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Fire and postfire compressive strength of recycled aggregate concrete made with ceramic stoneware

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ABSTRACT

This study investigated the possibilities of reusing ceramic stoneware (CS) waste, a particular type of tiles ceramic waste (TCW), provided by a treatment plant in Castellon (east Spain), as recycled aggregate in structural concrete, and its influence on concrete behaviour when exposed to elevated temperatures (200 °C, 400 °C, 600 °C, 800 °C; compressive strength determined in hot and after air and water cooling). Although all samples exhibited similar strength values at room temperature, their thermal conductivity reduced with increasing CS contents. Strength values progressively dropped with rising temperatures, with the highest results recorded in hot, followed by air and, finally, by water cooling. Smaller differences between the strength registered in hot and after cooling were generally recorded in the recycled aggregate concretes (RAC) than in the reference sample. The concrete prepared with 100 vol% CS gravel was the only one to generally provide better residual strength after air-cooling than in hot.

List of abbreviations:

| | |
|------|-------------------------------------|
| CS | ceramic stoneware |
| CSCA | ceramic stoneware coarse aggregates |
| CSFA | ceramic stoneware fine aggregates |
| CW | ceramic waste |
| CDW | construction and demolition waste |
| ITZ | interfacial transition zone |
| NA | natural aggregates |
| NCA | natural coarse aggregates |
| NFA | natural fine aggregates |
| PC | Portland cement |
| RAC | recycled aggregate concrete |
| TCW | tiles ceramic waste |
| vol% | percentage in volume |

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| | |
|----|------------------|
| WA | water absorption |
|----|------------------|

1. Introduction

Society's fast development and the growing world population have significantly raised demand for natural resources and energy, which has led to marked exploitation and the global warming of our planet [1]. It is estimated that the construction industry consumes more than 40 % of the energy worldwide, which generates more than 35 % of global CO₂ emissions [2]. Concrete use became widespread after the industrial revolution in the 19th century, and approximately 30 Gt/year are produced today, which are associated with 9 % of the worldwide greenhouse gas emissions and 3 % of the globally used energy [1,3]. This context has prompted the scientific community to develop more sustainable construction materials, such as recycled aggregate concrete (RAC), being necessary to investigate its performance in different scenarios, such as durability, adhesion or behaviour when exposed to elevated temperatures. Reusing industrial waste as a raw material would contribute to reduce the consumption of natural resources and energy and would also minimise the amount of landfilled waste and its associated impacts. This would imply an evolution towards circular economy.

Ceramic tiles are an industrial product consumed worldwide which production significantly rose in the last decade. Indeed 16,093 million m² were globally manufactured in 2020 [4], most of which in Asia (74.0 %), followed by the European Union (7.6 %). As reported by Baraldi [4], China was the top manufacturing country and Spain ranked fifth, with 52.7 % and 3 % of world production, respectively. Additionally, according to Medina et al. [5], a significant fraction of construction and demolition waste (CDW, from 8 % to 54 %) comprises ceramic materials, which implies large amounts of tiles ceramic waste (TCW), provided that 374 million tons of CDW were generated in the European Union in 2016 [6]. TCW is a good candidate to be reused in the production of new construction materials because it is inert, non-biodegradable, durable, and with high melting points and strength. However, its physico-mechanical properties and chemical and mineralogical composition very much depend on the composition of the clay minerals and the sintering process employed when manufacturing the ceramic product [7,8]. In particular, ceramic tiles are classified by ISO 13006 and EN 14411 specifications according to their production process (extruded, A; or pressed, B) and water absorption (WA: Ia, very low ≤ 0.5 %; Ib, low, $0.5 \leq WA \leq 3$ %; IIa, medium-low, $3 \leq WA \leq 6$ %; IIb, medium-high, $6 \leq WA \leq 10$ % and III, high, $WA > 10$ %). The Ceramic Tiles Guide developed by the Generalitat Valenciana [9] correlated these absorption ranges with the commercial denominations available on the market. Thus Ia products are commercially designed as porcelain stoneware tiles, those classified as Ib-IIa are commonly known as stoneware, and IIb-III products generally correspond to wall ceramic tiles. The TCW used in this study only contained stoneware and porcelain stoneware tiles, generally white-paste, with low water absorption, high strength and abrasion resistance, and therefore, with an inherent potential for its use in concrete. In contrast to other studies that generally used ceramic materials from construction and demolition waste (CDW), which are heterogeneous and contain a mixture of materials and impurities, the industrial by-product used herein was uniform, homogeneous and free of impurities.

Previous studies on the influence of TCW as recycled aggregate in concrete exposed to elevated temperatures provided relevant information on explosive spalling and fire-induced strength loss of the developed concrete samples [10,11]. Demir et al. [12] replaced up to 100 % of natural coarse aggregates (16–32 mm) with TCW, and observed a slight improvement in the residual compressive strength and modulus of elasticity of RAC after exposure to elevated temperatures. However, Li et al. [13], who conducted similar research, but included the analysis of residual tensile strength, reported better strength results when replacing natural sand with TCW. Talaei et al. [14] investigated how to prevent loss of strength in concrete containing TCW recycled aggregates when exposed to fire by adding polypropylene and steel fibres. Keshavarz and Mostofinejad [15] compared the use of red ceramic and porcelain tiles waste recycled aggregates in Portland cement (PC) and in calcium aluminate cement concretes exposed to elevated temperatures. The best results were obtained with PC and porcelain aggregates, as evidenced by the 62.5 % improvement in residual compressive strength compared to the reference mixture. Since the TCW used in the present study was only composed by ceramic tiles generally manufactured at temperatures in the 1100–1200 °C range, it is expected to improve the performance of the RAC when exposed to elevated temperatures. However, it is essential to explore the behaviour of the RAC developed combining these TCW recycled particles with natural aggregates, since the behaviour and transformations of the aggregates with rising temperatures will vary, given their different nature.

In addition to previous works on the influence of ceramic waste (CW) in RAC exposed to elevated temperatures, there is a large body of research that has explored the behaviour of RAC developed with other waste materials when exposed to fire. However, most of these studies have focused on employing coarse recycled aggregates obtained from concrete waste [10,16–19]. Kou et al. [16] observed that, although concrete strength at 25 °C decreased with increasing amounts of recycled concrete aggregates, the relative residual strength of RAC, which was determined after its exposure to elevated temperatures, was higher than that of the concrete prepared with natural aggregates (NA). In the study by Chen et al. [10], RAC performed better (in strength terms) than traditional concrete up to 200 °C, worse at 400 °C, and similarly after exposure to 600 °C. Fernandes et al. [19] conducted a review on the effect of elevated temperatures on the RAC developed using recycled aggregates obtained from concrete, and concluded that these types of aggregates were used mainly to replace natural gravel up to 100 %. The authors reported a wide dispersion of residual strength results after exposing RAC to elevated temperatures, which was attributed to different factors: testing conditions (heating rates, sample sizes, cooling, etc.) and material parameters (composition, moisture, etc.). In some analysed works, RAC performed better than limestone or siliceous concrete, similar properties were reported in others, and recycled concrete aggregates worsened strength in a third group of studies. Other research, such as that by Fernandes et al. [19], has observed lower thermal conductivity values with rising temperatures, and also in the RAC samples developed with recycled concrete aggregates (compared to NA concrete).

In short, CW properties make it a good candidate to be used as recycled aggregate in more sustainable concrete, with better performance at elevated temperatures. However, the results previously reported in the literature diverge, mainly due to the wide variety of ceramic products available on the market. To provide further relevant knowledge and a comprehensive understanding of this subject, this research focused on the potential use of ceramic stoneware tiles waste as recycled aggregate in concrete exposed to elevated temperatures. The TCW selected for the present study was provided by a treatment plant located in Onda, in the province of Castellon (east Spain). This company daily receives discarded CW directly provided by the ceramic tiles production companies located in the area, and classifies it into two groups: stoneware and porcelain stoneware; other types of tiles. In order to use a CW with relatively limited characteristics, only the CW from the first group (stoneware and porcelain stoneware, with water absorption (WA) lower than 3 %) was selected in this study. This specific CW type is called ceramic stoneware (CS) hereafter. Provided that this industrial waste is classified in a treatment plant and not obtained from CDW, it is highly homogeneous and does not contain impurities, such as gypsum or adhesives. Knowledge on the behaviour of RAC made with CS aggregates when exposed to elevated temperatures will be useful for designing and building more sustainable fire-resistant structures and will contribute to circular economy, reusing CW and minimising the consumption of natural resources.

