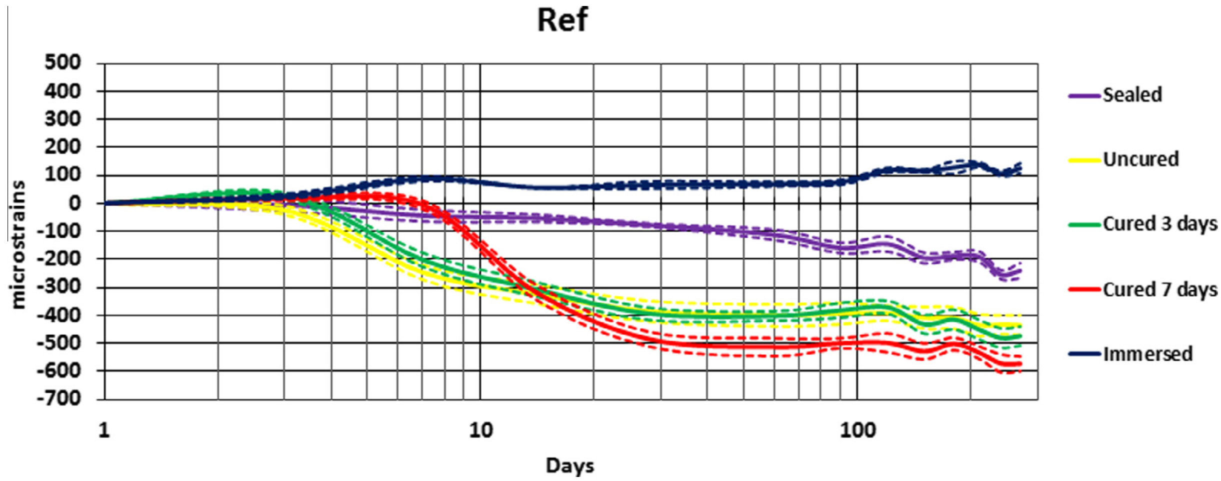

Fig. 8. Length variation of mixture Ref.

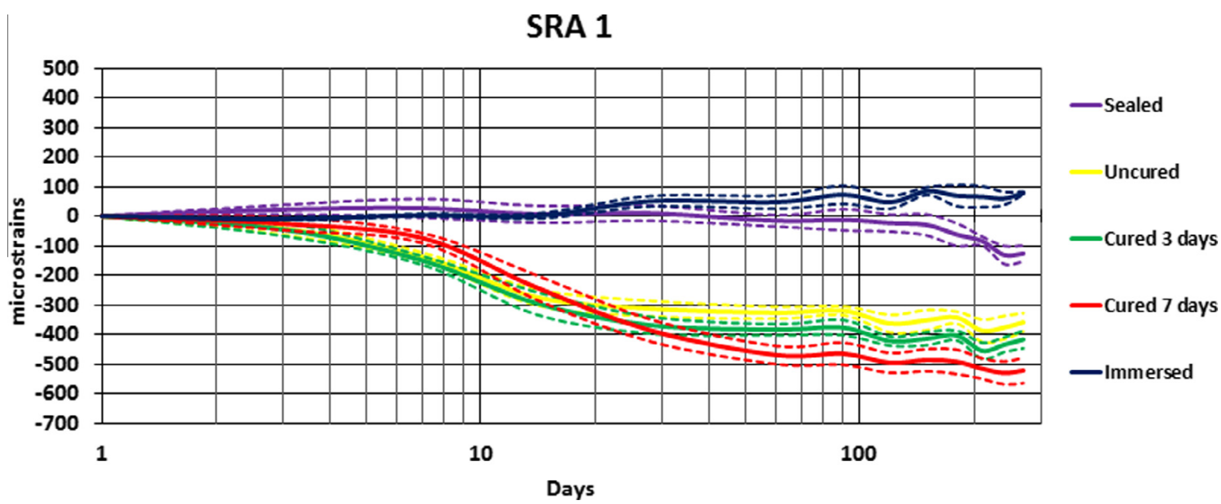

Fig. 9. Length variation of mixture SRA1.

