

Article

Carbon Emission Evaluation of CO₂ Curing in Vibro-Compacted Precast Concrete Made with Recycled Aggregates

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Abstract: The objective of the present study was to explore three types of vibro-compacted precast concrete mixtures replacing fine and coarse gravel with a recycled/mixed concrete aggregate (RCA or MCA). The portlandite phase found in RCA and MCA by XRD is a “potential” CO₂ sink. CO₂ curing improved the compressive strength in all the mixtures studied. One tonne of the mixtures studied could be decarbonised after only 7 days of curing 13,604, 36,077 and 24,635 m³ of air using natural aggregates, RCA or MCA, respectively. The compressive strength obtained, XRD, TGA/DTA and carbon emission evaluation showed that curing longer than 7 days in CO₂ was pointless. The total CO₂ emissions by a mixture using CO₂ curing at 7 days were 221.26, 204.38 and 210.05 kg CO₂ eq/m³ air using natural aggregates, RCA or MCA, respectively. The findings of this study provide a valuable contribution to carbon emission evaluation of CO₂ curing in vibro-compacted precast concrete with recycled/mixed concrete aggregates (RCA or MCA). The technology proposed in this research facilitates carbon capture and use and guarantees enhanced compressive strength of the concrete samples.

Keywords: carbon emission evaluation; CO₂ curing; waste recycling; CO₂ sequestration; construction and demolition waste



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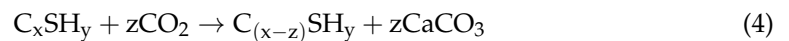
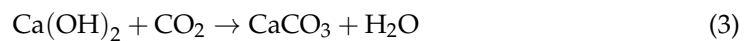
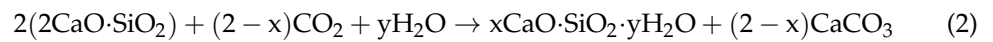
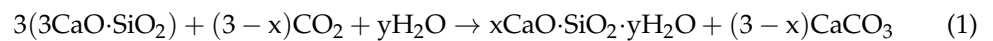
1. Introduction

Ordinary Portland cement (OPC) is the most widely used material in construction worldwide, with a global consumption between 1930 and 2013 of 76.2 billion tonnes [1,2]. The manufacture of OPC produces a large amount of CO₂ (1 tonne of cement emits approximately 1 tonne of CO₂), which means that 8% of the world’s total CO₂ emissions are related to the cement industry [3–5]. Scientists must therefore work together to reduce/reuse the CO₂ emissions produced by OPC manufacturing.

Global warming and climate change are growing problems. According to the Intergovernmental Panel on Climate Change (IPCC) [6], the increase in the earth’s surface temperature will lead to dire consequences, and CO₂ emissions are the main cause of global warming [7,8]. Carbon capture storage (CCS) and carbon capture utilisation technologies (CCU) are among the many ways to reduce CO₂ emissions [9]. The cost of emitting CO₂ in the European Union is approximately 80 €/t CO₂, although this will increase due to current policies [10]. With the implementation of CCU, instead of costing money, CO₂ would become a source of income.

It has been demonstrated recently that CO₂ curing of cement-based materials converts gaseous CO₂ into solid calcium carbonate [11–13], also called mineral carbonation

technology (MCT), and is considered to be a cost-effective and environmentally-friendly method of capturing and storing CO₂ [14]. CO₂ sequestration by mineral carbonation and its subsequent conversion into a product for the construction industry is one of the most representative examples of MCT [15]. Several studies have applied the CO₂ curing concept to produce commercial building material: Several authors [16–18] carbonated different types of slag into building material; Zhen et al. [19] directly used the CO₂ generated by a cement factory (flue gas carbonation) to cure cement products; Suescum-Morales et al. [5,12,20] studied the application of carbonation in different forms of mortars intended for use as unreinforced construction products up to 7 days of curing. There has been no application of carbonation for longer curing ages, for example, up to 28 days, and studies along these lines would fill many gaps in our current knowledge. Carbonation mainly involves a reaction between CO₂ and calcium silicate phases [21]. Equations (1) and (2) are usually related to accelerated carbonation at early ages. Equations (3)–(5) are normally related to the durable carbonation of concrete, although they also occur when there is forced carbonation [22,23]. The main equations are presented in Equations (1)–(5):



The rapid development and constant growth of the construction sector generate a large number of recycled aggregates from construction and demolition waste (RAC&D) [24,25], which is around 30 billion tonnes per year. The reuse of masonry waste, such as recycled aggregate, was a highly relevant topic to the scientific community from the early 1970s [26,27]. The main reasons for this interest include the avoidance of depositing such waste and the overexploitation of natural aggregates (NA) [28,29]. Within RAC&D, there are different types of aggregates, depending on their origin (in very short form) [12,20]: recycled ceramic aggregates (ceramic waste); recycled concrete aggregates (concrete waste) and mixed recycled aggregate (a mixture of the two above). RAC&D is usually of poorer quality than NA, which results in poorer properties of the resulting mixtures. This is due to RAC&D having adhered to the old mortar with lower density, higher porosity, lower crush resistance, higher water absorption value and weaker interfacial transition zones (ITZs) [5,12,30,31]. Therefore, the development of new techniques to improve the quality of RAC&D is on the rise [27,32–37], among which are those that improve the quality of RAC&D by using CO₂ (accelerated carbonation), which seems to be a very promising technology [38–41].

