Mechanical Characterisation and Shrinkage of Thermoactivated Recycled Cement Concrete

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Abstract: This paper investigates the mechanical and shrinkage behaviour of concrete with recycled cement (RC) thermoactivated from waste cement paste and waste concrete. Overall, compared to ordinary Portland cement (OPC), for the same water/binder ratio, the mechanical strength and ultrasonic pulse velocity were not significantly influenced by the incorporation of RC. The elasticity modulus decreased with the addition of RC and the shrinkage tended to increase at high RC content. The incorporation of up to 15% RC allowed the production of workable concrete with identical shrinkage and similar to higher mechanical strength than concrete with only OPC. RC proved to be a very promising more eco-efficient supplementary cementitious material.

Keywords: recycled cement; thermoactivated recycled cement concrete; mechanical characterisation; shrinkage; waste concrete

1. Introduction

The growing concern with the impact of the construction industry on the environment has prompted investigation works on concrete recycling to reduce the consumption of natural resources and the emissions of greenhouse gas, as well as to repurpose construction and demolition waste [1,2].

For various decades, concrete recycling studies have mainly addressed its use as coarse and fine aggregates as well as low-activity filler additions [3]. In recent years, the attention has shifted towards the production of recycled cement (RC) with rehydration capacity through thermal activation [1,4]. In this still relatively unexplored domain, studies have mostly focused on the microstructure and rehydration behaviour of RC [5–10], as well as the mechanical characterisation of cement pastes and mortars produced with this binder [11–18]. RC pastes have been reported to achieve compressive strengths of up to 32 MPa at 28 days [7], though generally presenting high water demand and low setting times [4,6]. Real et al. [9] recounted similar flexural and compressive strength up to 3 days of pastes with RC treated at 600–700 °C and reference ordinary Portland cement (OPC) paste of the same water/binder ratio (w/b), though, at 28 days, the compressive strength of the RC pastes was about 73% of the OPC paste. In mortars with fine thermoactivated recycled concrete aggregates, Shui et al. [11] also reported no significant evolution in the compressive strength between 7 and 28 days. The early strength development of RC pastes has been attributed to the high surface area of the RC particles [4,9,19]. Moreover, Bogas et al. [10] explained that a portion of the mixing water is directed to the formation of hydration products within the porosity of the RC particles, reducing the amount of water available between anhydrous particles. As such, the w/b becomes lower in bulk RC paste, increasing the proximity between particles, which, in turn, results in the acceleration of particle cohesion, improving the early strength of RC pastes.

Kalinowska-Wichrowska et al. [15] investigated the influence of the treatment temperature and residence period on the mechanical strength of mortars with 25% RC obtained from the cement fraction of waste mortar. The authors found that the incorporation of RC...
treated at 650 °C for 80 min led to the highest compressive strength. Real et al. [9] studied cement pastes with RC treated from 400 to 900 °C, having concluded that 650 to 700 °C led to the optimal rehydration capacity. Bogas et al. [14] reported higher performance for 650 °C.

Florea et al. [12] characterised mortars with 10–30% incorporation of RC treated at 500 and 800 °C, having found that the compressive strength decreased with the incorporation of RC. Carriço et al. [16] also evaluated the influence of different incorporation percentages of RC and of various thermal activation temperatures. The authors found that for up to 50% incorporation of RC treated at 700 °C, the water demand and setting time were not significantly affected. Moreover, the replacement of OPC with up to 20% RC led to minor variation of the workability and mechanical strength of the mortars. The authors also found that mortars with 100% RC achieved up to 27 MPa at 28 days, about 53% of that of OPC mortar with the same w/b. Yu and Shui [20] analysed the mechanical strength of pastes produced with RC from waste cement pastes treated at 650 °C. The authors found that the maximum compressive strength was obtained for an optimal amount of 5–10% RC incorporation. The highest improvement was attained when RC particles were previously dispersed with ethanol and sodium polyacrylate, underlining the relevance of adequate RC de-agglomeration on the mechanical properties.

The incorporation of RC in concrete has barely been studied, with only a few published papers [21,22]. Letelier et al. [21] investigated the combined effect of the incorporation of recycled aggregates and RC in concrete, having incorporated 5–15% RC of varying fineness (75–300 µm) treated at different temperatures (400–900 °C). The authors observed that the incorporation percentage of RC did not significantly influence the compressive and flexural strength, due to the low replacement percentages used in the study.

Qian et al. [22] conducted a study on the incorporation of RC treated at 650 °C in ultra-high performance concrete with a binder composed of lime powder (45.5%), silica fume (18.2%) and varying amounts of OPC and RC, for a w/b of about 0.2 and 3% of superplasticizer by weight of binder. The authors found that up to 25% OPC replacement with RC, the flowability decreased less significantly than for higher incorporation percentages, having attributed the workability reduction to the high surface area of the RC and CaO content. The compressive strength generally tended to decrease with the incorporation percentage of RC. The higher internal void volume owed to the reduction of the workability was pointed as the main reason for these findings. However, at 7 days, the authors observed a slight increase of 12.5% RC compared to the reference concrete, having justified the result with the more effective repolymerisation of RC hydration products at this age. The authors concluded that the optimal incorporation percentage of RC was 25%, promoting the hydration through the nucleation effect of the fine particles of RC, while not significantly reducing the workability.

The influence of RC incorporation on the volume stability of cement-based materials has barely been studied. Qian et al. [22] investigated the autogenous shrinkage of the above-mentioned high performance concrete, having found that the spontaneous shrinkage rate generally increased with the incorporation percentage of RC. This was attributed to the reduction of internal humidity of concrete due to the high surface area of RC, leading to self-drying and spontaneous contraction of concrete.

