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# Quantification and characterization of the microstructural damage of recycled aggregate self-compacting concrete under cyclic temperature changes

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#### ABSTRACT

Recycled Aggregate (RA) usually increases porosity and weakens Interfacial Transition Zones (ITZs) of concrete, which favors the appearance of internal thermal damage. Four Self-Compacting Concrete (SCC) mixes with coarse and fine RA were subjected to positive and negative cyclic temperature variations to characterize their thermal damage and quantify its effects. Two damage mechanisms were found. On the one hand, micro-cracks appeared in the ITZs. On the other hand, micro-cracks arose from the micro-pores and propagated through the cementitious matrix. Both damage mechanisms were promoted by the use of coarse and fine RA, respectively. The damage was most notable at sub-zero temperatures and when adding coarse RA. Furthermore, it primarily affected compressive strength, although ultrasonic pulse velocity and hardened density also decreased, which served as non-destructive indicators to indirectly quantify the level of thermal internal damage of SCC.

## 1. Introduction

Concrete sustainability can be increased by using industrial byproducts as raw materials. The use of aggregates from industrial processes to replace Natural Aggregate (NA) allows obtaining concrete mixes with an adequate balance between sustainability, mechanical response and durability [1].

Recycled Aggregate (RA) consists of crushed out-of-use concrete, which may be added as coarse and fine aggregate in any concrete type, such as Self-Compacting Concrete (SCC), which needs no vibration [2]. The adhered mortar in the coarse RA, and the mortar particles in the fine fraction reduce concrete workability, increase porosity, and weaken Interfacial Transition Zones (ITZs) [3], which generally reduce strength and durability [4]. However, an SCC with adequate behavior can be obtained by a mix design focused on RA properties [5].

RA also affects the thermal response of concrete, reducing its thermal conductivity [6] and increasing its thermal deformability and damage [7]. This letter supplements previous studies by the authors that address this issue in SCC containing RA. Firstly, the deformability of SCC with RA under cyclic temperature changes was evaluated. The appearance of a remaining strain as the cycles went by was found, which indicated the

existence of internal damage [8]. Subsequently, the evolution over the thermal cycles of the level of internal damage was analyzed [9]. This letter aims to complete this research by deepening the microstructural characterization of the damage experienced by SCC with RA under cyclic temperature changes, and by providing indicator properties to quantify it. Both aspects are novel in the scientific literature.

#### 2. Materials and methods

Four SCC mixes of slump-flow class SF3 [10] were produced. They incorporated 300 kg/m<sup>3</sup> of CEM I 52.5 R and 1855 kg/m<sup>3</sup> of aggregate, whose proportions, by volume, were 11.4 % limestone filler < 0.063 mm, 6.2 % limestone fines 0/1 mm, 31.0 % coarse aggregate 4/12.5 mm, and 51.4 % fine aggregate 0/4 mm. The effective water-to-cement ratio was 0.50–0.60, and the admixture content, 2.2 % of cement mass. Accordingly, mix *N* incorporated 100 % natural siliceous aggregate; mix *C*, 100 % coarse RA 4/12.5 mm; mix *F*, 100 % fine RA 0/4 mm; and mix *CF*, 100 % coarse and fine RA.

At an age of 180 days,  $75 \times 75 \times 275$ -mm prismatic specimens of each mix were subjected to freezing and heating conditionings, which consisted of 20 cycles of 12 h either at -15 °C in a freezer or at 70 °C in

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an oven, followed by 12 h at 20 °C in a climatic chamber. After conditioning completion, Ultrasonic Pulse Velocity (UPV), EN-12504-4 [11], and hardened density, EN-12390-7, were measured. Then,  $75 \times 75 \times 75$ -mm cubes were extracted from those prismatic specimens to determine their compressive strength, EN-12390-3, and modulus of elasticity, EN-12390-13. The values of the properties tested on the thermally stressed specimens were compared with those of other specimens of the same age that had not been subjected to thermal conditioning. A Scanning Electron Microscope (SEM) analysis was also conducted on the cut faces of the 75  $\times$  75-mm cubes before its mechanical testing.

## 3. Results and discussion

## 3.1. Characterization and effects of thermal internal damage

Table 1 details the compressive strength, modulus of elasticity, UPV, and hardened density of the four mixes in unconditioned and thermally conditioned specimens. Both thermal conditionings, especially the freezing one, caused significant internal damage in the mixtures, which worsened all four properties evaluated.

The thermal internal damage in SCC resulted from the appearance of micro-cracks in the ITZs, due to the different thermal deformability between the aggregate and the cementitious matrix, or micro-cracks that started in micro-pores and then propagated through the cementitious matrix. These two damage mechanisms can be observed in the SEM images in Fig. 1:

- Fig. 1a shows a zone of a mix-*C* specimen where an RA particle detached from the cementitious matrix, a phenomenon favored by the micro-crack that appeared due to thermal variation and propagated through the ITZ. Adding RA > 2 mm to concrete weakens the ITZs due to its adhered mortar [4], which is promoted by cyclic thermal variation.
- Fig. 1b shows a 500-µm-diameter pore from a mix-*F* specimen. It can be noted that a crack begins in that pore and propagates towards the cementitious matrix. The presence of mortar particles in fine RA causes a considerable increase in porosity [3], so this kind of micro-cracking was favored when adding this RA fraction.