To apply CO₂ curing at mixtures using RAC&D at a commercial scale, the environmental impact of accelerated carbonation treatment must be considered, since the energy consumption required is usually significant [41,42]. A life cycle assessment (LCA) can be carried out to quantify this impact [43–45], or a somewhat simpler form of CO₂ footprint assessment can be carried out [46–48]. There have been several studies conducting the comparative LCA analysis between NA and RAC&D [49], and, although the ISO 14040 standard regulates the definition and selection criteria of functional units, the units chosen for investigating concrete products are insufficient to reflect concrete in terms of its environmental impact [50]. Most of the studies found only calculate the CO₂ sequestration capacity of CO₂ cured samples, without taking into account the carbon footprint of the curing process itself [5,12,25,51]. However, CO₂ sequestration of cement-based waste materials is a multi-process activity with consumption of energy: demolition sector, recycling sector and carbon capture sector, among others [52]. There are no studies that calculate CO₂ sequestration in mixtures made with RAC&D through thermogravimetric analysis, nor are there studies that perform a carbon footprint assessment considering both the

materials used and the curing conditions and time. Studies along these lines can also fill this knowledge gap.

The objective of the present study was to explore three types of vibro-compacted precast concrete mixtures replacing fine and coarse gravel with two types of RAC&D. CO₂ curing for 1, 3, 7, 14 and 28 days was employed to improve the compressive strength and CO₂ sequestration. X-ray diffraction (XRD) of the hardened samples was measured to analyse the effect of CO₂ curing for the duration of 28 days of curing. The carbon emission evaluation was performed using thermogravimetric analysis for the CO₂ sequestration and, and a CO₂ footprint assessment for the different mixtures and time/curing conditions.

2. Materials and Methods

2.1. Raw Materials

In this study, two types of natural coarse aggregates and natural fine aggregates were used: coarse gravel (CG), fine gravel (FG), sand-1 (S1) and sand-2 (S2). Two types of recycled aggregate were also used: recycled concrete aggregate (RCA) and mixed concrete aggregate (MCA). Figure 1 shows the aspect of the aggregates used. These aggregates were taken from a quarry in Cordoba (Spain). The difference between RCA and MCA was that MCA included pieces of ceramic bricks. Figure 2 shows the original particle size distribution of all the aggregates used in this study [53]. The skeletal density and water absorption were measured according to UNE-EN-1097-6:2013 [54]. Table 1 shows the basic physical parameter indicators of the aggregates and a CEM II/A-V 42.5 R was used [55]. The mixing water was tap water with polycarboxylate-based product from BASF (Glenium 3030 NS) as a plasticizer (1210 kg/m³).



Figure 1. Images of the aggregates used (A) CG, (B) FG, (C) S1, (D) S2, (E) RCA and (F) MCA.

2.2. Aggregate Preparation and Mix Design

The aim was to replace coarse aggregates (CG and FG) with RCA and MCA. However, according to Figure 2, the particle sizes appear to be very different. Therefore, after an experimental and iterative process, it was decided to make a mixture of 22.22% CG and 77.78% FG (with respect to the sum of CG and FG). RCA had very large particle sizes (larger than 12.5 mm) and a very large proportion of finer particles (smaller than 2 mm). Therefore, in order to make the size of MCA and RCA similar, sieving was carried out. RCA material was sieved by 12.5 and 2 mm, rejecting the upper and lower parts, respectively. For MCA,

no sieving was required. With this combination, the particle sizes were very similar for the composition of GC + FG, RCA and MCA, as shown in Figure 3.

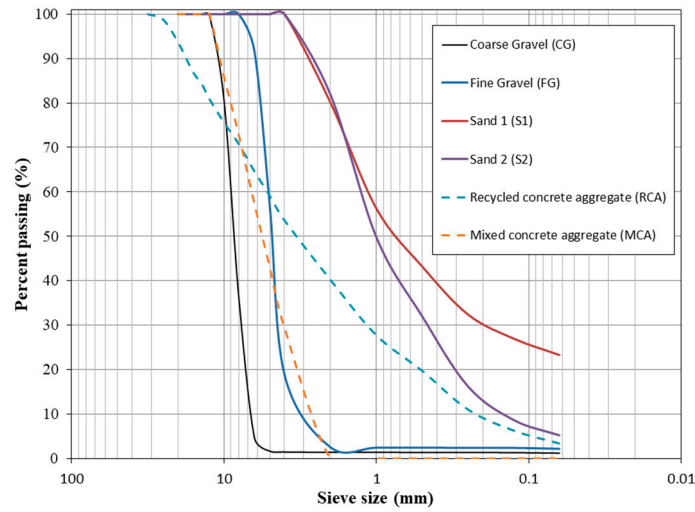


Figure 2. Original particle size distribution of aggregates used.

Table 1. Basic physical parameters of the aggregates used.

Type of Aggregates	Skeletal Density γ (g/cm ³)	Water Absorption (%)
Coarse gravel (CG)	2.47	3.13
Fine gravel (FG)	2.43	2.64
Sand 1 (S1)	2.65	2.40
Sand 2 (S2)	2.62	1.78
Recycled concrete aggregate (RCA)	2.21	7.42
Mixed concrete aggregate (MCA)	2.17	9.02

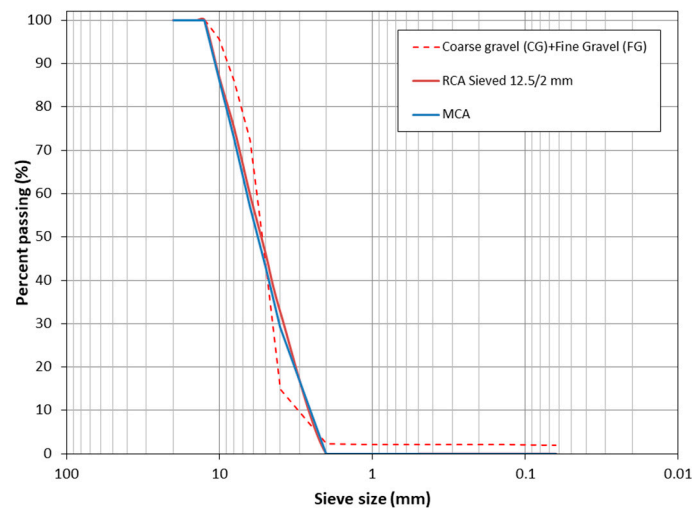


Figure 3. Particle size distribution of CG + FG, RCA sieving and MCA.

