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# Study of different granular by-products as internal curing water reservoirs in concrete

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## ABSTRACT

The use of three different waste-based materials, i.e. ceramic recycled aggregate (CRA), mixed recycled aggregate (MRA), and coal bottom ash (CBA), as internal curing water reservoirs was investigated. Their effects as fine aggregates in the mortar phase of a hypothetical concrete with a low water-to-binder ratio were studied. The used binders were ordinary Portland cement and high-volume fly-ash-blended cement. The addition of MRA and CBA significantly decreased the autogenous shrinkage, whereas their negative effects on drying shrinkage and the compressive strength were minimal. However, the CRA had the worst effect on autogenous shrinkage but increased the strength of the specimens.

## 1. Introduction

Several studies on using various waste-based porous materials (WASPORS) as internal curing water reservoirs (ICWRs) in concrete have been conducted. WASPORS have been used because of their potential suitability for this application, economic benefits, and environmental advantages [1]. Examples of WASPORS include ceramic-recycled aggregate [2–4] and coal bottom ash [5,6]. However, other granular by-products have been found to be unsuitable for their use as ICWRs. For example, recycled concrete aggregate exhibited inadequate desorption properties, owing to the tight pore structure of the adhered old mortar [7]. However, some studies have insisted on the potential feasibility of recycled concrete aggregate as ICWRs [8], and others have attributed part of their behaviour to the internal curing effect [9]. Furthermore, the characteristics of the old concrete from where the recycled aggregates are obtained and the processes involved in their transformation result in recycled aggregates of different qualities [10]. Some conventional lightweight aggregates also have disadvantages; for example, very lightweight aggregates tend to float [11]. Furthermore, their water absorption kinetics can be slow, and thus, they need to be vacuum-saturated to achieve their maximum capacities [12,13].

The comparison between different WASPOR types regarding their performance as ICWRs is challenging. Most researchers investigated them individually by performing different experimental tests (including non-standardised tests) on cement-based materials using different mix proportions. However, the results and findings of different studies have been collated [1]. Furthermore, a few studies on the performance comparison of different WASPORS using the same volume ratio [14,15] or amount of internal curing water (ICW) [16] have been conducted. Any of these two criteria leads to a lack of precise comparability. Efficiency factors can facilitate

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comparing different ICWRs regarding their ability to reduce autogenous shrinkage in materials with different compositions. An example is the efficiency factor derived by Zhutovsky et al. [17], as expressed in Eq. (1):

$$\eta = \frac{W_{ic}}{S \cdot \phi \cdot W_{LWA}} \cdot SR \quad (1)$$

where  $\eta$  is the efficiency factor,  $W_{ic}$  is the internal curing water required to eliminate self-desiccation (g),  $S$  is the degree of saturation (%),  $\phi$  is the water absorption capacity (wt%),  $W_{LWA}$  is the lightweight aggregate content (g), and  $SR$  is the autogenous shrinkage reduction (%).

Internal curing not only mitigates the self-desiccation of the cement paste but also sustains cement hydration. Furthermore, the rough texture in an ICWR can improve the paste–aggregate bond [1,18,19], referred to as the “nailing effect” by some authors [20]. However, these beneficial effects can be counteracted by the weakness of the ICWR [7]. Consequently, the internal stress generated by different phenomena (e.g. drying and self-desiccation) can induce larger deformations in internally cured concrete [19], contrary to the objective of internal curing. Nevertheless, a lower modulus of elasticity has also been pointed to reduce the cracking potential [21].

Research studies on the internal curing performance of different artificial aggregates can be found in the literature, however, the use of by-products for this purpose has been hardly studied [22–24]. In addition, the incorporation of supplementary cementitious materials to promote the sustainability of the new concrete may change the effect of the internal curing via pre-wetted WASPORS. The aim of this study is to analyse three fine WASPORS — coal bottom ash, ceramic-recycled aggregate, and mixed recycled aggregate — as ICWRs in two high-performance concretes (HPC), one with only Portland cement as binder and the other with fly ash as supplementary cementitious material. The efficiency of autogenous shrinkage mitigation and effects on drying shrinkage and compressive strength are also reported in this paper at different ages.

## 2. Material characterisation and mix design

All the materials used in this study had a maximum particle size of 4 mm. Thus, the experimental programme was conducted on mortar mixes that did not contain coarse aggregate but only the paste and fine aggregate of a hypothetical HPC.

Crushed conventional sand (S) with a 4 mm maximum size was partially substituted with three different WASPORS: coal bottom ash (CBA), ceramic-recycled aggregate (CRA), and mixed recycled aggregate (MRA). The CBA was produced during the combustion of pulverised Indonesian sub-bituminous coal at the As Pontes power station [25]. The CRA was produced by crushing rejected bricks supplied by Piera Ecoceramic. The MRA was obtained by sieving construction and demolition waste composed of ceramics (70%), concrete (20%), raw aggregates (9.4%), and other minor constituents, such as gypsum (0.5%). The photograph of each WASPOR is shown in Fig. 1.

The particle size distributions of S and the three WASPORS are shown in Fig. 2.

The physical properties are listed in Table 1. The water absorption capacity was obtained following a paper towel method based on the standard NY 703-19 E [26]. Two samples of each aggregate, with a volume of 1.2 l and free of particles with a size below 63  $\mu\text{m}$  were tested. They were saturated in water for 24 h and drained in a 63  $\mu\text{m}$  sieve. Then, the particles were dried with paper towels until no moisture remains on their surface (Fig. 3).