2. Experimental programme

The experimental work conducted to investigate the behaviour of RAC made with recycled CS aggregates was divided into the following steps: aggregates characterisation; concrete design and samples production; thermal conductivity of concrete at room temperature; heating-cooling methodology and tests. All these stages of the study are detailed in the following sections.

2.1. Aggregates characterisation

The aggregates used in the present study were characterised by means of particle size distribution, WA, density and microstructural studies (thermogravimetry; X-Ray diffraction).

2.1.1. Particle size distribution

The natural coarse aggregates (NCA) and natural fine aggregates (NFA) were replaced with two different fractions of the CS recycled aggregates: coarse (CSCA) and fine (CSFA). Fig. 1 shows all the aggregates used in this study.

Fig. 2 depicts the particle size distribution of the employed aggregates, determined according to Standard UNE EN 933-1. A distinction was made between fine aggregates ($D < 4$ mm, sand) and coarse aggregates ($D > 4$ mm, gravel), with a maximum particle size of 12 mm. As observed, the natural and CS coarse aggregates had similar particle sizes, with natural gravel being slightly finer than the recycled one. However, significant differences were observed between the natural and recycled sand, and CSFA were larger and less balanced than NFA. This was attributed to a greater hardness of the recycled CS aggregates because, as previously reported by Pitarch et al. [20], TCW particles like those herein used had lower mass loss than natural calcareous aggregates after MicroDeval tests (tests adapted from UNE-EN 1097-1 and UNE 83-115-89). Consequently, crushing stoneware and porcelain stoneware tiles to obtain a similar particle size to that of natural calcareous sand requires larger amounts of energy, which is counterproductive from both the environmental and economic perspectives.

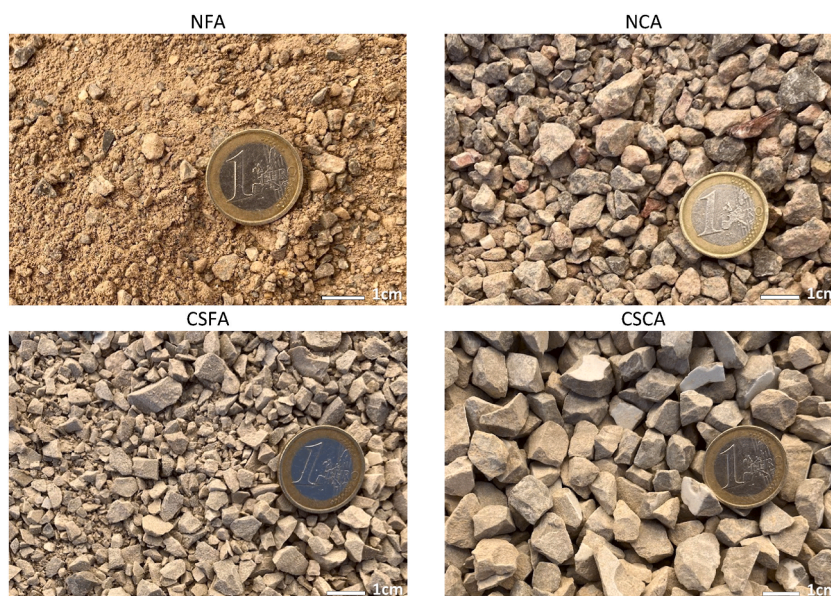


Fig. 1. Images of the aggregates used in concrete mixtures.

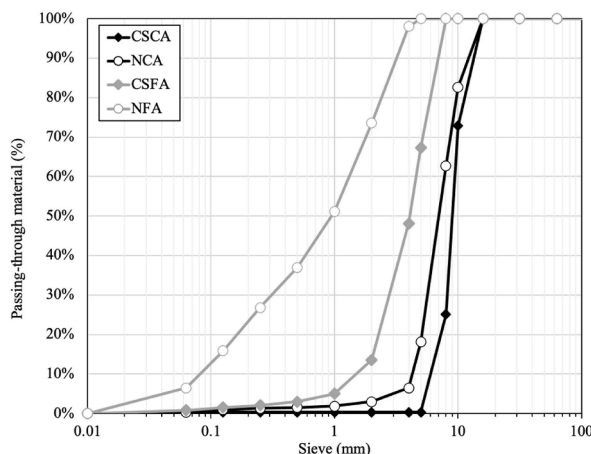


Fig. 2. Particle size distribution of the used natural and recycled aggregates.

2.1.2. Particle density and water absorption

The apparent density and WA of aggregates, tested according to EN 1097-6, are summarised in Table 1. The density of the CS aggregates was lower than that of the natural calcareous particles. For a given material, slightly higher results were obtained with finer particles (sand). Similar WA values were obtained for gravel, and were slightly higher for recycled (CSCA, 1.76 %) than NA (NCA, 1.60 %). On the contrary, natural sand had the greatest WA (NFA, 3.43 %), followed by recycled sand (CSFA, 2.49 %). These results are consistent with the review by Medina et al. [5], who reported a wide range of WA values for TCW used as recycled aggregate in concrete, which ranged from 1.4 to 11.6 % and from 2.0 to 17.2 % for coarse and fine aggregates, respectively. This broad range was attributed to the wide variation of ceramic tiles available on the market depending on their production process.

The density and WA values recorded for the CS particles are close to that reported by Silva et al. [21], who investigated the microstructure of CS tiles depending on their thickness and sintering temperatures. The authors observed a significant influence of these factors on the bulk density and WA of the developed tiles, insofar as the highest WA values (> 3 %) were reported when sintering at 1180 °C, while the absorption of the tiles sintered at 1,200 °C and 1,220 °C was lower than 0.5 %. In their study [21], bulk density varied from 2150 to 2300 kg/m³. The density and WA values of the CS waste used in the present study corroborate that it is composed of a combination of different types of ceramic tiles, which were mainly sintered at temperatures within the 1150–1,200 °C range.

The WA values obtained in the present study were considered to adjust the concrete mix proportions because, to keep constant the effective water available in the mix for PC hydration (total water, minus that absorbed by aggregates, as defined by UNE EN 206-1), the total water/cement ratio of concrete was modified depending on the water absorbed by aggregates. Given the different densities between natural and CS aggregates, the NA replacement percentages were specified in volume (vol%), which implied the same volume and a smaller mass of recycled CS particles for a given NA substitution.

2.1.3. Microstructural characterization

The X-ray diffractogram of the natural and CS aggregates is plotted in Fig. 3. Tests were run in a Bruker AXS D4 Endeavor diffractometer using Cu K α radiation at 40 kV and 20 mA within the 5–70 2 θ degrees range. The same peaks were distinguished in a given material regardless of whether it was sand or gravel. However, their intensity was slightly lower in gravel than in sand. Different phases were identified in CS and calcareous particles. Quartz (Q, SiO₂, PDFcard 77–1060), mullite (M, Al₆Si₂O₁₃, PDFcard 79–1455) and sodium feldspar albite (A, NaAlSi₃O₈, PDFcard 89–6423) were the main crystalline phases distinguished in the CS particles. Signals due to calcite (C, CaCO₃, PDFcard 85–849) and the calcium and magnesium carbonate dolomite (D, CaMg(CO₃)₂, PDFcard 71–1662) arose in the diffractograms of the limestone aggregates. As previously described by Pitarch et al. [22], the deviation from the baseline observed from 15 to 30 2 θ degrees in the ceramic particles denoted the presence of amorphous phases. According to Zanelli et al. [23], glassy phases in stoneware tiles begin to form at about 1,050 °C due to the melting of feldspars. Consequently, porcelain stoneware tiles are composed of some crystalline phases embedded in significant amounts of vitreous phases [24].