The proportions of concrete mixtures are given in Table 2. The aim of the mixtures was that they should be demoulded immediately. Therefore, a low w/c ratio was used (w/c = 0.4). For the reference mixture (named CONTROL), the water saturation was calculated according to the values of water absorption shown in Table 1. It can be observed that the composition of CG and FG represented 22.22 and 77.78%, respectively (with respect to the sum of CG and FG).

Table 2. Mixture proportion of concrete mixtures.

Notation	Mixture Proportions (kg/m ³)										
	Effective Water	Additional Water/Saturation water	Cem	Coarse Gravel (CG)	Fine Gravel (FG)	Sand 1 (S1)	Sand 2 (S2)	Recycled Aggregate Concrete (RAC)	Mixed Aggregate Concrete (MAC)	Sp	W/C
CONTROL	84	50	210	200	700	200	1200	-	-	0.5	0.4
M-100-RCA	84	86.70	210	-	-	200	1200	815.60	-	0.5	0.4
M-100-MCA	84	98.22	210	-	-	200	1200	-	798.95	0.5	0.4

For the mixture substituting 100% GC + FG by RCA (named M-100-RCA), the amount was calculated as shown in Equation (6). The volumes of both CG and FG (volumetric substitution) were taken into account. The same procedure was followed for the mixture substituting 100% GC + FG by MCA (named M-100-MCA), as shown in Equation (7). To calculate these quantities and the water absorption, the data shown in Table 1 was again used.

$$\text{Amount of RCA} = \frac{(\gamma_{\text{RCA}} \cdot \text{Amount}_{\text{CG}})}{(\gamma_{\text{CG}})} + \frac{(\gamma_{\text{RCA}} \cdot \text{Amount}_{\text{FG}})}{(\gamma_{\text{FG}})} \quad (6)$$

$$\text{Amount of MCA} = \frac{(\gamma_{\text{MCA}} \cdot \text{Amount}_{\text{CG}})}{(\gamma_{\text{CG}})} + \frac{(\gamma_{\text{MCA}} \cdot \text{Amount}_{\text{FG}})}{(\gamma_{\text{FG}})} \quad (7)$$

2.3. Concrete Mixing Procedure and Casting

The procedure for the mixing was as follows: the coarse aggregates (CG and FG or RCA or MAC) were added and mixed for 1 min, and then the fine aggregates (S1 and S2) were added and mixed for another minute. The saturation water and all aggregates were mixed for 10 min, the effective water together with the plasticizer was added and then the cement was added. A mixing time of 5 min was carried out. The mixer used was a professional electric mixer (Inhera X155, Inhera company, Castellón, Spain). For all the mixes, the results of the slump test were 0 mm [56], which indicated a dry consistency or S1 class, according to Eurocode 2 [57,58]. Cube samples of 100 mm were cast.

Due to the very dry consistency of the concretes obtained, vibro-compaction with a Kango vibration hammer (Milwaukee Kango 900 S) was applied. The 100 × 100 mm cubic moulds were filled in 2 batches, with a vibro-compaction of 10 s in each of these batches. The hammer used as well as the 3-D modelling of the specially fabricated steel part for this procedure by the authors is shown in Figure 4. The aim was to follow a procedure similar to that used in an unreinforced precast plant [59].

2.4. Curing Conditions and Test Methods

Once the samples were demoulded, they were subjected to:

- Conventional climatic chamber (CCC): 20 °C and 65% relative humidity. The CO₂ level under this environment was equivalent to atmospheric conditions (≈0.04%);
- CO₂ climatic chamber (CO₂CC): For this environment a Climacell 707-Evo (MMM Group, Planegg, München, Germany) with a CO₂ level of 5% (99.995% purity, supplied by Linde) and 20 °C with 65% relative humidity. The pressure was ambient.

According to the manufacturer, the maximum power of both pieces of equipment was 300 W, but with these conditions, a consumption of about 0.15 kW/h can be considered. This value will be used for the carbon footprint assessment.