The desorption capacity of the three ICWRs at different relative humidities (97, 90 and 60%) was tested based on ASTM C1498 [27] and the results are plotted in Fig. 4. The desorption capacity of the ICWRs is considered as one of the main parameters for the study of their efficiency [28] and other researchers have widely studied this property [29]. According to the obtained results, the desorption capacities of CRA and MRA are similar to one another and superior to that of CBA.

The chemical compositions of the four granular materials are listed in Table 2 and their crystalline phases are identified in Fig. 5. Silicon, aluminium and iron oxides are predominant in all the aggregates used (>85% in S and CRA). Quartz is detected in all the aggregates whereas mullite is present in CBA and CRA.

All the aggregates were wetted for 24 h before mixing with an amount of water equivalent to the water absorption capacities and then protected from moisture loss. This water was intended to cure the mortar paste internally; therefore, it was referred to as the internal curing water in the conventional sand (ICW-S) and internal curing water in the WASPOR (ICW-WASPOR). S was substituted with different volume proportions of the WASPORS: 15% and 30% of CBA, and 30% and 50% of both CRA and MRA. The different substitution ratios were selected so that the highest ones contain an amount of ICW-WASPOR close to that recommended by Bentz et

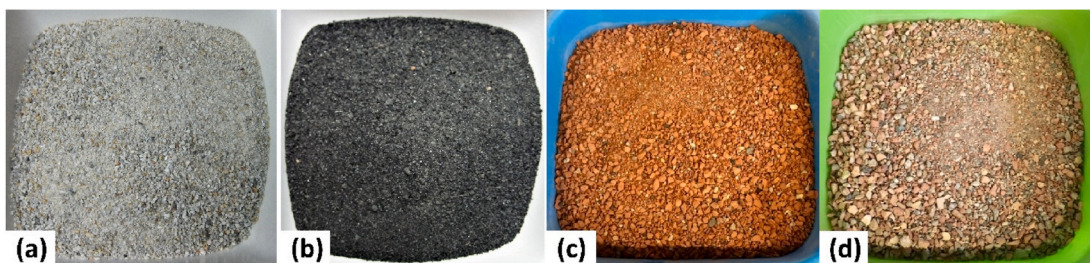


Fig. 1. Photographs of (a) S, (b) CBA, (c) CRA, and (d) MRA.

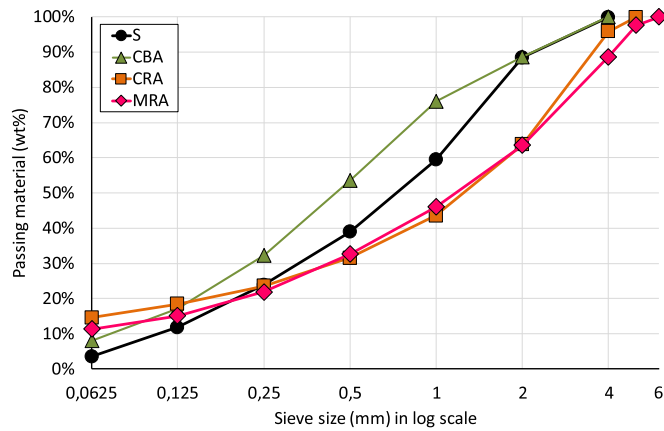


Fig. 2. Particle size distribution of the aggregates.

Table 1

Physical properties of aggregates.

	S	CBA	CRA	MRA
Maximum size (mm)	4	4	4	4
Fineness modulus	2.78	2.33	3.23	3.32
Specific gravity (g/cm <sup>3</sup> )	2.47	1.19	1.97	1.92
24 h mass water absorption capacity (%)	2.4	36.7	11.8	15.4
24 h volume water absorption capacity (%)	6.0	43.6	23.3	29.6

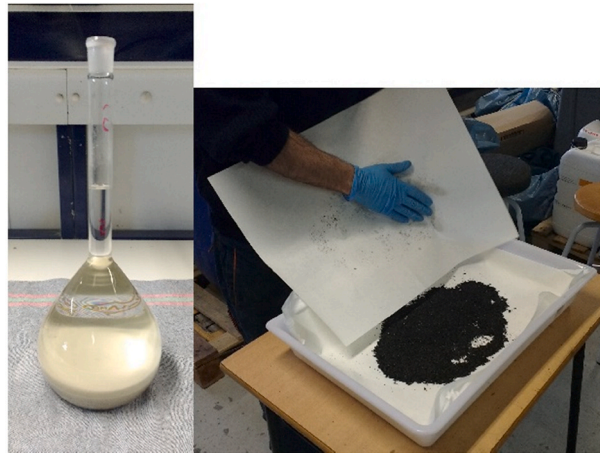


Fig. 3. Saturation and drying of the aggregates with the paper towels.

al. [30]. According to this recommendation, the ICW needed for the mitigation of autogenous shrinkage in a m<sup>3</sup> of mortar is 54 kg. For calculating that value, the next parameters have been considered: the content of cement (Cf), which is 925 kg per m<sup>3</sup> of mortar; the volumetric chemical shrinkage of cement (CS), which is estimated as 0.07; an expected degree of hydration ( $\alpha_{max}$ ) of 0.83, which is calculated as  $\frac{w/c}{0.36}$  as in Bentz et al. [30]; and the density of water (1000 kg/m<sup>3</sup>). The needed ICW-WASPOR results to be 54 kg/m<sup>3</sup> when applying Eq. (2). The lowest replacement ratios were selected so that some of them were the same in the three WASPORS (30%).