The differential thermogravimetric curves of the natural limestone and CS aggregates used in this study are plotted in Fig. 4. These tests were conducted in a Mettler Toledo SDTA851e/LF/1600 thermogravimetric analyzer from 35 °C to 1,000 °C at a heating rate of 20 °C/min, in alumina crucibles and in an air atmosphere. The CS particles exhibited minor mass loss within the 600–750 °C range

Table 1
Apparent particle density and water absorption of natural and recycled aggregates.

| ID | Apparent particle density (kg/m ³) | Water absorption (%) |
|------|--|----------------------|
| NCA | 2771.2 | 1.60 |
| NFA | 2843.0 | 3.43 |
| CSCA | 2373.7 | 1.76 |
| CSFA | 2423.3 | 2.49 |

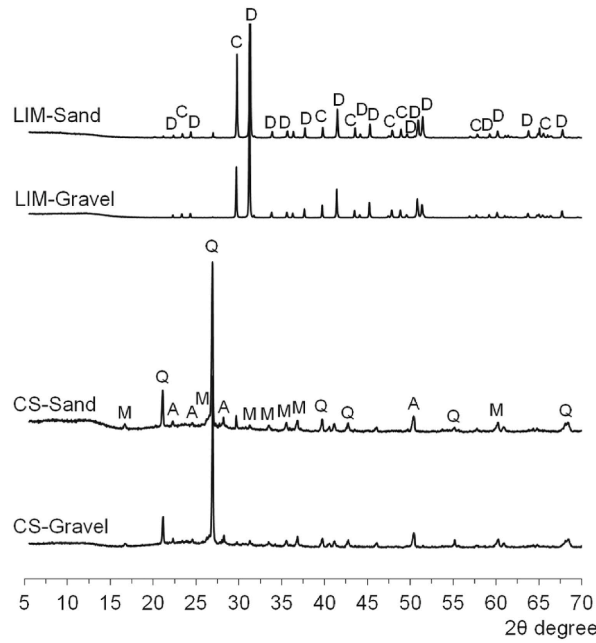


Fig. 3. X-ray diffractogram of limestone and CS particles. D: Dolomite ($\text{CaMg}(\text{CO}_3)_2$); C: Limestone (CaCO_3); Q: Quartz (SiO_2); A: Albite ($\text{NaAlSi}_3\text{O}_8$); M: Mullite ($\text{Al}_6\text{Si}_2\text{O}_{13}$).

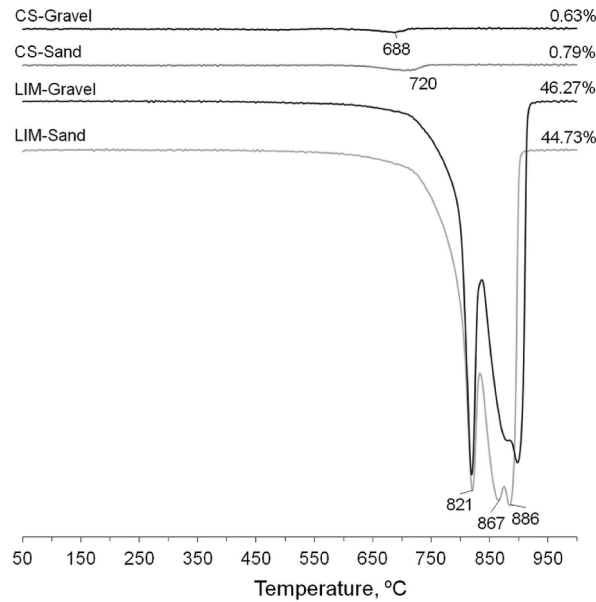


Fig. 4. Differential thermogravimetric curves of limestone and CS aggregates.

(0.46 wt% sand, 0.23 wt% for gravel), which led to total weight loss below 1 %. Contrarily, the dolomite particles ($\text{CaMg}(\text{CO}_3)_2$) showed significant weight loss within the 650-920 °C range, which was attributed to CaCO_3 and MgCO_3 decomposition, and led to total weight loss close to 45 wt%. According to Humienik [25], the first band is associated with the decomposition of magnesium carbonate and the second one with that of calcium. These reactions cannot be separately evaluated because they overlap.

These results fall in line with those previously reported by Li et al. [13], who replaced 50 % of limestone sand and gravel (separately) with porcelain stoneware aggregates, and also observed that NA exhibited significant weight loss within the 700-845 °C range, attributed to the decomposition of calcium and magnesium carbonates. In their study, the ceramic particles also exhibited minor weight loss of approximately 1.0 % from 600 °C to 700 °C, which was attributed to the partial transformation of quartz crystals from type α to β , and led to expansion, which brought about thermal stress. Zhang et al. [26] investigated the thermal degradation of dolomite when exposed to temperatures within the 100-800 °C range (2 °C/min) and slowly cooled to room temperature inside a furnace. The authors observed greater degradation with rising temperatures and, although the thermal cracking of the dolomite particles

started at 150–200 °C, no significant variations in both density and mass loss were recorded up to 600 °C. However from 600 °C, the thermal decomposition of dolomite resulted in significant degradation, with substantial mass and density losses.

Another property that may also determine the behaviour of aggregates in concrete exposed to high temperatures is the thermal expansion coefficient (α , in 10^{-6} K $^{-1}$), which represents length change with temperature variation. Luque et al. [27] determined the thermal expansion coefficient (α , in 10^{-6} K $^{-1}$) of different dolomitic marbles. They observed that it varied within the 8.0–16.6 $\cdot 10^{-6}$ K $^{-1}$ range depending on crystallographic orientation. In most ceramic tiles, this coefficient falls within the 4–8 $\cdot 10^{-6}$ °C $^{-1}$ range [28], and this value generally varies between 9.2 $\cdot 10^{-6}$ and 11 $\cdot 10^{-6}$ in concrete, depending on factors like the type of employed cement and aggregates, dosage, and temperature at which it is determined. Regarding the type of recycled aggregate herein used, TA Instruments [24] investigated the thermal expansion of porcelain tiles when heated up to 1,000 °C and slowly cooled. The allotropic transformation of quartz at 573 °C brought about dimensional changes to tiles during heating and cooling, and transformations of the glassy phase were also observed at 764 °C and 904 °C. However, the authors concluded that, as crystalline phases were embedded in a vitreous matrix, this reduced expansion-shrinkage compared to pure quartz or siliceous particles.

By way of conclusion, given that aggregates occupy 50–75 % of the concrete volume [29], they strongly influence concrete behaviour, especially when exposed to elevated temperatures. Therefore, properties like density, WA, thermal decomposition or expansion of aggregates may significantly influence the degree of stress inside concrete and, consequently, its residual compressive strength.

2.2. Concrete design and sample production

After characterising the NA and recycled aggregates, concrete mixtures were designed and concrete samples were prepared using a pan concrete mixer. As summarised in Table 2, four different RAC mixtures were designed, plus a reference one (Ref.) prepared only with natural calcareous aggregates, which was used as a control to evaluate the effect of the recycled CS aggregates on the properties of the concrete exposed to elevated temperatures. The nomenclature of each batch was based on the employed aggregate types and their substitution percentage: i.e. 'CSFA50' indicates that 50 vol% of the fine NA were replaced with CS fine aggregates, while 'CSCA100' refers to a mixture in which 100 vol% of the coarse NA was replaced with CS coarse aggregates. An effective w/c ratio of 0.44 was selected in this study, which is a common ratio used in concrete design to ensure the final product's adequate strength and durability. As reported in Table 2, to keep the effective water constant in the different concrete mixtures, the WA of aggregates was considered and their moisture content was measured before being used (NFA, 1.9%HR; CSFA, 1.3%HR; NCA, 0.4%HR; and CSCA, 0.2%HR).

Cement type CEM II/B-L 32.5 N, supplied by the local company Élite Cementos S.L. (Castellón, Spain), was employed in all the concrete mixes. As established by European Standard EN 197-1, this PC contains 65–79 % clinker and up to 20 % limestone, being more sustainable and environment-friendlier than the CEM I type. The fluidity and workability of mixtures were improved by adding a constant amount of superplasticiser MC-Powerflow 3200, supplied by the German company MC-Bauchemie.

2.3. Concrete's thermal conductivity

Provided that concrete is composed mainly of aggregates (60–80 %) [30], they significantly influence the heat conduction in material, which influences the behaviour of the developed concrete when exposed to elevated temperatures. In the present study, the thermal conductivity of each concrete mixture was determined by the Transient Line Source method according to UNE EN 12664-1. Conductivity was measured at room temperature under dry conditions using 100 mm concrete cubes. To ensure that measurements were representative of the whole specimen, each sample was drilled 5 times in different positions. The mean thermal conductivity values, together with standard deviation, are summarised in Fig. 5. As observed, lower conductivity values were recorded with increasing CS contents and, for a given percentage of NA substitution, lower results were obtained with CS gravel than with CS sand. So the thermal conductivity of the CSCA100 concrete lowered by 28 % compared to the Ref. sample. The conductivity of the Ref. concrete was consistent with the values set out by Eurocode 2 Part 1.2 (EN 1992-1-2) at room temperature because fell within the 1.33–1.95 W/m·K range. Values also fall in line with those reported in the review by Fernandes et al. [19], according to whom the thermal conductivity values of normal-strength concrete developed with NA varied within the 1.4–3.6 W/m·°C range at room temperature. The lower thermal conductivity values of the developed RAC samples were attributed to the lower conductivity of the recycled CS particles. These results are expected to improve the fire resistance of concrete structures because, as explained by Xing et al. [30], although lower λ values increase the thermal gradient in concrete exposed to fire, which leads to higher thermal stresses, they improve the protection of steel reinforcements.