In sum, there is still insufficient knowledge on the influence of the incorporation of RC in the fresh and hardened behaviour of concrete, which needs to be addressed. Bearing this in mind, this paper aims at characterising the mechanical and shrinkage behaviour of concrete produced with various replacement percentages of OPC with RC, for distinct w/b. To this end, an extensive experimental campaign was carried out, involving the fresh (slump, density) and physical and mechanical (density, compressive strength, tensile splitting strength, ultrasonic pulse velocity, modulus of elasticity) characterisation, as well as the long-term drying shrinkage of concrete produced with partial or total incorporation of recycled cement obtained from waste cement paste or waste concrete.
2. Experimental Programme

2.1. Materials

For this study, CEM I 42.5R, crushed limestone coarse aggregate and natural siliceous sand were selected to produce concrete. Tables 1 and 2 present the main properties of these constituents.

| Parameter                              | Standard   | OPC      | NT       | RCP       | RCC       | CF       |
|----------------------------------------|------------|----------|----------|-----------|-----------|----------|
| Absolute density (g/cm³)               |            | 3.07 (a) | 2.445 (b)| 3.005 (b) | 2.964 (b) | -        |
| BET surface area (cm²/g)               |            | 18134    | 156853   | 78673     | -         | -        |
| Loss on ignition (950 °C) (%)          |            | 2.48     | 20.65    | 8.41      | -         | 28.71    |
| Insoluble residue (%)                  |            | 0.89     | 0.8      | <0.52     | -         | 18.77    |
| SiO₂ + Al₂O₃ + Fe₂O₃ (%)               | EN 196-2 [26] | 19.64 + 5.34 + 3.05 | 14.78 + 3.97 + 2.49 | 19.14 + 5.13 + 3.00 | - | 20.61 + 2.55 + 0.97 |
| CaO + MgO (%)                          | EN 196-2 [26] | 62.80 + 1.80 | 50.08 + 1.29 | 60.79 + 1.77 | - | 40.55 + 0.77 |
| Free CaO (%)                           | EN 451-1 [27] | 0.70     | -        | 13.94     | -         | -        |
| Water requirement (w/b)                | EN 196-3 [28] | 0.31     | -        | 0.74      | 0.50      | -        |
| Setting time (min) initial              | EN 196-3 [28] | 170      | -        | 290       | 375       | -        |
| Setting time (min) final               |            | 280      | 385      | 415       | -         | -        |

(a) Determined according to LNEC E 64 [29]; (b) Determined through helium pycnometry.

The origin paste (OP) was produced with a w/b of 0.55, having reached an average 28 days compressive strength of about 41 MPa. After production, the OP was cured in a controlled environment at 95% relative humidity (RH) up to 7 days and then kept under laboratory environmental conditions for over 90 days. After curing, the OP was subjected to several crushing, grinding and milling processes, in order to decrease their average grain size. First, the OP was reduced into approximately 7 cm particles by means of two jaw crushers with distinct jaw openings. Afterwards, the OP ran twice through a roller mill, leading to particle sizes smaller than 2 mm, and then was further ground in a horizontal steel ball mill for about 2 h. Finally, the fine OP particles were passed through a 250 µm sieve. The thermal activation of the waste cement paste was accomplished in a rotary tube furnace from Thermolab Scientific Equipments. The waste material was heated at 10 °C/min, up to 150 °C, where it remained for 1 h, followed by heating at 10 °C/min until the treatment temperature of 650 °C, where it remained for 3 h. Afterwards, the binder was cooled inside the oven until ambient temperature. The main phase composition of the binders was analysed by means of x-ray diffraction (XRD) and thermogravimetric (TG) analyses. For the XRD analysis, the samples were scanned at a 5–60 °C 2θ range, with a step size of 0.03° and a step time of 100 s, using a PANalytical X’Pert Pro diffractometer with CuKα radiation. The TG analysis was performed using a PC LUXX Thermobalance with a heating ratio of 10 °C/min, from ambient temperature to 950 °C. The degree of hydration and the calcium hydroxide (CH) content were determined according the procedure described in Real et al. [9]. The obtained recycled cement paste (RCP), with rehydration capacity, was mainly composed of α’H₂C₂S and CaO, as well as traces of calcite (Figure 1). Moreover, the XRD and TG analyses demonstrated that the thermal treatment at 650 °C enabled the decomposition of the hydration products of the non-treated hydrated cement paste (NT)—namely, calcium silicate hydrates (C-S-H) and CH—without
significant decarbonation (Figures 1 and 2). The amount of CH estimated from TG analysis (11%) suggested the occurrence of pre-hydration during cooling and storage of anhydrous RCP (Figure 2). The TG analysis of NT also indicated that the OP had a sufficiently high hydration degree, over 75%, being representative of well-hydrated old waste concrete.

Figure 1. X-ray diffraction analysis of (a) OPC, (b) NT, (c) RCP and (d) RCC. Legend: Δ—CH; †—CaCO₃; *—ettringite; ∞—gypsum; ©—CaO; ■—C₃S; ▼—C-S-H; ×—β-C₂S; ▲—α’H-C₂S; □—calcium aluminum iron oxide; ◆—C₄AF; Φ—alumohydrocalcite; θ—calcium aluminum oxide carbonate hydrate; +—quartz.

Figure 2. Thermogravimetric analysis of OPC, recycled cement paste (RCP), non-treated cement paste (NT) and non-treated concrete cement paste (NTC).

The origin concrete (OC) was produced with the same composition of the reference OPC concrete, with w/b of 0.55 (R55), presented in Table 3. In an initial phase, the OC was cured and subjected to crushing and grinding stages in a similar manner to that used for the OP. Then, the cementitious fraction was separated from the remaining concrete constituents, according to a new procedure patented by the authors [30]. Finally, the separated waste concrete cement was subjected to the same thermal treatment of waste cement paste. The obtained recycled cement from concrete (RCC) presented similar crystalline compounds to those of the RCP, except for quartz, which is attributed to some residual sand from the OC (Figure 1). Furthermore, in the RCC, the calcite peaks were more intense than in the RCP, also due to the presence of residual limestone aggregate from the OC (Figure 1). Based on TG analysis and a simple acid attack procedure, RCC was estimated to be composed of about 74% cement fraction, 16% sand and 10% limestone by weight of binder.
Table 3. Concrete mixture compositions.

