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How the Carbonation Treatment of Different Types of Recycled Aggregates Affects the Properties of Concrete

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Abstract: In this research work, two types of recycled aggregates were used: (1) the recycled concrete aggregate, RCA, obtained by crushing the parent concrete produced using limestone cement and (2) the recycled concrete aggregate RCA-FA produced by crushing parent concrete made with FA blended cement. After the carbonation treatment process, the carbonated RCA-C and RCA-FA-C recycled aggregates were produced. The recycled concrete mixtures were prepared using the four types of recycled aggregates (RCA, RCA-FA, RCA-C, and RCA-FA-C) in 50% (by volume) to replace natural coarse aggregates. The physical and mechanical properties and durability (sorptivity, chloride ion penetration, and carbonation resistance) were determined and analysed. The obtained results were also compared with those of conventional concrete (CC). It was concluded that the physical and mechanical properties of recycled concrete improved when RCA-C were employed in concrete production. In contrast, the recycled concrete produced with RCA-FA-C was found to have the worst property values. According to durability properties, the concrete made with RCA-C and RCA-FA aggregates achieved the highest chloride resistance, similar to CC concrete. Nevertheless, the concrete produced with uncarbonated RCA acquired carbonation resistance equivalent to CC concrete.

Keywords: carbonation treatment; parent concrete with FA; recycled aggregate concrete; mechanical properties; MIP; durability; carbonation; sorptivity; chloride

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1. Introduction

Demolition of concrete buildings generates a considerable amount of waste that finishes in landfills. For example, in 2018, approximately 806 million tons of construction and demolition waste were generated in the European Union, of which only 54.2% were recycled [1]. Consequently, with respect to concrete production, the use of recycled concrete aggregates (RCA) obtained by crushing concrete structures can help reduce the environmental impact caused by concrete waste [2,3].

The employment of recycled aggregate concrete (RAC) as a structural material has been widely analysed and validated in many applications [3–6]. In addition, it has been determined that the use of 50% of RCA as a substitute for coarse natural aggregates had no effect on the lowering of compressive strength [7,8]. However, the durability of RAC is usually lower than that of natural aggregate concrete (NAC) because durability is influenced by the connectivity of the porous network and water content [4,9–12].

According to chloride ion diffusion resistance, it was found that the RAC had lower resistance than the NAC when concretes were produced with ordinary Portland cement (OPC) [9,12–14].

The studies' conclusions tend to be inconsistent with the carbonation resistance of RAC. Several researchers [12,14] concluded that the RAC prepared with a high proportion of RCA had a lower carbonation resistance than the NAC. In contrast, several researchers [9] determined that the RAC mixes had similar or higher carbonation resistance than the NAC because of the old attached mortar. Leemann and Loser [15] determined that the impact on the carbonation coefficient of RAC, independent of the replacement levels of RCA and if they are already carbonated, is low (around 10%) at a given compressive strength. According to Zeng [8], using up to 50% of the RCA in place of natural coarse aggregates avoided a decrease in carbonation resistance. However, Pedro et al. [16] documented that when the quality of the RCA's attached mortar was lower than that of the new paste, the RAC carbonation coefficient increased when higher percentages of RCA were employed in place of natural aggregates.

Over the last decade, many studies have been conducted based on RCA quality improvement via the carbonation process [17,18]. Natural or accelerated carbonation using gas in contact with RCA is a possible way to improve the properties of RCA [19–21]. Indeed, hydrates such as portlandite or C–S–H, which are contained in the cement paste adhered to the natural aggregates, could react with CO_2. The reaction has slow kinetics in natural conditions. Still, it could be accelerated using a gas with a higher CO_2 content, such as flue gas from cement factories, and a higher temperature or gas pressure. Accelerated carbonation increases the density of the attached cement paste and decreases the water absorption of the RCAs thanks to the formation of $CaCO_3$ [19,22]. Portlandite carbonation usually reduces pore size and meso- and macropore volume due to $CaCO_3$ precipitation [23]. In addition, the capillary porosity decreases due to the clogging of the pores.

The performance of mortars and concrete made with carbonated RCA improved [18]. According to Liang et al. [17], concrete with carbonated RCA has better workability, mechanical properties and durability than untreated RCA. Xuan et al. [24] found that concrete achieved lower water absorption and permeability when RAC was prepared with carbonated RCAs as well as improved the extent of the durability of RAC. They found that concrete prepared with 100% carbonated RCAs reduced chloride ion permeability by 36.4% for concrete produced with uncarbonated RCA.

More recently, in the trend of CO_2 emission reduction, fly ash (FA) has been used more widely to reduce cement consumption in building materials. In addition, using FA can result in concrete with better durability [25]. However, due to the reduced portlandite content in supplementary cementitious material (SCM)-containing systems, carbonation will occur more rapidly in the main CO_2-binding phases. When using blast furnace slag and FA as SCM, the C-A-S-H phases can induce coarsening of the pore structure upon carbonation [23]. Consequently, it is believed that when the structures produced using FA are demolished, the carbonation treatment will not be satisfied with the improvement of the RCA properties due to the results of coarsening of the pores of the attached mortar, resulting in the increased permeability of RCA.

This study aimed to determine how the properties of concrete are affected by the carbonation treatment on different compositions of recycled aggregates. The recycled aggregates were obtained by crushing two types of parent concretes. Firstly, the recycled concrete aggregates named RCA were obtained by crushing the parent concrete produced using cement type CEM IIAL. Secondly, the recycled concrete aggregates named RCA-FA were produced by crushing parent concrete produced with FA blended cement (50% OPC and 50% FA). After the carbonation treatment process, the RCA-C (carbonated aggregates) and RCA-FA-C (carbonated FA aggregates) were produced, respectively. Finally, the recycled concrete mixtures were prepared using the four types of recycled aggregates (RCA, RCA-FA, RCA-C, and RCA-FA-C) in 50% (by volume) to replace natural coarse aggregates. The physical, mechanical, and durability (sorptivity, chloride ion penetration, and carbonation resistance) properties of the concrete produced using the four recycled aggregates were determined and analysed. The consequent results were also compared with those obtained by conventional concrete (CC).

2. Materials and Methods

2.1. Materials

2.1.1. Cement and Admixture

The cement CEM II A-L 42.5R (88% clinker, 10% limestone, and 2% set regulator, gypsum) was used in all the concrete production. The composition and physical properties (loss of ignition, LOI; Blaine specific surface, Blaine; and density) of the cement are given in Table 1. In addition, a superplasticiser (S) that is polymer-based on PAE compound was used for concrete production.

Table 1. Chemical composition of cement.

