

Durability of concrete produced with recycled cement from waste concrete

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Abstract. In the present study, concretes produced with recycled cement (RC) obtained from the thermoactivation of concrete waste (RCC) or laboratory cement paste (RCP) were analysed in terms of some of their main transport properties (capillary absorption, oxygen permeability) and carbonation and chloride penetration resistance. Recycled cement was incorporated at various percentages, between 5 and 100%, taking into account concretes of different w/b. RCC was obtained from an innovative process of concrete waste separation recently patented by the authors, allowing the retrieval of cement waste with almost 90 vol% purity. RCP and RCC showed high rehydration capacity and concrete produced with them reached comparable durability to that of reference ordinary Portland cement (OPC) concrete. For up to 15% RCC replacement the concrete durability was not significantly affected and the cement retrieval from concrete waste was very effective. Overall, RCP and RCC actively contributed to the densification of the microstructure and the improvement of concrete durability. The recycled cement, as obtained in the present study, showed high potential to be used as an eco-efficient clinker substitute.

1 Submitting the manuscript

Today, the concrete industry is facing three major environmental challenges, namely the significant emission of greenhouse gases, the huge generation of construction and demolition waste (CDW) and the excessive consumption of natural resources [1,2]. Indeed, concrete production is responsible for the consumption of as high as 2.4 tonnes of natural resources per cubic meter of concrete, over 30% of the global CDW and over 7% of the world carbon emissions, essentially derived from the clinker manufacture [1,3]. This scenario is even expected to be aggravated with a predicted 12-23% increase of cement production by 2050 [4]. Therefore, various strategies have been explored by the concrete industry and scientific community towards the circularity and sustainability of concrete, including the reuse of CDW and the significant reduction of the carbon emissions during clinker production, by means of carbon capture and storage, more energy efficient technologies, alternative fuels and the development of alternative low-carbon binders [5,4]. Regarding this last target, relevant research has been carried out aiming the development of recycled cement (RC) from hardened concrete waste [6-10]. The idea is to recover the hydration capacity of cement waste at low thermoactivated temperatures, leading to a more eco-efficient binder associated to over 60% reduction of CO₂ emissions compared to conventional clinker [1,11]. In addition, reusing concrete waste contributes to the

reduction of natural resources depletion and the CDW disposal [1,4].

However, recycling cement is a recent domain that is still poorly explored. Relevant works are essentially related to the product development, RC rehydration capacity [6,7,12,13] and its behaviour when incorporated in cement pastes or mortars, including, mechanical characterization [14-17] and microstructure development [9]. A detailed review on the current state-of-art of RC is presented elsewhere [1].

Two obstacles that have hindered the affirmation of RC are its high water demand and the efficient retrieval of cement waste from old concrete, avoiding the significant aggregate contamination [1,7]. In this regard, an innovative process for concrete separation was recently patented by the authors, allowing to recover waste cement with almost 90 vol% purity [18]. In addition, studies on concrete with RC and especially regarding its durability are almost non-existent [1]. Therefore, further research is justified in this domain.

In the present study, concrete produced with RC is characterized in terms of transport properties (capillary absorption, oxygen permeability) and accelerated carbonation and chloride penetration behaviour. To this end, RC obtained from concrete waste (RCC), after employing the novel separation methodology of the authors, and reference RC obtained from laboratory cement paste (RCP) of equal w/b were considered.

2 Materials, compositions and methods

The origin concrete (OC) was produced with CEM I 42.5, a water/binder (w/b) ratio of 0.55 and two types of natural aggregates (coarse limestone gravel with 2/8, 4/11.2 and 11.2/20 mm; siliceous sand with 0/1 and 0/2 mm). In parallel, origin paste (OP) was produced with the same w/b of OC. The objective of OP is the production of a reference recycled cement with 100% cement waste, without contamination of aggregate phases. Therefore, the maximum potentiality may be achieved with RCP from OP and the efficiency of RCC from OC can be better accessed. The 28 days compressive strength of OC and OP was about 41 MPa and 52 MPa, respectively.