Table 2
Concrete mix proportions.

| ID | Cement (kg) | Fine aggregates | | Coarse aggregates | | Water | | | Super-plasticiser (kg) |
|---------|-------------|-----------------|-----------|-------------------|-----------|---------------------|-----------------|-----------------|------------------------|
| | | NFA (kg) | CSFA (kg) | NCA (kg) | CSCA (kg) | Effective water (L) | Effective (w/c) | Total water (L) | |
| | | Natural | Stoneware | Natural | Stoneware | | | | |
| Ref. | 425 | 651 | – | 1082 | – | 185 | 0.44 | 207.7 | 2.89 |
| CSFA50 | | 325.5 | 277.3 | 1082 | – | | | 206.2 | |
| CSFA100 | | – | 554.6 | 1082 | – | | | 204.6 | |
| CSCA50 | | 651 | – | 541 | 460.9 | | | 208.6 | |
| CSCA100 | | 651 | – | – | 921.7 | | | 209.5 | |

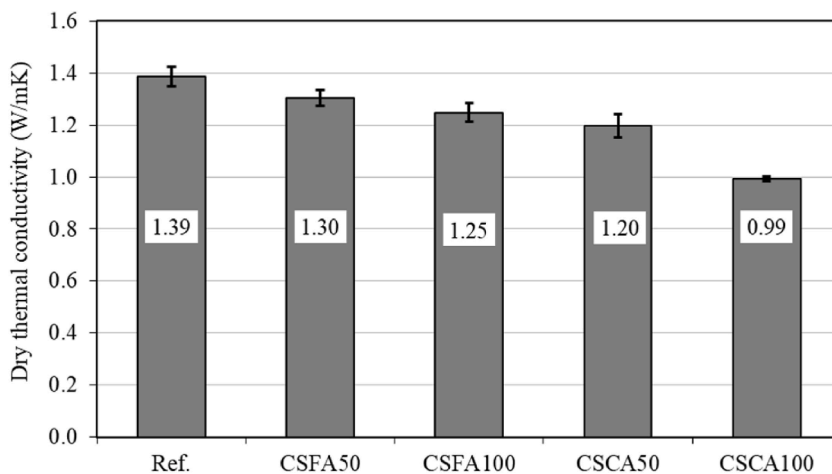


Fig. 5. Thermal conductivity of the developed concrete mixtures.

2.4. Heating-cooling methodology and tests

The concrete samples were tested under three different conditions: hot air, air-cooled and water-cooled. For each one, 16 cubes (100 mm side) were prepared. Three of these cubes were tested after exposing concrete to each temperature (20 °C, 200 °C, 400 °C, 600 °C, 800 °C). A thermocouple was inserted in the remaining cube to control the temperature of the samples inside the furnace. An additional cube was prepared for each mixture to evaluate its conductivity at room temperature. This involved 235 samples: 75 were tested in hot, 75 after air cooling, 75 after water cooling, five samples were used to control temperatures when heated inside the furnace and the remaining five samples were employed to determine thermal conductivity. The large number of samples and conducted tests enabled a comprehensive evaluation of the RAC developed under different temperature and cooling conditions.

Once mixed, the concrete specimens were cured under controlled laboratory conditions. They were covered with plastic sheets to prevent water evaporation during the first hours of the curing process. After 24 h, samples were demoulded and left inside a curing chamber for 28 days at 20 °C and 100 % relative humidity.

As described by Kodur [31], explosive spalling generally occurs within the 480-510 °C range when concrete is exposed to rapidly rising temperatures. It is influenced mainly by the density and moisture content of concrete because dense microstructures may bring about high vapour pressures inside capillary pores when heating [11]. Although, according to Peng and Huang [32], no spalling occurred in concrete with strength values lower than 60 MPa no matter what the moisture content, to work safety and to minimise the possibility of spalling, the samples in the present study were dried in an electric furnace at 100 °C for 8 h before being exposed to high temperatures. The furnace used to heat the concrete samples has a nominal power of 7.5 kW and may operate at temperatures of up to 1,300 °C (Fig. 6).

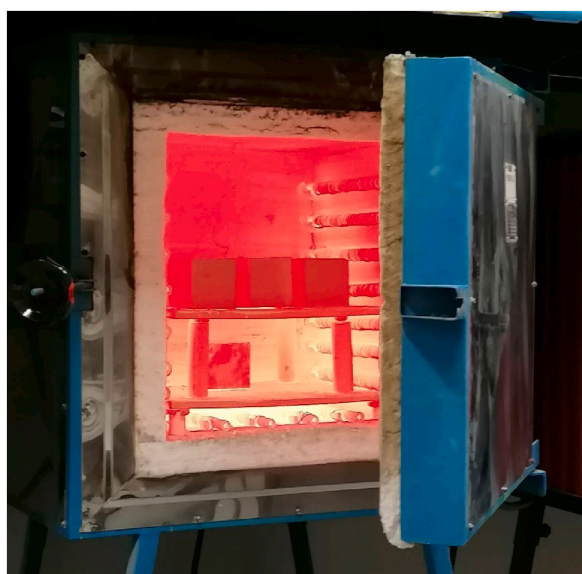


Fig. 6. Furnace used to heat the concrete samples.

As a reference to be compared to the samples exposed to elevated temperatures, the compressive strength of each concrete mixture was determined at room temperature (20 °C) without being heated in the furnace. Fig. 7 shows the heating methodology followed to guarantee that concrete samples were exposed to the desired temperatures. The furnace was set at 50 °C higher than that desired temperature and, when the thermocouple inserted into the corresponding sample indicated that the specimen centre had reached the pertinent temperature, three samples were taken out of the oven to be tested. The furnace was then set to the next temperature and the process was repeated. Samples were exposed to each temperature for approximately 2 h, which allowed homogeneous heat distribution throughout them.

To ensure that the temperature of samples during hot tests came as close as possible to that at which specimens were inside the furnace, these compression tests were conducted immediately after removing samples from the furnace (Fig. 8).

In addition to hot tests, two different cooling conditions were applied: in water, which simulates the method used by firefighters; in air, which simulates real field conditions where concrete that has been exposed to high temperatures naturally cools. To rapidly cool specimens, they were placed inside a water tank for 30 min, and then removed and left at room temperature for 24 h. The naturally cooled samples were left in the laboratory at room temperature outside the oven for 24 h. Fig. 9 shows photographs of the CS-FA50 specimens, prepared by replacing 50 vol% of the natural sand with recycled CS sand after being exposed to different temperatures and cooled in water (left) and in air (right), and before determining their compressive strength.

The compressive strength tests were conducted according to Standard EN 12390-3 in a hydraulic press with a maximum capacity of 1500 kN and at 6 kN/s until the specimen fails. As previously explained, each compressive strength value provided in the Results and Discussion section was obtained as an average of three tested samples.

To ensure that variations in the properties of the different concrete samples developed were exclusively attributed to the type of aggregate used, all the stages of the process were exhaustively controlled: design, mixture, curing, drying, heating in the furnace, cooling and testing. This was essential to guarantee that all the samples were prepared under homogeneous conditions.

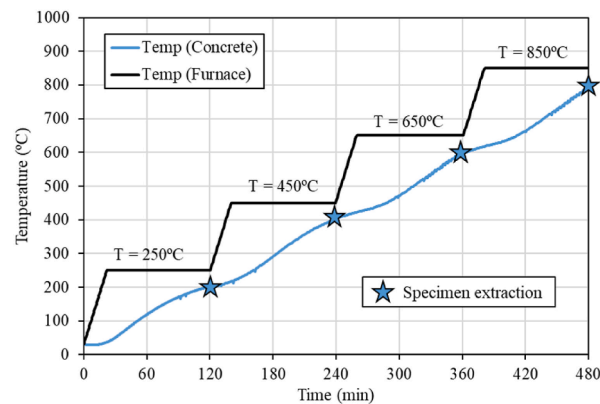


Fig. 7. Temperature evolution in the furnace and inside concrete samples.



Fig. 8. Broken sample after hot testing at elevated temperature.



