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# Waste-Based porous materials as water reservoirs for the internal curing of Concrete. A review



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# ABSTRACT

This review collates findings from more than 100 scientific publications regarding the performance of several waste-based porous materials (WASPORs) as water reservoirs for the internal curing of concrete. Results obtained by using recycled concrete aggregates, crushed ceramics, coal bottom ash, artificial waste-based aggregates, different powder materials and porous fibres were included. The influence of these WASPORs on the consistence, hydration, setting, microstructure, density, strength, modulus of elasticity, autogenous deformation, drying shrinkage and durability properties of concrete were analysed. General recommendations for suitable characterization of WASPOR and mix design are also given. The differences in water absorption capacity between the different porous materials studied have been used for explaining several of the observed phenomena. A moderate water absorption capacity together with a quick water desorption capacity were found to be among the key factors that define the internal curing efficiency of the proposed WASPORs.

# **1. Introduction**

Concrete is vastly used for the construction of building and civil structures due to its high strength and durability. It is the artificial material that mankind uses the most and approximately 10 billion cubic metres are produced each year. However, its negative impacts on our environment have been of increasing concern during the last decades [\[1\].](#page-13-0)

The production of Portland cement (PC), which is one of the key ingredients of conventional concrete, generates about the 8% of the greenhouse gas emissions in the world  $[2]$ . Furthermore, PC is an expensive component of concrete. As a consequence, intense efforts have been focused on finding less costly binder materials with a lower carbon footprint. Fortunately, other industries generate large amounts of powder wastes that have been shown to work in combination with PC, the so called supplementary cementitious materials (SCMs). Limestone powder  $[3]$ , coal fly ash  $[4-6]$  $[4-6]$ , ground granulated blastfurnace slag (GGBS) [\[7,8\]](#page-13-0) and silica fume [\[9\]](#page-13-0) are among the most common and are already included in cement and concrete standards [\[10\]](#page-13-0). The success of these materials is not only due to their suitability for replacing part of

the PC, and consequently reducing environmental and economic costs, but also due to their ability to improve the technical performance of concrete. The specific particle shape, particle size distribution, hydraulic and or pozzolanic effects etc. of SCMs can have a positive effect on different concrete properties.

However, the concrete industry has not been so motivated to find a replacement for conventional aggregates despite them not being easy to obtain in some areas  $[11-13]$  $[11-13]$  and its exploitation always implies a certain land impact [\[14\]](#page-14-0). In fact, the price and the carbon footprint of conventional aggregates is much lower than that of PC [\[15\]](#page-14-0). The main motivation for the reduction in the use of conventional aggregates comes therefore from industries that generate granular by-products that could work as their substitutes. Thus, the performance of many types of waste-based alternative aggregates in concrete has been investigated and general conclusions regarding their influence on different properties have been drawn [\[16](#page-14-0)-19]. However, value added applications, i.e. concretes with specific improvements achieved thanks to the incorporation of the by-products, should be identified to decisively encourage the use of any alternative aggregate out of the laboratory. In this direction, the use of different waste-based porous materials (WASPORs) as

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<span id="page-1-0"></span>internal curing water reservoirs (ICWRs) have been proposed in different studies that are collated in the present review. Pellets made of powdery by-products, discarded natural fibres and porous SCMs, which can also work as ICWRs, have also been included.

Internal curing is the supply of water within a cementitious mixture using pre-wetted lightweight aggregates, or other materials that readily release water from within the particles, thereby mitigating selfdesiccation and sustaining hydration [\[20\]](#page-14-0). Self-desiccation is the reduction in the internal relative humidity of concrete due to the progressive hydration of cement and it is considered as the main driving force of autogenous shrinkage [\[21\].](#page-14-0) This phenomenon is a remarkable problem in high performance concrete (HPC) with a low water to binder ratio [\[22,23\]](#page-14-0). Furthermore, the sustainment of hydration is important not only in HPC, but also in concretes with higher water to binder ratios and where external curing is difficult. Via internal curing, poor cement hydration caused by limited water supply during the external curing process can be offset [\[13,24](#page-13-0)–26].

# **2. Research significance**

The effects of using conventional lightweight aggregates as ICWRs have been extensively studied and the obtained results have already been reviewed [\[27\]](#page-14-0). The use of WASPORs with the same purpose has been claimed to be economically and environmentally advantageous [\[28\]](#page-14-0). Indeed, the internal curing effect via WASPORs is a topic of growing interest (Fig. 1).

The effect of internal curing has been detected not only in conventional concrete and HPC, but also in fibre reinforced concrete [\[29,30\]](#page-14-0), earth-moist concrete [\[31\]](#page-14-0) and even in 3D printing concrete [\[32,33\]](#page-14-0). Furthermore, the combination of internal curing via WASPORs with other shrinkage mitigating strategies such as the use of expansive agents has already been proposed [\[34,35\]](#page-14-0) and the use of these alternative particles with purposes other than internal curing has been studied. In this direction, some pre-wetted WASPORs have been proven to promote autogenous self-healing in concrete [\[36\]](#page-14-0) and some authors are already exploring the internal supply of substances other than water via WAS-PORs, such as sulphate activators [\[37\]](#page-14-0) and shrinkage reducing admixtures [\[38\]](#page-14-0). Although all these new approaches are continuously developing, there is a need to review and consider current results obtained using WASPORs as ICWRs. Therefore, the present study aims to collate the findings provided by different works regarding the use of several WASPORs as ICWRs and identify the key factors that influence their internal curing efficiency. A comparison between their characteristics and their influence on different concrete properties has been carried out and general recommendations for suitable characterisation of WASPOR and mix design are proposed.



*Construction and Building Materials 299 (2021) 124244*

# **3. WASPOR classification and characteristics**

The internal curing effect has been detected in concrete when using a wide diversity of pre-wetted WASPORs. A classification based on their source is proposed in Table 1 in order to facilitate the comparison between the results obtained in different studies.

The physical and chemical characteristics of the different types of WASPOR are collated in the next sections.

# *3.1. Physical properties*

A wide range of particle sizes can be found among the studied WASPORs [\(Fig. 2](#page-2-0)). Usually, CON and CER can be classified as coarse aggregates and CBA can be classified as fine aggregate. Moreover, coarse and fine ART can be found. The size of Lechuguilla fibre is defined by its mean length and diameter, which are 5.0 and 0.3 mm respectively [\[82\]](#page-15-0). The WASPORs that are classified as POW have a particle size below 0.2 mm or are used as a substitute for the conventional binder.