After 7 days in water and over 90 days in the laboratory environment, the OC and OP were subjected to crushing, grinding and milling down to lower than 1 mm particle size. Then, OC was further subjected to the patented method of Bogas et al. [18] in order to individualize the cementitious fraction. It was found that the retrieved cement waste obtained from OC was composed by 74 wt% of cement, 16 wt% of sand and 10 wt% of limestone, showing high purity. This was determined from thermogravimetric (TG) analysis and acid attack [18].

The last phase involved the extra milling of powder OP and separated OC down to lower than 250 μ m particle size, by means of a horizontal ball mill, and the subsequent thermoactivation in a rotary kiln (10 °C/min up to 650 °C, 3 hours at the maximum temperature; cooling inside the kiln). The thermoactivation procedure was defined according to [8,15].

From previous studies of the authors [8,19], based on TG and X-ray diffraction (XRD) analysis, it was confirmed the effective dehydration of RC, without relevant decarbonation. Contrary to ordinary Portland cement (OPC), the anhydrous RCP was essentially composed by the high temperature polymorph α' -C₂S,

CaO, CaCO₃ and aluminate phases [19]. Moreover it was estimated a 75% hydration degree of OP, confirming the maturity of this cement waste, which increases the validity of this study.

Other main properties of CEM I 42.5 (OPC), RCP and RCC are summarized in Table 1. Noteworthy is the significant higher surface area of RCP (8.7x) and RCC (4.3x) compared to OPC, which contributes to the faster reactivity and higher water demand of recycled cement. This is confirmed by the significant higher water demand of RCP in Table 1, also explained by its porous nature, free lime content and particle agglomeration [8,12,17,20]. The presence of aggregates in RCC reduces its surface area and water demand compared to RCP.

Table 1. Binder properties.

Binder	Absolute density (g/cm ³)	BET surface area (cm ² /g)	Water demand (w/b) EN196-3[21]	Initial/final setting time (mins) EN196-3[21]
CEMI 42.5R (OPC)	3.07 ^a	18134	0.31	290/385
RCP	3.01 ^b	156853	0.74	375/415
RCC	2.96 ^b	78673	0.5	290/385

a - LNEC E 64 [22]/ b - helium pycnometry

Concrete compositions are indicated in Table 2. About 10 concrete compositions were analysed, taking into account two w/b ratios (0.55, 0.65), different percentage replacements of OPC with RCP (5, 15, 30, 40 and 100 wt%) and RCC (15 and 30 wt%), as well as reference mixtures with only OPC (prefix "R" in Table 2 followed by the w/b ratio). Concretes were produced with a target slump S3 (100-150 mm, EN 206 [23]). A polycarboxylate based superplasticizer (SP) was added in concrete with high water demand, namely those with over 15% RCP.

Table 2. Binder Concrete compositions, fresh and dry density, air entrainment (V_{voids}) and 28 days compressive strength ($f_{cm,28d}$)

Designation	effective w/b	M _{OPC} (kg/m ³)	M _{RC} (kg/m ³)	V _{aggregate} (L/m ³)	SP (wt% _{binder})	Slump (mm)	ρ_{fresh} (kg/m ³)	V _{voids} (L/m ³)	$\rho_{dry,28d}$ (kg/m ³)	$f_{cm,28d}$ (MPa)
R55	0.55	360	-	666	-	150	2330	13	2220	52.2
R65	0.65	360	-	630	-	Fluid	2280	8	2100	39.8
5RCP55	0.55	342	18	665	-	130	2320	17	2220	53.2
15RCP55	0.55	306	54	665	-	90	2290	27	2190	52.4
30RCP55	0.55	252	108	664	0.4	110	2280	30	2170	47.8
40RCP55	0.55	216	144	657	1.5	190	2290	19	2160	52.5
15RCP65	0.65	306	54	629	-	190	2230	28	2090	40.1
100RCP65	0.65	0	360	621	3.8	100	2220	24	2090	33.2
15RCC55	0.55	306	54	663	-	120	2290	28	2190	52
30RCC55	0.55	252	108	662	-	130	2270	33	2160	45