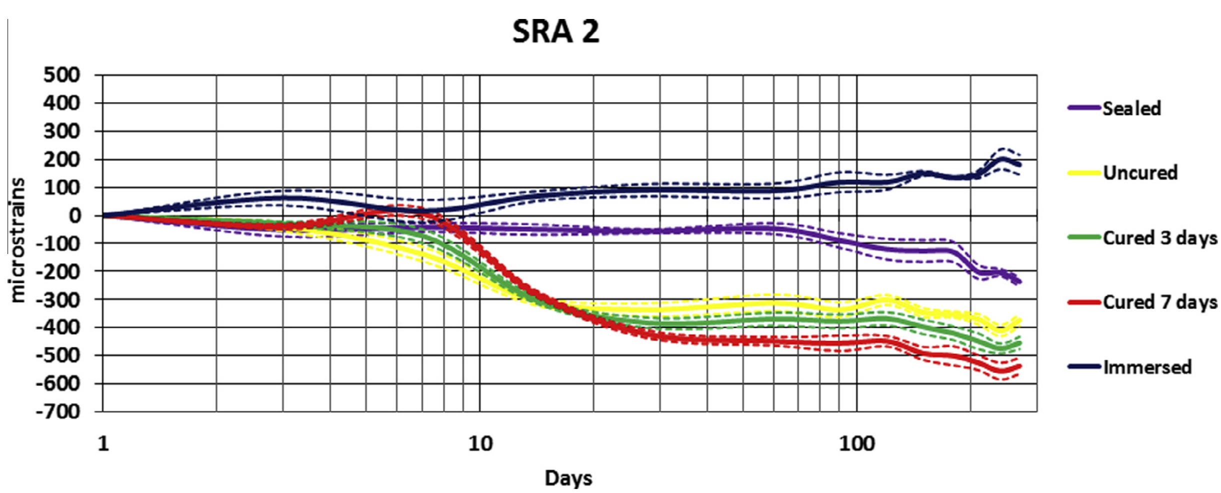

Fig. 10. Length variation of mixture SRA2.

However, for specimens subjected to drying, from 10 days until the final measurement there is an inversion in the relative positioning of uncured and 7 days cured curves. Unlike the case with the mass loss, at long term, the shrinkage of uncured specimens is higher than the shrinkage of the 7 days cured specimens. The cause for this contradictory result will be discussed later in the porosity section.

Figs. 9 and 10 present the results obtained with mixtures containing SRA. At early age there are small differences between the specimens made with SRA1 (Fig. 9) and the specimens made with