CON ([Fig. 3](#page-2-0)**a**), AC, CER [\(Fig. 3](#page-2-0)**b**) and CBA [\(Fig. 3](#page-2-0)**c**) are irregular in shape and rough in texture. However, ART ([Fig. 3](#page-2-0)**d, e & f**) with rounded shape and smooth surface can be found. FIB, presented in [Fig. 3](#page-2-0)**g**, has a rough and porous texture that reflects its vegetal origin. Regarding POW, it shows a wide variety of shapes and textures depending on its nature ([Fig. 3](#page-2-0)**h-l**).

The water absorption capacity of the WASPORs is a key property for them to be used as ICWRs. It is typically assessed after 24 h of water immersion because the wetting of WASPORs during that period of time is normally sufficient for it to reach a high saturation degree and it is a reasonable time for a real field situation. However, longer wetting periods [\[29,43,54,55,60\]](#page-14-0) or even vacuum saturation [\[73\]](#page-15-0) have also been proposed. The precise determination of the water absorption capacity can be challenging. Indeed, some standardized test methods where the particles are dried with an air flow after soaking [\[86\]](#page-15-0) are inapplicable due to the low density of the particles. Procedures where particles are dried with paper towels  $[87]$  or the retainability method  $[71,88,89]$  are preferred for granular WASPORs. In case of POW, retainability methods have also been proposed using pre-wetted filter paper to drain the surplus water after soaking [\[78\].](#page-15-0) It must be noted that different procedures can lead to different results and they can consider surface-bond or even inter-particle water as absorbed.

The water absorption capacity of different types of WASPOR are plotted against the corresponding specific gravities in [Fig. 4](#page-3-0)**a**. Note that the water absorption capacity is presented by volume and not by mass. This criterion is considered as more appropriate for studying the water storage of an ICWR [\[62\].](#page-14-0) The water absorption capacity-to-specificgravity ratio follows a similar trend in CON and CER, whereas the curves that correspond to AC and CBA are moved to the left, i.e. lower



**Table 1** 



 $<sup>(1)</sup>$ Includes one case of CBA that is cold bonded with cement and coal fly ash</sup>

[\[69\].](#page-15-0) (2)Includes one case of bottom ash from a paper factory boiler where coal is burnt together with other wastes from the paper production [\[72\]](#page-15-0).

**Fig. 1.** Cumulative number of papers regarding internal curing and internal curing with some types of water reservoirs.

<span id="page-2-0"></span>

specific gravities are needed for obtaining similar water absorption capacities. Furthermore, ART and POW show only slight changes in the water absorption when their specific gravities are different. The latter,

together with FIB, are much more absorptive than the others. This difference can be partially attributed to the much higher specific surface of POW, but also to the specific test methods used for determining the water absorption capacity of this type of WASPOR. The difference in specific surface between fine and coarse particles, however, does not lead to a significant change in the trend [\(Fig. 4](#page-3-0)**b**). The higher specific surface of fine aggregates might be compensated with for by a lower porosity due to the exposure of internal pores after crushing processes involved in their production ([Fig. 5\)](#page-3-0).

An effective ICWR must show not only a high water absorption capacity but also a high water desorption capacity at high relative humidity (RH) [\[90\].](#page-15-0) Different procedures for its determination can be found in the literature. For instance, the particles can be submitted to water immersion for different times or even vacuum saturated before the desorption test. Furthermore, the desorption test itself can be carried out at different temperature conditions. [Fig. 6](#page-3-0)**a** collates the results obtained following methods based on ASTM C1761 [\[20\]](#page-14-0) and ASTM C1498 [\[91\]](#page-15-0). In these cases, the desorption at a specific RH is considered as the proportion of absorbed water that is released at that RH until an equilibrium is achieved. However, different criteria have been used for the definition of the equilibrium. Actually, some authors establish arbitrary time periods for the particles to remain at each RH step ([Fig. 6](#page-3-0)**b**). Furthermore, a



**Fig. 3.** Images of different WASPORs: (a) CON [\[84\]](#page-15-0), (b) CER [\[58\]](#page-14-0), (c) CBA [\[67\]](#page-15-0), (d) GGBS based ART [\[74\]](#page-15-0), (e) fly ash based ART [\[75\],](#page-15-0) (f) red mud and clay based ART  $[76]$ , (g) lechuguilla fibre  $[82]$ , (h) biochar 0.125 to 0.25 mm  $[78]$ , (i) coal ash perforated cenospheres  $[79]$ , (j) DWTW  $[80]$ , (k) red mud  $[81]$  and (l) rice husk ash (RHA) [\[85\]](#page-15-0). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

<span id="page-3-0"></span>

**Fig. 4.** Relationship between specific gravity and water absorption capacity of several WASPORs. Results grouped by (a) type of WASPOR and (b) particle size [\[24,25,43,45,46,48,52](#page-14-0)–57,29,58,60–62,64–69,34,70–76,78–80,35,81,82,36,39–42]



**Fig. 5.** Particle crushing and reduction in the volume of pores.



**Fig. 6.** Desorption capacity of WASPOR: (a) Results grouped by type of WASPOR [\[13,29,35,53,72,73,92,93\],](#page-13-0) (b) example of CBA in arbitrary time intervals [\[68\]](#page-15-0), (c) example of CER [\[62\]](#page-14-0) and (d) example of ART [\[76\]](#page-15-0).

different approach has been proposed by Zou et al. ([Fig. 6](#page-3-0)**c & d**) where the time needed for the wetted particles to completely dry at different RH is recorded. The latter method is considered as more appropriate as it does not only determine the amount of desorbed water but also the desorption rate at different RH.

From results collated in [Fig. 6](#page-3-0)**a**, it can be inferred that ART has the highest desorption capacity, what is understandable as ART is specifically designed with that purpose. The POW might have a narrower pore network that tends to retain water more tightly. The smaller the size of a particle, the smaller its largest pores can be. This justifies the results shown in [Fig. 5](#page-3-0), where POW, which is a biochar, shows the lowest desorption capacity. Furthermore, the performance of CON, CER and CBA is similar.