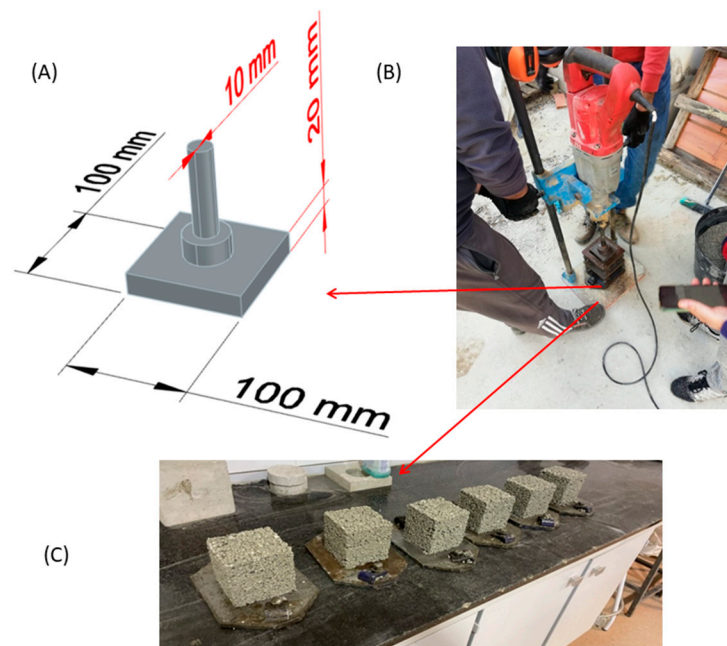


Figure 4. (A) 3-D modelling of the special part used to compact the manufactured concrete, made by the authors; (B) Hammer used and (C) Specimens demoulded immediately after being manufactured.

The raw materials were subjected to X-ray Fluorescence (XRF) in order to determine their chemical composition. For this purpose, ZSX PPRIMUS IV (Rigaku) equipment with a power of 4 kW was used, as well as X-ray diffraction (XRD) for all raw materials and for the samples hardened at the age of 7, 14 and 28 days in the different hardening environments. For XRD, a Bruker D8 Discover A25 instrument with $\text{CuK}\alpha$ ($\lambda = 1.54050 \text{ \AA}$, 40 kV and 30 mA) was used. A speed of $0.018 \text{ } 2\theta \cdot \text{s}^{-1}$ was used from 10° to 70° (2θ). The library used to compare the crystalline peaks was the JCPDS library [60]. Thermogravimetric analysis and differential thermal analysis (TGA/DTA) were applied for all raw materials and for the hardened samples at the same ages for XRD. TGA/DTA was carried out with Setaram Setys Evolution 16/18 apparatus. Before XRF, XRD and TGA/DTA, all samples were properly powdered. The heating rate of the TGA/DTA test was 5° min^{-1} , and the temperature range was approximately $20\text{--}1000^\circ \text{C}$. At the indicated ages and for XRD and TGA/DTA, the samples were immersed in pure absolute ethanol (PanReac.AppliChem) for 48 h to “stop” the setting reactions. The same recommendations and procedures indicated by RILEM TC-238 SCM were followed, except for the immersion time (in this case 48 h) [61].

“All raw materials and hardened concretes were prepared by crushing them in advance to obtain a representative sample of each material (for XRD and TGA/DTA). The powder was quartered and all measurements were carried out in triplicate. A similar procedure was carried out in other investigations [4,62–64]. The compressive strength [65] was determined at 1, 3, 7, 14 and 28 days of curing in the two hardening environments presented. Dry bulk density and accessible porosity for water were determined at 28 days of age, according to UNE 83980 [66], for both environments.

2.5. Carbon Footprint Assessment

The calculation of the carbon footprint was made for each mixture: CONTROL, M-100-RCA and M-100-MCA. As can be seen in Table 2, the main difference was the use of CG, FG, RAC or MAC. The CO_2 emission system for the production of the different blends is presented in Figure 5.

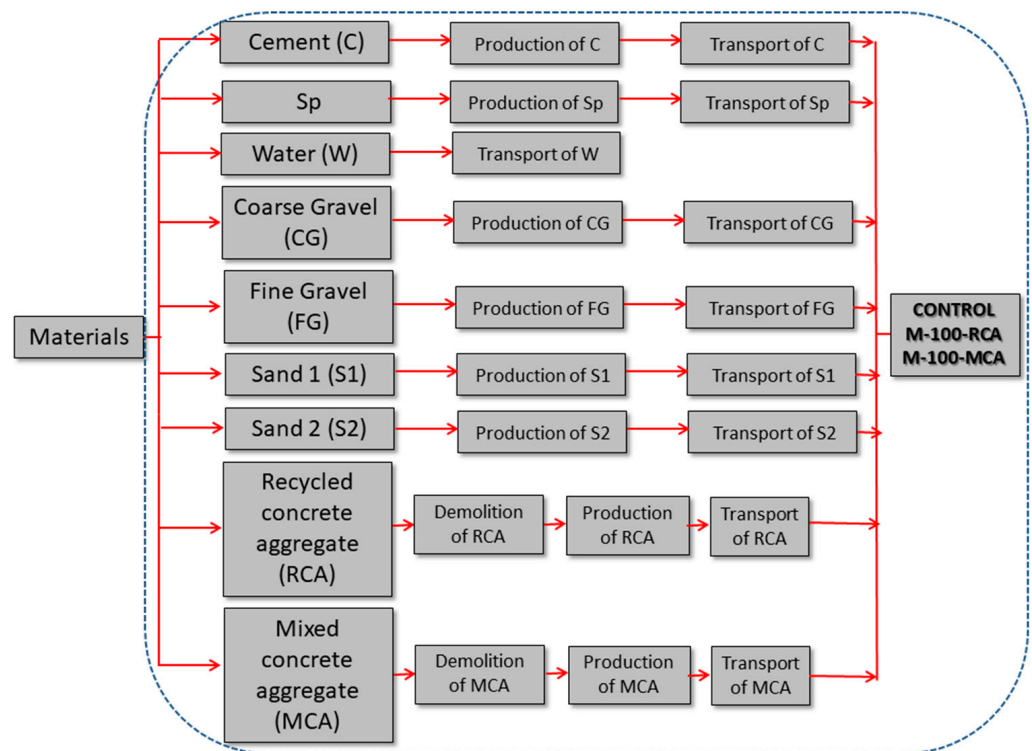


Figure 5. CO₂ emission system for CONTROL, M-100-RCA and M-100-MCA mixtures.