The obtained slump, fresh (ρ_{fresh}) and 28 days dry density ($\rho_{dry,28d}$) were determined from EN 12350-2 [24], EN 12350-6 [25] and EN 12390-7 [26], respectively, and are also indicated in Table 2. As expected from the higher water demand of RCP in Table 1, concretes with over 15%

RCP required extra SP to meet the intended slump. Concrete with RCC presented higher slump than concrete with the same amount of RCP, which is explained by the less cement fraction in RCC, and hence, lower porosity and free lime content.

Density was only slightly affected by the incorporation of RC (Table 2). However, as the workability tended to be lower in RC concrete, the compaction was less effective and the air entrainment slightly increased compared to OPC concrete (V_{voids} , Table 2). This was more evident in concrete with 15 to 30% RCP, where a low content or even no SP was used.

Concretes were tested for 28 days compressive strength according to EN 12390-3 [27], using 150 mm cubic specimens cured in water until testing. As found in a previous study regarding the mechanical characterization of concrete with RC [19], the incorporation of RCP did not significantly affect the compressive strength of concretes with similar w/b (Table 2). Even the total replacement of OPC with RCP in concrete with w/b of 0.65 led to only 17% reduction in compressive strength (Table 2). This confirms the effective rehydration capacity of RCP [8,9]. Actually, probably due to filler and nucleation effects, the incorporation of up to 15% RCP slightly increased the compressive strength, regardless of the w/b ratio. The slightly reduction of compressive strength found for 30% RCP suggests the formation of less external products in this binder than in OPC, as found in previous studies [8,9]. This may be also related to the higher air content attained in this concrete, since an inverse trend was observed in concrete with 40% RCP. Indeed, the addition of high amounts of SP was effective in counteract the increased water demand and lower dispersibility of RCP. Finally, the compressive strength was not significantly affected when OPC was replaced by up to 30% RCC instead of the same amount of RCP (Table 2). The lower amount of cement fraction in RCC (74%), which led to an effective water/cement fraction (w/cf) of 0.57 and 0.6 for 15% and 30% RCC, respectively (higher than reference w/b of 0.55), was compensated by the better workability attained in RCC concrete. Nevertheless, comparing to RCP concrete, a reduction of 6% in the compressive strength was obtained for 30% RCC incorporation.

The durability characterization of concretes, which was the main focus of this study, was carried out through capillary absorption, oxygen permeability, chloride migration and carbonation resistance accelerated tests. The capillary absorption and oxygen permeability tests were carried out at 28 days in $\phi 150 \times 50$ mm specimens cut from 300 mm cylinders. For both tests, the specimens were cured for 7 days in water, followed by oven-dried at 50°C for 3 days and then by 17 days in the oven, sealed by a plastic film that aimed to avoid moisture changes (LNEC E 464 [28]; Rilem TC116-PCD [29]). After 1 more day at ambient temperature, the specimens were finally tested for capillary absorption and oxygen permeability, according to LNEC E 393 [30] and E 392 [31], respectively.

Regarding the capillary absorption test, the cylindrical specimens were laid in 5 ± 1 mm water and the mass gain was measured after 10, 20 and 30 minutes and 1, 3, 6, 24 and 72 hours upon contact with water. The capillary absorption coefficient, C_{abs} , corresponded to the slope of the linear regression between $\sqrt{20}$ minutes and $\sqrt{6}$ hours.