| Designation | w/b | M_{binder} (kg/m^3) | Additions (%) | V_{coarse aggregate} (L/m^3) | V_{fine aggregate} (L/m^3) | V_{water} (L/m^3) | SP (% of M_{binder}) |
|-------------|-----|---------------------|--------------|-------------------------------|---------------------------|------------------|--------------------|
| R35         | 0.35| 450                 |              | 406                          | 270                       | 157.5            | 0.5                |
| R55         | 0.55| 360                 |              | 400                          | 266                       | 198              | -                  |
| R65         | 0.65| 360                 |              | 378                          | 252                       | 234              | -                  |
| 5P55        | 0.55| 360                 | 5%RCP        | 399                          | 266                       | 198              | -                  |
| 15P35       | 0.35| 450                 | 15%RCP       | 404                          | 269                       | 157.5            | 1.0 (a)            |
| 15P55       | 0.55| 360                 | 15%RCP       | 399                          | 266                       | 198              | -                  |
| 15P62       | 0.62| 360                 | 15%RCP       | 384                          | 256                       | 223.2            | -                  |
| 15P65       | 0.65| 360                 | 15%RCP       | 377                          | 252                       | 234              | -                  |
| 30P55       | 0.55| 360                 | 30%RCP       | 398                          | 266                       | 198              | 0.4                |
| 40P55       | 0.55| 360                 | 40%RCP       | 394                          | 263                       | 198              | 1.5 (a)            |
| 100P65      | 0.65| 360                 | 100%RCP      | 373                          | 248                       | 234              | 3.8 (a)            |
| 15C55       | 0.55| 360                 | 15%RCC       | 398                          | 265                       | 198              | -                  |
| 30C55       | 0.55| 360                 | 30%RCC       | 397                          | 265                       | 198              | 0.2                |
| 15NT55      | 0.55| 360                 | 15%NT        | 397                          | 264                       | 198              | -                  |
| 15CF55      | 0.55| 360                 | 15%CF        | 398                          | 265                       | 198              | -                  |
| 30CF55      | 0.55| 360                 | 30%CF        | 396                          | 264                       | 198              | -                  |

(a) Water content of SP accounted for in the V_{water}, according to EN 206 [31].

For comparison purposes, non-treated waste cement paste (NT), sieved at 250 µm, and waste concrete filler (CF), sieved at 150 µm, were also considered as nearly inert additions. The CF was obtained from the above mentioned method of waste concrete separation, corresponding to the fine fraction below 150 µm, that could not be processed. The properties of these materials are presented in Table 2. As shown in Figure 3, RCP was produced with slightly coarser particle size distribution than OPC, especially over 10 µm. Moreover, the RCC presented about 50% less surface area than RCP, due to the contamination of non-porous natural aggregate (Table 2). The particle size distribution of CF was markedly coarser and less extensive than the other selected materials. Finally, a polycarboxylate based superplasticizer (SP) was also adopted in mixtures with a low w/b (0.35) or high water requirement.

![Figure 3. Particle size distribution of the cements and additions.](image_url)

2.2. Composition and Production of Recycled Cement Concrete

For this study, 16 compositions were selected, covering concrete with different incorporation percentages of RCP (0, 15, 30, 40 and 100%), RCC (0, 15 and 30%), NT (0 and 15%) and CF (0, 15 and 30%), as well as w/b between 0.35 and 0.65, as presented in Table 3. The compositions in Table 3 were designated by “R”, “P”, “C”, “NT” or “CF”, for reference
concrete with CEM I and concrete produced with RCP, RCC, NT or CF, respectively. This nomenclature was preceded by the replacement percentage and followed by the w/b of concrete. For 15% incorporation of RCP, an extra concrete was produced with a w/b of 0.62, in order to have the same workability of the reference OPC concrete with a w/b of 0.55 (R55). For the same w/b, SP had to be added in concrete with more than 15% RCP.

The concrete was produced in a vertical shaft mixer. The aggregates were mixed with part of the water for about 2 min and then rested for about 1 min. Afterwards, the binder was added with the remainder mixing water. For mixtures with low w/b or high incorporation percentages of RCP, the SP was slowly incorporated in the mixture with about 10% of the water, 1 min later after the addition of other components. The total mixing time was about 8 min.

2.3. Test Methods

The produced concretes were tested in the fresh state, for slump and fresh density, and in the hardened state, for dry density, compressive strength, splitting tensile strength, ultrasonic pulse velocity, modulus of elasticity and shrinkage. The slump and fresh density were determined according to EN 12350-2 [32] and EN 12350-6 [33], respectively. After demoulding, all produced specimens, except those for drying shrinkage, were kept in an environment of over 95% RH until the testing age (28 days). The dry density was determined from two 100 mm cubic specimens per composition, according to EN 12390-7 [34]. The compressive strength and ultrasonic pulse velocity were tested in three 150 mm cubic specimens per composition and testing age, according to EN 12390-3 [35] and EN 12504-4 [36], respectively.

The ultrasonic pulse velocity (UPV) was determined by direct transmission, using a PUNDIT portable ultrasonic non-destructive digital indicating tester. The time taken by a pulse to travel through the concrete was determined with an accuracy up to 0.1 µs, using 54 kHz transducers positioned in the middle of each opposing concrete cast face. Finally, UPV is the ratio between the length traveled by the pulse (150 mm) and the measured time.

The splitting tensile strength and the modulus of elasticity were performed on three φ150 × 300 mm cylinders each per composition, according to EN12390-6 [37] and LNEC E 397 [38], respectively. For the modulus of elasticity, two linear variable displacement transducers of 25 mm stroke were applied, operating over an initial gauge length of 150 mm. The test involved, at least, 8 cycles of loading at a rate of about 0.5 ± 0.01 MPa/s, and unloading, with an applied stress varying between 1 MPa and 1/3 of the estimated compressive strength, until the difference between the average strain for consecutive cycles was lower than 10%.

The drying shrinkage was determined from two 350 × 100 × 100 mm prisms per studied composition, according to LNEC E 398 [39]. Two steel pins were glued to each specimen, 200 mm apart, and the drying shrinkage was monitored over time by means of a DEMEC mechanical strain gauge with a precision of 1 µm and a gauge length of 5 mm. After demoulding, the specimens were kept in a controlled chamber with 20 ± 2 °C and 50 ± 5% RH, over the measurement period, which lasted up to 90 days.