Alternative methods to study the desorption of water by WASPORs in cement paste have also been proposed. For instance, Kim et al. measured the distance that a fluorescent ink could reach in cement paste when desorbed from CBA (Fig. 7**a**) and Lura et al. visualized the movement of water inside mortars with pre-wetted ART by neutron tomography (Fig. 7**b**).

The desorption capacity of any WASPOR depends on the pore structure of its particles, with larger pores leading to higher desorption, but also on their specific surface, being higher when the ratio "specific surface to stored water" is higher [\[62\]](#page-14-0). This phenomenon is illustrated in Fig. 8, where two particles with the same specific volume of pores with the same diameter but different specific surface are illustrated. The one on the right shows a higher desorption capacity due to the shorter depth of the pores.

Some authors have proposed the study of the pore structure of WASPORs by mercury intrusion porosimetry (MIP) as an indirect, fast method to get an approximation to the desorption properties [\[73\]](#page-15-0). They have also stated that the proportion of ink-bottle shaped pores in WASPORs can be high (Fig. 9). However, some authors have pointed out that WASPORs can suffer deformations or even break during a MIP test due to their weakness [\[78\].](#page-15-0)

Certainly, a low crushing strength of some WASPORs has been reported [\[29,74,75\]](#page-14-0), as a direct consequence of their porous structure. However, it must be noted that the crushing value of CER can be higher than that of other conventional light-weight aggregates [\[56,58\].](#page-14-0) On the other hand, the stiffness of natural organic fibres used as ICWRs has been found to be so low that they deform during changes in their water content [\[82\]](#page-15-0).

#### *3.2. Chemical composition*

The chemical composition of WASPORs is less important than their physical properties in regard to their role as ICWRs. However, it can have relevant consequences on the development of the interfacial transition zone (ITZ) [\[94,95\]](#page-15-0). Some examples of chemical compositions of WASPORs are shown in Fig. 10. The most common elements are



**Fig. 8.** Particles with the same volume of pores with the same diameter but different specific surface.



**Fig. 9.** Pore size distribution of ART, measured by MIP [\[73\]](#page-15-0) 



[\[51,54,85,57,66](#page-14-0)–68,70,79–81].



**Fig. 7.** (a) desorbed fluorescent ink from CBA to cement paste  $[68]$ ; (b) ART (contours in yellow) and water movement in mortar (red = water losing region, blue = water gaining region) [\[73\].](#page-15-0) (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

silicon and aluminium. However, some WASPORs can be very different in nature. That is the case of DWTW, which has a high content of calcium and a high loss on ignition (LOI), or biochar, which is mainly composed of carbon, hydrogen and nitrogen [\[78\].](#page-15-0)

# **4. Design of internally cured HPC via pre-wetted WASPOR**

Experimental programmes with the specific objective to study the ability of WASPORs to mitigate self-desiccation are usually carried out in high performance cement based materials designed as per the specification shown in Table 2. The most significant characteristics are the high content of binder, often incorporating SCMs, and the low water to binder ratio. The common maximum substitution rates of conventional aggregates or binders by the different WASPORs are shown in [Table 3](#page-6-0). It must be noted that the internal curing effect has been detected in concretes with other design characteristics, for instance with higher water to binder ratios, where the principal objective was not to study the effect of this technique on the autogenous shrinkage [\[39,43,49,51,66,70,96\]](#page-14-0). Those works have not been considered either in Table 2 nor in [Table 3](#page-6-0).

These replacement ratios are commonly established based on the equations proposed by Bentz et al.  $[97]$  Eqs.  $(1)$  and  $(2)$ . It must be noted that these equations were specifically designed to calculate the amount of LWA that would replace the same volume of normal weight aggregates in concrete. For the few cases where the ICWR substitutes part of the binder system, they are to be taken only as a broad approximation and the effects on the amount of reactive components should be carefully considered.

$$
V_{LWA} = \frac{V_{wat}}{S\hat{\mathbf{A}} \cdot \boldsymbol{\phi}_{LWA}}\tag{1}
$$

Where:

VLWA is the amount of LWA needed for the effective internal curing of concrete

Vwat is the internal curing water needed for an amount of concrete (vol%) (see Eq. (2))

S is the saturation degree of LWA (%)

ФLWA is the absorption capacity of LWA. This should be corrected by a factor based on the desorption capacity of the LWA at a high RH (92% − 97%) [\[98\]](#page-15-0).

$$
V_{wat} = \frac{Cf\hat{A} \cdot CS\hat{A} \cdot a_{max}}{\rho}
$$
 (2)

Where:

Cf is the amount of cement in concrete  $\frac{\text{kg}}{m^3}$ 

CS is the chemical shrinkage associated to the cement (vol%)

 $\alpha_{\text{max}}$  is the maximum expected degree of hydration (%)

 $ρ$  is the density of water (kg/m<sup>3</sup>)

The replacement of aggregates or binders with any WASPOR is commonly applied by volume due to the notable difference between the specific gravities of the conventional material and its substitute. Replacement by mass must be avoided, otherwise significant distortions in the mix proportions may occur. Thus, the results obtained in studies where the substitution rates are fixed by mass are not collated in the comparative graphs shown in next sections. Furthermore, the

**Table 2** 

Mix design parameters of internally cured high performance cement based materials via WASPORs.

|  | Concrete                 | Mortar phase  |
|--|--------------------------|---------------|
| Binder dosage $(kg/m^3)$                         | $600 \pm 200$            | $800 \pm 150$ |
| Water to binder ratio $(g/g)$                    | $0.32 \pm 0.05$          |               |
| Paste to aggregate ratio $(m^3/m^3)$             | $1.8 \pm 0.5$            | $1.0 \pm 0.5$ |
| Coarse to fine aggregate ratio $(m^3/m^3)$       | $1.1 + 0.5$              |               |
| Most common supplementary cementitious materials | None/fly ash/silica fume |               |

substitution of highly reactive SCMs by inert ICWRs can lead to a lack of comparability with the plain counterparts. Therefore, only the works where the POW-type WASPORs replace inert materials or low reactive binders have been included in the comparative graphs. However, representative findings of any study regarding any property linked with the internal curing effect are commented upon.

# **5. Properties of internally cured concrete via WASPOR**

The main objectives of internal curing are the mitigation of selfdesiccation and the sustainment of hydration. The effectiveness for the former can be evaluated by studying the shrinkage of concrete. The latter is also related to the mechanical and durability properties. Furthermore, the influence of pre-wetted WASPORs on other properties is addressed in order to confirm the feasibility of its use.