$$ICWWASPOR = \frac{Cf \cdot CS \cdot \alpha_{max}}{\rho} \quad (2)$$

Two different binders were used: ordinary Portland cement (OPC) with a density of 3.12 g/cm<sup>3</sup>, and blended cement composed of equal volume proportions of OPC and Class F fly ash (FA) with a density of 2.21 g/cm<sup>3</sup>. Then, two series of mortars were developed. The series with OPC as binder is referred to as cement mortar (CM) and the series with the blended cement is referred to as blended-cement mortar (BCM). A high-range water-reducing admixture (HRWRA) with a density of 1.05 ± 0.02 g/cm<sup>3</sup> and a solid residue of 20.3% ± 1% was dosed at 0.80% in terms of solid residue/OPC to enhance the mortar fluidity and achieve self-compaction. The mix-

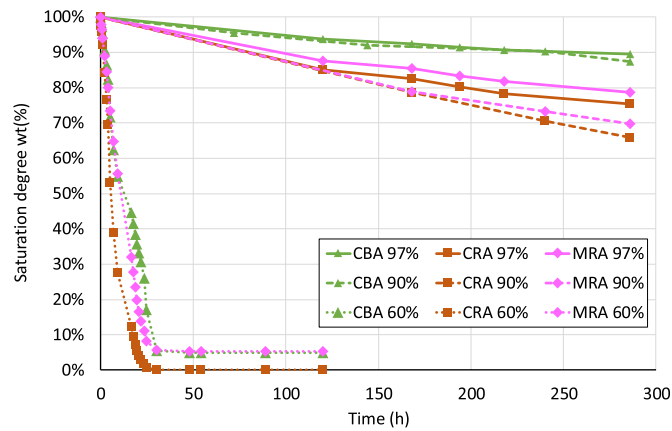


Fig. 4. Desorption of CBA, CRA and MRA in environments with 97%, 90% and 60% relative humidity.

Table 2

Chemical compositions obtained by XRF.

Oxides (wt%)	S	CBA	CRA	MRA
SiO <sub>2</sub>	68.90	35.9	55.2	38.2
Al <sub>2</sub> O <sub>3</sub>	17.70	11.3	24.2	12.4
Fe <sub>2</sub> O <sub>3</sub>	0.82	12.3	7.8	4.1
CaO	0.41	7.4	2.7	22.3
MgO	0.09	2.5	2.7	2.4
SO <sub>3</sub>	–	0.9	0.1	2.8
Na <sub>2</sub> O	5.50	0.7	0.8	1.0
K <sub>2</sub> O	4.80	0.8	3.6	2.3
TiO <sub>2</sub>	–	0.6	1.0	0.5
P <sub>2</sub> O <sub>5</sub>	0.43	0.1	0.3	0.1
Other	0.45	0.7	0.8	0.3
LOI	0.90	27.1	1.0	13.6

ing water- (W) to-binder ratio by volume remained constant for all mixes. The mixing water did not include the ICW referred to in previous paragraphs.

Combining the two different binders with the three WASPORS and the corresponding substitution ratios resulted in 14 different mortar mixes (Table 3).

### 3. Testing methods

The compressive strength and strain of the mortar specimens were determined based on UNE-EN 196-1 [31] and UNE 80112 [32], respectively. All the fabricated specimens were protected from moisture loss while enclosed in moulds. The specimens were demoulded 18 h after casting so that the first records could be obtained as close as possible to the time when the mixture is transformed from liquid-like fresh mixture to solid. After de-moulding, the specimens were separated into two groups (equal number of specimens in each group) and cured in two different conditions: sealed condition (covered with aluminium foil) and an air-drying 60% relative humidity environment. The temperature was constant in both curing environments at  $22 \pm 2$  °C.

The compressive strengths of the mortars were determined using the average value of four specimens per mix, curing condition, and age. Results at seven different ages from 1 to 90 days after casting were obtained. The shrinkage of the mortars was measured from the age of 18 h, just after demoulding. Although some test procedures are useful for measuring the very early age shrinkage after set of the paste, they have not been applied in this study. Then, the shrinkage of the mortars were measured using three specimens per mix and curing condition, and each specimen had a size of 25 mm × 25 mm × 285 mm (250 mm of gauge length) (Fig. 6). The changes in length at different ages were divided by the initial length; thus, the microstrains ( $\mu\epsilon$ ) were obtained. The strain in the sealed condition (autogenous shrinkage) was recorded while a remarkable self-desiccation occurred. This is 14 days in case of CM, where self-desiccation is due to the Portland cement hydration, and 56 days in case of BCM, where pozzolanic reactions could cause long-term self-desiccation [33]. Furthermore, the weights of the specimens cured in air-drying conditions were recorded over time. The weight loss was divided by the surface of the specimens.