The aforementioned thermal-damage mechanisms worsened all the

#### Table 1

Average property values.

|  |                             | Mix N | Mix C | Mix F | Mix<br>CF |
|--|-----------------------------|-------|-------|-------|-----------|
| Compressive<br>strength (MPa)            | Without<br>conditioning     | 62.6  | 49.2  | 45.8  | 32.7      |
|  | After freezing conditioning | 52.3  | 34.6  | 36.7  | 21.6      |
|  | After heating conditioning  | 59.4  | 45.7  | 43.5  | 29.9      |
| Modulus of elasticity<br>(GPa)           | Without<br>conditioning     | 37.2  | 33.1  | 29.2  | 21.2      |
|  | After freezing conditioning | 36.8  | 32.6  | 28.9  | 20.3      |
|  | After heating conditioning  | 37.1  | 33.0  | 29.2  | 21.0      |
| UPV (km/s)                               | Without<br>conditioning     | 4.04  | 3.69  | 3.41  | 2.76      |
|  | After freezing conditioning | 3.45  | 3.03  | 2.89  | 2.21      |
|  | After heating conditioning  | 3.88  | 3.51  | 3.27  | 2.61      |
| Hardened density<br>(Mg/m <sup>3</sup> ) | Without<br>conditioning     | 2.35  | 2.27  | 2.22  | 2.14      |
|  | After freezing conditioning | 2.33  | 2.23  | 2.20  | 2.09      |
|  | After heating conditioning  | 2.32  | 2.19  | 2.17  | 2.01      |



**Fig. 1.** SEM images of thermally conditioned specimens: (a) mix *C* with a crack in an ITZ; (b) mix *F* with a cracked micro-pore.

properties evaluated (Table 1). However, the decrease in each property was different, as detailed in Fig. 2. The highest reduction was in compressive strength, which showed decreases of up to 15-35 % after the freezing conditioning. However, the modulus of elasticity experienced an almost negligible reduction. Therefore, the thermal microcracking suffered by SCC mainly affected its mechanical behavior under failure conditions. On the other hand, thermal micro-cracking increased the discontinuity in the cementitious matrix, which in turn led to a decrease in UPV of around half of the percentage loss of compressive strength. UPV is a non-destructive measure of the continuity and compactness of concrete [9], and both features were reduced due to cyclic temperature variations. Finally, the remaining strain that appeared in the concretes after thermal stress [8] caused a decrease in hardened density of 1–7 %. Those reductions were always higher in the freezing conditioning for all properties. It can therefore be stated that the micro-cracking produced by the freezing of the capillary water of concrete was more severe than the micro-cracking experienced by SCC in the heating conditioning, which was mainly caused by the different thermal deformability of the aggregate and the cementitious matrix.

RA also influenced the thermal performance of SCC. Regardless of the thermal conditioning, the decrease in the properties was always larger in mix C than in mix F. In fact, mix F showed a similar behavior to mix N in many cases. Hence, it appears that the damage mechanism found in mix C, micro-cracking in the ITZs, was more relevant for the behavior of SCC than the micro-cracking originated in the pores, which was favored by fine RA. Logically, the simultaneous use of both RA fractions led to a combination of both damage mechanisms, so mix CFexhibited the highest performance deterioration.



Fig. 2. Average property percentage variations: (a) freezing conditioning; (b) heating conditioning.

## 3.2. Indicator properties of thermal internal damage

The direct quantification of the thermal damage by measuring the compressive strength in cores extracted from the concrete element may not be feasible. Therefore, indicator properties that allow indirectly estimating the thermal damage may be of great interest. Thus, Fig. 3 shows the linear simple regression between the loss of compressive strength (the conventional property that best reflects the thermal damage experienced by SCC [7]) and the variation of the other measured properties. Modulus of elasticity did not show a high correlation (R<sup>2</sup> coefficients below 80 %), so its use is not adequate for estimating the thermal damage of SCC. However, UPV and hardened density did reach R<sup>2</sup> coefficients higher than 90 % in both thermal conditionings. Thus, a traditional indirect measurement such as UPV [2] would be a good way to quantify the thermal damage. In addition, the hardened density measured in small samples extracted from the surface of the damaged concrete elements could be a successful alternative.

## 4. Conclusions

Cyclic thermal variations produced internal damage in SCC, which was increased when using RA. This internal damage consisted of micro-



**Fig. 3.** Linear simple regression between loss of compressive strength and decrease of indicator properties: (a) freezing conditioning; (b) heating conditioning.

cracks in the ITZs, a phenomenon favored by the use of coarse RA, or micro-cracks that arose in the micro-pores and propagated through the cementitious matrix, damage mechanism promoted when adding fine RA. Micro-cracking fundamentally decreased the compressive strength of concrete. However, UPV and hardened density of SCC were also negatively affected and could serve as indirect indicators to successfully estimate the level of that thermal damage (loss of compressive strength) by a linear simple regression. The internal thermal damage was most notable when applying sub-zero temperatures and adding coarse RA, although the joint use of both RA fractions had the most detrimental effect.

## CRediT authorship contribution statement

Víctor Revilla-Cuesta: Conceptualization, Methodology, Investigation, Data curation, Writing – original draft. Marta Skaf: Conceptualization, Investigation, Supervision, Writing – review & editing. José A. **Chica:** Conceptualization, Methodology, Supervision. **Vanesa Ortega-López:** Supervision, Writing – review & editing, Resources, Funding acquisition. **Juan M. Manso:** Supervision, Writing – review & editing, Resources, Funding acquisition.

## **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.

## Data availability

Data will be made available on request.

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