	SiO_2	Fe_2O_3	Al_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	LOI	Blaine (cm^2/g)	Density (g/cm^3)
CEM II A-L	18.27	3.29	4.09	61.82	1.35	2.9	0.10	0.77	5.07	3900	3.16
FA	58.4	7.3	21.6	2.3	1.9	0.2	0.9	2.1	3.1	3400	2.16
CEM I 52.5R	19.4	3.4	4.2	63.5	1.4	3.0	0.12	0.53	3.7	4900	3.15

2.1.2. Natural Aggregates

Three fractions of crushed limestone aggregates (gravel 10–20 mm, gravel 4–10 mm, and sand 0–4 mm) were used as raw aggregates (NA) to produce conventional concrete (CC) (see Figure 1). The fineness modulus of sand was 2.9. Figure 2 shows their grading distribution. It was determined following EN 933-1 specifications and complied with EN 12620 specifications.

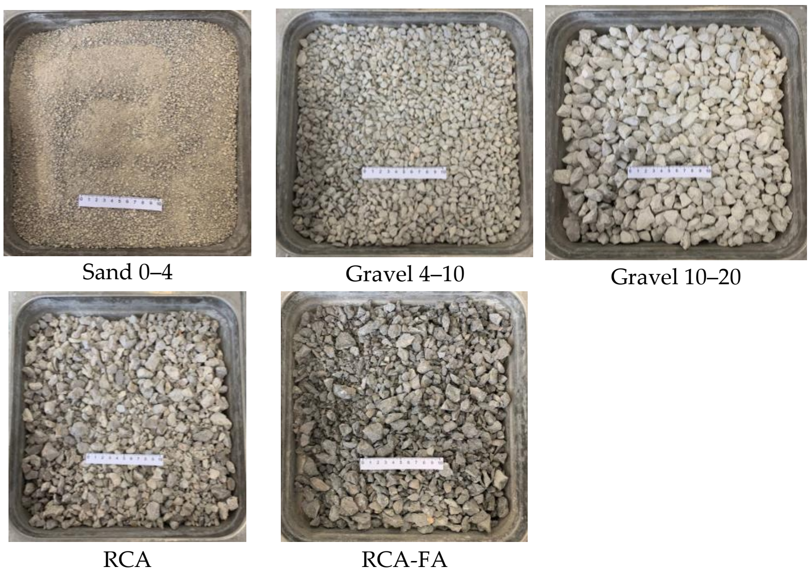


Figure 1. The raw and recycled aggregates (each line in the ruler is 1 cm).

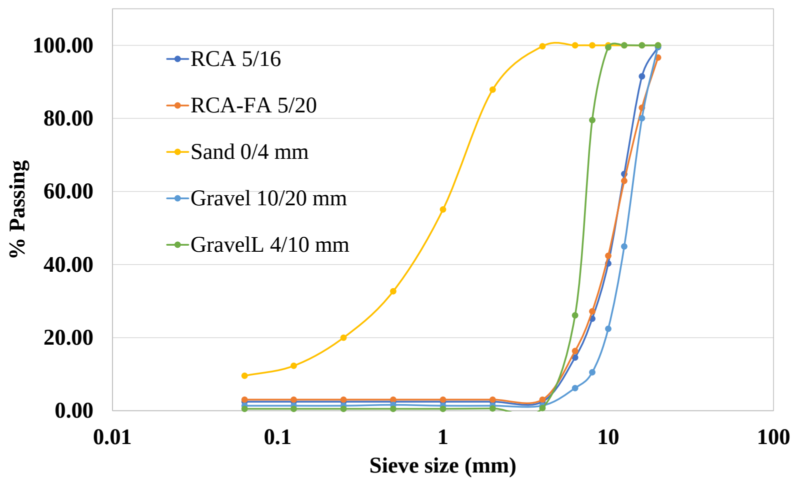


Figure 2. Particle size distribution of raw and recycled aggregates.

The physical properties of dry density (ρ_{rd}) and water absorption value (WA_{24}) were determined according to EN 1097-6 specifications (see Table 2).

Table 2. Physical properties of raw, recycled aggregates and carbonated recycled aggregates.

	Raw Aggregates, NA			Recycled Aggregates, RA		Carbonated RA			
						Process 1		Process 2	
	0/4 mm	4/10 mm	10/20 mm	RCA	RCA-FA	RCA-C	RCA-FA-C	RCA-C	RCA-FA-C
ρ_{rd} (kg/m^3)	2590	2640	2650	2310	2240	2360	2310	2370	2310
WA_{24} (%)	1.79	0.75	0.57	5.8	6.8	4.85	5.64	4.8	5.68

2.1.3. Recycled Aggregates

RCA and RCA-FA recycled aggregates (see Figure 1) were produced by crushing two different parent concretes. The RCA aggregate was produced by crushing the parent concrete made with CEM II A-L cement (see Table 1), and it had 30 MPa of compressive strength at 28 days. In addition, the RCA-FA aggregate was obtained by crushing concrete made using a blended cement: 50% of CEM I 52.5R and 50% of FA (equivalent to ASTM class F) (see Table 1 for their composition). The concrete had a compressive strength of 32 MPa after 28 days. Both parent concretes were produced with limestone aggregates, and they were approximately nine months old when they were crushed.

The RCA and RCA-FA aggregates were sieved to achieve a particle size distribution similar to that of coarse natural aggregates. Figure 2 and Table 2 describe the particle size distribution and the physical properties (ρ_{rd} and WA_{24}) of RCA and RCA-FA, respectively.

The recycled aggregates (RA) obtained a lower density and a higher absorption coefficient than NA due to the attached mortar presented in RA, which was found to provide more pores, causing a decrease in density and an increased absorption capacity.

Carbonated Recycled Aggregates

The carbonation treatment of the RCA and the RCA-FA aggregates was carried out in two processes (process 1 and 2). In process 1, the RCA and the RCA-FA were subjected to a carbonation process using an automatic carbonation control chamber with a concentration of 3% CO_2, 57% relative humidity (RH), and 20 °C for 24 days until they were fully carbonated. It was carried out following the conditions defined by UNE-EN 12390-12 specification. On the other hand, in process 2, the RCA and RCA-FA were submitted to a carbonation process in 20% CO_2, 57% RH, and 20 °C conditions for four days to achieve a complete carbonation. Process 2 was carried out following the GB T50082-2009 [26] standards. The physical properties (ρ_{rd} and WA_{24} were determined following EN 1097-6 [27] specifications) of the carbonated RA in both processes are described in Table 2.

Table 2 shows that the RCA-C and the RCA-FA-C achieved similar ρ_{rd} and WA_{24} values after they were submitted to carbonation processes 1 and 2. Consequently, due to the findings of carbonation process 2, using a reduced period (4 days), it was decided that all the RA volume required for concrete production would be carbonated under this process. As described by Liang et al. [17], an excessive concentration of CO_2 gas does not result in an evident increase in the carbonation percentage of RCA. However, the increased concentration of CO_2 gas also leads to the decalcification of C–S–H and even the complete disappearance of C–S–H under the condition of a 100% CO_2 concentration [28], resulting in an adverse effect on the properties of RCA.