Fig. 9. Photographs of the CSFA50 specimens after being exposed to different temperatures and cooled in water and in air.

3. Results and discussion

3.1. Compressive strength

The compressive strength of the different concrete mixes defined in Table 2, determined at room temperature, is presented in Fig. 10. For every mixture, the mean strength values of the three tested specimens, together with standard deviation (error bars), were plotted. The mean strength of all the samples fell within the 35–45 MPa range. This implies that, at room temperature, replacing natural calcareous aggregates with recycled CS aggregates had no significant effect on the strength of the developed concrete samples. As observed, after 28 curing days the strength of RAC was similar or slightly better than that of the Ref. sample. Although these results are attributed mainly to common concrete dispersion, they could also be partially explained by the greater hardness of the ceramic particles (see Section 2.1.1) and by some pozzolanic reaction between the ceramic particles and the binding matrix, which would improve the interfacial transition zone (ITZ) between them. Indeed Mas et al. [7] and Pitarch et al. [22], who used this specific CW type to partially replace PC in mortars, observed pozzolanic reactions with the Portlandite released during PC hydration, which started being significant after 28 curing days.

Room temperature strength values were used as a reference to determine samples' relative compressive strength after exposing them to elevated temperatures (Figs. 11 and 12). As every mixture used as a reference its corresponding compressive strength at room temperature, the differences observed among concrete samples were eliminated. Variations in residual strength depending on the testing methodology can be clearly observed by grouping results according to the different concrete mixes (see Fig. 11). In line with the previous reviews by Kodur [31] and Ma et al. [33], the Ref. sample exhibited progressive strength reduction with temperature. According to Kodur [31], no significant strength loss was generally observed up to 400 °C for the normal strength concretes

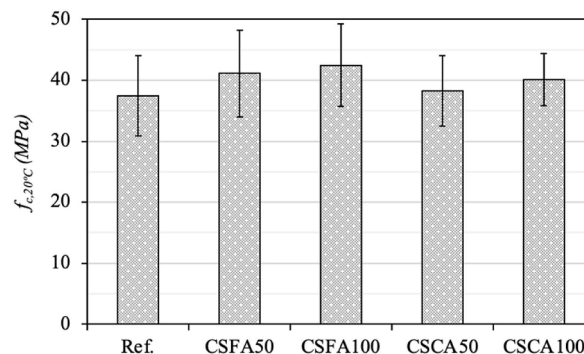


Fig. 10. Compressive strength of each concrete mix at room temperature.

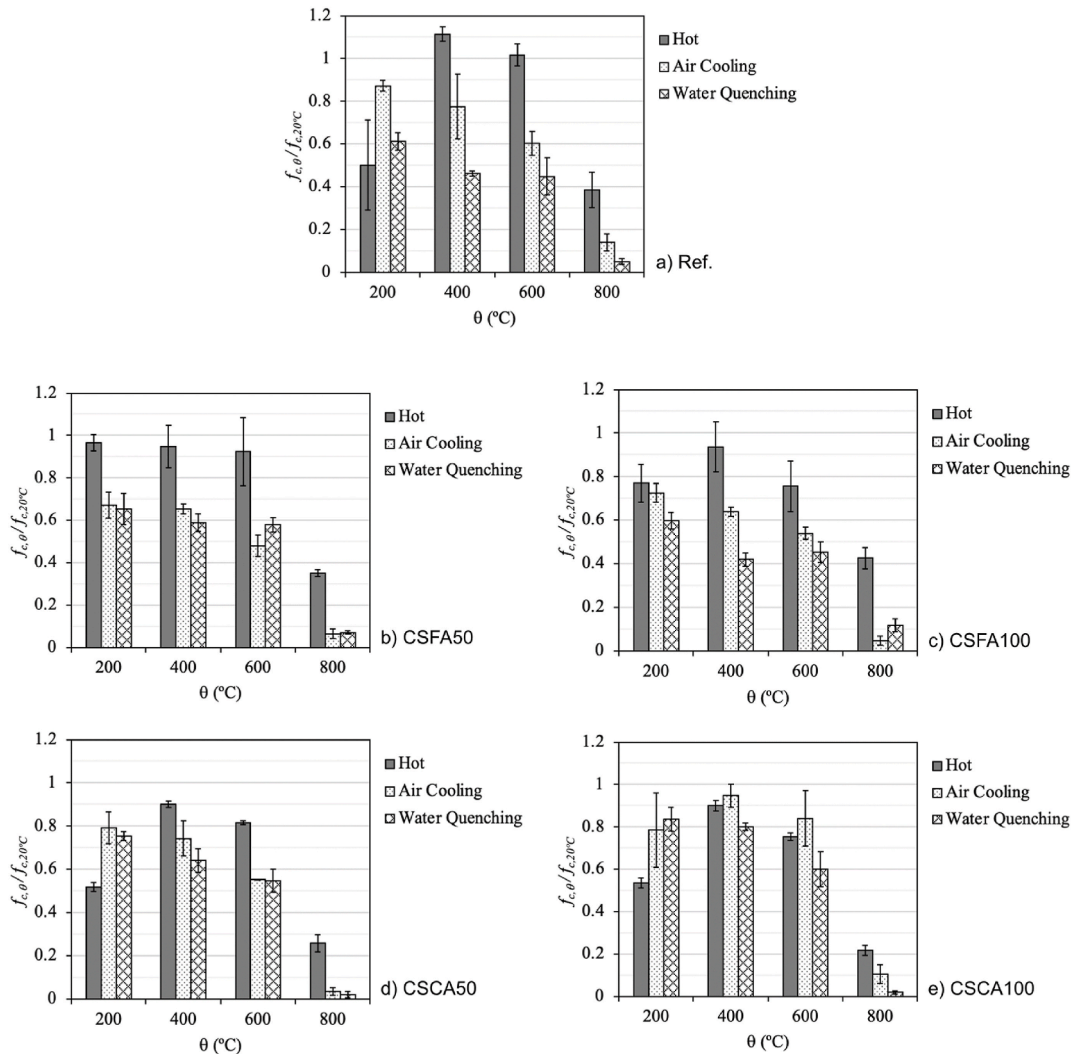


Fig. 11. Relative compressive strength of specimens after exposure to elevated temperatures. Grouped depending on the concrete mix: a) Ref.; b) CSFA50; c) CSFA100; d) CSCA50; e) CSCA100.

(20–50 MPa after 28 curing days), and residual relative strengths varied within the 0.25–0.8 and 0.2–0.55 range after exposure to 600 °C and 800 °C, respectively. In their review, Ma et al. [33] differentiated three main regions, with minor variations in compressive strength up to 300 °C; a marked reduction from 300 °C to 800 °C; and complete strength loss after 800 °C. Both reviews reported a wide variation in the residual compressive strength values, which was explained by the large number of results reported in the literature, and by differences in experimental processes: shape of specimens, loading and heating rates, the w/c ratio, the mixing process, curing conditions, type of used aggregates or employed admixtures.

As observed in Fig. 11, the compressive strength obtained in hot was generally higher than that recorded after cooling. These results agree with the previous reviews by Fernandes et al. [19] and Ma et al. [33], who also reported higher strength values in hot than in cooled samples. In the present study however, the strength obtained in hot at 200 °C in samples Ref., CSCA50 and CSCA100 was lower than that recorded in the cooled specimens, and was also lower than the strength values obtained in hot at higher temperatures. Although all the concrete samples were dried for 8 h at 100 °C before being introduced into the furnace to be exposed to elevated temperatures, the rupture of these specimens under compression was accompanied by a sudden release of steam. Additionally, according to previous research on the microstructural evolution of concrete at elevated temperatures, no significant microstructural changes occur up to 300 °C [33,34]. So, the unusual behaviour observed at 200 °C is attributed to the pressure generated by the evaporation of the residual moisture inside these concrete samples, which could not completely evaporate when drying the sample and generated further internal pressure in pores. The review by Ma et al. [33] also observed that up to 400 °C, the strength values tested in hot were lower than those obtained in cooled samples, and they also attributed this behaviour to free water evaporation, which generated vapour pressure in pores. As in the present study, the tendency reversed after 400 °C, and better results were obtained in hot than in cooled samples. The authors [33] attributed this behaviour primarily to CaO rehydration during cooling because this is a highly expansive reaction. Additional thermal stresses are also generated by shrinkage of aggregates during cooling.