#### *5.1. Consistence*

There are two main factors that affect the consistence of concrete with WASPORs: (i) the shape of the particles and (ii) the absorption/ desorption of water from/to the paste. A decrease in fluidity has been recorded in some studies where CBA and POW were used due to their irregular shape together with the ongoing water absorption while mixing [\[66,81\]](#page-15-0). It must be noted that, despite the use of the WASPORs in pre-wetted condition, they may not be completely saturated due to the difficulties in precisely determining their water absorption capacities. The adsorption of superplasticizer on the surface of CON and CER has also been considered as a cause for the increase in the consistence of concrete [\[51\].](#page-14-0) This trend can have an especial impact on cement based materials with high flowability requirements, such as 3D printing concrete, where reductions in the extrudability when incorporating CON have been reported [\[32\].](#page-14-0) However, the release of water while mixing has been considered the cause for a fluidity increase in cases of concrete incorporating CON [\[24\]](#page-14-0), AC [\[52\]](#page-14-0) and CBA [\[67\]](#page-15-0). The centrifugal force caused by the mixing process can contribute to this phenomenon [\[99\]](#page-15-0). A precise water dosage can lead to negligible effects on the consistence of concrete [\[43\].](#page-14-0) Nevertheless, it must be noted that the precise determination of the absorption and desorption properties is challenging and different test methods have been proposed for its determination due to the lack of a standardized procedure (section 3.1).

## *5.2. Hydration and setting*

Cement based materials show a lower rate of hydration when their water to cement ratios are increased due to the higher dilution of alkali and hydroxide ions in the pore solution [\[100\]](#page-15-0). This also seems to be applicable to internally cured concrete [\[101\].](#page-15-0) Thus, the hydration peak, normalized by mass of cement, is lower and delayed when using prewetted WASPOR ([Fig. 11](#page-6-0)**a**) due to the increase in the total water content. It must be noted that this figure compares the results obtained in samples with the same amount of cement. However, the cumulative heat released by internally cured materials with low water to cement ratio is normally slightly higher than the reference ([Fig. 11](#page-6-0)**b**) and a higher de-gree of hydration at late ages has been reported when using CER [\[56\]](#page-14-0), CBA [\[102\]](#page-15-0) and POW [\[80\]](#page-15-0) in cement-based materials with a water to cement ratio as low as 0.3. This enhancement of the cement hydration can have a positive impact on the strength and the durability properties. Therefore, it is considered as one of the main advantages of the internal curing technique.

A parallel delay in setting has also been attributed to the higher total content of water in HPC [\[35,53\].](#page-14-0) Nevertheless, other possible causes related with the chemical composition of the WASPOR have also been discussed [\[79,82\].](#page-15-0) Some examples of the influence of different WAS-PORs on the initial and final setting times are shown in [Fig. 12](#page-6-0)**a** & [Fig. 12](#page-6-0)**b**.

#### <span id="page-6-0"></span>**Table 3**

Common maximum substitution rates of conventional constituents by type of WASPOR.



 $(1)$  Most works that detect the effect of internal curing via CON use coarse particles. However, the only two works found where concrete is specifically designed for the study of the internal curing effect use fine parti

<sup>(2)</sup> The references [\[79\] and \[80\]](#page-15-0) are studies where the fine aggregate is substituted by cenospheres and drinking water treatment waste, respectively. The reference [\[81\]](#page-15-0) corresponds to a study where fly ash is substituted by red mud with pozzolanic properties.



**Fig. 11.** (a) Early age heat of hydration rate for concrete with POW [\[79\]](#page-15-0) and (b) cumulative heat released in mortars with CBA [\[72\].](#page-15-0) In both cases, the heat is normalized by mass of cement. In (b) the CBA (BA or BTA) substitutes the 40% of the sand by volume.



**Fig. 12.** Examples of the initial and final setting of cement based materials with (a) CER [\[53\]](#page-14-0) and (b) FIB [\[82\]](#page-15-0) 

#### *5.3. Microstructure and density*

An increase in the air content of concrete has been detected when increasing the content of CON [\[24\]](#page-14-0). In general, the incorporation of any WASPOR increases its porosity. A higher porosity leads to a lower density, which can have some positive side effects such as the reduction of the dead load in structures or the reduction in thermal conductivity. The latter phenomenon has been reported when using CBA [\[103\]](#page-15-0). However, the internal curing effect, which enhances cement hydration and pozzolanic reactions, has resulted in denser concrete when using CER [\[55,56,60\].](#page-14-0)

Positive effects of internal curing on the ITZ, (where cement hydration and pozzolanic reactions are promoted), have been described when using CON [\[51\],](#page-14-0) CER [\[51\]](#page-14-0) and CBA [\[67\]](#page-15-0). The idea of a denser ITZ when using LWAs as ICWRs in HPC has been generally supported [\[27,104\]](#page-14-0). Furthermore, Kim et al. [\[67\]](#page-15-0) explained how CBA slightly absorbs the surrounding cement paste so that the hydration products and the WASPOR properly merge across the ITZ. The rough surface of some LWA has also been considered to enhance the bond with the paste due to a noticeable mechanical interlocking [\[105\]](#page-15-0). Nevertheless, it must be

noted that detrimental effects on the ITZ may be observed if the surface of the pre-wetted particles is not completely dry [\[106\]](#page-15-0). Again, the correct determination of the water absorption capacity of the ICWR is considered a matter which must be carefully addressed.

# *5.4. Mechanical properties*

The effectiveness of internal curing is influenced by the external curing conditions and the curing sensitivity of the specimens. Thus, the internal curing may not have noticeable positive effects next to the surface of specimens cured at a low RH [\[52\].](#page-14-0) However, some studies have concluded that internal curing reduces the curing sensitivity of concrete, at least regarding the RH curing conditions [\[40,71\].](#page-14-0) Nevertheless, given the variety of curing methods among the collated studies, their results are compared without taking into account the differences regarding the size or shape of the specimens nor their external curing conditions.

The introduction of low doses of internal curing water in concrete  $( $0.1 \text{ m}^3$  per m<sup>3</sup> of binder) via WASPOR leads to only slight changes$ (decreases or increases) in the 28-day compressive strength [\(Fig. 13](#page-7-0)**a**).