According to Table 2, the ρ_{rd} increased and WA_{24} decreased when the recycled aggregates were submitted to carbonation process treatment. Table 2 shows that RCA and RCA-FA (uncarbonated) aggregates achieved a 5.8% and 6.8% absorption capacity, respectively. Once the aggregates were carbonated, the RCA-C and RCA-FA-C achieved absorption capacities of 4.8% and 5.7% with 17% and 16% reductions, respectively.

The Pore Structure of Recycled Aggregates

Porosity and pore structure were tested by mercury intrusion porosimetry (MIP) using a "Micromeritics Poresize 9320" mercury intrusion porosimeter following BS7591 Part 1. This test was performed on small RA samples weighing approximately 5.5 g. The samples were first soaked in acetone for four days to stop the hydration of the cement and then placed in a vacuum drier for 2 h to remove the remaining acetone. Before testing, the samples were dried in an oven at 50 °C for four days. The total porosities were 13.5% for RCA, 10.7% for RCA-C, 11.8% for RCA-FA, and 9.6% for RCA-FA-C.

Figure 3a shows the pore size distribution development of the RA before and after carbonation. As shown in Figure 3b, the pore size diameter can be divided into four pore size ranges following the Mindess [29] classification: >10 µm (air), 10–0.05 µm (macropores), 0.05–0.01 µm (mesopores), and <0.01 µm (micropores). Certain researchers [30,31] emphasised that concrete's mechanical and permeability properties depend on the mesopores and macropores. The RCA aggregate reduced the porosity from 13.5% to 10.7% upon carbonation. In addition, no increase in the pore volume of any size was appreciated. Xiao et al. [32] concluded that carbonated RAs (when OPC was the constituent of the attached mortar) decreased pore volume and pore amount. Although RCA-FA aggregates suffered a porosity reduction from 11.8% to 9.6%, after the carbonation process, the pore size distribution of carbonated RCA-FA-C aggregates was also found to have changed with respect to that of RCA-FA. An increase in mesopores and macropores from 0.05 to 0.2 µm was appreciated in the RCA-FA-C aggregate. In addition, the RCA-C and RCA-FA-C aggregates achieved very similar pore distributions.

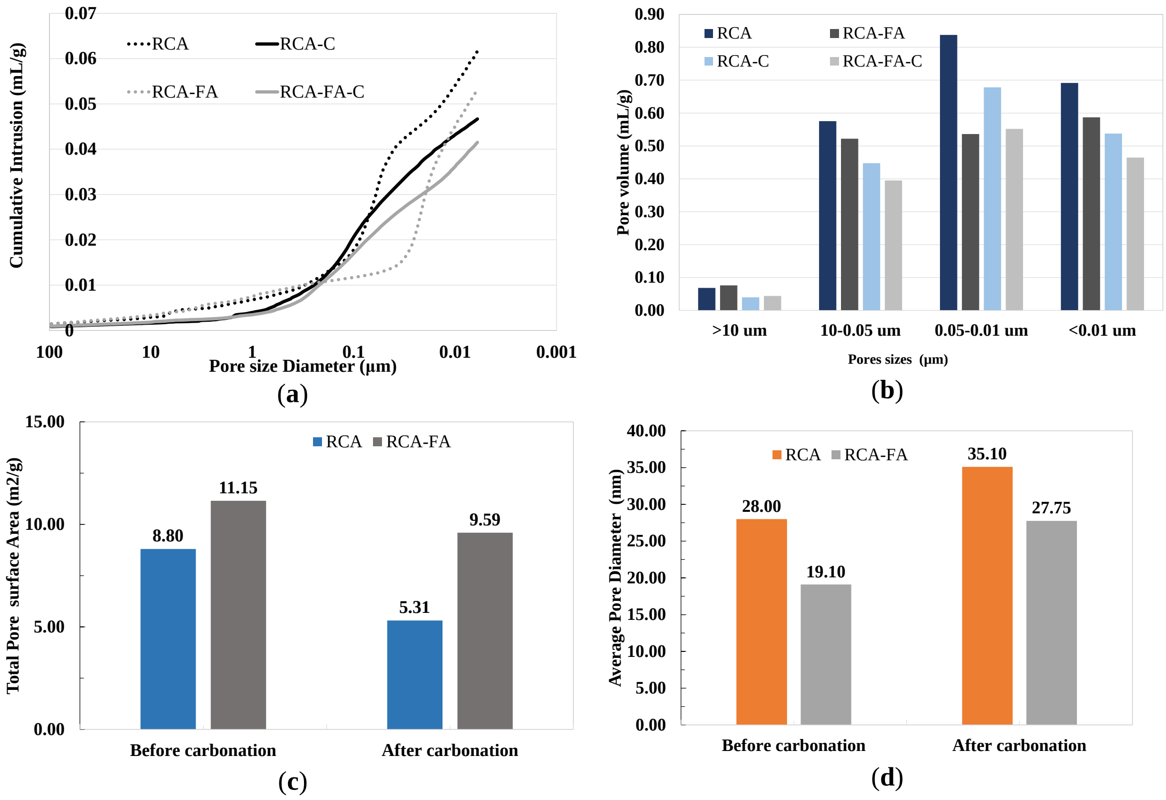


Figure 3. (**a**) Development of pore size distribution, (**b**) distribution of pore diameters, (**c**) total pore surface area before and after the carbonation process, and (**d**) average pore diameter before and after the carbonation process.

Figure 3c,d show the pore structure parameters of RA before and after carbonation. The pore structure of the RA improved when carbonated. The total pore surface area decreased by approximately 30% in the case of RCA and 14% in the case of RCA-FA.

Liang et al. [33] reached a similar conclusion regarding the pore surface area of RA (when the OPC was a composition of the attached mortar) after carbonation, which decreased by approximately 36%.

2.2. Concrete Production

All concrete mixes were prepared and manufactured in the laboratory. A total of five types of concrete mixes were prepared (see Table 3). Firstly, the CC concrete was produced with 100% raw fine and coarse aggregates. In addition, four recycled concretes (RAC50, RAC50-C, RAC50-FA, and RAC50-FA-C) were produced, replacing 50% (by volume) of both raw coarse aggregate fractions by the RCA, the RCA-C (carbonated), the RCA-FA, and the RCA-FA-C (carbonated) aggregates, respectively.

Table 3. Mix proportions of produced concretes (kg of material/m^3). Superplasticizer (S) is given in % with respect to cement weight.