The oxygen permeability test was performed under different pressures (0.5, 1.5, 2.5 bar) and the coefficient of oxygen permeability, K_{O_2} (m^2) was estimated by Eq. (1),

where V is the volume of the flowmeter (m^3), t is the average flow time (s), L and A are the thickness (m) and cross-sectional area of the specimen (m^2), respectively, and p_{test} and p_{atm} the absolute input and atmospheric pressures (N/m^2), respectively [32]. At the end of permeability tests the relative humidity (RH) was additionally measured in broken specimens using a moisture meter probe.

$$K_{O_2} = [4.04 \times 10^{-5} (V/t) \cdot p_{atm} \cdot L] / [A \cdot (p_{test} - p_{atm})] \quad (1)$$

The rapid chloride migration test (RCMT) was performed under $\phi 100 \times 50$ mm sawn specimens according to NT Build 492 [33]. Three specimens were tested per composition, after curing 7 days in water followed by 21 days in a controlled chamber with 20 ± 2 °C and $50 \pm 5\%$ RH. After curing, the specimens were vacuum saturated in calcium hydroxide during 24 hours prior testing. According to this test, an external electrical potential, U (V), was applied for a period t (hours) and the chloride ions were forced to migrate from the anodic solution, at temperature T (°C), into the concrete specimen with thickness L (mm). After testing, the specimen were broken into two halves and the chloride penetration depth, X_d (mm), was measured by a colorimetric method with the aid of a 0.1M silver nitrate solution. The chloride migration coefficient, $D_{cl,RCMT}$ ($\times 10^{-12}$ m^2/s) is given by Eq. (2).

$$D_{cl,RCMT} = \frac{0.0239(273+T)L}{(U-2)t} \cdot \left(x_d - 0.0238 \times \sqrt{\frac{(273+T)L \cdot x_d}{U-2}} \right) \quad (2)$$

The accelerated carbonation test was carried out in $\phi 100 \times 40$ mm sawn specimens, based on LNEC E 391 [34]. Three specimens were tested per composition after following the same curing conditions adopted in chloride migration tests. About 7 days before testing, the cross-sectional surfaces of the specimens were sealed with an epoxy resin, ensuring the unidirectional diffusion of CO_2 . At 28 days, the specimens were subjected to forced carbonation in a controlled environment with 23 ± 3 °C, $65 \pm 5\%$ RH and $2.5 \pm 0.1\%$ of CO_2 . The carbonation depth was then measured after 28, 56 and 91 days, by broking one specimen per composition into four parts and spraying a 0.2% phenolphthalein solution. Assuming constant conditions over time [35], the carbonation coefficient, K_{ca} , was estimated by the slope of the linear regression of the carbonation depth up to $\sqrt{90}$ days.

3 Results and discussion

3.1 Capillary absorption

The capillary absorption was affected by the RC content. Up to 15% RCP the $C_{abs,28d}$ increased up to 33%, but then decreased for higher RCP contents. Indeed, a reduction of 26% and 56% was found for 40% and 100% RCP in concrete with w/b of 0.55 and 0.65, respectively (Fig. 1).

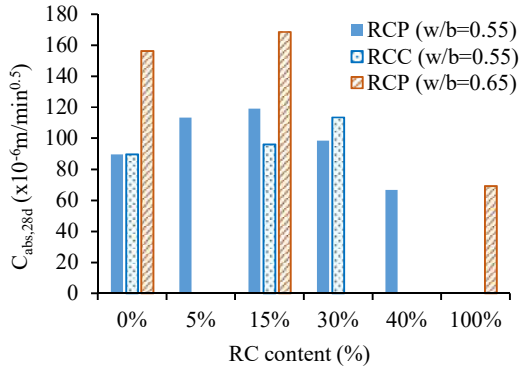


Fig. 1. Capillary absorption coefficient, $C_{abs,28d}$, of concrete with different RC contents.