3. Results and Discussion

The average results of slump, fresh, \( \rho_{\text{fresh}} \), and dry, \( \rho_{\text{dry}} \), density, compressive strength, \( f_{\text{cm}} \), at 3–90 days, splitting tensile strength at 28 days, \( f_{\text{ctm},28d} \), ultrasonic pulse velocity at 28 days, \( \text{UPV}_{28d} \), and modulus of elasticity at 28 days, \( E_{\text{cm},28d} \), of the produced concretes, as well as the respective coefficients of variation (CV) are presented in Table 4. The long-term drying shrinkage results are presented in Section 3.6. The coefficients of variation of all studied properties were not significantly affected by the type of binder.
Table 4. Test results of the produced concretes.

| Designation | w/b | Slump (mm) | Designation | w/b | Slump (mm) |
|-------------|-----|------------|-------------|-----|------------|
| R35         | 0.35| 150        | R55         | 0.55| 150        |
| R65         | 0.65| Fluid      | 2280        | 8   | 2100       |
| R65         | 0.65| Fluid      | 2280        | 8   | 2100       |
| 30P55       | 0.55| 110        | 30P55       | 0.55| 110        |
| 15C55       | 0.55| 120        | 15C55       | 0.55| 120        |
| 30C55       | 0.55| 130        | 30C55       | 0.55| 130        |
| 15NT55      | 0.55| 110        | 15NT55      | 0.55| 110        |
| 15CF55      | 0.55| 130        | 15CF55      | 0.55| 130        |
| 30CF55      | 0.55| 140        | 30CF55      | 0.55| 140        |

| Characteristics | 28d | 3d | 7d | 28d | 90d | 28d | 28d | 28d |
|-----------------|-----|----|----|-----|-----|-----|-----|-----|
| $\rho_{\text{fresh}}$ (kg/m$^3$) |     |    |    |     |     |     |     |     |
| $V_{\text{voids}}$ (L/m$^3$)    |     |    |    |     |     |     |     |     |
| $\rho_{\text{dry}}$ (kg/m$^3$)  |     |    |    |     |     |     |     |     |
| $f_{\text{cm}}$ (MPa)           |     |    |    |     |     |     |     |     |
| $f_{\text{cm}}$ (MPa)           |     |    |    |     |     |     |     |     |
| $f_{\text{cm}}$ (MPa)           |     |    |    |     |     |     |     |     |
| $f_{\text{cm}}$ (MPa)           |     |    |    |     |     |     |     |     |
| $f_{\text{cm}}$ (MPa)           |     |    |    |     |     |     |     |     |
| UPV (m/s)                    |     |    |    |     |     |     |     |     |
| $f_{\text{cm,sp}}$ (MPa)       |     |    |    |     |     |     |     |     |
| $E_{\text{cm}}$ (GPa)         |     |    |    |     |     |     |     |     |

| R35         | 0.35| 150 | 2360 | 34  | 2330 | 63.6 | 3 | 72.1 | 3 | 84.4 | 1 | 93.5 | 4 | 5180 | 0 | 5.5 | 12 | - | - |
| R55         | 0.55| 150 | 2330 | 13  | 2220 | 33.7 | 1 | 42.0 | 3 | 52.2 | 1 | 59.2 | 0 | 4716 | 0 | 3.6 | 5 | 40.0 | 1 |
| R65         | 0.65| Fluid | 2280 | 8      | 2100 | 27.9 | 1 | 33.4 | 2 | 39.8 | 3 | -   | -   | 4620 | 0 | 2.7 | 2 | - | - |
| 5P55        | 0.55| 130 | 2320 | 17  | 2220 | 35.8 | 1 | 44.5 | 1 | 53.2 | 3 | -   | -   | 4762 | 0 | 4.0 | 7 | - | - |
| 15P35       | 0.35| 100 | 2340 | 35  | 2310 | 76.3 | 3 | 86.1 | 1 | 90.0 | 4 | -   | -   | 5268 | 0 | 5.5 | - | - | - |
| 15P55       | 0.55| 90  | 2290 | 27  | 2190 | 34.7 | 1 | 42.9 | 3 | 52.4 | 1 | 57.9 | 2 | 4771 | 1 | 4.1 | 2 | 38.8 | 3 |
| 15P62       | 0.62| 140 | 2260 | 26  | 2110 | 31.1 | 2 | 35.8 | 1 | 44.6 | 2 | -   | -   | 4650 | 1 | 3.5 | 11 | - | - |
| 15P65       | 0.65| 190 | 2230 | 28  | 2090 | 27.9 | 1 | 33.5 | 1 | 40.1 | 1 | -   | -   | 4580 | 0 | 3.5 | 1 | - | - |
| 30P55       | 0.55| 110 | 2280 | 30  | 2170 | 33.7 | 2 | 40.1 | 0 | 47.8 | 2 | 50.7 | 5 | 4646 | 0 | 3.3 | 36.4 | 2 |
| 15P62       | 0.62| 140 | 2260 | 26  | 2110 | 31.1 | 2 | 35.8 | 1 | 44.6 | 2 | -   | -   | 4650 | 1 | 3.5 | 11 | - | - |
| 15NT55      | 0.55| 110 | 2270 | 38  | 2130 | 29.0 | 2 | 35.8 | 3 | 43.1 | 3 | -   | -   | 4520 | 1 | 3.2 | 11 | - | - |
| 15CF55      | 0.55| 130 | 2300 | 21  | 2150 | 29.5 | 2 | 35.6 | 3 | 43.1 | 3 | -   | -   | 4618 | 0 | 2.7 | 10 | - | - |
| 30CF55      | 0.55| 140 | 2300 | 21  | 2130 | 22.3 | 1 | 26.8 | 3 | 32.8 | 0 | -   | -   | 4524 | 0 | 2.4 | 14 | - | - |
3.1. Slump and Density

In general, the concretes were produced with S3 consistency, according to EN 206 [31], for slumps between 100–150 mm (Table 4). However, as discussed in the literature [4,8,9,19,40], recycled cement has a higher water demand than OPC, which leads to a significant slump reduction for the same w/b. This can be attributed to the high surface area (Table 2), high porosity and subsequent water absorption [4,8,9,19,40], particle agglomeration [16] and free CaO content [9,13]. Therefore, for incorporation percentages of RCP over 30%, high amounts of SP had to be added in order to comply with the S3 consistency class (Table 4). As mentioned, to achieve the same slump, the w/b had to be increased from 0.55 to 0.62 (13%), when OPC was replaced with only 15% RCP.