The CO₂ emissions: “CO₂ emitted materials” from the materials shown in Figure 5 for 1 m³ of the mixture can be calculated according to Equation (8) [46].

$$\text{CO}_2 \text{ emitted materials} = \sum_{i=1}^n N_i \cdot I_i \tag{8}$$

where *n* represents the number of raw materials used (see Figure 5); *N_i* is the weight of material used to make 1 m³ of concrete (kg); and *I_i* is the CO₂ emission of material *i* per kilogram (kg CO₂ eq/kg).

The CO₂ emissions produced by the curing of the samples “CO₂ emitted by curing”, both in the CCC and CO₂CC environment can be calculated according to Equation (9) to make 1 m³ of concrete [52].

$$\text{CO}_2 \text{ emitted by curing} = (E_{\text{ele}} \cdot t) + (E_{\text{CO}_2\text{-cur}} \cdot t) * 1 \text{ m}^3 \tag{9}$$

where *E_{ele}* is the CO₂ emission of electricity (kg CO₂ eq/h) for conventional climatic chamber (CCC); *E_{CO₂-cur}* is the CO₂ emission of electricity for CO₂ climatic chamber (CO₂CC) and *t* hours for curing (h). If the sample was cured in CCC, only the first term of Equation (9) was taken into account. If it was cured in CO₂CC, only the second term was considered. The emission factor for the Spanish electricity grid was considered to be 200 g CO₂/KWh [67]. The difference between *E_{ele}* and *E_{CO₂-cur}* is that the CO₂ emission factor of *E_{CO₂-cur}* is, albeit insignificant, slightly higher because it also considers the CO₂ emission necessary to capture the CO₂ used in the curing process. Normally, this capture is carried out in industries that generate CO₂, usually using monoethanolamine (MEA) as a capture device, because of its many advantages [68,69]. Therefore, for this rough estimate, both *E_{ele}* and *E_{CO₂-cur}* would be considered equal, although we want to clarify their differences. Finally, the total CO₂ emissions would be the sum of Equations (8) and (9), as shown in Equation (10). These emission factors may differ for other investigations, as

the efficiency of the equipment used may be different. However, they are usually very close to each other.

$$\text{Total CO}_2 \text{ emissions} = \text{CO}_2 \text{ emitted materials} + \text{CO}_2 \text{ emitted by curing} \quad (10)$$

Table 3 shows the carbon emission coefficient for the different materials (I_i) and for both curing chambers (E_{ele} and $E_{\text{CO}_2\text{-cur}}$) [41,46,47,49,70–72]. Emissions from the mixing process were not taken into account. The I_i factor depends, among other factors, on the fineness of the material (if it is obtained by crushing) and whether it comes from the natural or recycled aggregate.

Table 3. Carbon emission coefficient for materials and curing used.

Materials	Factor I_i	Unit	References
Cement	1.002	Kg CO ₂ eq/kg	[47,49,70]
Superplasticizer	1.150	Kg CO ₂ eq/kg	[49]
Water	3.47×10^{-4}	Kg CO ₂ eq/kg	[70]
Coarse gravel	4.10×10^{-3}	Kg CO ₂ eq/kg	[47]
Fine gravel	9.87×10^{-3}	Kg CO ₂ eq/kg	[49]
Sand 1	2.79×10^{-3}	Kg CO ₂ eq/kg	[71]
Sand 2	3.21×10^{-3}	Kg CO ₂ eq/kg	[49]
Recycled concrete aggregate	1.50×10^{-3}	Kg CO ₂ eq/kg	[46,49]
Mixed concrete aggregate	1.30×10^{-3}	Kg CO ₂ eq/kg	[46,49]
Curing	Factor $E_{\text{ele}}/E_{\text{CO}_2\text{-cur}}$	Unit	
Conventional Chamber (0.15 kW/h)	0.03	kg CO ₂ eq/h curing	[52,67–69]
CO ₂ Chamber (0.15 kW/h)	0.03	kg CO ₂ eq/h curing	[52,67–69]

3. Results and Discussion

3.1. Raw Materials

Table 4 presents the chemical composition. Figures 6 and 7 show the mineralogical composition of all the raw materials used in this research. The fundamental oxide of both gravels (CG and FG) was CaO. The main phase for CG and FG was calcite (CaCO₃) (05-0586) [60]. A very small intensity of dolomite (CaMg(CO₃)₂) (36-0426) [60] was also found in both gravels. For S1, the amount of CaO decreased while MgO increased, which was reflected in XRD (Figure 6), where the dolomite phase had a higher intensity than that of CG and FG. For S2, again, the major oxide was CaO and, in this case, only the calcite phase was found in XRD.

For RCA and MCA, the chemical composition was very similar. Slightly higher SiO₂ and Al₂O₃ contents were found in MCA, which might be related to the content of ceramic brick pieces in MCA [73] and could improve the pozzolanic reactions in the resulting concrete [74]. The main phases for RCA and MCA were quartz (SiO₂) (05-0490) [60] and calcite (CaCO₃) (05-0586) [60]. Other minority phases were also found: albite (Na(Si₃Al)O₈) (10-0393) [60]; illite ((Na,K)Al₂(Si₃AlO₁₀)(OH)₂) (02-0042) [60]; larnite (Ca₂SiO₄) (09-0351) [60], gypsum (CaSO₄·H₂O) (21-0816) [60] and portlandite (Ca(OH)₂) (44-1481) [60]. The portlandite phase found in RCA and MCA using X-ray diffraction is a “potential” CO₂ sink according to Equation (3), which may be due to the fact that both RCA and MCA had been in storage for a very short time and were “fresh”. The feldspar phase (NaSiAl₃O₈) [60] was found in MCA and not in RCA, which may have come from the pieces of ceramic brick that present MCA [75].