As found in previous studies, a more refined porosity is usually attained in RCP than in OPC pastes [8,9], which is explained by the reduction of the interparticle w/b due to the water absorption by RCP particles. This effect tends to be diluted over time, because OPC pastes develop more long-term external hydration products [9,17]. This may explain the reduction of capillary absorption for high RC contents, but do not explain the inverse trend for lower RC amounts, as well as the poor correlation with the mechanical strength. The relative humidity (RH) of concrete specimens before testing is also relevant (Table 3). Despite the pre-condition procedure adopted to minimize RH variations (section 2), this parameter varied between 67 and 85%, being usually lower in concrete with RC. This may explain the higher capillary absorption in RC concrete with up to 30% RCP (Fig. 1). This also helps explaining the significant reduction of $C_{abs,28d}$ in 100%RCP concrete, with 85% RH. The higher air entrainment found in RC mixtures (Table 2) may also contributes to the increase of $C_{abs,28d}$.

For high RC contents the addition of SP was important to improve the dispersion of RC particles and to allow the adequate compaction of concrete, which promotes denser microstructures [36]. In fact, despite the lower RH in 40% RCP concrete, the $C_{abs,28d}$ was lower in this concrete than in the reference one with OPC. Nevertheless, it is clear that the addition of RC may contribute to the early age microstructure densification and that the RC concrete tends to present, at least, the same performance of OPC concrete regarding this analysed property. In addition, from Fig. 1 it is evident that the w/b generally assumed more relevance than the type and amount of binder. However, the reduction of interparticle porosity attained in 100% RCP counteracted the higher w/b of this concrete

(0.65) compared to reference OPC concrete with w/b of 0.55 (Fig. 1).

The incorporation of up to 15% RCC only slightly increased the $C_{abs,28d}$ comparing to reference OPC concrete (Fig. 1). The difference was lower than that found for 15% RCP concrete, because the RH was more close to that of OPC concrete (Table 3). However, for 30% RCC the $C_{abs,28d}$ highly increased, being 15% higher than that of concrete with equal amount of RCP and similar RH. As mentioned, the w/cf is slightly higher in RCC concrete than in RCP concrete (0.6 versus 0.55), which leads to a coarser microstructure.

3.2 Oxygen permeability

In general the influence of RC in the oxygen permeability followed the same trend found in capillary absorption (Fig. 2). This property is also highly affected by the RH, which tended to be higher in RC concrete (Table 3). In fact, it is reported that the oxygen permeability at 75% RH may be twice of that at 80% RH [37]. It was confirmed the increase of $K_{O_2,28d}$ up to 15% RCP and then a progressive reduction for higher RCP contents. For high RCP content, over 30%, the $K_{O_2,28d}$ could be as low as about 42% (40% RCP) to 70% (100%RCP) lower than that of OPC concrete.

Despite the test variability and RH influence, it may be concluded that concrete with high content of RC, associated to lower interparticle w/b, develops a finer microstructure. These results confirm the reduction of transport properties in concretes of high RCP content and high amount of SP, associated with less air entrainment and better dispersion properties. The improvement after RC addition was higher in oxygen permeability than in capillary absorption because permeability is more affected by the critical pore diameter [38].

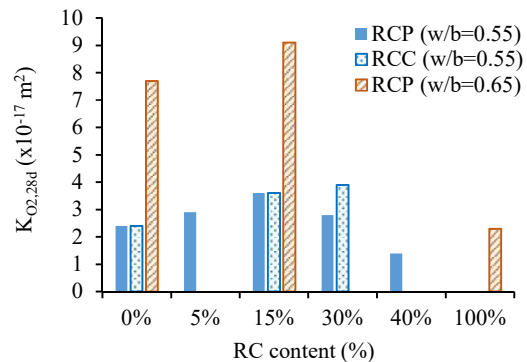


Fig. 2. Oxygen permeability, $K_{O_2,28d}$, of concrete with different RC contents.