The incorporation of 15% NT or 15% CF resulted in concretes with higher slumps than that with 15% RCP, but lower than that with only OPC of equal w/b, which can be explained by the intermediate porosity, nearly inert nature and absence of free CaO of well hydrated NT and CF particles.

The partial replacement of OPC with RCC led to higher slumps than those obtained for the same substitution percentage of OPC with RCP. In fact, as mentioned, RCC was contaminated with about 26% non-porous aggregates, which corresponds to a higher water/cementitious fraction ratio (w/cf) for the same w/b. This led to a significant reduction of the water required for the normal paste consistency (about 32%) (Table 2). In other words, the porosity, surface area and CaO content are lower in RCC than in RCP.

Figure 4 displays the workability over time of 15P65. Contrary to what is usually observed in OPC mixtures, a quick workability reduction occurred during the first 15 min, due to the exothermic hydration of free CaO, as well as the RCP absorption [9,14,22]. This loss of workability is expected to increase with the incorporation percentage of RCP. Then, up to 45 min, the slump was less altered. The fresh concrete faces an induction period, as shown by isothermal calorimetry analysis conducted by the authors [9,10] and also documented in the literature [8,19,41]. After 45 min, the workability gradually decreased, due to the progressive hydration and setting of the binder.

The SP was effective in improving the workability of concretes with RCP, having been able to compensate the significantly higher water requirement of RCP than of OPC. However, compared to OPC concretes, greater SP dosages (as high as 3.8%) were necessary for a given percentage water reduction, i.e., the SP saturation point was increased. This was attributed to the porous nature [9] and significantly higher surface area of RCP particles, which was about 8.6 times higher than that of OPC particles (Table 2), increasing the surface adsorption of SP molecules.

Figure 5 shows the fresh density of the produced concretes as a function of the incorporation percentage of additions. As expected, the fresh density was more affected by the w/b than by the incorporation of RCP, which had only slightly lower density than OPC.
(Table 2). This and the correspondent increase in paste volume, for the same w/b (Table 3), contributed to the decrease in the fresh density, up to 2.6%, with the incorporation of RCP. Moreover, as mentioned, due to their higher water demand, RCP concretes tended to present lower workability than OPC concretes of the same w/b, which tended to promote lower compactness and higher air entrainment (Figure 6). This may explain the sharp density reduction of concretes produced with up to 15% RCP, without SP.

![Figure 5. Fresh density, $\rho_{\text{fresh}}$, of concrete with different replacement percentages of OPC with RCP, RCC, NT and CF.](image)

![Figure 6. Slump and volume of voids, $V_{\text{voids}}$, as a function of the incorporation percentage of RCP in concrete with a w/b of 0.55.](image)

Knowing that RCC, NT and CF have lower particle density than OPC, the fresh density of concrete decreased with the replacement percentage of OPC (Figure 5). The incorporation of 15% of RCP, RCC, NT and CF resulted in a 1.7, 1.7, 2.6 and 1.3% reduction of the fresh density, respectively.

In general, the reduction of dry density as a function of the incorporation of RCP followed the same trend of fresh density. Similarly, the dry density was more affected by the w/b than by the amount of RCP (Figure 7). For the same w/b, the dry density only decreased up to 2.7% with the incorporation of RCP. Moreover, the average difference between the fresh and dry density was similar in RCP and OPC concrete of equal w/b (Table 4). This suggests that RCP and OPC tended to bind similar amount of hydration water.
The incorporation of 15% RCP, RCC, NT and CF led to a 1.4, 1.4, 4.1 and 3.2% decrease in the dry density, respectively (Figure 7). That is, contrary to concrete with RCP or RCC, in concrete with NT or CF, the reduction of dry density was higher than that of fresh density. In fact, besides the lower density of NT and CF, the replacement of OPC with these additions led to an overall reduction of the hydration products, due to their low reactivity.

3.2. Compressive Strength

Figure 8 shows the compressive strength at 28 days of concretes with different w/b as a function of the incorporation percentage of RCP. Depending on the incorporation percentage of RCP and w/b, the compressive strength varied between 33.2 and 90 MPa at 28 days. These concretes ranged strength classes from C20/25 to C70/85, according to EN 206 [31], making them suitable for the production of common structural concrete.

For the same w/b, the compressive strength was not significantly influenced by the RCP content, even for high incorporation percentages, demonstrating the potential of this new binder. In fact, as also demonstrated in previous studies [9,10], RCP could effectively rehydrate and generate similar hydration products to those produced by OPC. Therefore, RCP is an active binder that clearly distinguishes itself from other non-hydraulic additions.

For the same w/b, the compressive strength tended to increase slightly for incorporation percentages of RCP up to 15%. The maximum increase of about 6.7% was found...
in concrete with a w/b of 0.35. It is possible that the finer RCP particles promoted filler and nucleation effect, improving the hydration development and microstructure refinement, due to their high surface area [16,20,22]. This should be more relevant at early age strengths, as discussed later. For up to 15% replacement of OPC with recycled cement, Yu and Shui [20], Letelier et al. [21] and Qian et al. [22] also reported no significant changes in compressive strength. In this study, the maximum compressive strength in concrete with w/b of 0.55 was attained for 5% RCP. In cement pastes, Yu and Shui [20] reported that the optimum strength was achieved for 5–10% recycled cement in pastes with a w/b of 0.3.

However, the incorporation of more than 15% RCP tended to decrease the compressive strength. This may be attributed to the diluted effect of the OPC amount, as well as the reduction of the workability and consequent compactness of concrete with high incorporation percentage of RCP. As documented by Real et al. [9] in a previous study with an equal recycled cement thermoactivated at 650 °C, the compressive strength at 28 days of RCP pastes was about 28% lower than that of OPC pastes of equal w/b, displaying less long-term development of outer hydration products than OPC. In fact, part of the mixing water is rapidly consumed in the hydration of internal products within the porous structure of recycled cement particles [10].