<span id="page-7-0"></span>

Fig. 13. Variation in the compressive strength of internally cured cement based materials when increasing the amount of internal curing water [24,29,44–46,48,52–56,58,34,59–[65,67,69,70,35,71,72,74](#page-14-0)–76,79–82,36,39–43]. Results grouped by (a) type of WASPOR and (b) WASPORs with different water absorption capacity.

This phenomenon is generally attributed to the coexistence of the detrimental effect of the weakness of any WASPOR and the beneficial effect of internal curing on the paste and the ITZ, i.e. the promotion of hydration and the consequent densification. However, the strength generally decreases when the content of internal curing water increases. Collated results regarding the use of ART indicate that this type of WASPOR has a lower effect on compressive strength than others. CON and CBA are the most prone to decrease it. CON might be relatively weak due to the presence of an old ITZ between the recycled aggregate and the old paste [\[39,48\].](#page-14-0) In contrast, CER and POW are more prone to contribute to the increase in compressive strength, at least when using low volumes. This could be related with possible pozzolanic reactions on the surface of CER, as other research suggest [\[94,95\]](#page-15-0). Moreover, the partial replacement of Portland cement by POW with different particle size distributions and shapes could lead to a better packing [\[107,108\]](#page-15-0). A higher strength gain at late ages has been attributed to the sustainment of hydration and improvement of the ITZ via pre-wetted CON [\[96,109\]](#page-15-0), CER [\[54,61\],](#page-14-0) CBA [\[70\]](#page-15-0) and POW [\[79\].](#page-15-0) This distinctive performance should be found in concretes with a low water to binder ratio, as more cement remains unhydrated. However, this differentiation was not possible to draw in the collated data (Fig. 14).

Furthermore, the strength of the WASPORs themselves is believed to be the most decisive characteristic for determining their effect on the strength of concrete. This property can be considered as inversely proportional to the pore volume in the particles. However, similar trends are found in the compressive strength when using WASPORs with different water absorptions / porosities. This is due to the fact that fewer particles of WASPOR are generally used for the internal curing of concrete when they are more porous or absorptive. Thus, similar strength reductions are found for the same content of internal curing water



**Fig. 14.** Variation in the ratio between the compressive strength at 7 days and at 28 days when increasing the amount of internal curing water [\[24,34,](#page-14-0)  52–55,58–[63,35,64,65,75,76,78](#page-14-0)–80,82,39–41,43,45,46,48].

(Fig. 13**b**).

Mechanical properties other than compressive strength have been less studied. However, they might have a decisive influence on the ability of concrete to withstand shrinkage and avoid cracking. The flexural strength generally decreases when WASPORs are used, similarly to compressive strength, due to the weakness of the alternative particles [\[39,51,62,75,102\].](#page-14-0) However, the internal curing effect and pozzolanic reactions on the surface of the aggregates can lead to similar results to baseline mixes  $[76,81]$  or even slightly superior ones  $[24,51]$ . The same phenomena explain changes in splitting-tensile strength, where values obtained when increasing the content of WASPOR can be worse [\[29,40,48,58,74,102\]](#page-14-0) or similar to those of the conventional concrete [\[36,76\].](#page-14-0)

The reduction of the modulus of elasticity is clearly predominant (Fig. 15), with only a few exceptions when WASPORs with a specific gravity similar to that of conventional aggregates are used [\[46,74\]](#page-14-0). It must be noted that this reduction in the modulus of elasticity does not have to be necessarily unfavourable, at least from the cracking propensity viewpoint, as less stiff mixtures tend to better relax the internal shrinkage stresses.

Finally, lower abrasion resistance has been reported in concrete with CON, again due to the weakness of the WASPOR [\[24\].](#page-14-0)

In short, it can be stated that the use of low contents of WASPOR with a specific gravity approximately between 1.5 and 2.0  $\frac{g}{cm}$  can lead to slight improvements in the strength of concrete. In general, for lower specific gravities, the effect is detrimental due to the low crushing value of these particles. The modulus of elasticity is the property that suffers the clearest reduction due to the low modulus of elasticity of the WASPORs.



**Fig. 15.** Variation in the modulus of elasticity at 28 days when increasing the amount of internal curing water [\[29,36,72,74,76,110,40,48,51,56,57,63,65,67\]](#page-14-0)

#### *5.5. Autogenous deformation*

The autogenous strain of concrete is defined as its bulk deformation under sealed and isothermal conditions, with no external forces applied [\[111,112\].](#page-15-0) Before setting, the autogenous strain is equivalent to the chemical shrinkage (settlement excluded). The volume reduction from reactants to products in cement hydration is translated into an external deformation due to the lack of stiffness of the fresh mix [\[112\].](#page-15-0) After setting, autogenous shrinkage is mainly caused by paste self-desiccation. The progressive consumption of water by cement hydration reduces the RH in the pores of concrete, i.e. the paste self-desiccates [\[113\],](#page-15-0) and generates internal pressures leading to an external contraction [\[114\]](#page-15-0). These internal pressures are more intense in narrow pores of cementbased materials with a low water to binder ratio. Different phenomena can cause pressures with the opposite sign of those caused by selfdesiccation and even surpass them causing autogenous swelling. Some of those phenomena are the reabsorption of internal and external bleeding [\[115,116\]](#page-15-0), the ingress of internal curing water in unsaturated C-S-H gel sheets in pastes with a low water to cement ratio [\[53\]](#page-14-0), the migration of water stored in pre-wetted aggregates to the paste pores [\[117\],](#page-15-0) the growth of portlandite crystals [\[22\]](#page-14-0) or the formation of ettringite [\[118\].](#page-16-0)

A few works have concluded that internal curing via WASPORs decreases the early autogenous shrinkage in high performance concrete (Fig. 16**a and b**). Delsaute et al. [\[119\]](#page-16-0) points out that the effect of CON coarse aggregate is more beneficial than that of CON fine aggregate due to the more detrimental effect of the latter on the modulus of elasticity of concrete. Indeed, Qadri et al. [\[47\]](#page-14-0) obtained inconsistent autogenous deformations when using fine CON.

As stated earlier, the RH inside sealed concrete decreases over time due to the consumption of water by hydration and pozzolanic reactions, i.e. self-desiccation occurs [\[113\]](#page-15-0). Internal curing has been proven to mitigate the self-desiccation of paste, contributing to the sustainment of the internal RH [\[27,120\]](#page-14-0). Several works have concluded via direct measurement of RH inside HPC that internal curing with WASPORs is valid for this purpose via ([Fig. 17](#page-9-0)).