Additionally, the internal humidity of the mortars was evaluated by measuring the humidity in a hole in sealed specimens (Fig. 7). The specimens used in this test are cylindrical with a volume of 0.79 l (10 cm in diameter and 10 cm high). The hole is cylindrical with a volume of 0.03 l (25 mm in diameter and 60 mm high) and was made by appending a solid tube to the mould top. Therefore,

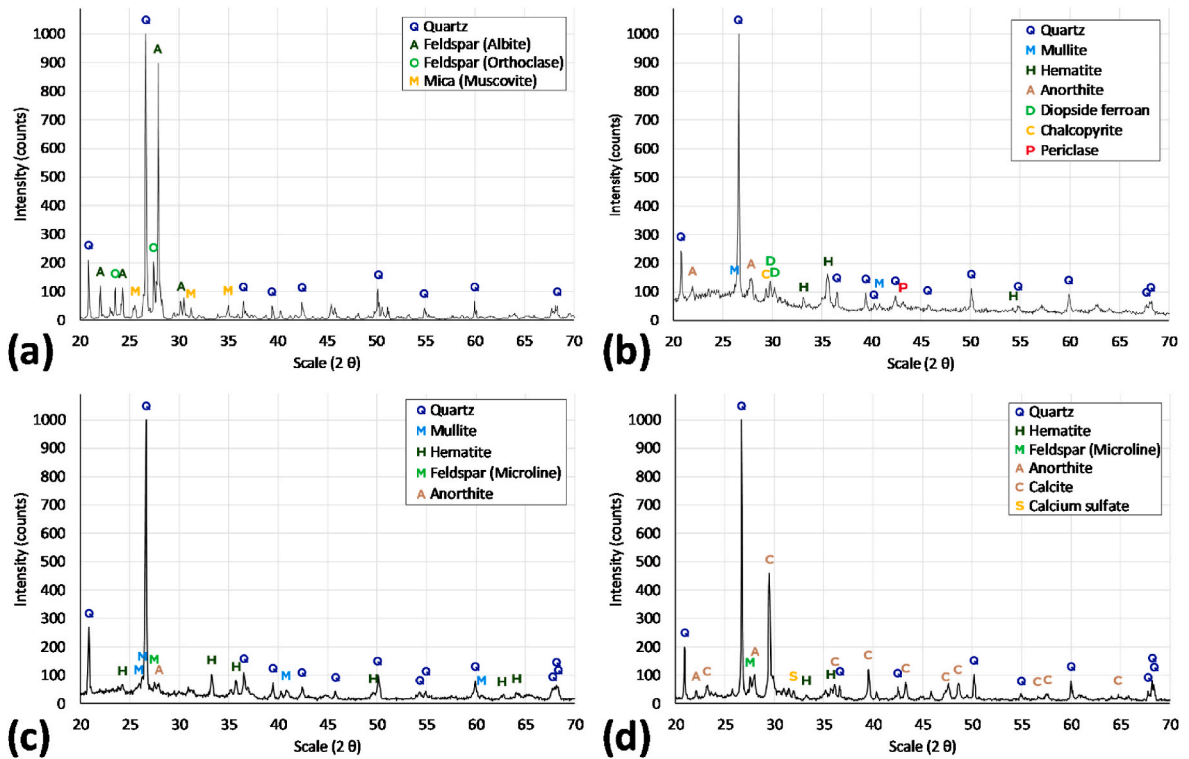


Fig. 5. X-Ray diffractograms of (a) S, (b) CBA, (c) CRA and (d) MRA.

Table 3  
Mix proportions [kg/m<sup>3</sup>].

	OPC	FA	S	ICW-S	WASPOR	ICW-WASPOR	W	HRWRA
CM-0	925	0	1112	27	0	0	239	15
CM-CBA15	925	0	945	23	80	29	239	15
CM-CBA30	925	0	778	19	161	59	239	15
CM-CRA30	925	0	778	19	266	31	239	15
CM-CRA50	925	0	556	14	443	52	239	15
CM-MRA30	925	0	778	19	259	40	239	15
CM-MRA50	925	0	556	14	432	67	239	15
BCM-0	463	327	1112	27	0	0	246	8
BCM-CBA15	463	327	945	23	80	29	246	8
BCM-CBA30	463	327	778	19	161	59	246	8
BCM-CRA30	463	327	778	19	266	31	246	8
BCM-CRA50	463	327	556	14	443	52	246	8
BCM-MRA30	463	327	778	19	259	40	246	8
BCM-MRA50	463	327	556	14	432	67	246	8



Fig. 6. Curing conditions of specimens for determining mortar strain: (a) in moulds, (b) in sealed condition (b) and (c) in air drying environment.



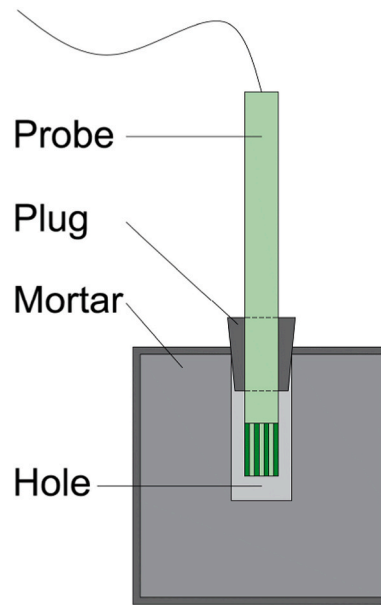


Fig. 7. Setup for the measuring of internal humidity in the mortars.

the hole is formed in fresh state and not by drilling the sample in hardened state. The thermo-hygrometer was a probe with a precision of  $\pm 0.8\%$ . It was put into the hole and adjusted with a rubber plug. The test was carried out in 2 specimens. Other researchers have measured the internal relative humidity in concrete following similar procedures [5,34–36].