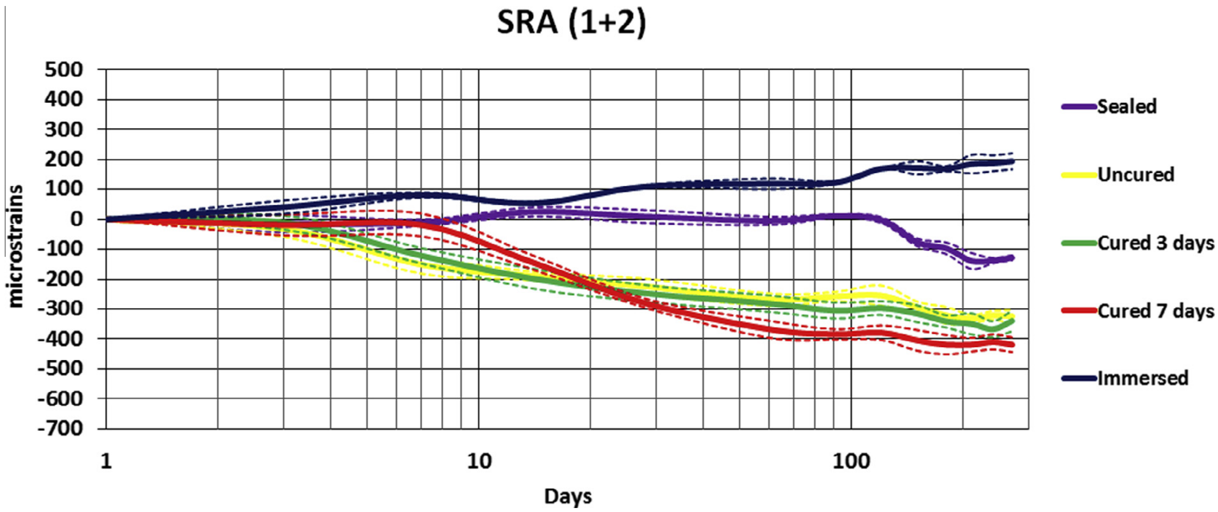

Fig. 11. Length variation of mixture SRA (1 + 2).

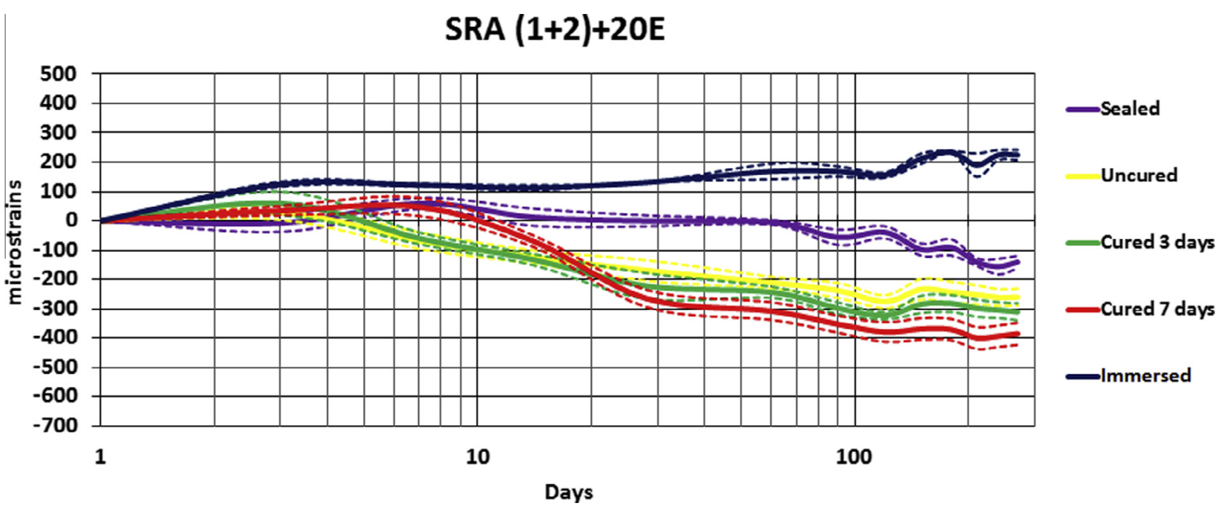

Fig. 12. Length variation of mixture SRA (1 + 2) + 20E.