The use of pre-wetted WASPORs prevents self-desiccation and therefore decreases autogenous shrinkage at different ages in concrete with a low mixing water to binder ratio ([Fig. 18](#page-9-0)**a**–**c**). The reduction of shrinkage over 100% means swelling.

The type of WASPOR does not generally govern its effectiveness in reducing autogenous shrinkage. Only especially poor results have been reported for CON [\[92\],](#page-15-0) which may suggest that the internal curing via this type of WASPOR is ineffective. The pores in the old carbonated paste might be narrower than the pores in the surrounding new paste. This hinders the migration of water from CON and the consequent mitigation of self-desiccation. This idea has been supported in some studies [\[40\]](#page-14-0)  whereas some others suggest that CON could properly work as an ICWR [\[109\].](#page-15-0) Furthermore, other authors state that the internal curing effect

can be detected in concrete with CON even when it is not used specifically used for that purpose [\[40\].](#page-14-0) It must be noted that WASPORs with pores larger than those in the surrounding paste desorb water more easily and therefore contribute to maintain the high RH. Furthermore, particle size does not seem to be a decisive parameter for the distribution of the results. Smaller particles guarantee a low spacing between them, i. e. a better distribution of the internal curing water [\[97\].](#page-15-0) However, they can also show a finer porosity (see [Section 3.1\)](#page-1-0). Therefore, the particle size of an ICWR has been identified by other authors as unimportant for determining the efficiency for reducing autogenous shrinkage [\[121\]](#page-16-0). However, the anticipated advantages of the possible restraining effect of FIB have not been detected in the collated results. The WASPORs with lower water absorption capacity are linked to higher reductions in autogenous shrinkage (with the exception of CON), as can be seen in [Fig. 18](#page-9-0)**d**.

A lower water absorption capacity means a higher volume of WAS-POR is needed to provide the same amount of internal curing water, i.e. better distribution and lower spacing ([Fig. 19\)](#page-10-0). Therefore, this approach seems to be preferable to using fewer particles with higher water absorption.

#### *5.6. Shrinkage in air-drying conditions*

Shrinkage in air-drying conditions is triggered by different phenomena such as drying, i.e. moisture loss to the environment, selfdesiccation and carbonation. The degree of contribution of each of these to the recorded shrinkage depends on many factors. One of them is the size and shape of the specimen. Thus, shrinkage in air-drying conditions in specimens with a high surface to volume ratio (H) is composed of a higher proportion of drying shrinkage than specimens with a low surface to volume ratio (L). Thus, H and L specimens are differentiated when representing the variation of shrinkage in air-drying curing conditions in [Fig. 20.](#page-10-0)

Shrinkage reductions for an increasing content of pre-wetted WAS-POR are more common among L specimens, as they show a higher selfdesiccation that can be mitigated by internal curing. However, H specimens mainly shrink due to drying. Internally cured specimens suffer more severe drying due to the higher initial total water content and, therefore, are more prone to suffer drying shrinkage. It must be noted that, at earlier ages, some L specimens containing CER show an increase in shrinkage, whereas some H specimens containing CON show a decrease [\(Fig. 20](#page-10-0)**a**). This might be directly related with the desorption properties of these two WASPOR. Internal curing water stored in CER migrates to the paste easily. Thus, it is available for mitigating selfdesiccation but also to leave the specimen in case it is not sealed. In contrast, internal curing water stored in CON might be retained in the particles, therefore neither contributing to mitigation of self-desiccation nor to external drying. In any case, all H specimens had increased shrinkage at late ages (56 or 90 days) when they contain higher amounts



**Fig. 16.** Early age autogenous shrinkage in (a) concrete with CON [\[119\]](#page-16-0) and (b) concrete with CER [\[60\].](#page-14-0) In (b), the number after "HF" denotes the percentage of fly ash on the binder and the number after "G" the volume proportion of the coarse aggregate substituted by CER.

<span id="page-9-0"></span>

**Fig. 17.** Examples of internal RH in HPC with different types of WASPOR: (a) CER [\[53\],](#page-14-0) (b) CBA [\[69\],](#page-15-0) (c) CBA [\[72\]](#page-15-0) and (d) ART [\[73\]](#page-15-0). In (c), BA-B and BA-P denote two different CBAs obtained from different sources and substitute the 40% of the sand by volume.



**Fig. 18.** Variation in the autogenous shrinkage at (a) 1 and 3 or 4 days, (b) 28 days and (c) and (d) 7 days, of internally cured cement based materials when increasing the amount of internal curing water [29,34,64,65,68,69,72–[74,76,79,80,35,81,82,45,53,54,57](#page-14-0)–59,62]. Results grouped by (a, b, c) type of WASPOR and (d) WASPORs with different water absorption capacity.

of internal curing water.

When grouping the results by the water absorption capacity of the WASPORs [\(Fig. 20](#page-10-0)**d**), it can be seen that specimens, both L or H type,

with higher water absorption capacity led to higher shrinkage in airdrying conditions. The presence of more absorptive particles, although in a lower volume, could have a more negative impact on the drying

<span id="page-10-0"></span>

**Fig. 19.** Relationship between the volume of WASPOR and the volume of internal curing water used in different studies [\[24,29,46,48,51](#page-14-0)–58,34,60–62,64–70,35,71–76,78–81,36,82,39,41–43,45]. Results grouped by (a) type of WASPOR and (b) WASPORs with different water absorption capacity.



**Fig. 20.** Variation in the shrinkage in air-drying conditions at (a) 7 days, (b) 28 days and (c) and (d) 56 or 90 days, of internally cured cement based materials when increasing the amount of internal curing water [24,34,64,65,74,80–[82,36,41,43,50,54,55,60,63\]](#page-14-0). Results grouped by (a, b, c) type of WASPOR and (d) WASPORs with different water absorption capacity.

rate. Nevertheless, more data should be analysed to draw clearer conclusions, as variations in factors such as the composition of the binder, the mixing water to binder ratio, the precise surface to volume ratio of the specimens or the specific RH in the curing environment might have a significant influence on the results.