It should be noted that the experimental programme of this study was conducted in two different laboratories. The baseline mixes (CM-0 and BCM-0) were fabricated and subjected to tests at both laboratories using the obtained results to contrast possible scatter of the experimental data.

## 4. Results and discussion

### 4.1. Internal relative humidity and autogenous shrinkage

The internal relative humidity in the mixes with Portland cement as the only binder is shown in Fig. 8. It can be seen that the performance of the three internal curing reservoirs correlates with their desorption capacities in environments with a high relative humidity (see Fig. 3). Among the lowest content of WASPORS, those of CBA and CRA are unable to maintain high the relative humidity inside the mortar.

The highest contents of CBA and MRA partially mitigated the autogenous shrinkage of CM (Fig. 9a). The difference between the behaviours of CM-MRA50 and CM-C.

BA30 are mainly attributed to the higher desorption capacity of MRA and the better distribution of the internal curing due to the use of a higher volume of material for providing the same amount of ICW. The key role of the desorption capacity in the internal curing of concrete has been previously pointed out by many authors [28,29]. The lowest content of any of the WASPORS did not reduce

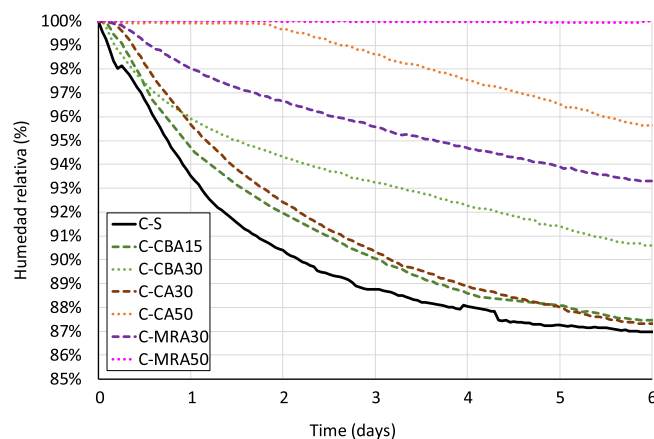


Fig. 8. Internal relative humidity inside the mortars.

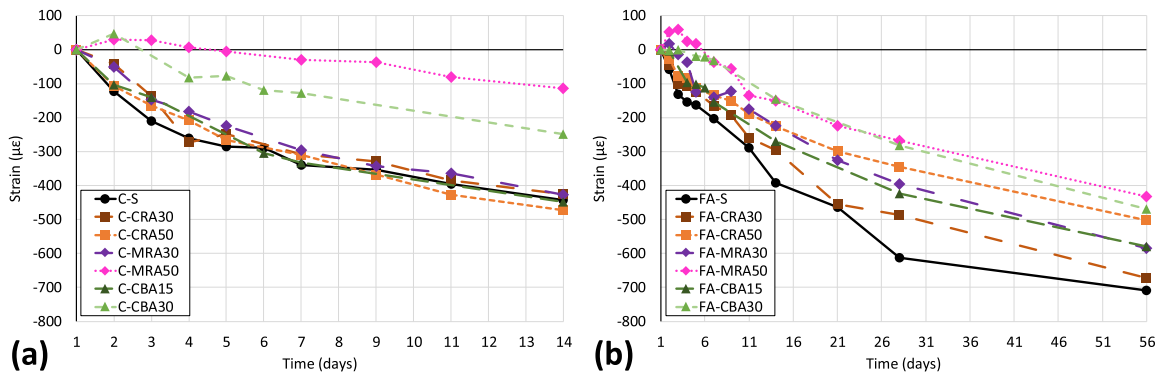


Fig. 9. Autogenous strain for (a) CM and (b) BCM.

the autogenous shrinkage. Furthermore, the addition of CRA did not cause any significant improvements in CM. This phenomenon is attributed to the loss of the micro-reinforcement provided by Portlandite, as this compound could have been consumed in pozzolanic reactions with the CRA. It must be noted that a large amount of silica in form of Quartz and Mullite is present in CRA (Table 2 & Fig. 5). Furthermore, the phenomenon of the loss of micro-reinforcement due to the consumption of Portlandite in pozzolanic reactions was pointed out before by Jensen & Hansen [37] and some researchers detected pozzolanic properties in ceramic aggregates [38–40] and fillers [41–44]. It should be noted that the CRA contained a high proportion of particles with sizes smaller than 63 µm (15%). These fine particles were expected to be more reactive because of their higher specific surface. This effect was not reported in studies where coarse CRA was successfully used as an ICWR [2,3]. Further research should be carried out in order to confirm the low efficacy of fine CRA with a high volume of filler particles to work as an ICWR.

In BCM, all the contents of all the WASPORS contributed to the partial mitigation of autogenous shrinkage (Fig. 9b). The pore network characteristics of the blended paste eased water migration from the WASPOR, producing larger spheres of influence in this case. Therefore, a sufficient volume of paste is protected for remarkable reductions in the autogenous shrinkage to be detected. In this case, the CRA performance was better than in CM. This is believed to occur because the pozzolanic reactions on the surfaces of the CRA particles were lower, owing to competition for Portlandite with the FA. In BCM, the highest contents of MRA and CBA follow parallel tendencies less for the initial autogenous swelling registered in BCM-MRA50. This difference is again attributed to the higher desorption capacity of MRA.

It should be noted that concrete containing these proposed mortar phases would attain lower shrinkage values because of the restraining effect of the coarse aggregate.