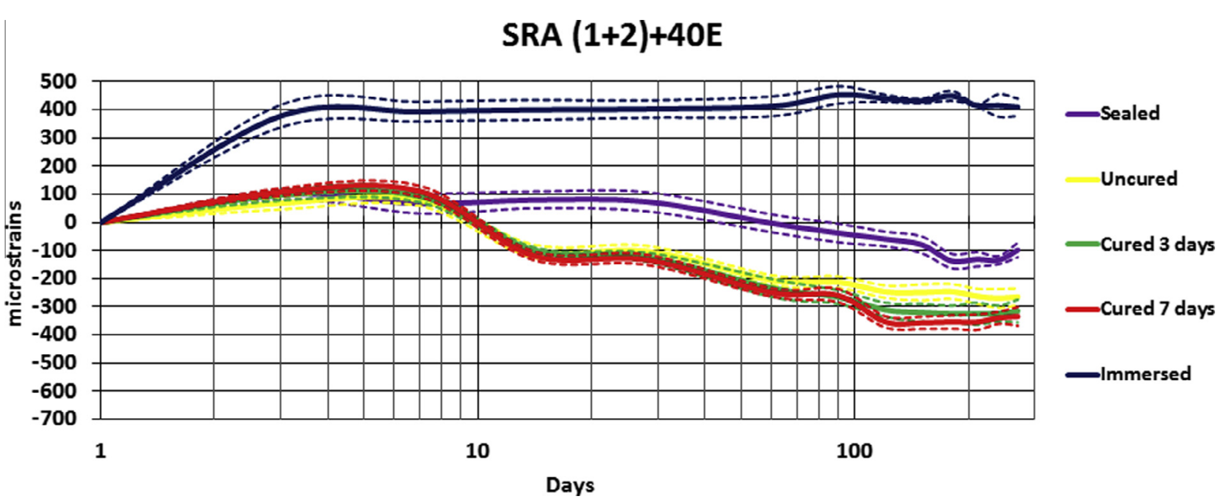

Fig. 13. Length variation of mixture SRA (1 + 2) + 40E.

SRA2 (Fig. 10), which may not be attributed to differences on mass losses, but rather are related with the influence of SRA. For both mixtures, and for specimens subjected to drying, from 10 days until the final measurement there is an inversion in the relative positioning of uncured and 7 days cured curves, as observed for the reference mixture, which means higher shrinkage for longer curing period, is therefore an unexpected tendency.

Fig. 11 presents the shrinkage results obtained in the mixture with both SRA. The curves reveal a pattern similar to the curves presented in Figs. 9 and 10, but with a synergic effect, in concordance with previous work [6]. The inversion in the relative positioning of uncured and 7 days cured curves is also evident, which reveals a consistent influence of the different curing procedures.

Fig. 12 presents the shrinkage results obtained in the mixture with both SRA and with expansive powder at the lower dosage (20 kg/m^3). The benefit associated with the use of expansive powder is visible, at young ages. In fact, at 10 days the shrinkage of specimens cured during 7 days is null, and the shrinkage of uncured and 3 days cured specimens is only 100 microstrain. At long term, we can also observe the inversion in the relative positioning of uncured and 7 days cured curves.

Fig. 13 presents the shrinkage results obtained in the mixture with both SRA and with expansive powder at the higher dosage (40 kg/m^3). The initial expansion associated with the use of more expansive powder is higher, and, at 10 days, none of the curves show shrinkage. However, the submerged specimens show a significant expansion, 400 microstrain, and the cracking risk increases for specimens in similar conditions. For specimens subjected to drying, at long term, we observe again the highest shrinkage on specimens cured 7 days and the lowest shrinkage on uncured specimens.

Table 9 shows the results for the total shrinkage obtained at 1, 3, 6 and 9 months, with no cure and subjected to the maximum level of cure.

In Table 9, the values in brackets indicate the difference for uncured specimens. Giving as an example, the value (+32%) presented in the cell which corresponds to the measurement at 9 months of the reference mixture is obtained by:

$$\frac{-573}{-435} \times 100 - 100 = +32$$

−573 microstrain is the shrinkage at 9 months of the specimens of the reference mixture cured 7 days;
−435 microstrain is the shrinkage at 9 months of the specimens of the reference mixture with no cure;
+32 is the percentage of the shrinkage increase due to the 7 days curing.

It is found that the cured specimens have a long-term total shrinkage higher than the uncured ones, and the difference is very significant for this low strength concrete.

Besides comparing the effect of long-term cure, it is also important to analyse the shrinkage at early ages (Table 10). The analysis of the results shows that the curing procedures lead to a significant reduction in the total shrinkage.