On the other hand, the estimated volume of entrapped air tended to increase with the replacement of OPC with RCP (Table 4). The high water requirement of RCP reduces the fluidity of concrete and this affects its compactness and, as a consequence, the mechanical strength. The full replacement of OPC with RCP in concrete with a w/b of 0.65 led to an increase of about 12 L/m$^3$ in the volume of voids and to a 17% reduction of the compressive strength at 28 days (Table 4). In addition, the increase in air entrainment for 15% RCP incorporation also contributed for the optimal compressive strength found in 5% RCP concrete (Figure 6).

The exception occurred for 40% RCP concrete, in which an inversed trend was observed. This may be explained by the addition of high amounts of SP that effectively dispersed, not only the agglomerated RCP particles, but also the OPC particles. Moreover, the estimated air entrainment was reduced (Figure 6). Therefore, a more effective and homogeneous growth of hydration products should have been attained and the above mentioned side-effects were compensated. This may also explain the lower compressive strength reduction obtained in 100% RCP concrete than in the recycled cement pastes produced by Real et al. [9] without the addition of SP. This demonstrates the effective contribution of SP in reducing the negative effect of the high water requirement of RCP.

As mentioned, the RCP tended to be more efficient in concretes with lower w/b. The incorporation of 15% RCP resulted in a 6.7%, 0.5% and 0.8% increase in the compressive strength at 28 days compared to OPC concrete, for concrete with w/b of 0.35, 0.55 and 0.65, respectively. This trend may be attributed to two main reasons: the similar entrapped air found in reference OPC and 15% RCP concrete with a w/b of 0.35 (Table 4); the addition of SP in concrete with a w/b of 0.35, promoting the de-agglomeration and more effective contribution of RCP particles.

In spite of the aggregate contamination of RCC and the consequent reduction of the cementitious fraction, the compressive strength was not significantly different from that of RCP concrete of similar composition, especially up to 15% replacement (Figure 9). Actually, taking into account that the cementitious fraction is 74% by weight of RCC (Section 2.1), the respective w/cf is about 0.57 and 0.60 for concrete with 15% and 30% RCC, respectively. Therefore, up to 15% replacement, the filler and nucleation effect of RCC should have compensated the slight increase in w/cf. However, for 30% replacement, the higher w/cf assumed more relevance in the compressive strength reduction.
Figure 9. Compressive strength at 28 days, $f_{cm,28d}$, of concrete with a w/b of 0.55 and different incorporation percentages of additions.

The incorporation of NT and CF resulted in a significant reduction of the compressive strength at 28 days, having been up to 37% lower than that of reference OPC concrete with the same w/b (Figure 9). This confirms the low reactivity of NT and CF additions, with an overall reduction of the hydration products. In fact, considering the compressive strength at 28 days as a function of the water/OPC ratio (w/c), the addition of NT and CF had little contribution to the compressive strength (Figure 10). The slightly higher compressive strength, for a given w/c, of concrete with 15% CF/NT may be attributed to an eventual filler and nucleation effect.

Figure 10. Compressive strength at 28 days, $f_{cm,28d}$, of the produced concretes as a function of the w/c.

Overall, the compressive strength increased over time (up to 90 days), independently of the type of binder and w/b (Figures 11–13). However, contrary to the general trend, a tendency for stabilization of the compressive strength between 28 and 90 days was observed for concretes with 30%RCP (w/b of 0.55, Figure 11) and 100%RCP (w/b of 0.65, Figure 12).
Various authors have reported that for high percentages of recycled cement, the long-term mechanical strength may be limited by a ceiling strength related to the high porosity and lower hardness of RCP particles \([8,16]\). However, for concrete with 40% RCP, this phenomenon was not observed, which can be partly explained by the greater de-agglomeration attained in this high SP concrete. The faster development of compressive
strength of recycled cement of higher surface area than OPC has been recognised [4,9,19]. As mentioned, since part of the mixing water is retained in the porous RCP particles, the w/b of bulk paste between anhydrous particles is reduced, which accelerates their proximity and cohesion, contributing to the early strength. Moreover, the nucleation and filler effect are more relevant at early age. In fact, the relation between the compressive strength of concrete with RCP and OPC tended to be higher at early ages (Figure 13, Table 4), especially for high incorporation percentages of RCP. Actually, concrete with RCP consistently displayed higher compressive strength at 3 days than that with OPC of equal w/b (Table 4).

Comparing the reference OPC concrete with a w/b of 0.55 (R55) to that with 15% RCP of the same workability (w/b of 0.62), but without SP, the required increase in w/b led to a respective reduction of the compressive strength, of about 14.5% (Table 4). As mentioned, for the same workability, more water is required by RCP with higher particle absorption, surface area and CaO content [4,8,9,19,40]. This strength reduction was very similar to that found for 30% RCC concrete, in which the estimated w/cf of 0.6 was about the same.

Regarding the addition of NT and CF, the significant reduction of compressive strength was confirmed, which tended to increase over time compared to the incorporation of the same percentage of RCP (Figure 13). As suggested, the filler and nucleation effects provided by these mineral additions are more relevant at early ages.

A very good correlation was found between the compressive strength and the dry density at 28 days (Figure 14). This can be explained by the fact that both properties are affected by concrete porosity, which primarily depends on the w/b and secondly on the entrapped air and type of binder.

![Figure 14. Compressive strength, $f_{cm}$, as a function of dry density, $\rho_{dry}$, at 28 days.](image)

### 3.3. Tensile Splitting Strength

The tensile splitting strength at 28 days of the produced concretes varied between 2.2 and 5.5 MPa, having essentially been affected by the w/b (Figures 15 and 16).

Overall, the tensile splitting strength at 28 days followed a similar trend to that observed for the compressive strength at the same age. In this case, the maximum tensile splitting strength was obtained for 15% RCP, both in concrete with a w/b of 0.55 and 0.65. For the same w/b, the tensile splitting strength of concrete with up to 15% RCP was up to 30% higher than that with OPC. For 30% RCP concrete, the tensile splitting strength was about 8% lower than that of OPC concrete with the same w/b. Once again, concrete with 40% RCP and high dosage of SP was able to invert the descending trend of tensile splitting strength (Figure 15). The highest tensile splitting strength reduction relative to the reference OPC concrete was found for 100% RCP concrete (18.5%).
Comparing mixtures with the same workability and without SP, the tensile splitting strength was only 3% higher in concrete with OPC (w/b of 0.55) than with 15%RCP (w/b of 0.62) (Table 4). A similar reduction was found for concrete with 30% RCC (w/cf of 0.60). This indicates that the tensile splitting strength was less affected by the w/b than the compressive strength, which corroborates the well-known lower proportional increase in the tensile splitting strength with the compressive strength [42,43]. Similarly to the compressive strength, the NT and CF did not significantly contribute to the tensile splitting strength (Figure 16). As mentioned, the incorporation of these admixtures decreases the overall amount of hydration products. In fact, considering the tensile splitting strength at 28 days as a function of the w/c (Figure 17), the contribution of NT and CF to this property was not significant.