Other works have analysed this property after a period of sealed/ humid curing longer than one day, obtaining comparable results. For example, Liu et al. [\[51\]](#page-14-0) concluded that H specimens containing CON or CER showed a higher shrinkage due to the low stiffness of the WASPOR, although CER seemed to have a better performance due to an improved ITZ as a result of pozzolanic reactions. Furthermore, Kadhim Al-Saad et al. [\[59\]](#page-14-0) detected a reduction in shrinkage of L type specimens with CER and Qadri et al. [\[47\]](#page-14-0) concluded that the performance of L specimens with CON show similar drying shrinkage after being moisture cured for 14 days.

In conclusion, it can be stated that internal curing increases the

drying shrinkage whereas it decreases the self-desiccation shrinkage. Therefore, its influence on the total shrinkage depends on the proportion between those two phenomena. In any case, it must be noted that the shrinkage performance of a specimen in air-drying curing conditions is only comparable with the surface of a poorly cured real scale element, so a good performance will be found as long as external curing is applied in combination with internal curing.

# *5.7. Durability*

The higher porosity of concrete via WASPORs can lead to a higher carbonation depth, as has been reported when using CON [\[36\]](#page-14-0) and CBA [\[71\]](#page-15-0). However, densification of paste due to internal curing when using CER has been argued to decrease the carbonation depth in some cases [\[55,60\].](#page-14-0)

The higher porosity also explains the increase in water absorption by

immersion when using any WASPOR [\[59,63,67,76,81\]](#page-14-0). The effect on water capillary uptake cannot be so directly explained, as the size and shape of the pores also play an important role in this property. Actually, some works have obtained worse results than for the baseline mixes when using CER [\[63\]](#page-14-0), whereas others obtained similar or better results when using CBA [\[103,122\]](#page-15-0). Water ingress in concrete with WASPORs has also been assessed by its water permeability, which resulted to increase due to the higher pore volume [\[36\].](#page-14-0) Likewise, the chloride ion permeability increased when using CON [\[24\]](#page-14-0) and CER [\[63,65\]](#page-14-0) due to the increased volume of pores, whereas the internal curing effect led to a decrease in this parameter when using CBA [\[70,122\]](#page-15-0).

Regarding resistance to freezing and thawing cycles, some studies have remarked that the water stored in WASPORs must evaporate before the concrete is submitted to freezing. Otherwise, the water inside the particles expands and their low stiffness allows them to swell causing pop-outs [\[44\]](#page-14-0). Therefore, external water curing of concrete with WAS-PORs when there is a risk of freezing is especially not advised [\[109\].](#page-15-0) It must be noted that this recommendation is also applicable to concrete without ICWRs. Acceptable results have been obtained for the freezing and thawing test when using low amounts of CON [\[43,46,47\]](#page-14-0).

# **6. Discussion**

The internal curing of concrete via pre-wetted WASPORs has important environmental and economic advantages. However, the wide variety of WASPOR types forces a careful study of each of them regarding their influence on concrete properties. In general, the incorporation of these particles by substituting conventional aggregates or powder materials leads to a reduction in autogenous shrinkage and an increase in drying shrinkage. However, only a few works [\[34,74,80](#page-14-0)–82] have studied the performance of any WASPOR in both sealed and airdrying conditions, which could be very useful to better understand their influence on the different types of shrinkage.

In contrast, the study of the compressive strength together with the autogenous shrinkage (Fig. 21**a**) or together with the shrinkage in airdrying conditions (Fig. 21**b**) is more common. In fact, this is also a very important analysis to be carried out. WASPOR are weak particles, and the potential advantages regarding shrinkage mitigation and sustainment of hydration are valueless if the strength is jeopardized. The influence of any WASPOR on other mechanical properties such as flexural strength and tensile strength might be more decisive regarding the resistance to cracking. However, fewer findings can be collated from scientific literature. Nevertheless, it is reasonable to expect that they follow a similar trend to the compressive strength when increasing the amount of internal curing water (section 5.4). Actually, it must be kept in mind that the final objective of the internal curing in HPC is to reduce the cracking risk and therefore enhance its durability characteristics. Reducing the shrinkage has no benefit if the concrete cannot withstand any deformation.

From Fig. 21**a**, it can be inferred that CER is the most effective WASPOR when reducing the cracking risk in sealed conditions, i.e. it significantly reduces the autogenous shrinkage without remarkably affecting the strength. CON, on the contrary, show the worst performance. When analysing the data collated in Fig. 21**b**, it can be observed that no WASPOR can effectively mitigate the shrinkage in air-drying conditions and enhance the strength at the same time. CON and CER show the opposite performance regarding their influence on shrinkage and strength when cured at low RH. However, it must be noted that this curing condition might only occur close to the surface of a concrete element and can be avoided if external conventional curing is applied.

Furthermore, a few works have assessed the cracking risk of concrete with WASPORs by a specific test. One of the most popular procedures for the study of this property is the ring test based on ASTM C1581 [\[123\]](#page-16-0). For instance, Wei et al [\[75\]](#page-15-0) reported that concrete with a low water to cement ratio with a fly-ash-based ART developed no cracks in a sealed ring. Gesoglu et al. [\[74\],](#page-15-0) using the same test on high performance concrete but in air-drying curing conditions, concluded that internal curing via ART delays the appearance of cracks. They propose the lower stress concentration around the less stiff WASPORs, similar to that of the paste, as a partial explanation for this performance. Other procedures have also been used for assessing the cracking in severe air-drying conditions. For instance, Salgues et al. [\[42\]](#page-14-0) submitted a large surface of concrete with a high water to binder ratio and different contents of CON to a fan air flow, based on ASTM C1579 [\[124\],](#page-16-0) and detected larger and earlier fissures than in baseline mixes. However, Corinaldesi et al. [\[41\]](#page-14-0) submitted an elongated specimen of concrete, also with a high water to binder ratio, and with different volumes of CON to air-drying and warming conditions and obtained better results than in reference concretes. It must be noted that the results obtained in the latter experiences strongly depend on the severity of the curing conditions and level of exposure of the specimens. In general, it can be concluded that external curing should be applied together with internal curing for ensuring the absence of cracks on the surface of concrete.

For sure, a WASPOR must show some specific characteristics for it to be used as ICWR and effectively reduce the autogenous shrinkage of concrete without excessively jeopardizing the drying shrinkage and the strength. Among those characteristics, two seem to be the most important:

- The capacity to quickly desorb considerable amounts of water after the setting of concrete. This is possible when the WASPOR has a high water absorption capacity, a wide porous network (wider than that of the paste) and a high specific surface.
- A low crushing value. This is important for not excessively jeopardizing the modulus of elasticity and the strength of concrete and therefore be effective in reducing the shrinkage and the cracking risk.