Concrete Reference	CEM	Raw Aggregates			RA	Total Water	S (%)	SLUMP (mm)	DENSITY (kg/m^3)
		0–4 mm	4–10 mm	10–20 mm					
CC	350	900	275.2	644.6	-	192.5	0.5	170	2420
RAC50	350	900	137.6	322.3	399.6	211.1	0.5	150	2310
RAC50-C	350	900	137.6	322.3	411.8	208.9	0.5	170	2340
RAC50-FA	350	900	137.6	322.3	389.2	217	0.5	170	2310
RAC50-FA-C	350	900	137.6	322.3	401.3	211.8	0.6	190	2360

The concrete mix proportions were defined according to their maximum volumetric compaction. This mix proportion for the CC was 50% fine aggregates and 50% coarse aggregates. The distribution of coarse aggregate was 30% coarse 4–10 mm and 70% coarse 10–20 mm. The recycled concretes were produced using 50% of RA in substitution of coarse NA aggregates. All concrete production was subjected to a constant effective water–cement ratio (effect w/c) of 0.50 and a cement content of 350 kg/m^3. They were defined following the Spanish structural code [34] in order to guarantee the compressive strength of 44 MPa in cubic specimens and durability properties for reinforced concrete to be exposed to XC4 and XS 1 environment conditions. (A characteristic compressive strength of 30 MPa was required in cylindrical specimens.) Following Neville's [35] definition of the effective amount of water in the mixture occupying the space outside the aggregate particles, the effect w/c ratio was kept constant in all concretes. This was in order to achieve the same conditions with respect to the hydration of the cement paste caused by the high absorption of RA. As a result of the high absorption capacity of RA, all four types of RA were used with a humidity of up to 70–80%, thus reducing their water absorption value [36]. However, the most important aspect was that the aggregates employed were moist to reduce their absorption capacity [37]. Due to the moderate initial moisture content, the recycled aggregate absorbed some amount of free water, which lowered the initial w/c ratio in the interfacial transition zone (ITZ) and thus improved the interfacial bond between the aggregates and cement [38]. Table 3 describes the mix proportions used for concrete production. The weight of the aggregates is expressed as dry weight. In addition, the total amount of water was considered, including the effective water, the water absorbed by aggregates, and the humidity of RA at concrete production. Finally, 0.5 to 0.6% of superplasticisers were used relative to cement weight of all concretes to achieve the desired workability.

The consistency (slump test) of the concretes was determined in the fresh state following the standard UNE-EN 12350-2:2020 [39]. All the concretes achieved a slump value between 150–170 mm employing 0.5% of superplasticiser. The use of 0.6% of S in the RAC50-FA-C concrete increased the slump value to 190 mm. Although the total water content of the RAC was higher than that of the CC concrete, it did not affect the workability of concretes, as the RA were used with high humidity.

The fresh density was determined following UNE-EN 12350-6 [40] specification. Table 3 shows that the use of RA, carbonated or not, did not affect the properties of the concrete in the fresh state. Although the density of RAC was reduced by 100 kg/m^3 compared to CC concrete, all the concretes reached adequate density for structural concrete [35].

All the concrete mixtures were produced in a vertical axle mixer. The materials were always added manually in the same order. First was the aggregates (from coarser to finer); after they were mixed for 30 s, the cement was added. While the solid components were mixed for 1 min, water was added, followed by a superplasticiser. The complete mixture was then mixed for 1 min more. Once the concrete was produced, concrete specimens were produced after the slump test, and fresh density was determined. The concrete specimens were produced and cured according to the UNE-EN 12390-2: 2020 [39] standard and were manually compacted using a metal bar, then covered with a plastic sheet, and left to cure in the air for 24 h. The concrete specimens were kept in moulds for 24 h. After demoulding, they were stored in a humidity room with an RH of $\geq$95% and a constant temperature of 20 $\pm$ 2 °C until one hour before testing. All test elements were subjected to the same conditions before testing.

Three types of specimens were produced. Three cubic samples of 100 $\times$ 100 $\times$ 100 mm for each test were used to determine the physical properties, compressive strength, and sorptivity. Two cylinders with a 100 mm diameter and 200 mm height were employed to determine the properties of splitting tensile strength, modulus of elasticity, and chloride ion penetrability. In addition, two prismatic specimens of 100 $\times$ 100 $\times$ 400 mm were used in each concrete to determine carbonation resistance.

2.3. Test Procedure

2.3.1. Physical Properties

The density, absorption and voids were measured following the ASTM C 642-21 "Standard Test Method for Density, Absorption, and Voids in Hardened Concrete" [41] at 28 days. Three cube specimens of 100 $\times$ 100 $\times$ 100 mm were used for each type of concrete produced.

2.3.2. Mechanical Properties

The compressive strength, splitting tensile strength, and modulus of elasticity (E) were determined for each concrete. The compressive strength of concrete was determined using a 3000 kN loading capacity machine. The compressive strength was determined at 7, 28, and 56 days following UNE-EN 12390-3 [42] specifications. Three 100 $\times$ 100 $\times$ 100 mm cubic specimens were used for each age. The elastic modulus (E) and tensile strength were tested at 28 days following UNE 12390-13 [43] and UNE-EN 12390-6 [44] specifications, respectively. Two specimens (cylinders of 100 mm in diameter and 200 mm in height) were used in each test for each type of concrete produced.

2.3.3. Durability

The sorptivity value, the chloride ion penetration test, and the accelerated carbonation resistance of each concrete were determined. The capillary water absorption of concrete was assessed at 28 days using two of the 100 $\times$ 100 $\times$ 100 mm cubic specimens according to ISO 15148:2002(E) [45]. To determine the amount of capillary water absorption content, the bottom face of the specimens was submerged in water 5 mm. (The lateral surfaces were impregnated with an impermeable resin.) The cumulative water absorbed was recorded at different time intervals up to 48 h by weighing the specimen after removing the surface water using a dampened cloth. Sorptivity is the slope of the regression curve of the quantity of water absorbed by a unit surface area versus the square root of the elapsed time from the initial instant to 120 min. The capillary water absorption results are the average of the three measurements.

The chloride penetrability of concrete was measured according to ASTM C1202-22e1 [46] using a 100Ø mm concrete disc of 50 mm thickness cut from the 100∅/200 mm concrete sample. According to Ghanem et al. [47], this test could represent the true long-term chloride penetration when the concrete was produced with the cement employed in this study. The resistance of concrete to chloride ion penetration is described by the total charge (in Coulombs) passed during a test period of 6 h. In this study, the chloride ion penetrability test was performed on the concrete specimens at 28 and 56 days of age, and each result was the average of the two measurements taken.