Table 4. Chemical components of raw materials.

Components (Mass% as Oxide)	Coarse Gravel (CG)	Fine Gravel (FG)	Sand 1 (S1)	Sand 2 (S2)	Recycled Concrete Aggregate (RCA)	Mixed Concrete Aggregate (MCA)	Cement
Na ₂ O	-	-	-	-	0.82	0.82	0.24
MgO	0.88	0.96	37.98	0.78	2.77	3.11	1.33
Al ₂ O ₃	0.20	0.73	0.06	0.96	7.78	10.49	3.73
SiO ₂	0.39	2.12	0.91	2.16	51.40	52.08	15.58
P ₂ O ₅	-	-	-	-	0.11	0.12	0.09
SO ₃	0.07	0.10	0.11	0.11	1.14	1.35	4.79
Cl ₂ O ₃	-	0.05	0.21	-	0.06	0.10	0.18
K ₂ O	0.03	0.09	0.05	0.18	1.80	2.38	1.21
CaO	98.31	95.75	60.44	90.32	30.62	25.15	70.03
TiO ₂	-	-	-	-	0.43	0.55	0.23
MnO ₂	-	-	-	-	0.09	0.08	0.06
Fe ₂ O ₃	0.10	0.18	0.23	5.50	2.76	3.59	2.44
CuO	-	-	-	-	-	-	-
ZnO	-	-	-	-	-	-	0.02
SrO	0.03	-	-	-	0.03	0.04	0.08
Rb ₂ O	-	-	-	-	-	0.01	-
Cr ₂ O ₃	-	-	-	-	0.21	0.15	-

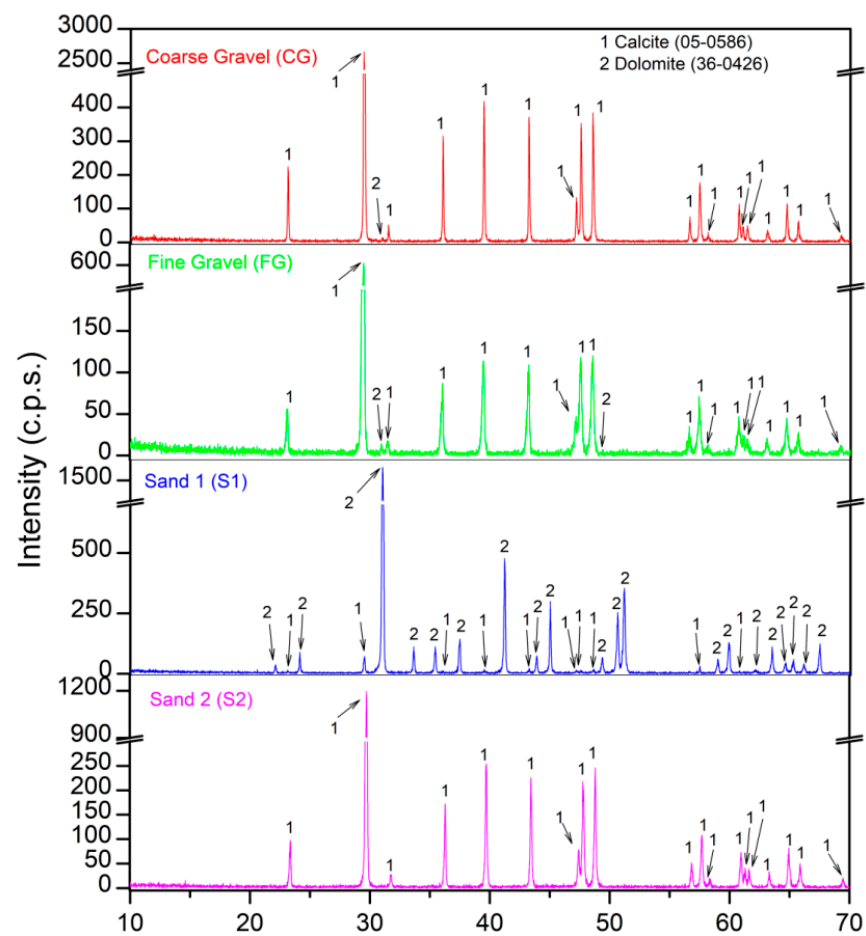


Figure 6. XRD patterns of natural aggregates used.