Table 3. Relative humidity of concrete specimens tested for capillary absorption and permeability

Composition	R55	R65	5RCP55	15RCP55	30RCP55	40RCP55	15RCP65	100RCP65	15RCC55	30RCC55
Relative humidity (%)	81	81	69	67	73	77	73	85	76	73

The oxygen permeability was not significantly affected by the incorporation of up to 15% RCC, showing similar behaviour to that of 15%RCP concrete. However, once more, the incorporation of 30% RCC led to a 37% increase

of K_{O_2} compared to concrete with equal amount of RCP and similar RH (Fig. 2). As mentioned, this is explained by the higher w/cf of RCC concrete. Nevertheless, from both capillary and permeability tests it may be concluded

that up to 30% RCC replacement the transport properties were not significantly affected and the cement retrieval from concrete waste, based on the patented method of the authors, was effective.

3.3 Rapid chloride migration

The chloride migration coefficient, $D_{cl,RCMT,28d}$, obtained in this study were within those reported in literature for conventional OPC concrete of similar w/b ratios [39], being little affected by the RC content (Fig. 3).

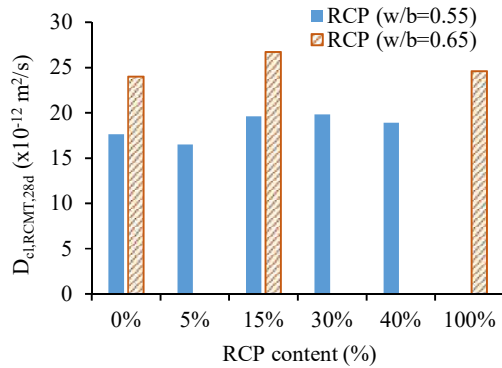


Fig. 3. Chloride migration coefficient at 28 days, $D_{cl,RCMT,28d}$, of concrete with different RC contents.

The chloride migration was essentially affected by the w/b, with the type of binder assuming less relevance. However, the $D_{cl,RCMT,28d}$ of RC concrete tended to be slightly higher than that of reference OPC concrete, especially over 5% and up to 30% RCP (Fig. 3).

Contrary to the analysed transport properties, the $D_{cl,RCMT,28d}$ is independent of the RH, because the specimens were tested in saturated conditions. Moreover, the critical pore diameter has less influence on diffusion than in permeability [38]. In this case, the $D_{cl,RCMT,28d}$ is less affected by the eventual coarser microstructure of OPC concrete than RC concrete. Thus, the $D_{cl,RCMT,28d}$ is essentially affected by the open porosity, which is expected to be higher in RCP than in OPC concrete, due to the less development of external hydration products overtime [9].

Nevertheless, the maximum increase of $D_{cl,RCMT,28d}$ was lower than 13% for 30% RCP, and lower than 10% for other RC contents. It was even found a slight reduction of $D_{cl,RCMT,28d}$ for 5% RCP (6%) and about the same chloride penetration behaviour for 100% RCP (3% higher $D_{cl,RCMT,28d}$). For 5% RCP, the water requirement and workability was not significantly affected, which led to a denser microstructure and the subsequent increase of compressive strength (section 2) and chloride penetration resistance. On the other hand, the use of high amounts of SP in concrete with over 30% RCP, improving the workability and dispersibility of cementitious particles, benefited the reduction of $D_{cl,RCMT,28d}$.

3.4 Carbonation

As per compressive strength, the carbonation coefficient was little affected by the incorporation of up to 30% RCP.

In fact, it was found that, except for 30% incorporation, the replacement of OPC with RCP improved the carbonation resistance, especially for over 40% RC (Fig. 4). As mentioned, the recycled cement tends to develop a lower volume of hydration products than OPC over time [8,9]. Moreover, as RC is obtained from old concrete, which is partly carbonated, the amount of carbonatable products tends to be lower than that of OPC [1,7-9]. Therefore, it would be expected a lower carbonation resistance of RC concrete than reference OPC concrete.