The accelerated carbonation coefficients were determined according to the UNE-EN 12390-12 [48] specification. Two prismatic specimens of 100 × 100 × 400 mm were used for each produced concrete. The specimens subjected to an accelerated carbonation process were removed from the humidity chamber after 28 days and preconditioned in the laboratory for 14 days. Pre-conditioning was performed at a CO_2 concentration of 400 ppm, 21 ± 1°C, and 50–55% RH before placing them in the CO_2 chamber. After pre-conditioning for 14 days, the samples were placed in a chamber at 3% CO_2 and 57% RH at 20 °C. The carbonation depth of each sample was determined after 0, 14, 28, 56, and 91 days of exposure to the chamber environment. To determine the carbonation depth, a phenolphthalein indicator with a solution of 1 g phenolphthalein in 70 g ethanol and 30 g water was applied to the fractured concrete surface, following UNE-EN 14630 [49] specification.

3. Results

3.1. Physical Properties

Table 4 shows the physical properties obtained from the produced concretes. In addition, the dispersion of the results is described between brackets. All the concretes produced with RA had a higher absorption capacity than the CC concrete due to the high porosity of the adhered mortar. As a result, the RAC50 concrete achieved a 6.5% higher absorption capacity than that of the RAC50-C concrete, in which the carbonated RCA-C aggregates were employed. However, the absorption capacity of the RCA50-FA was 8% lower than that of the RCA50-FA-C concrete, in which the carbonated RCA-FA-C recycled aggregates were used. Thus, the CC concrete achieved the lowest absorption capacity, followed by RAC50-FA, RAC50-C, RAC50-FA-C, and RAC50 concrete. The RAC50 concrete achieved the highest percentage of accessible voids. It was noted that the standard deviation of all the properties was low.

Table 4. Physical properties of concretes (Standard deviation in brackets).

Concrete Reference	Water Absorption (%)	Dry Density (Kg/m^3)	Accessible Voids (%)	Sorptivity (mm/min$^{0.5}$)
CC	4.86 (0.13)	2290 (20)	11.13 (0.23)	0.035
RAC50	5.75 (0.14)	2240 (10)	12.85 (0.23)	0.039
RAC50-C	5.38 (0.14)	2240 (10)	12.06 (0.27)	0.036
RAC50-FA	5.2 (0.07)	2240 (10)	11.66 (0.13)	0.032
RAC50-FA-C	5.62 (0.05)	2230 (10)	12.53 (0.09)	0.048

3.2. Mechanical Properties

Table 5 shows the mechanical properties obtained for each type of concrete. In addition, the standard deviation value of the results is given in parenthesis. Regarding compressive strength, the RAC50-FA-C concrete reached the lowest strength at any age: 12% lower than the CC concrete at seven days. At 28 days, RAC50-C reached the strength of the CC concrete, and tests on the other three recycled concretes determined a 5% lower value than CC concrete. In addition, all the concretes achieved the compressive strength required by Spanish structural code [34]. At 56 days, RA concrete reached a similar strength: 7–9% lower than CC concrete. The standard deviation value obtained by all the concretes' properties was acceptable.

Table 5. Mechanical properties of the produced concretes.

Test	Time (Days)	CC	RAC50	RAC50-C	RAC50-FA	RAC50-FA-C
Compressive strength (MPa)	7	44.63 (0.31)	42.23 (0.19)	42.76 (0.52)	42.26 (0.33)	39.19 (2.76)
	28	45.92 (0.21)	44.10 (1.83)	45.71(1.48)	45.09 (1.22)	43.82 (2.22)
	56	54.0 (1.52)	50.29 (0.38)	50.65(1.30)	49.90 (1.22)	49.07 (1.76)
Splitting tensile (MPa)	28	3.19 (0.08)	2.89 (0.25)	3.36 (0.16)	3.14 (0.17)	3.02 (0.44)
E (GPa)	28	34.1 (0.87)	30.9 (0.35)	31.9 (0.33)	31.9 (0.71)	30.6 (0.19)

Regarding splitting tensile strength, the RAC50-C concrete achieved the highest strength: 18% higher than the RAC50. In contrast, while the RAC50-FA concrete achieved a similar strength to the CC concrete, the RAC50-FA-C concrete achieved a 5% lower strength value. However, all the recycled concrete achieved an adequate strength value for structural concrete [35]. Kou et al. [50] determined that the splitting tensile strength at 28 days and 90 days of concrete produced with carbonated recycled aggregates (when the recycled aggregates' attached mortar was composed with OPC) increased by 6% and 12%, respectively, compared to concrete produced with uncarbonated RA. In addition, the tensile strength with carbonated RA at 90 days was even higher than that of the reference concrete. Moreover, Liang et al. [17] determined that the effect of CO_2 treatment of RA on the increase in splitting tensile strength was more significant than that of compressive strength. Table 5 also shows that the standard deviation of the obtained values was low for all the produced concretes. Figure 4 describes the relationship between compressive strength and splitting tensile strength of each concrete at 28 days. The findings described indicate that a higher compressive strength value also resulted in an increase the splitting strength value. It is clear that the use of RCA-C aggregates improved the mechanical properties of concrete and, more considerably, the splitting tensile strength. However, the concrete produced with carbonated RCA-FA-C aggregates, the RAC50-FA-C, achieved lower values than those of the RAC50-FA concrete.

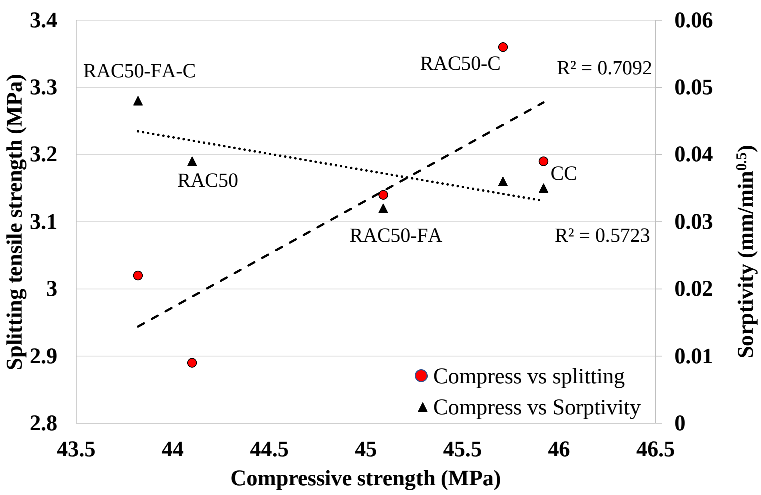


Figure 4. Compressive strength vs. splitting tensile strength and sorptivity.

Consistent with the modulus of elasticity test, all the concrete produced using the RA suffered a reduction in the values obtained for the CC concrete. However, while the RAC50-C and the RAC50-FA concrete mixtures values decreased by 6%, the values of the RAC50 and the RAC50-FA-C decreased by 10% compared to that of CC concrete. Table 5 also described that the standard deviation obtained in all the values was low.