3.3. Porosity

In the previous section it was observed that the specimens cured during 3 or 7 days exhibited higher shrinkage than the specimens without cure. As the results point in the opposite direction than is recommended as good practise in construction [1,6], it is important to assess whether this increase in shrinkage with curing time was related to the refinement of pore structure, as expected in cementitious materials. The graphs of Fig. 14 show the pore size of two samples using the mercury intrusion technique. Nine tests were tested per specimen. The results can be found in Table 11.

Both specimens belong to the mixture SRA2. One sample was not subject to a cure period beyond the additional inherent to the mould release, while the other was subject to an additional cure period of 7 days.

Analysing Fig. 14 and the data presented in Table 11, it may be concluded that the average pore diameter is lower in the specimens subjected to the additional curing period of 7 days.

The test was done in only one mixture, due to limited funding, and because it aimed at confirming an expected change in microstructure due to better cure.

Table 9
Effect of long-term cure in total shrinkage.

Mixture		Total shrinkage (×10^{-6})								Compared average
		Uncured				Cured 7 days				
		Age (months)				Age (months)				
		1	3	6	9	1	3	6	9	%
SCC	Ref	−386	−394	−409	−435	−486 (+26%)	−500 (+27%)	−502 (+23%)	−573 (+32%)	+27
	SRA1	−313	−320	−343	−359	−387 (+24%)	−465 (+45%)	−493 (+44%)	−522 (+45%)	+40
	SRA2	−336	−336	−353	−374	−424 (+26%)	−455 (+35%)	−500 (+42%)	−536 (+43%)	+37
	SRA (1 + 2)	−224	−258	−321	−324	−280 (+25%)	−384 (+49%)	−419 (+31%)	−419 (+29%)	+34
	SRA(1 + 2) + 20E	−170	−238	−244	−260	−265 (+56%)	−353 (+48%)	−371 (+52%)	−385 (+48%)	+51
	SRA (1 + 2) + 40E	−103	−213	−246	−265	−133 (+29%)	−260 (+22%)	−354 (+44%)	−334 (+26%)	+30

Table 10
Effect of curing at early ages in the total shrinkage.

Mixture		Total shrinkage (×10^{-6})						Compared average
		Uncured			Cured 7 days			
		Age (days)			Age (days)			
		3	7	14	3	7	14	%
SCC	Ref	−24	−246	−324	+17 (−170%)	−5 (−98%)	−318 (−2%)	−90
	SRA1	−42	−138	−274	−26 (−38%)	−74 (−46%)	−241 (−12%)	−32
	SRA2	−40	−137	−304	−39 (−3%)	+8 (−106%)	−277 (−9%)	−39
	SRA (1 + 2)	−32	−151	−189	−19 (−41%)	−19 (−87%)	−145 (−23%)	−50
	SRA (1 + 2) + 20E	+28	−68	−123	+35 (−25%)	+47 (−169%)	−69 (−43%)	−79
	SRA (1 + 2) + 40E	+68	+79	−102	+108 (−59%)	+111 (−41%)	−118 (+16%)	−28