However, as mentioned, the microstructure of OPC pastes is typically coarser than that of RC pastes. This was confirmed in a recent study of the authors, regarding the microstructural characterization of RC pastes [9], where mercury intrusion porosimetry analysis confirmed a higher volume of small micropores (<50 nm) in these pastes than in OPC pastes of equal w/b and similar total porosity. This aspect assumes more relevance in carbonation than in chloride diffusion, because the specimens were tested in semi-dried conditions, where only the very small pores are clogged with water. Therefore, the carbonation rate is essentially affected by the coarse pores, which volume is higher in OPC concrete than in RC concrete. In addition, as also discussed, the RC concrete is benefited by the incorporation of high dosages of SP, improving the dispersion of cement particles and facilitating the concrete compaction. To sum up, combining all this factors, it may be concluded that the carbonation resistance is similar or even improved with the incorporation of RC.

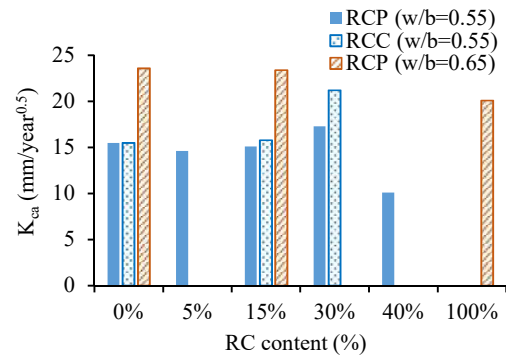


Fig. 4. Carbonation coefficient, K_{ca} , of concrete with different RC contents.

As found in other studied properties, the carbonation resistance was little affected by the incorporation of up to 15% RCC. However, for 30% replacement, the K_{ca} was 23% higher than that of RCP concrete (Fig. 4). This is again explained by the higher w/cf and lower volume of cement fraction in RCC than in RCP, which implies a smaller volume of hydration products and carbonatable substances.

Finally, regarding all analysed properties (capillary absorption, permeability, carbonation, chloride penetration) it may be concluded that RCP and RCC actively contributed to the densification of the microstructure and the improvement of concrete durability, behaving as a cementitious material. Therefore, the recycled cement showed to be a very promising eco-efficient clinker substitute.

4 Conclusions

The durability of concrete produced with different contents of recycled cement from waste cement paste (RCP) and waste concrete (RCC) was analysed in terms of capillary absorption, permeability, chloride migration and carbonation resistance. The following main conclusions have been drawn:

- RCC and RCP revealed adequate hydration capacity and actively contributed to the densification of the microstructure, improving the concrete durability;
- The workability was significantly affected by the high water demand of RC. Therefore, the addition of large amounts of SP was necessary to compensate this negative effect in concrete with over 15% incorporation of RC.
- The mechanical strength was only little affected by the incorporation of RCP. Even for up to 100% replacement, the strength reduction was lower than 17%;
- The analysed transport properties (capillary absorption, oxygen permeability) were significantly influenced by the RH. However, it may be concluded that over 30% RCP promoted the microstructure refinement, due to the lower interparticle w/b and the addition of SP;
- The chloride migration and carbonation coefficients were little affected by the incorporation of RCP;
- The slight increase of $D_{cl,RCMT,28d}$ in RC concrete is attributed to its higher total open porosity than that of reference OPC concrete. Nevertheless, the maximum increase of $D_{cl,RCMT,28d}$ was lower than 13%;
- The carbonation resistance tended to increase with the incorporation of RCP. The results suggest that the microstructure refinement promoted by the addition of recycled cement may have compensated the reduction of the volume of hydration products and carbonatable substances in RC concretes;
- It is concluded that up to 15% RCC replacement the analysed durability properties were not significantly affected and the cement retrieval from concrete waste, based on the patented method of the authors, showed to be very effective. For 30% RCC replacement, the 12 vol% contamination of aggregate increased the w/cf that slightly reduced the concrete durability;

Overall, RCP and RCC actively contributed to the densification of the microstructure and the improvement of concrete durability. The recycled cement showed high potential to be used as an eco-efficient clinker substitute.

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