Consequently, it can be concluded that the RAC50-C concrete achieved better mechanical properties than the RAC50. Furthermore, other researchers also found that in the concrete produced with carbonated RCA (when OPC is a component of attached mortar), the mechanical properties of concrete improved compared to those of the recycled concrete produced with uncarbonated recycled aggregates [17,24,50]. In contrast, the RAC50-FA-C concrete achieved lower mechanical properties than the RAC50-FA. When the composition of the attached mortar of RA included FA, the carbonation process worsened the properties of the RCA-FA-C with respect to those of the RCA-FA aggregates and, consequently, the mechanical properties of the RAC50-FA-C concrete with respect to the RAC50-FA, too.

3.3. Durability

3.3.1. Capillary Water Absorption—Sorptivity

Figure 5 shows the capillary absorption capacity of all the produced concretes. All the obtained data are described with error bars. While the RAC50-FA concrete proved to achieve the lowest absorption capacity value, the RAC50-FA-C achieved the highest. In addition, RAC50-FA reached a lower capillary absorption capacity than the CC concrete. Moreover, RAC50-C concrete achieved lower capillary absorption capacity than RAC50 concrete. Similarly, Table 4 shows that the RAC50-FA concrete achieved the lowest sorptivity value of 0.032 mm/min$^{0.5}$, followed by the CC concrete with 0.035 mm/min$^{0.5}$ and the RAC50-C with 0.036 mm/min$^{0.5}$. The concretes RAC50 and RAC50-FA-C achieved the highest values, with 0.039 and 0.048 mm/min$^{0.5}$, respectively. Similar to previously analysed properties, the RAC50-FA-C concrete produced using RCA-FA-C aggregates (carbonated aggregates with FA in attached mortar) achieved 50% higher sorptivity than the concrete produced with the RCA-FA aggregate.

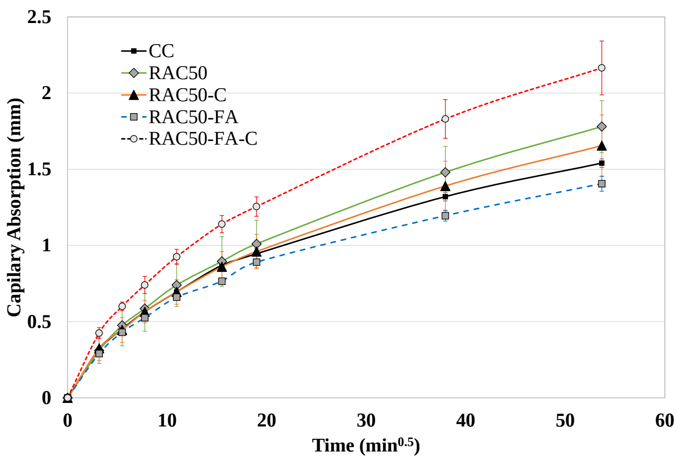


Figure 5. Capillary absorption of all the concrete.

Figure 4 shows the relation between compressive strength and sorptivity at 28 days of age. While the RAC50-FA-C concrete achieved 2% lower compressive strength than that of the RAC50-FA concrete, the sorptivity of the RAC50-FA-C was 50% higher than that of the RCA50-FA. In contrast, the RAC50-C concrete improved compressive strength and sorptivity values compared to those of the RAC50. While the RAC50-C concrete achieved a 4% higher compressive strength than the RAC50, the sorptivity of the RAC50-C was reduced by 8% with respect to that of the RAC50.

According to several researchers [51], concrete with a sorptivity value lower than 0.10 mm/min$^{0.5}$ is considered durable. However, other researchers [52] proposed lowering that value to 0.05 mm/min$^{0.5}$ for safety reasons. Pedro and Brito [53] found that the quality of concrete is low if the sorptivity value is higher than 0.2 mm/min$^{0.5}$, medium if the coefficient is between 0.2 mm/min$^{0.5}$ and 0.1 mm/min$^{0.5}$, and good if the coefficient is lower than 0.1 mm/min$^{0.5}$. Based on those criteria, all the concretes produced with 50% RA achieved a durable category according to the sorptivity property.

3.3.2. Chloride Ion Penetration

Table 6 shows the chloride ion penetration resistance value of the produced concretes at 28 days and 56 days of curing. According to the results of the ASTMC1202 test, every concrete had a high chloride ion penetrability, as the obtained electrical conductance values were higher than 4000 coulombs, which is the maximum admitted value to categorise as moderate resistance to the penetration of chloride ion concretes. In addition, resistance was reduced when RA was employed. However, the RAC50-FA concrete obtained only a 4% higher value than the CC concrete. Therefore, the use of RAC-FA aggregates (uncarbonated and FA in attached mortar) improved the property of recycled aggregate concrete. As known, the resistance to chloride ion penetration is higher when FA is used [54,55]. Consequently, the presence of FA in RA's attached mortar improved the concrete's property.

Table 6. Penetration of chloride ion resistance coulombs.

Concrete Reference	28 Days (Coulombs)	56 Days (Coulombs)	Increase of Resistance (%)
CC	6731 (49.2)	5180 (821.1)	23
RAC50	8799 (582.2)	6377 (810.2)	27
RAC50-C	7407 (411.0)	6166 (319.7)	17
RAC50-FA	7006 (352.5)	5412 (300.1)	23
RAC50-FA-C	9296 (1088.2)	7221 (270.2)	22

However, the RAC50-FA-C concrete achieved the lowest value to chloride penetration resistance. The carbonation process of the RCA-FA-C aggregate caused the coarsening of the pore structure of the C-A-S-H phase [23] presented in FA attached mortar. Table 6 shows that the RAC50-FA-C and RAC50-FA concretes achieved 38% and 4% higher values than the CC concrete, respectively. In contrast, the RAC50-C concrete reached higher chloride resistance than the RAC50 concrete. The RAC50-C and RAC50 achieved 10% and 30% higher values, respectively, than the CC concrete. At 56 days, the chloride ion penetration resistance of all the concretes increased compared to the values obtained at 28 days. The RAC50 achieved the highest increase with 27% and the RAC50-C the lowest with 16%. However, at 56 days, the CC concrete achieved the lowest chloride penetration resistance, followed firstly by the RAC50-FA, then the RAC50-C, the RAC50, and lastly the RAC50-FA-C concrete with the lowest resistance. Moreover, the RAC50-FA and the RAC50-FA-C achieved 4% and 39% higher values, respectively, than that of the CC concrete. However, it must be mentioned that the standard deviation of each of the properties was relatively high, requiring as a consequence more than two specimens to determine the average value.

3.3.3. Carbonation Resistance

Table 7 depicts the accelerated carbonation coefficient (k_{acc}) obtained in the testing of all the concretes. The standard deviation of each average carbonation depth value is also described. The k_{acc} was calculated assuming a steady-state condition (i.e., a constant carbonation coefficient) defined by Fick's first law of diffusion, as shown in Equation (1).

$$X_c(t) = k_{acc} \cdot (t)^{0.5} \quad (1)$$

where X_c is the carbonation depth (mm) determined experimentally, k_{acc} is the carbonation coefficient, and t is the time (day).