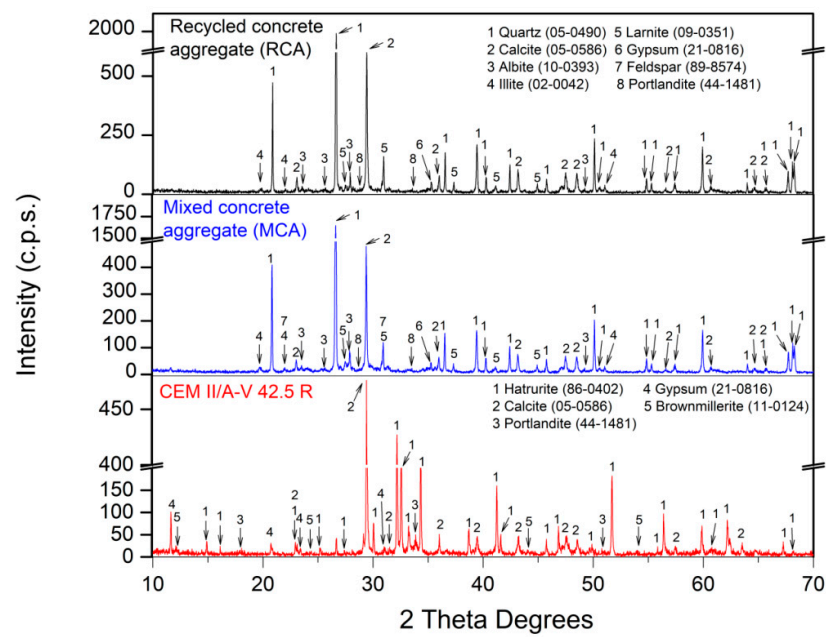


Figure 7. XRD patterns of recycled aggregates and cement used.

TGA and DTA for the natural aggregates used in this study are presented in Figure 8. For CG and FG, the main mass loss started at approximately 700 °C. From this range, the decomposition of calcite started, according to Equation (11), which was the main phase found in XRD. The low dolomite intensity found in the gravels was not detected by TGA/DTA. Similar results were obtained in other research studies [12,30,76].

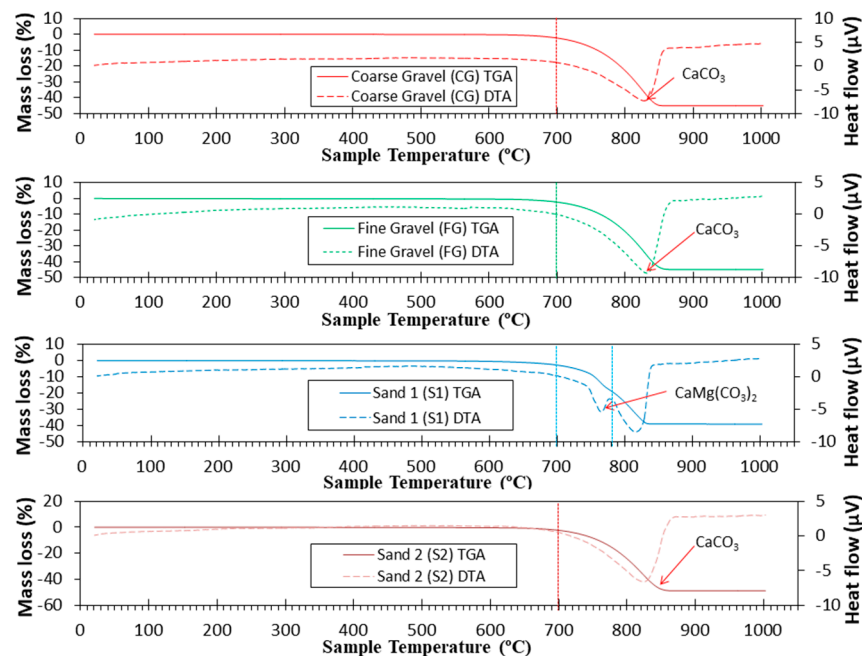
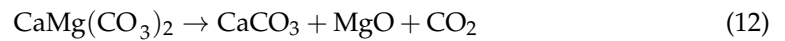


Figure 8. TGA (Solid lines) and DTA (dotted lines) curves for natural aggregates used.

However, for S1, a change in DTA was observed, although it did not start to lose mass noticeably until 700 °C, because dolomite was the main phase found in S1. The thermal

decomposition of dolomite includes two stages [77,78]. The first stage (from 700 to 780 °C) is shown in Equation (12) and the second stage (from 780 to 1000 °C) in Equation (11).



A very similar result was found for S2 as for FG and CG. This confirms the purity of calcite found for S2.

TGA and DTA for RCA, MCA and cement used in this study are presented in Figure 9. For RCA and MCA, the TGA/DTA result was very similar: (i) up to 105 °C of the physically absorbed water was lost [30]; (ii) from 105 to 380 °C, the loss of hydrated calcium silicates and aluminates occurred (CSH and CASH, respectively) [79,80]; (iii) from 380 to 480 °C, an endothermic peak (in DTA) related to the loss of portlandite was found which was identified in XRD (Figure 7) [23,81] and (iv) from 640 to 1000 °C, decomposition of calcium carbonate occurs, according to Equation (11) [4,82]. For cement, a typical result was found, with ranges of weight loss already extensively described in other research studies [5,12,30].