4.2. Weight loss and shrinkage in air-drying conditions

Generally, the weight loss of the specimens placed in a low RH environment increased with the WASPOR content (Fig. 10). Several researchers found that this trend was due to the higher initial water content, which is the sum of the mixing water and the ICWR [4,45]. When comparing the effect of each of them, the correlations with the desorption capacities are similar to those detected in the autogenous shrinkage. The slight increase in the weight of the BCM specimens after 56 days was caused by carbonation.

The shrinkage in air-drying conditions was generally higher in CM and BCM when any of the WASPORS was used (Fig. 11). However, slight reductions in the shrinkage of the BCM specimens were observed during the first few days when the highest CRA and MRA contents were added. This reduction in shrinkage indicated that these aggregates partially minimised the self-desiccation that co-existed with external drying in the short term. It is believed that CRA increases the drying shrinkage of CM due to the loss of micro-

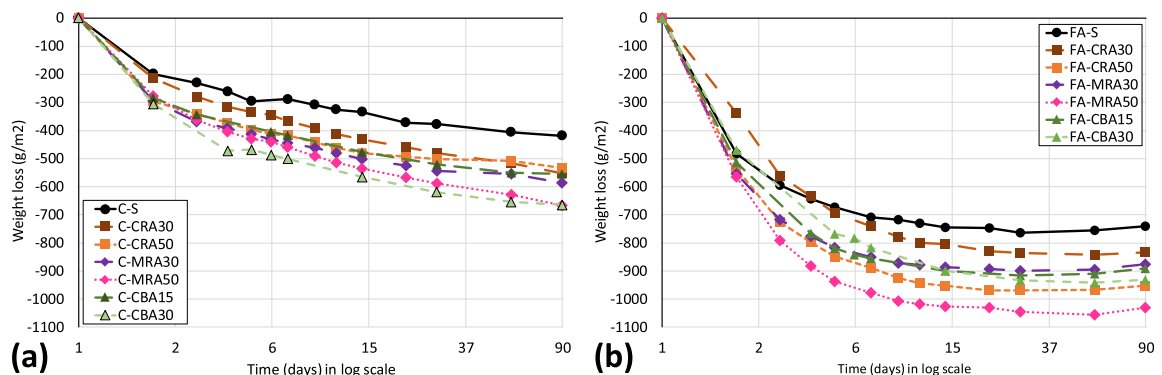


Fig. 10. Weight loss for (a) CM and (b) BCM.

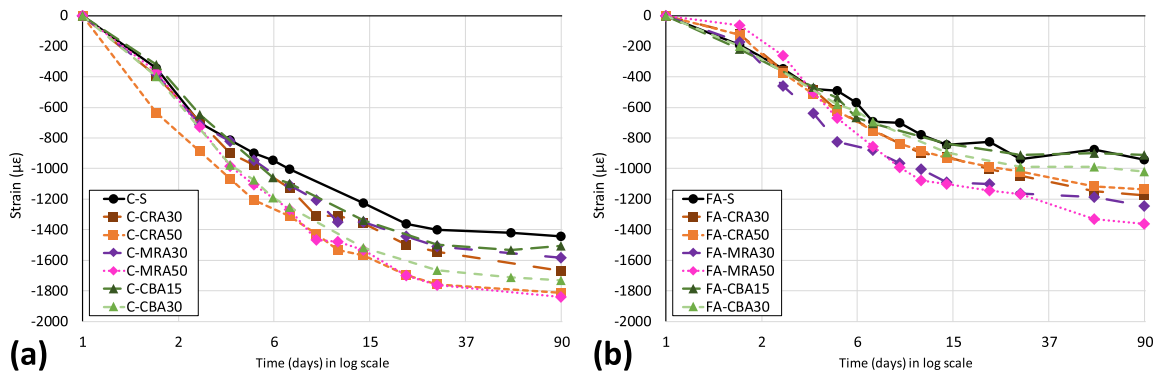


Fig. 11. Strain in air-drying conditions for (a) CM and (b) BCM.

reinforcement together with external drying. On the contrary, the consumption of Portlandite and consequent loss of micro-reinforcement occur at a low rate in BCM due to the consumption of that compound by the fly ash. Because the specimens used in this study had high surface-to-volume ratios, the shrinkage attributed to external drying, that is, drying shrinkage, might be more dominant than the self-desiccation shrinkage in the long term.

For the same weight loss, lower shrinkage values were observed when any WASPOR was used (Fig. 12). The specimens were affected by self-desiccation and external drying but only the latter implies a reduction in weight. Hence, the ratio between these two phenomena was comparatively lower in internally cured mixtures. In other words, the internally cured mixes required higher weight losses to attain the same shrinkage value because they experienced low self-desiccation. This behaviour also explained the significant difference in shrinkage between the CM and BCM specimens, with the CM specimens more significantly influenced by the self-desiccation of the paste.

Because the specimens used for measuring the shrinkage had high surface-to-volume ratios, their drying rates were higher than those of actual construction elements [46]. This factor should be considered to evaluate the mixes correctly, as drying only affects the element surface and can be minimised through conventional external curing [6].