Table 7. Carbonation depth and carbonation coefficient of the various concretes.

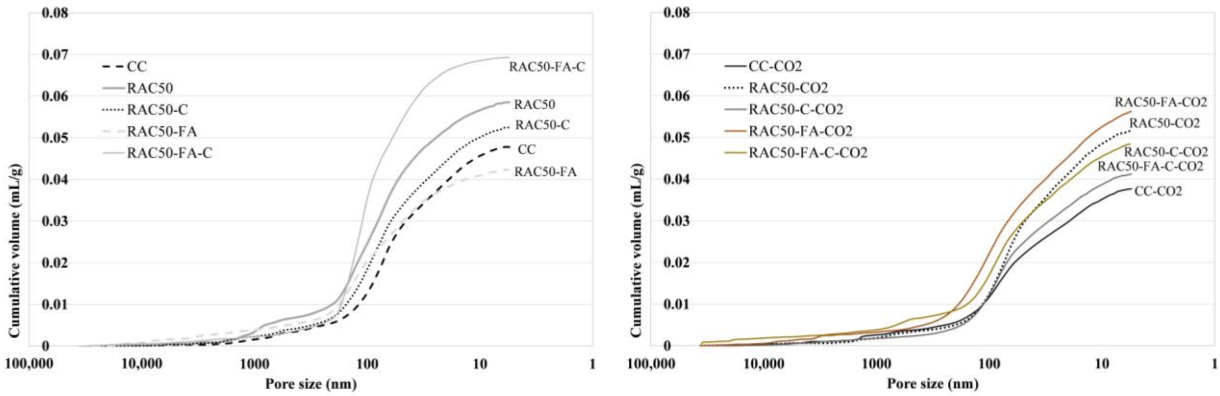
Concrete Reference	Carbonation Depth (mm) at Exposure Time					Carbonation Coefficient	
	0 Days	**14 Days**	**28 Days**	**56 Days**	**91 Days**	k_{acc} (mm/day$^{0.5}$)	$K_{natTHEO}$ (mm/year$^{0.5}$)
CC	0 (0.00)	3.5 (0.29)	5.2 (0.77)	7.0 (0.82)	8.8 (0.27)	0.926	2.105
RAC50	0 (0.00)	4.4 (0.18)	5.4 (0.53)	7.0 (0.00)	9.5 (0.35)	0.958	2.180
RAC50-C	0 (0.00)	5.1 (0.62)	7.4 (0.44)	9.9 (0.32)	12.9 (1.18)	1.338	3.043
RAC50-FA	0 (0.00)	4.5 (0.53)	6.9 (0.29)	9.9 (0.74)	11.8 (1.41)	1.269	2.886
RAC50-FA-C	0 (0.00)	5.0 (0.06)	8.7 (1.36)	10.0 (0.18)	13.9 (1.97)	1.432	3.257

After 91 days of CO_2 exposure, the CC concrete reached the lowest carbonation depth, with an 8.8 mm value, followed by the RAC50 concrete, which achieved a carbonation depth of 9.5 mm. As several researchers have determined, the concrete produced with 50% of uncarbonated RCA, i.e., the RAC50 concrete, achieved a similar carbonation rate to that of the CC concrete [7,8,15]. In contrast, the RAC50-C concrete (produced with carbonated RCA-C) achieved 12.9 mm of carbonation, reaching an accelerated carbonation rate, kacc, of 1.35 mm/day$^{0.5}$. The RAC50-C concrete achieved a 35.5% higher carbonation rate than that of the RAC50 concrete. In addition, the recycled concrete produced with RCA-FA (obtained from FA parent concrete), i.e., the RAC50-FA and RAC50-FA-C concretes, achieved a carbonation rate of 1.27 and 1.44 mm/day$^{0.5}$, respectively. As expected, the concretes produced with the RCA-FA aggregates (composed with FA) achieved a higher carbonation rate than those produced with the RCA aggregates. In addition, the concrete made with uncarbonated RCA-FA aggregates achieved a lower carbonation rate than the carbonated aggregates. The CO_2- treated recycled aggregates worsened the carbonation resistance of recycled concrete.

The total porosity of the carbonated (nominated as CO_2) and uncarbonated area (No) of the five produced concretes mixtures after being submitted to the 91 days of accelerated carbonation attack were measured using a porosimetry mercury intrusion (MIP) tester (see Table 8). The porosity of the RAC50-FA concrete increased from 9.36 to 12.67%, probably due to the coarsening pore diameter of the attached mortar of recycled aggregates, which was composed of FA. In contrast, the total porosity of the RAC50-FA-C decreased after the carbonation process (RA were already carbonated) was concluded. Consequently, the new cement paste was only carbonated. All the concrete mixtures were produced using CEM IIAL cement, which did not have SCM; therefore, the carbonation of the new cement paste caused the decrease in pore size and the total volume of meso- and macropores due to $CaCO_3$ precipitation [23]. Thus, the porosity and average pore diameter of the CC, the RAC50, and the RAC50-C concretes also decreased after the carbonation process. Figure 6 shows that the RAC50-FA concrete was the only concrete that increased its cumulative volume due to the coarsening of the macro- and mesopores of the attached mortar composed of FA.

Table 8. Total porosity and average pore diameter of produced concrete submitted to the carbonation process.

	CC		**RAC50**		**RAC50-C**		**RAC50-FA**		**RAC50-FA-C**	
	No	**CO_2**	**No**	**CO_2**	**No**	**CO_2**	**No**	**CO_2**	**No**	**CO_2**
Porosity (%)	11.12 (1.6)	8.95 (0.98)	13.18 (1.3)	11.64 (2.01)	12.05 (1.1)	11.84 (1.45)	9.36 (0.8)	12.67 (1.6)	14.93 (2.2)	12.92 (1.9)
Average pore D (nm)	35.61 (1.1)	32.74 (0.7)	46.29 (2.3)	32.57 (1.5)	38.48 (1.8)	34.25 (1.4)	47.67 (1.9)	34.45 (1.7)	61.92 (2.6)	35.35 (0.7)



(**a**) (**b**)

Figure 6. Development of pore size distribution of (**a**) uncarbonated area (No) and (**b**) carbonated area (CO_2) of concrete after exposure to the accelerated carbonation attack.

The k_{acc} can be used to estimate the theoretical natural carbonation coefficient ($k_{natTHEO}$)) [56,57] process of each produced concrete using Equation (2).

$$k_{acc}/k_{natTHEO} = (\varnothing_{acc})^{0.5}/(\varnothing_{natTHEO})^{0.5} \quad (2)$$

where $\varnothing_{acc}$ and $\varnothing_{natTHEO}$ are the CO_2 concentrations in the accelerated carbonation (3%) and natural carbonation processes (425 ppm, in Barcelona), respectively. Table 7 describes the $k_{natTHEO}$ of each concrete.