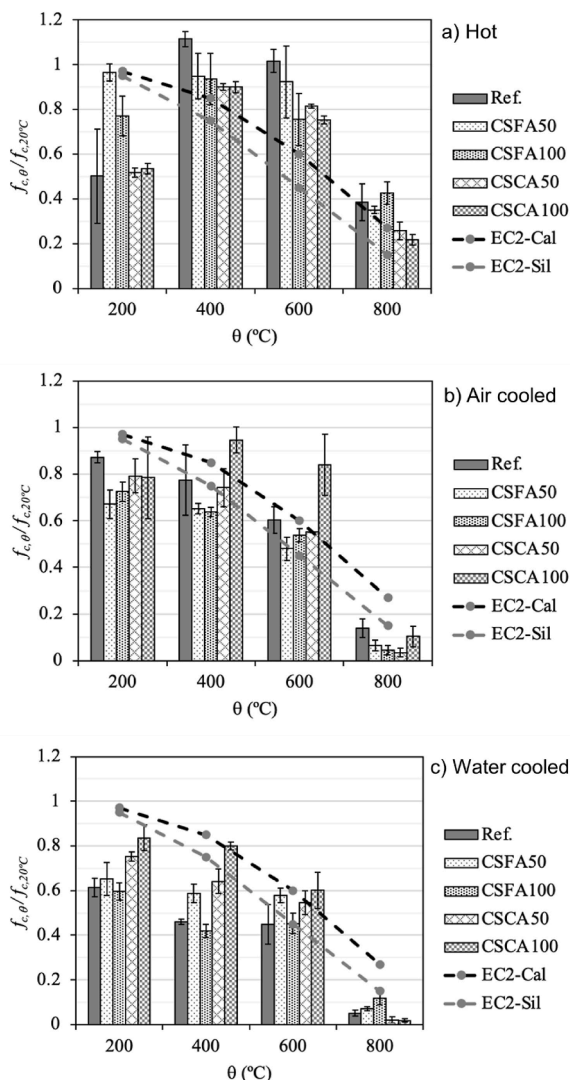


Fig. 12. Relative compressive strength of specimens after exposure to elevated temperatures. Tested: a) Hot; b) after air cooling; c) after water cooling.

In general, all the samples exhibited progressive strength reduction with rising temperatures. Tantawy [34] associated concrete strength loss with elevated temperatures to five different factors: i) coarsening of pores as a result of water loss in the cementitious matrix; ii) the vapour pressure that originated in closed pores; iii) stress in the ITZ due to the different thermal behaviour between aggregates, which usually expand during heating, and the cementitious matrix, which shrinks due to the dehydration of cementitious products; iv) decomposition of portlandite and C–S–H gel; v) cracking as a result of lime rehydration.

As described by Ma et al. [33], up to 100 °C, free and physically absorbed water evaporates, chemically bonded water starts being lost at 105 °C, and ettringite and monosulfate (Aft/AFm) dehydration occurs within the 110–150 °C range. According to the authors [33], these changes result in the progressive increase in both porosity and pore size, which leads to strength reduction. However, Tantawy [34] observed that the vapour pressure originated by water evaporation and the dehydration of PC compounds promoted the reaction of non-hydrated PC phases and the formation of new hydration products that derived from pozzolanic reactions. So, before portlandite decomposition, it may convert into C–S–H gel if accelerated pozzolanic reaction occurs due to the internal vapour pressure that originates in the system. The newly-formed phases would fill some pores and, consequently, could reduce total porosity.

Several studies [29,32–34] agree that no significant microstructural changes and strength loss were generally observed up to 400 °C because, at this temperature, CaCO_3 and the main phases of the binding PC matrix (C–S–H gel and $\text{Ca}(\text{OH})_2$) remain unaltered. However, coarsening of the pore structure occurs, which may be linked with the formation of cracks that reduce compressive strength. From 450 °C, the main changes to occur in the binding matrix are the decomposition of portlandite and calcium silicate hydrates. While portlandite dehydration is a single-step reaction that initiates at approximately 450 °C, C–S–H gel dehydrates within the 110–450 °C range and its decomposition to $\beta\text{C}_2\text{S}$ and C_3S starts at approximately 560 °C, occurring over a wide range of temperatures up to approximately 750 °C [32–34]. The thermal decomposition rate of C–S–H significantly rises with temperature, and is the main

parameter to determine concrete strength loss from 600 °C. Calcite decarbonation occurs within the 580-900 °C range and, as explained by Peng and Huang [32], while portlandite completely decomposes after 20 min, calcite decomposes more slowly. Above 1,000 °C, concrete sintering occurs with complete loss of strength. As highlighted by Peng and Huang [32], temperatures within the 400-800 °C range are critical for the compressive strength of concrete, and most strength is lost from 600 °C to 800 °C.

In addition to the strength loss observed with rising temperatures, the residual strength obtained after cooling specimens was generally lower than that recorded in hot. Accordingly, Tantawy [34] detected secondary portlandite formation when rehydrating PC that had been previously fired to 600 °C or 750 °C. As explained by Ma et al. [33], although $\text{Ca}(\text{OH})_2$ decomposition is not generally harmful for the mechanical properties of concrete, the rehydration of the CaO released while exposing concrete to elevated temperatures brings about significant expansion, and leads to the formation of cracks and strength reduction. The deterioration caused by CaO rehydration is influenced by factors, such as the WA rate or the initial mineralogy of PC. This, together with the thermal-shock produced by water cooling, which augments temperature differences between the centre and the surface of samples, explains the greater strength loss generally recorded in water-cooled than in air-cooled samples. The obtained results fall in line with those previously reported by Fernandes et al. [19], who also observed a more significant strength reduction in water-than in air-cooled samples. Although this tendency seems broken in the CSPA100 samples exposed to 200 °C, at this temperature it was the only series that showed higher average strength when water-cooled instead of air-cooled, but the differences are not significant and fall within the standard deviation. Additionally, at this temperature, there was no decomposition of the aggregates or the main compounds of the binding matrix [29,32-34], which implies that no expansive rehydration reactions occurred during cooling.

It must be noted that the differences between the strength obtained in hot and after cooling were generally more significant in the Ref. sample than in the RAC specimens, especially when coarse recycled aggregates were employed. As, according to Fig. 4, dolomite decomposition initiated at approximately 560 °C, no significant amounts of CaO from the decomposition of NA were expected at 600 °C. So, at 600 °C, the smaller strength differences observed in RAC series depending on the testing condition, are partially explained by the presence of smaller amounts of portlandite, due to its partial consumption during pozzolanic reactions between the CS recycled aggregates and the binding matrix. At 800 °C, the improved residual strength observed in the water-cooled CSFA100 samples as compared to those air-cooled is explained by the combination of both effects, portlandite consumption and lower CaCO_3 contents in the system. The rehydration of CaO was limited in this sample due to the absence of natural calcareous sand, which typically decomposes from 580 °C, and to lower amounts of portlandite, due to pozzolanic reactions with the CS recycled sand. This behaviour is consistent with that observed in CSFA50 samples, which contained 50 % natural dolomitic sand and displayed an intermediate behaviour between CSFA100 and Ref. series. CSFA100 results differ from those observed in the CSPA100 mixture because the latter was prepared with 100 % natural sand (see Table 2) and the larger size of the recycled CS gravel reduces its reactivity.

CSPA100 was the only sample to generally provide better residual strength after air cooling than when tested in hot (Fig. 11e). In this sample, the differences between the results obtained in hot and after water cooling were significantly lower than in the other developed concrete samples. Given that the volume of particles was kept constant, differences were attributed mainly to different aggregates behaviour when cooling the concrete sample. This was associated with more CSPA100 sample homogeneous behaviour compared to those prepared by replacing 50 vol% of NA (CSPA50 and CSFA50), together with greater stability of the CS recycled gravel compared to CS sand.

Strength results have also been presented in Fig. 12 depending on the methodology followed to determine compressive strength: a) in hot; b) after air cooling; c) after water cooling. The strength reduction factors after exposing concrete to elevated temperatures provided by Eurocode 2 part 1-2 (EN 1992-1-2) for calcareous and siliceous aggregates are also plotted in Fig. 12 for comparison purposes. As Eurocode curves are used to determine the residual strength of concrete structures after exposure to fire, the air-cooled results were those that most closely matched the expected values. As observed, Eurocode already assumes strength differences depending on aggregate type, so that the residual strength expected in concrete prepared with siliceous aggregates is lower than that expected with calcareous aggregates. Although quartz is harder than calcite, the larger strength loss with temperature expected with siliceous particles is attributed mainly to the greater thermal expansion of quartz ($12.5 \cdot 10^{-6} \text{ }^\circ\text{C}$) and its allotropic transformation, from α -quartz (density of 2.65 g/cm^3) into hexagonal β -quartz (density of 2.53 g/cm^3), which generally occurs at 573 °C and is accompanied by intensive thermal expansion. Limestone particles degrade further with temperature due to their decomposition into CaO plus CO_2 , which initiates at approximately 600 °C and leads to the formation of cracks inside particles [29].