#### 4.3. Compressive strength

The compressive strength of CM at one day using any WASPOR was lower because of the weakness of the porous particles (Fig. 13). However, this decrease in strength tended to decline over time. The sealed specimens with the lowest MRA content and highest CRA content rapidly resulted in a higher strength than in the reference specimen. In air-drying conditions, a similar trend was observed for CM-CRA50, and in this case, CM-MRA30 also attained strength higher than that of the reference at late ages. The distinct behaviours of these mixes could be attributed to the pozzolanic reactions on the surface of the CRA particles (it should be noted that MRA also contained ceramic particles), which were still possible in CM in air-drying curing conditions. Furthermore, the internal curing effect appeared not to significantly influence the compressive strength of specimens produced with CRA, as this WASPOR did not mitigate the autogenous shrinkage (section 4.1). Nevertheless, the sustained hydration due to ICW desorption from any WASPOR may have partially compensated for the negative effect of their weakness.

The only significant difference between the CM mixes cured in sealed and air-drying conditions is the strength development after 28 days. The strength development in specimens exposed to low RH environments was minimal.

The weakness of the WASPOR also contributed to the strength reduction of the BCM (Fig. 14). The specimens produced with CBA had worse values, as it was generally observed in CM. However, no disruptive performances were observed among the specimens con-

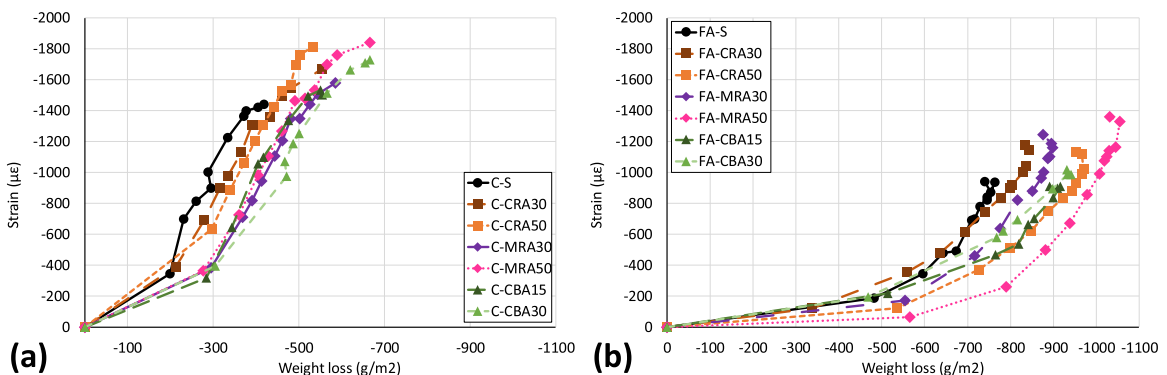


Fig. 12. Relationship between weight loss and shrinkage in air drying curing conditions for (a) CM and (b) BCM.



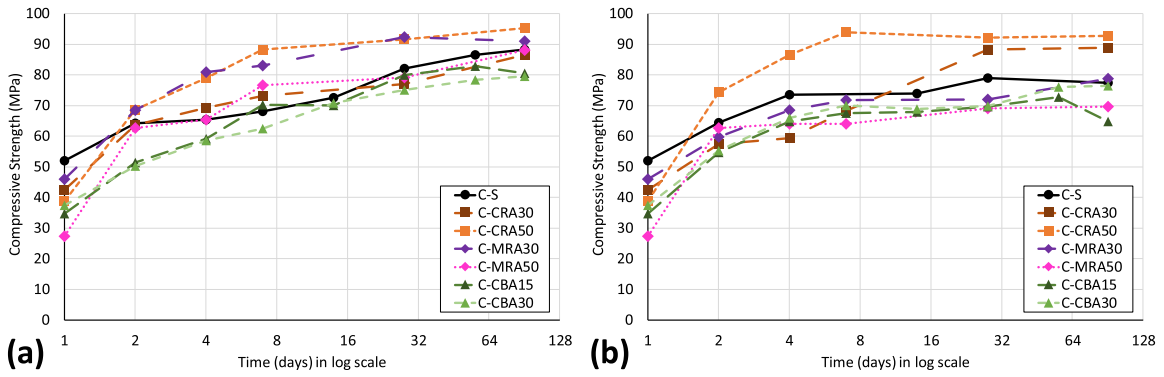


Fig. 13. Compressive strength of CM in (a) sealed and (b) air drying curing conditions.

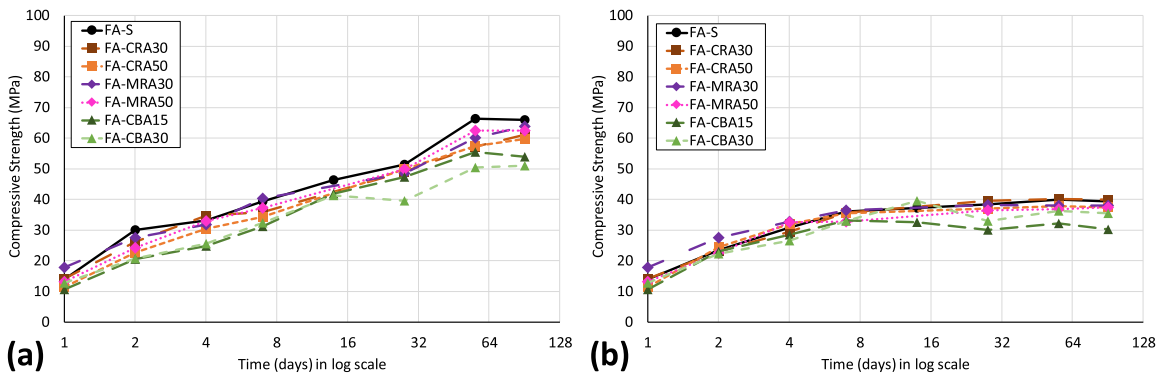


Fig. 14. Compressive strength development of BCM in (a) sealed and (b) air drying curing conditions.

taining CRA and MRA because of the unavailability of calcium hydroxide for the pozzolanic reactions on the ceramic particles. The lack of this indispensable compound was caused by its consumption during the pozzolanic reaction of the FA, resulting in the significant strength development of sealed specimens between 28 and 56 days. However, the contribution of FA to late strength development was negligible in air-drying curing conditions [47].