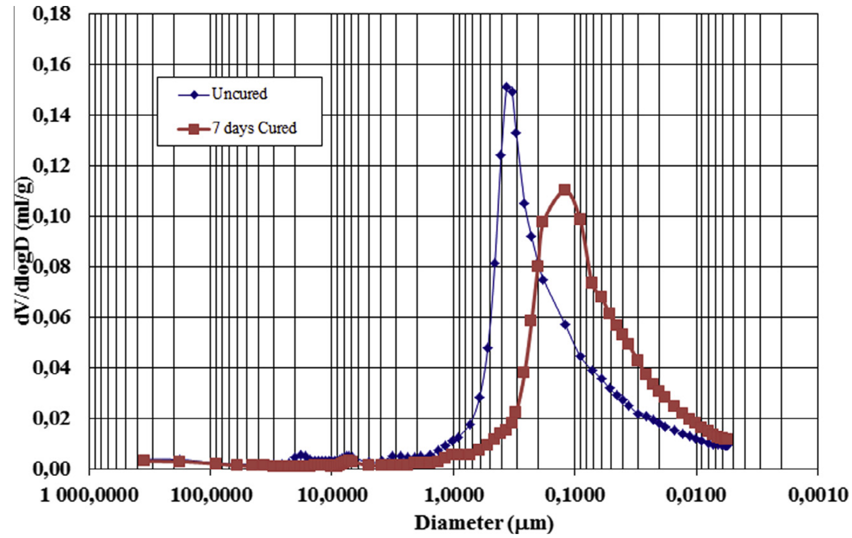

Fig. 14. Pore diameter of the section 1 for the uncured and 7 days cured samples.

Table 11
Pore diameter of the samples, for different sections.

Mixture: SRA2			
Uncured		Curing period of 7 days	
Sections	D_M (µm)	Sections	D_M (µm)
1	0.35	1	0.14
2	0.30	2	0.09
3	0.32	3	0.15
4	0.30	4	0.19
5	0.37	5	0.14
6	0.30	6	0.16
7	0.30	7	0.17
8	0.38	8	*
9	0.32	9	0.12

* Not measured.

Drying shrinkage is usually referred as mainly consequence of capillary tension and disjoining pressure, for usual environmental relative humidity values. As concrete dries the process begins by emptying the larger pores, followed by successively smaller pore sizes, which leads to liquid tension increase in the capillary pores. The release of gel water in the very small pores also increases shrinkage due to disjoining pressure.

As an example for capillary pressure of a cylindrical ideal pore, liquid tension may be quantified by the Laplace equation. (Eq. (1))

$$p'' - p' = \frac{2\sigma\cos\theta}{r} \quad (1)$$

where: p″ – steam tension; p′ – liquid tension; σ – surface tension of the interstitial liquid; r – radius of curvature of the meniscus; θ – wetting angle.

If it is assumed that the liquid moistens all the pore walls and perfect wetting ($\theta = 0°$) is applicable, then the radius of curvature of the meniscus is equal to the radius of the pore. Applying Eq. (1) we may estimate the capillary tension for the two average pore radius of the tested samples. Considering a surface tension of the interstitial liquid of 60×10^{-3} N/m:

$$dP_{\text{Air dry}} = \frac{2\gamma\cos\theta}{r} = \frac{2 \times 60}{0.35 \times 10^{-6}} = 0.34 \text{ Mpa}$$

$$dP_{\text{Cured}} = \frac{2\gamma\cos\theta}{r} = \frac{2 \times 60}{0.14 \times 10^{-6}} = 0.86 \text{ Mpa}$$

The pressure difference is more than double from one case to another. So, the existence of smaller pores is in agreement with the occurrence of a greater shrinkage.

4. Conclusions

The reduction of concrete shrinkage due to the presence of SRA was observed, as expected. This reduction is improved using two SRA products in synergy, as previous put in evidence, and further shrinkage reduction is observed when expansive powder are used in combination with two SRA, especially at early age.

However, this work was mainly focused on the influence of the curing process in the total shrinkage of a lower strength self-compacting concrete. Tests were carried out in specimens uncured, subjected to 3 days cure, and 7 days cure. The results obtained indicate that in all the concrete mixtures tested (with or without shrinkage reducing admixtures and expansive product) the highest long-term total shrinkage was obtained on the 7 days cured specimens, and the lowest long-term total shrinkage was obtained on the uncured specimens.

The higher total shrinkage was related to the refinement of structural porosity.

The results also show that the curing time is essential to minimise the shrinkage at early ages. It causes a lower drying mass loss causing a delaying effect in the development of shrinkage. This is important for early cracking proneness.

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