In hot better results (Fig. 12a) were generally obtained for the Ref. sample, and the relative strength of RAC was similar. The most irregular behaviour was observed at 200 °C, with bigger differences among the RAC samples and better results obtained when replacing natural sand than gravel (CSFA50 and CSFA100 series). As previously explained, this was attributed to the vapour pressure generated inside specimens. At this temperature, the strength of the RAC samples was similar or better than that recorded for the Ref. concrete. According to Savva et al. [35], who investigated the influence of elevated temperatures on concrete developed with three different types of pozzolans (0 %, 10 % and 30 % each), using limestone and siliceous aggregates, up to 200 °C intensive water evaporation generated steam pressure in the pores, which promoted additional pozzolanic reactions and further hydration of unhydrated cementitious phases. In short, at up to 200 °C, steam may enhance pozzolanic reactions between the CS recycled aggregates and the binding matrix.

The residual strength obtained after air cooling (see Fig. 12b) was generally quite homogeneous. The values obtained for the recycled sand concrete (CSFA50 and CSFA100) were slightly lower than those of the Ref.; those of the concrete prepared replacing 50 % gravel were similar; and the strength of the samples prepared replacing 100 % natural gravel were similar to or higher than those obtained for traditional concrete, especially after exposure to 400 °C and 600 °C. In other words, the beneficial behaviour of the recycled CS coarse aggregate was more significant with the total replacement of natural gravel (CSPA100), and became less evident with the 50 vol% replacement (CSPA50). These results were attributed to the higher stability of the recycled aggregates and their lower ther-

mal expansion. Compared to the natural siliceous and calcareous aggregates commonly employed in concrete, no decomposition of the herein used CS recycled aggregates was expected up to 800 °C because, as reported in Section 2.1.3, no significant mass loss occurred up to 1,000 °C. Additionally, lesser thermal expansion-contraction was expected in these ceramic particles because crystalline phases, such as quartz, mullite and feldspar albite, are embedded in an amorphous matrix. The lower thermal expansion coefficient generally reported for ceramic tiles ($4\text{--}8\cdot 10^{-6} \text{ }^\circ\text{C}^{-1}$), compared to that of NA, was also expected to reduce stress in the ITZ because, when exposing concrete to elevated temperatures, aggregates usually expand, while the cementitious matrix shrinks due to the dehydration and decomposition of PC hydrates. These results fall in line with those previously reported by Tufail et al. [29], who investigated the influence of three different types of aggregates (quartzite, limestone and granite) on the properties of concrete exposed to elevated temperatures (up to 650 °C). The best results were obtained with granite, followed by quartzite and limestone. The authors assumed similar changes in the binding matrix, and attributed strength loss differences to the different behaviour of aggregates with rising temperatures. Similarly to the present study, granite particles exhibited greater stability, with lower phase conversion and thermal expansion. The authors explained that the loss of strength recorded with rising temperatures was due to the decomposition of the binding matrix, the different thermal behaviour between aggregates and the cementitious paste, and degradation of aggregates.

Contrary to the hot-strength results, the RAC water-cooled samples (Fig. 12c) exhibited higher strength values than the Ref. As previously explained, this was attributed to both lower CaO content (due to higher portlandite consumption and lower amounts of CaCO_3) and the greater stability of the CS recycled aggregates, which better resisted thermal shock. As, according to Tufail et al. [29], the allotropic transformation of quartz is only reversible when temperatures change very slowly, residual β -quartz is expected to exist in the cooled samples and lower contraction is expected in the CS particles during water cooling.

3.2. Formation of cracks

A systematic analysis of the quantity and size of the cracks that formed after cooling the concrete samples was conducted. Both Figs. 13 and 14 show the photographs of the different concrete mixes after being exposed to the selected temperatures, and air- and water-cooled, respectively. Four cube faces were analysed because the lower and upper cube faces were not considered representative due to their heterogeneity, which could generate variations in crack characteristics compared to the other cube faces.

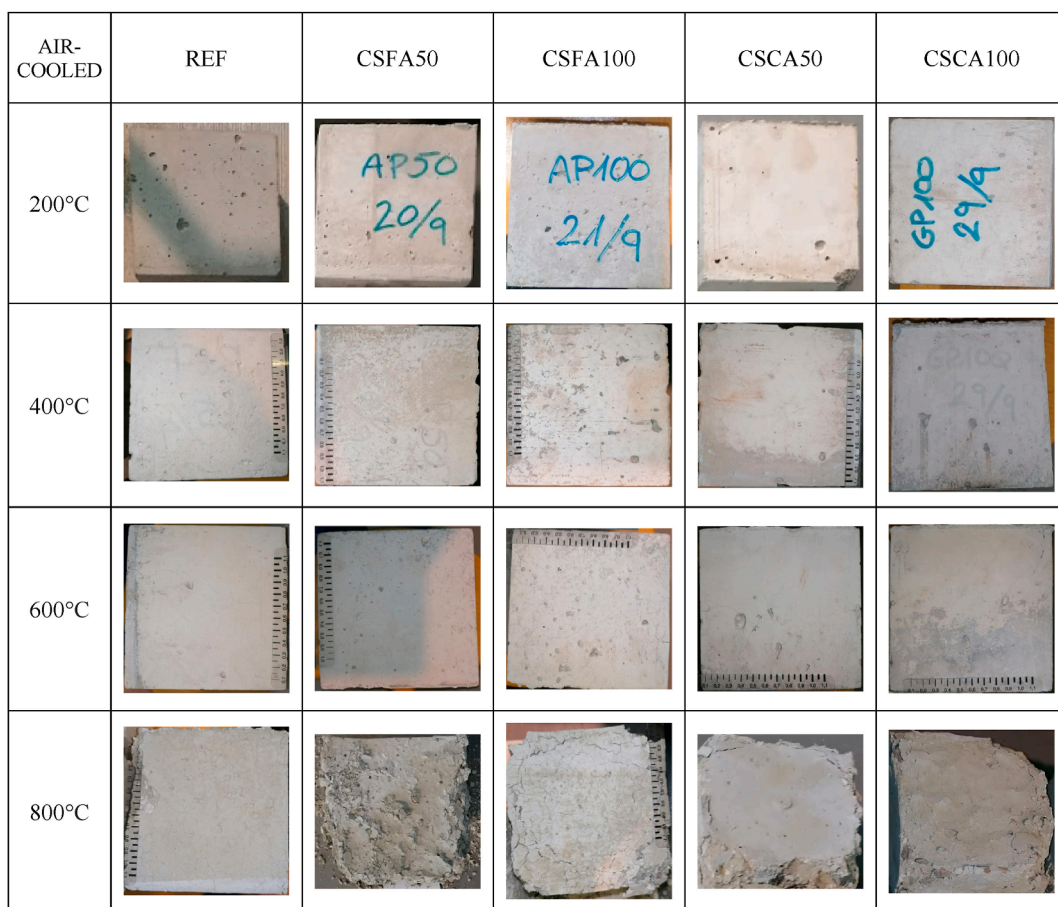


Fig. 13. Photographs of the different concrete mixtures after being subjected to all the temperatures and air-cooled.