### 5. Internal curing efficiency

The internal curing efficiency (ICE) of mixes containing the three WASPORs was evaluated using the equation derived by Zhutovsky et al. (Section 1) to better compare their performance regarding autogenous shrinkage. The results obtained at seven days and the last recorded date are shown in Fig. 15.

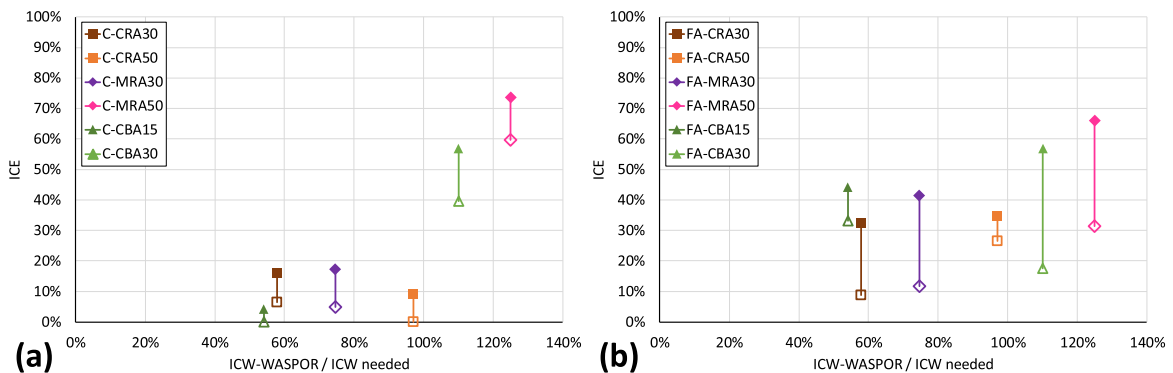


Fig. 15. Internal curing efficiency of (a) CM and (b) BCM. The full bullets represent the efficiency for CM and BCM specimens at 7 days, and the empty bullets represent the efficiency for CM and BCM specimens at 14 and 56 days, respectively.

The ICE of all the mixes decreased with time, owing to the progressive consumption of the ICW. None of the ICEs reached the 100% because of the inability of the WASPORS to desorb all the ICW effectively, or the amounts needed were infra-estimated. Furthermore, the ICE of the specimens containing high substitution ratios in the CM was significantly higher than that of the low ratios. CRA was an exception, owing to its negligible effect on the autogenous shrinkage, even when the highest percentage contents were added. Overall, the ICE of the mixes with low contents of WASPOR was higher in the BCM because of the higher permeability of the blended paste, which eased the extension of the spheres of influence of the WASPOR.

## 6. Conclusions

The effects of three WASPORS on the shrinkage and compressive strength of the mortar phase of a hypothetical high-performance concrete under different curing conditions were investigated. The following conclusions can be drawn.

- The results obtained in this research indicate that the studied CRA is ineffective for reducing the autogenous shrinkage but significantly improves the strength of the CM. The absence of this positive effect on the strength of BCM supports the idea that pozzolanic reactions on the surface of CRA particles consume Portlandite and therefore reduce the micro-reinforcement provided by this compound. The chemical characterisation and content of filler particles in CRA should be carefully evaluated before use as ICWR. However, further research should be carried out in order to confirm the low efficacy of fine CRA with a high volume of filler particles. Furthermore, the use of CRA moderately increases the shrinkage in air-drying curing conditions.
- MRA and CBA exhibit similar performance. However, MRA shows the best results as ICWR due to its higher desorption capacity and the fact that a higher volume of material is used for providing the same amount of ICW, so its particles are better distributed. The highest contents of the two aggregates significantly mitigated the self-desiccation of CM and BCM. However, the lowest contents were only effective in BCM. The effects of any replacement ratio on shrinkage in air-drying curing conditions and the compressive strength are slightly unfavourable.

Therefore, the substitution of conventional sand with 30% CBA or 50% MRA is considered adequate for the internal curing of high-performance OPC or FA-blended cement-based materials, whereas lower contents might only have a perceptible effect on the blended materials. The practical use of these WASPORS will improve concrete properties and prevent waste discarding, which will contribute to the circular economy. Among the WASPORS used in this study, CBA is the least investigated. Thus, the performance evaluation of CBA as an ICWR is recommended for future research. The possible synergies between internal curing using CBA and pozzolanic materials such as FA should be investigated in detail.

## Credit authorship contribution statement

**Roberto Rodríguez-Álvarez:** Conceptualization, Formal analysis, Investigation, Writing – original draft, Visualization. **Sindy Seara-Paz:** Formal analysis, Investigation, Writing - review & editing, Supervision. **Belén González-Fontebo:** Formal analysis, Investigation, Writing - review & editing, Supervision.

## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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