However, in practice these two characteristics seem to be



**Fig. 21.** Relationship between the variation in the compressive strength and the variation of (a) the autogenous shrinkage [29,34,80–[82,35,45,53,54,58,59,65,74\]](#page-14-0)  and (b) the shrinkage in air-drying conditions [24,34,74,80–[82,36,41,43,54,55,60,64,65\].](#page-14-0) Results grouped by type of WASPOR.

contradictory, and therefore an optimum intermediate situation should be found.

Furthermore, the spacing between the particles of WASPOR in concrete should be small in order to protect the largest possible volume of paste from self-desiccation. It must be noted that despite the high absorption and desorption properties of an ICWR, its sphere of influence in the paste becomes limited in size depending on the paste characteristics [\[97\]](#page-15-0). Therefore, the use of particles with a smaller size or a higher volume of particles with a lower absorption capacity contributes to increase the volume of internally cured paste (Fig. 22).

The studies collated in this review have led to the conclusion that internal curing via WASPORs can be very useful for avoiding selfdesiccation shrinkage and the potential for associated early-age cracking. However, the use of conventional external curing must be applied to avoid drying shrinkage at the surface of concrete. In addition, the implementation of restraining strategies such as the use of fibres or the use of expansive agents can complement the internal curing effect.

The discussed parameters that influence the internal curing of high performance concrete or that are related to it, because of the common fundamental objective of improving this material, are illustrated in [Fig. 23.](#page-13-0) Another deep review of the factors affecting the effectiveness of internal curing has been published by Yang, Li et al. [\[125\]](#page-16-0).

Concrete with a low water-to-cement ratio suffers self-desiccation. This negative phenomenon can be mitigated with internal curing via pre-wetted porous particles with a moderate absorption capacity, a high desorption capacity, and well space distributed. However, the internal curing might increase the external drying of concrete so external curing, use of fibres or expansive agents are recommended as complementary shrinkage reduction strategies. Furthermore, the use of porous particles as ICWRs have a moderate impact on strength. Therefore, internal curing can improve the durability by mitigating the autogenous shrinkage and preventing the associated cracks whereas the mechanical properties are maintained to obtain high performance concrete.

#### **7. Conclusions and future research**

The following conclusions can be made out after analysing the findings provided by more than 100 scientific articles regarding the internal curing effect in concrete via pre-wetted WASPOR. General recommendations for suitable characterization of WASPOR and mix design are also given:

1. There are WASPORs with very different origins, sizes, shapes and chemical compositions. However, they are generally characterized by a high porosity, a high water absorption and a high water desorption. The testing of the water absorption capacity is challenging as conventional procedures are not applicable to such porous particles. Some alternative methods are already established but their applicability in waste powder materials needs further research. Furthermore, only a few works study the desorption of the WASPORs, although it is, joint to the desorption rate, a valuable parameter to be determined.

- 2. As the precise evaluation of the water absorption capacity is difficult, WASPORs may be use in different wet conditions (over or infrasaturated), which will lead to the increase or decrease of consistence of concrete, respectively.
- 3. A higher content of WASPOR decreases the concrete density, and this can be considered as an advantage in terms of thermal conductivity. The internal curing effect, which promotes the cement hydration, can partially compensate the decrease in strength and modulus of elasticity due to the weakness of the WASPOR.
- 4. Internal curing via pre-wetted WASPORs contributes to the sustainment of the internal relative humidity in high performance concrete leading to a decrease in self-desiccation and in resulting autogenous shrinkage. The coexistence of self-desiccation and drying in the shrinkage measured in air-drying curing conditions leads to different trends depending on the size and shape of the specimens used. The collated results support the idea that the presence of pre-wetted WASPORs promotes drying shrinkage. Therefore, internal curing does not eliminate the need for appropriate external curing under some conditions. When incorporating the same amount of internal curing water, the use of WASPOR with a moderate water absorption capacity leads to a higher reduction in the autogenous shrinkage and a lower increase in the shrinkage in air-drying curing conditions.
- 5. The durability of concrete with different WASPORs is positively influenced by the internal curing effect whereas negatively influenced by the high porosity of the WASPORs themselves. The amount of collated findings is not enough to state general conclusions regarding any type of WASPOR in terms of durability.

When combining the collated findings regarding different properties, it can be concluded that coarse crushed ceramics and recycled concrete aggregates show the best and the worst performance as ICWRs, respectively. Their desorption properties and possible pozzolanic reactions on the ITZ might be involved in this differential performance.

The next future research lines are proposed:

- Further study of the less documented WASPORs, such as coal bottom ash or other types of ashes. The collated data indicate that they are potentially suitable for working as an internal curing water reservoir.
- Development of new types of artificial waste based porous materials, which combine a low carbon footprint with good absorption and desorption capacities.
- Investigations regarding other types of powder waste materials that can effectively work simultaneously as supplementary cementitious materials and internal curing water reservoir.
- Investigations regarding more fibres that can effectively work simultaneously as restraining agents and as internal curing water reservoirs.



**Fig. 22.** Protected paste in a cement-based system with (a) large ICWRs, (b) the same volume of small ICWRs and (c) a lower volume of ICWRs with that same small size but a higher water absorption capacity.

<span id="page-13-0"></span>

**Fig. 23.** Aspects of concrete related with internal curing.

- New procedures should be proposed for determining the water absorption capacity of powder porous materials.
- Further study regarding the influence of any type of WASPOR on the shrinkage of concrete in fresh state and durability properties.

Finally, the knowledge attained by studying the internal curing function of WASPORs have opened the door for new applications. The possibility for these alternative particles to carry substances other than water such as admixtures, activators or self-healing agents should be studied in deep.

# **CRediT authorship contribution statement**

**Roberto Rodríguez-Álvaro:** Conceptualization, Formal analysis, Investigation, Writing – original draft, Visualization. **Sindy Seara-Paz:**  Formal analysis, Investigation, Writing - review & editing, Supervision. Belén González-Fonteboa: Formal analysis, Investigation, Writing review & editing, Supervision. Verónica Ferrándiz-Mas: Formal analysis, Investigation, Writing - review & editing, Supervision. **Kevin**  Paine: 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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*Construction and Building Materials 299 (2021) 124244*

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