A good correlation was found between the tensile splitting strength and the compressive strength at 28 days (Figure 18). This is due to the fact that both properties are influenced by the characteristics of the cement matrix, which are primary affected by the w/b. The reduction of the $f_{ctm}/f_{cm}$ ratio in high strength concrete was confirmed.
Figure 17. Tensile splitting strength at 28 days, $f_{ctm,sp,28d}$, of the produced concretes as a function of the w/c.

Figure 18. Tensile splitting strength at 28 days, $f_{ctm,sp,28d}$, as a function of compressive strength at the same age, $f_{cm,28d}$.

EN 1992-1 [42] proposes Equations (1) and (2) to determine the tensile splitting strength, $f_{ctm}$, as a function of the compressive strength and the dry density of concrete. Overall, Equations (1) and (2) were able to render a good approximation to the experimental results, only slightly underestimating the tensile splitting strength for high strength concrete, independently of the type of binder (Figure 19). The determination of $f_{ctm}$ in Equations (1) and (2) took into account the compressive strengths and dry densities, $\rho_{dry}$, indicated in Table 4, assuming the relation between the compressive strength in cubic specimens and in cylindrical specimens, $f_{cm,cyl}$, of EN 1992-1 [42]. Furthermore, the axial tensile strength, $f_{ctm}$, was considered to be about 90% of the splitting tensile strength [44].

\[
\begin{align*}
    f_{ctm} &= 0.30 \times \left( f_{cm,cyl} - 8 \right)^{0.5} \times \left[ 0.4 + \frac{0.6\rho_{dry}}{2200} \right] \text{ for } 20 < f_{cm,cyl} < 68 \text{ MPa} \\
    f_{ctm} &= 2.12 \times \ln \left( \frac{1 + f_{cm,cyl}}{10} \right) \times \left[ 0.4 + \frac{0.6\rho_{dry}}{2200} \right] \text{ for } f_{cm,cyl} > 68 \text{ MPa}
\end{align*}
\]
3.4. Modulus of Elasticity

The static modulus of elasticity decreased with the increase in RCP content (Figure 20). This trend was different from that obtained for the compressive strength (Section 3.3), in which the mechanical strength increased up to 15% RCP. As reported by Yu and Shui [20], the porous RCP particles have lower hardness and stiffness than those of OPC. This explains the slightly lower modulus of elasticity obtained in 15% RCP concrete compared to OPC concrete with the same compressive strength (Table 4). A non-proportional more significant reduction of the modulus of elasticity was found for 30% RCP concrete (three times higher than that with 15% RCP), because, for the reasons mentioned in Section 3.3, this concrete also had lower compressive strength. In addition, the RCP concrete was also produced with slightly higher volume of paste than OPC concrete with the same w/b (Table 2), which also contributed to the reduction of the modulus of elasticity. However, this variation was not significant, especially for low incorporation percentages of RCP.
considered for the tensile splitting strength were adopted for the estimation of $f_{cm,cyl}$ and $\rho_{dry}$ in Equation (3).

$$E_{cm} = 22000 \times \left( \frac{f_{cm,cyl}}{10} \right)^{0.3} \times \left( \frac{\rho_{dry}}{2200} \right) \text{ for } 20 < f_{cm,cyl} < 98 \text{ MPa} \quad (3)$$

### 3.5. Ultrasonic Pulse Velocity

The ultrasonic pulse velocity of the produced concretes ranged from 4415 to 5268 m/s, having been more influenced by the w/b than by the incorporation of RCP (Figure 21) or other additions (Figure 22). In fact, for the same w/b, the UPV only varied up to about 2% with the incorporation of RCP (Figure 21).

![Figure 21. Ultrasonic pulse velocity at 28 days, UPV$_{28d}$, of concrete with different incorporation percentages of additions.](image)

![Figure 22. Ultrasonic pulse velocity, UPV, of concrete as a function of compressive strength, $f_{cm}$, and dry density, $\rho_{dry}$.](image)

The UPV of concrete is essentially affected by its density and stiffness, which are both related to the microstructure porosity. For this reason, the UPV as a function the incorporation of RCP showed a similar evolution to that found for the compressive strength (Section 3.2).

As shown in Section 3.3, a good correlation was obtained between the compressive strength and the dry density. However, up to 15% RCP incorporation, the modulus of elasticity showed an opposite trend to that of the compressive strength (Section 3.5). This may explain the descending trend of UPV as a function of the incorporation of RCP in concrete with a w/b of 0.65, as well as the modest variation of UPV in concrete with a w/b
of 0.55 and distinct amounts of RCP (Figure 21). Nevertheless, except for low incorporation percentages of RCP, in which the microstructure can be densified, the UPV tended to decrease with the amount of RCP, due to the increase in air entrainment and decrease in the cement matrix stiffness.

For the same workability, the UPV of OPC concrete (w/b of 0.55) was only 1.4% higher than that of 15% RCP concrete (w/b of 0.62, without SP) (Table 4). This may be explained by the fact that UPV is approximately related with the sixth root of compressive strength, according to Equation (4) [45]. In this scenario, the 14.5% reduction of compressive strength obtained in Section 3.3 would correspond to a reduction of UPV lower than about 2%, as found in this study.

\[
UPV = K_{UPV} \times f_{cm}^{0.15} \times \rho_{dry}^{0.5}
\] (4)

The UPV of RCC concretes was similar to those of RCP concretes with the same incorporation percentage of recycled cement (Figure 21). The higher w/cf and the slightly lower compressive strength of concrete with RCC was offset by its higher cement particle stiffness, due to the 26% aggregate fraction within the RCC. On the other hand, the UPV of concrete with NT or CF was lower than that of concrete with the same incorporation percentage of RCP (Figure 21), which may be attributed to the lower density of NT and CF, the reduction of the amount of hydration products and their slightly higher volume of paste.