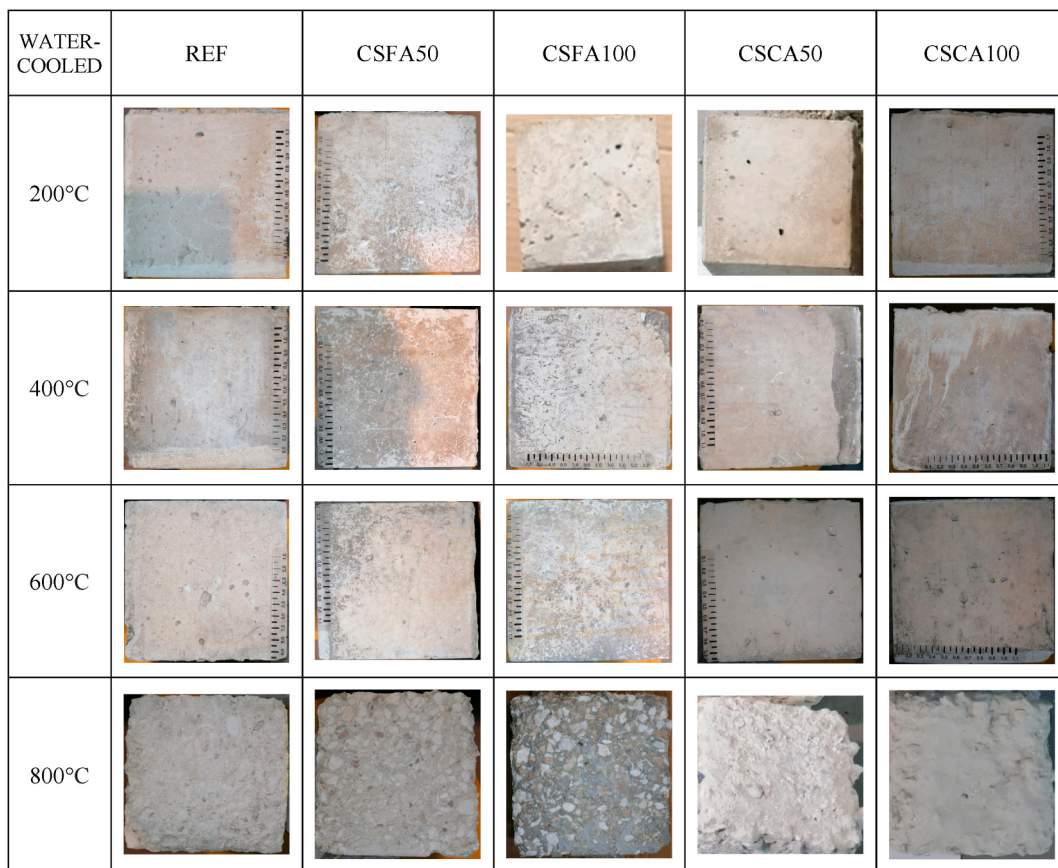


Fig. 14. Photographs of the different concrete mixtures after being subjected to all the temperatures and water-cooled.

As observed in Fig. 13, in line with the compressive strength results, the cracking maps observed after air cooling were homogeneous in all the samples, with less cracking observed in the CSCA100 concrete. As expected, the number and size of cracks increased with temperature. At 200 °C, no cracking was observed in any of the concrete samples. At 400 °C, all the samples, except CSCA100, displaced one slightly cracked face or more. Although almost all the cubes displayed cracking on all four faces at 600 °C, only two of the cubes of sample CSCA100 showed minor cracking, and no cracks were distinguished in the third one. All the samples completely cracked and crumbled when touched after being exposed to 800 °C.

In the water-cooled samples (Fig. 14), no significant cracks were generally observed after exposure to 200 °C (only some cubes had minor cracks on one face). Contrarily to the generally observed trend, at this temperature the CSC100 sample had more cracks than the other concrete mixtures. Although this behaviour was attributed to the vapour pressure generated in pores, no strength reduction was observed compared to the other mixes, as it gave the maximum residual strength (Fig. 12c). At 400 °C, no significant differences were observed in the different concrete mixtures, and some samples displayed cracks on one face or two, while others showed no visible cracks. As after air cooling, water quenching from 600 °C generated cracks on all four faces of all the samples, except CSCA100, which only had cracks on one face of one of the specimens. In both cooling methods, concrete samples completely degraded after being exposed to 800 °C.

In short, although the residual strength of the water-cooled samples was generally lower than that recorded after air-cooling (Fig. 11), at the macroscopic level no significant differences were observed between the cracking maps that formed after air or water cooling. Accordingly, it is worth noting that the strength differences observed between both cooling methods were much less significant in the recycled concrete specimens than in the Ref. sample, and the latter was much more sensitive to the cooling rate. Minor cracks formed in the water-cooled samples at 200 °C, and cracking progressively increased with temperature until total sample degradation after being exposed to 800 °C. The results fall in line with those previously reported by Tantawy [34], who only observed minor cracks on the surface of cementitious samples up to 300 °C, which became wider and deeper at higher temperatures. The authors attributed these findings to the combination of different factors, such as the internal pore pressure caused by water evaporation; thermal shock that originated when cooling the sample; and the increased volume produced by lime rehydration. Ma et al. [33] neither observed microcracks up to 200 °C, but they were distinguished in both the binding matrix and the ITZ from 400 °C, and propagated and intensified with rising temperatures. The authors [33] attributed these microcracks to the stresses caused by different strains between aggregates and the binding matrix because, while the binding matrix shrinks due to the dehydration and decomposition of PC hydrates, aggregates expand with higher temperatures. As explained by Tufail et al. [29], microcracking occurs when the stress gener-

ated in the system exceeds the strength of aggregates or PC hydrates. Consequently, the lower strength of the binding matrix at elevated temperatures, due to changes in its microstructure, facilitates cracking. Given that new surfaces generated in cracks are directly exposed to high temperatures, the temperature gradient may significantly accelerate cracking deterioration.

In line with the compressive strength results, less cracking was generally observed in the CSCA100 concrete compared to the other samples no matter what the temperature was, nor the applied cooling method. Thus, the total natural gravel replacement with the CS recycled aggregates improved residual strength and significantly reduced RAC concrete cracking after exposure to elevated temperatures. These results agree with those previously reported by Xing et al. [30], who investigated the physico-thermal properties of concrete developed with three different types of aggregates (calcareous, silico-calcareous, siliceous) when exposed to elevated temperatures. The authors observed the most cracking when combining siliceous and calcareous aggregates, which was explained by the differential behaviour between both aggregate types that increased stresses. So compared to NA, the lower thermal expansion coefficient of ceramic aggregates reduces their dimensional changes and, consequently, the stress generated in the ITZ. Additionally, decomposition of dolomite particles from 560 °C (see Section 2.1.3) also increases crack formation because, as reported by Xing et al. [30], the carbonates fragmented after exposure to 750 °C due to the rehydration of the CaO released during CaCO₃ decarbonation.

4. Conclusions

This research assessed the performance of RAC developed by replacing natural calcareous aggregates with CS sand and gravel when exposed to elevated temperatures. The used CS was provided by a treatment plant that classifies industrial waste from ceramic tile companies. Based on the obtained results, the following conclusions are drawn:

- Using ceramic waste as a recycled aggregate in concrete has the potential to increase both the sustainability and performance of concrete exposed to elevated temperatures.
- Compared to natural dolomitic aggregates, the CS particles used in this study exhibit greater stability at elevated temperatures, with no thermal decomposition and lower thermal expansion.
- The lower thermal conductivity recorded with increasing CS contents and particle sizes is expected to better protect steel reinforcements, enhancing the fire resistance of concrete structures.
- Replacing natural calcareous aggregates with CS gravel generally improves the residual strength of the concrete exposed to elevated temperatures, and enhancement is more significant with total gravel replacement (CSCA100).
- Aggregates' more homogeneous behaviour and the presence of smaller amounts of CaO in the CSCA100 concrete after its exposure to elevated temperatures, reduce cracking and improve its thermal stability during cooling.

This research proved that the TCW used in the present study may improve the behaviour of traditional calcareous aggregates concrete when exposed to elevated temperatures, especially if high percentages of natural gravel are replaced. Besides improving the fire resistance of the developed concrete, reusing this CW contributes to circular economy, reducing its accumulation in landfills and the consumption of natural resources.

CRedit authorship contribution statement

V. Albero: Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Supervision, Writing – original draft, Writing – review & editing. **L. Reig:** Data curation, Formal analysis, Investigation, Methodology, Supervision, Writing – review & editing. **D. Hernández-Figueirido:** Conceptualization, Funding acquisition, Investigation, Methodology, Project administration, Resources, Supervision, Writing – review & editing. **M. Roig-Flores:** Conceptualization, Funding acquisition, Investigation, Methodology, Project administration, Writing – review & editing. **A. Melchor-Eixea:** Data curation, Formal analysis, Investigation, Writing – original draft. **A. Piquer:** Funding acquisition, Methodology, Project administration, Resources, Writing – review & editing. **A.M. Pitarch:** Methodology, Resources, Writing – review & editing.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: The authors reports financial support was provided by Government of Valencia. The authors reports financial support was provided by University Jaume I. The company Élite Cementos S.L. provided the Portland cement required to conduct this study.

Data availability

Data will be made available on request.

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