According to the Spanish concrete structural code (SC) [34], a concrete structure exposed to XC3 and XC4 environments (corrosion by carbonation) with a useful life of 50 and 100 years has limited the minimum cover depth. Therefore, in XC3 conditions, a minimum cover of 20 mm and 30 mm would be required. In addition, a minimum cover depth of 25 and 35 mm, respectively, would be required for concrete exposed to the XC4 environment conditions. Table 9 describes the carbonation depth achieved by each concrete according to the $k_{natTHEO}$ rate after a lifespan of 50 and 100 years.

Table 9. Carbonation depth after a lifespan of 50 and 100 years.

	CC	RAC50	RAC50-C	RAC50-FA	RAC50-FA-C	XC3	XC4
	Carbonation Depth (mm)					Min. Cover (mm)	
50 years ($k_{natTHEO}$)	14.9	15.5	21.6	20.5	23.1	20	25
100 years ($k_{natTHEO}$)	21.1	21.9	30.5	28.9	32.6	30	35

The obtained results from this study concluded that all the concretes achieved a lower carbonation depth value than the minimum cover depth value required for the defined useful lifespan expected in XC4 environment conditions. However, for exposure in XC3 environment conditions, the CC and RCA50 concretes would only be adequate for a lifespan of 50 years, and the CC, the RCA50, and the RCA50-FA would be adequate for exposure in XC4 environmental conditions. Thus, none of the recycled concrete produced with CO_2-treated recycled aggregates would achieve the governmental minimum requirements.

3.3.4. Chloride Ion Penetration vs. Carbonation Resistance

Figure 7 describes the chloride ion penetration (in coulombs) at 28 days (Table 7) and carbonation resistance (K_{acc}, Table 8) of recycled aggregate concretes with respect to the CC concrete. Figure 7 shows that the treatment and composition of recycled aggregates influenced the concrete's chloride and carbonation resistance in a different form.

While the RAC50-C concrete (concrete produced with carbonated RCA-C) and RAC50-FA concrete (concrete produced with uncarbonated RCA-FA) achieved the highest chloride resistance and similar values to the CC concrete, the RAC50 concrete (concrete produced with uncarbonated RCA) achieved carbonation resistance equivalent to that of the CC concrete.

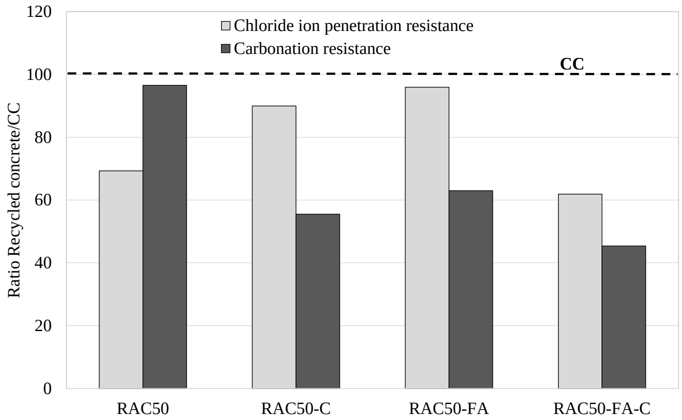


Figure 7. Influence of recycled aggregate concrete in Chloride ion penetration and carbonation resistance with respect to CC concrete.

4. Conclusions

The following conclusions can be deduced from the results obtained by testing how the properties of concrete are affected by the carbonation treatment on different compositions of recycled aggregates:

- The CO_2 treatment increased the density and reduced the absorption capacity of recycled aggregates. However, when the attached mortar of the recycled aggregates was composed with FA (RCA-FA), after carbonation, the pore size distribution was transformed, increasing in the mesopores and macropores. Consequently, the RCA-FA-C (carbonated) aggregate achieved higher permeability than the RCA-FA;
- Therefore, according to the properties of recycled aggregates, the carbonate treatment was effective when the attached mortar of the recycled aggregates was composed of OPC. In contrast, the RCA-FA-C (carbonated) aggregates achieved higher permeability than those of the RCA-FA aggregates;
- According to physical and mechanical properties, the following is true:
    - ○ All the recycled concretes achieved adequate properties as structural concrete. They reached the compressive strength of the CC concrete at 28 days and a lower modulus elasticity than that of the CC concrete;
    - ○ The recycled concrete produced with 50% of the RCA-C and RCA-FA aggregates achieved the most efficient properties of all the recycled concretes:
        - ■ They only achieved a 7% higher absorption capacity than that of the CC concrete;
        - ■ The concrete produced with RCA-C achieved the highest strength;
        - ■ The recycled concrete produced with RCA-C and RCA-FA reached similar or higher splitting tensile strength to that of the CC concrete;
        - ■ The recycled concrete produced with RCA-C and RCA-FA suffered a lower decrease of 6% in modulus elasticity compared to the CC concrete.

According to durability, the following is true:

- All the recycled concrete produced with 50% of RA achieved the durable category defined with a sorptivity value lower than 0.05 mm/min$^{0.5}$. The concrete made with 50% of the RCA-FA achieved the lowest sorptivity value of 0.032 mm/min$^{0.5}$, followed by the CC concrete with 0.035 mm/min$^{0.5}$ and the recycled concrete produced with 50% of the RCA-C with 0.036 mm/min$^{0.5}$;

- The recycled concrete achieved higher chloride ion penetration than that of the CC concrete. However, the concrete produced with the RCA-FA aggregates achieved the best value with only 4% higher penetration than the CC concrete, followed by the concrete produced with the RCA-C aggregates. In contrast, the concrete produced with the RCA-FA-C aggregates achieved the highest penetration: 38% higher than that of the CC concrete;
- The CC concrete achieved the lowest carbonation rate, followed by the concretes produced with 50% RCA and 50% RCA-FA. The CO_2-treated recycled aggregates worsened the carbonation resistance of the recycled concrete, with this worsening condition increasing when the RCA-FA-C aggregates were used;
- It was found that the CC and RAC50 concretes were the only concretes adequate for both XC3 and XC4 environment expositions.

It can be concluded that carbonation treatment was inefficient when the recycled aggregates were obtained by crushing a parent concrete composed of FA blended cement. The concretes made with the RCA-FA-C aggregates obtained the worst physical, mechanical, and durability property values. In contrast, the concrete produced with 50% of the RCA-C aggregates obtained the best property values except for carbonation resistance, which was higher in concrete produced with the RCA aggregate. In addition, the concrete properties made with the RCA-FA aggregates achieved better sorptivity and chloride ion penetration resistance than those produced with the RCA-C aggregates.

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