Figure 22 presents the UPV of the produced concretes as a function of the compressive strength and dry density, according to the methodology suggested by Bogas et al. [45]. A very good correlation was found between these properties, which validates the results. Besides the variability associated to the UPV test method, this relation is also influenced by the fact that these properties are affected by the water content in different ways.

### 3.6. Drying Shrinkage

Figure 23 displays the drying shrinkage of concrete with different incorporation percentages of RCP and w/b of 0.55. In general, the long-term 90 days drying shrinkage followed the same trend of the compressive strength. The drying shrinkage decreased slightly up to 15% RCP, but over this replacement percentage increased with the incorporation of RCP, except for concrete with 40% RCP, which was produced with a high dosage of SP.

For the same composition, i.e., the same aggregate/paste ratio, the drying shrinkage is primarily affected by the volume of evaporable water and w/b [46]. Given that the concretes were compared for the same w/b, the drying shrinkage essentially depended on the microstructure porosity and stiffness of the cement matrix.

As previously discussed, the concretes with up to 15% RCP were able to develop denser microstructures with lower porosity. However, due to the higher porosity and lower stiffness of the RCP particles, the modulus of elasticity was lower in these concretes.
Therefore, up to 15% RCP incorporation, the drying shrinkage was similar to slightly lower than that of the reference OPC concrete. Despite some variability, it may be concluded that the drying shrinkage over time was similar in both concretes.

The 30%RCP concrete developed a less dense microstructure (Section 3.3) and stiffness was further reduced (Section 3.5). Moreover, the RCP concretes tended to have slightly higher volumes of paste than the reference OPC concrete. Therefore, in this case, the drying shrinkage increased about 15% at 90 days.

An inverted trend was observed for concrete with 40% RCP, because as discussed in previous sections, the addition of high dosages of SP improved the dispersion and hydration of RCP and OPC, leading to denser microstructures, associated with higher stiffness and lower amounts of evaporable water.

Based on mercury intrusion porosimetry, scanning electron microscopy and nitrogen adsorption analyses, Bogas et al. [10] reported that, up to 28 days, the RCP pastes may be associated to more refined microstructure for the same total porosity of reference OPC pastes with equal w/b. This was explained by the water absorption of porous RCP particles that reduces the w/b of the bulk cement paste and promotes the development of finer porosity. Therefore, the total volume of evaporable water may be similar, but the capillarity forces of narrower pores are increased, which can also increase the drying shrinkage, at least, at early ages, when the hydration degree is still lower in OPC pastes than in RCP pastes. This may explain the slightly higher drying shrinkage observed in concrete with RCP during the first 7 days (Figure 23).

Concrete with 30%RCC showed similar long-term drying shrinkage to that of 30%RCP concrete (Figure 24). On the one hand, the RCC presented higher w/cf and hence lower stiffness and higher volume of evaporable water than the RCP. On the other hand, since RCC had about 26% aggregate fraction (Section 2.1), the volume of paste in RCC concrete was reduced.

![Figure 24](image-url)
As expected, for the same workability, the long-term drying shrinkage of 15% RCP concrete (w/b of 0.62) was slightly higher than that of reference concrete with OPC (Figure 24). In this case, the higher w/b of the RCP concrete reduced the aggregate/paste ratio and increased the amount of evaporable water, as well as the microstructure porosity. Therefore, the source of shrinkage was increased and the restraining action promoted by the aggregate skeleton and paste microstructure was reduced [46].

4. Conclusions

In this paper, the fresh and mechanical performance, as well as the long-term shrinkage of concrete produced with total or partial incorporation of thermoactivated recycled cement from waste cement paste and waste concrete, were characterised.

For the same workability, concrete with RCP required more mixing water or SP than that with OPC, due to the higher water demand of recycled cement than OPC. Nonetheless, up to 15% RCP incorporation, the workability was not significantly affected and recycled cement concrete could be produced without correcting the mixture composition. The SP was effective in compensating the lower workability of concretes with high amounts of RCP.

The concrete density was more affected by the w/b than by the incorporation of recycled cement. However, for over 15% incorporation of RCP, the reduction of workability for the same w/b tended to increase the air entrainment of concrete.

Contrary to non-treated waste cement and concrete filler, the recycled cement showed high rehydration capacity, retaining similar binding water than OPC and effectively contributing to improve the mechanical strength of concrete.

Overall, for the same w/b, the compressive and tensile splitting strength were not significantly influenced by the substitution of OPC with RCP, even for high incorporation percentages, demonstrating the potential of this more eco-efficient binder as a supplementary cementitious material. The optimum incorporation percentage was attained for up to 15% RCP, with concrete showing similar to better mechanical strength than reference OPC concrete. The performance of thermoactivated recycled cement was significantly affected by its effective dispersion and the fresh concrete workability, which were improved by the addition of SP.

In addition, concrete with RCP consistently displayed higher compressive strength at 3 days than that with OPC of equal w/b, which was attributed to the higher surface area and porous nature of RCP, improving the particle cohesion and the nucleation and filler effect at early age.

The modulus of elasticity decreased with the increase in the percentage incorporation of RCP, which presents lower hardness and stiffness than OPC. A more significant non-proportional reduction was observed for the addition of more than 15% RCP.

The equations suggested in EN 1992-2 [42] for OPC concrete, regarding the estimation of tensile splitting strength and modulus of elasticity, can also be adopted in concrete with recycled cement. Over 15% RCP, the UPV tended to decrease with the amount of RCP, due to the increase in air entrainment and stiffness reduction.

The mechanical strength and drying shrinkage were not significantly reduced when RCC was used instead of RCP, especially up to 15% replacement. Thus, the recycled cement derived from waste concrete, according to the patented method developed by the authors, may be as effective as that directly obtained from waste cement paste of equal composition.

For the same w/b, the drying shrinkage of concretes followed the same trend of the compressive strength. In concrete with up to 15% RCP incorporation, the lower stiffness of RCP particles and the slightly higher volume of paste were compensated by the development of denser microstructures. For higher amounts of RCP, the trend was inverted.

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