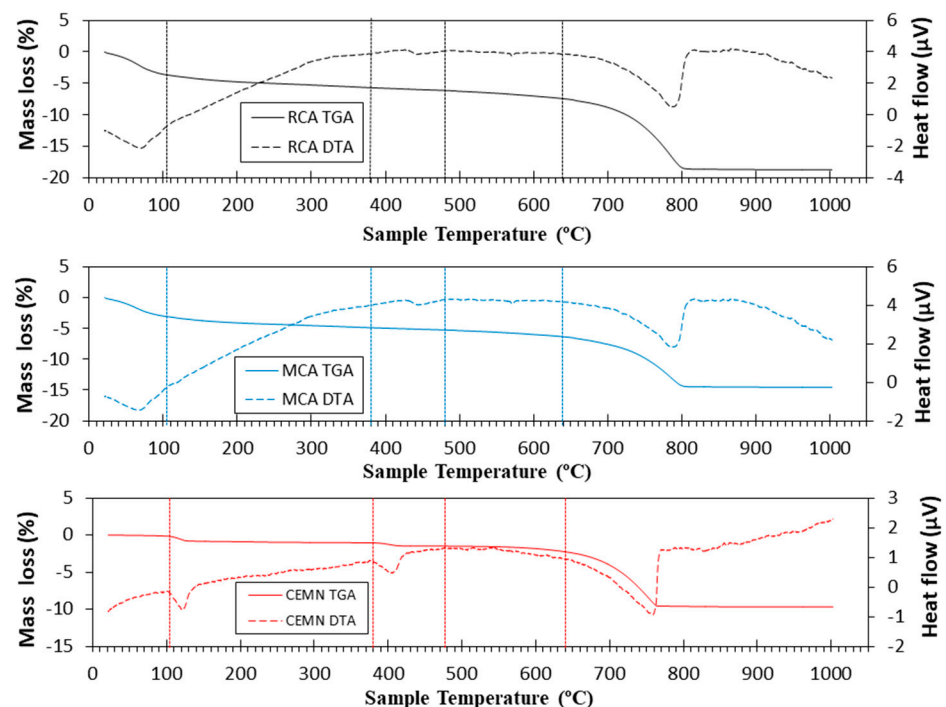


Figure 9. TGA (Solid lines) and DTA (dotted lines) curves of recycled aggregates and cement used.

3.2. Compressive Strength

Figure 10 shows the compressive strength of all the mixes studied, under both curing environments for 1, 3, 7, 14 and 28 days of age. The substitution of CG and FG for RCA using CCC (i.e., CONTROL vs. M-100-RCA) improved the compressive strength at all curing ages. Similarly, the substitution of CG and FG by MCA using CCC (i.e., CONTROL vs. M-100-MCA) also improved the compressive strength at all curing ages. These increases compared to CONTROL for the age of 28 days were 29.81 and 5.22% for M-100-RCA and M-100-MCA, respectively. While this is a very good result, it is unusual. The compressive strength of recycled aggregate was lower than conventional concrete [28,83]. There are many factors that influence the relationship between compressive strength and the use of recycled aggregates [84]: (a) recycled aggregate replacement level; (b) recycled aggregate size; (c) quality of recycled aggregate; (d) influence of the mixing procedure; (e) environmental conditions; (f) chemical admixtures and (g) additions incorporation. In this case, the use of very similar sizes between the FG and CG. Both recycled aggregates were very important factors (see Figure 3, factor (b)) [85], as well as the use of saturated aggregate before mixing (see Table 2, factor (d)) [86]. The quality of the recycled aggregate is also very important

(factor (c)), as the portlandite and larnite phases found (see Figures 7 and 9) can lead to improvements in compressive strength [87]. This indicates the feasibility of replacing CG and FG with RCA and MCA, which maximises the circular economy concept and minimises the use of non-renewable natural resources.

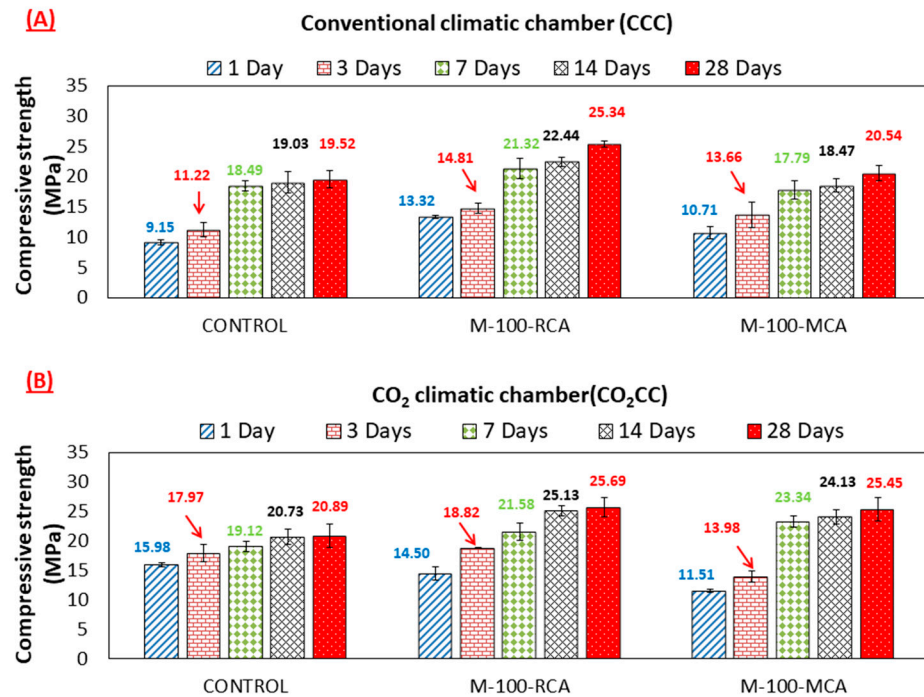


Figure 10. Compressive strength development of CONTROL, M-100-RCA and M-100-MCA under (A) CCC and (B) CO₂CC at the ages of 1, 3, 7, 14 and 28 days.

Curing with a CO₂ climatic chamber (CO₂CC) improved compressive strength at all ages, compared to the conventional climatic chamber (CCC). This result, which is very common, is recognised in most research [25,39,88] and is caused by a “densification” of the sample due to carbonation according to Equations (1)–(5) [21,89]. For the control mixture, 7 days of curing in CO₂CC was similar to 28 days of curing in CCC. For the M-100-RCA mixture, 14 days of curing in CO₂CC was similar to 28 days of curing in CCC. For the M-100-MCA mixture 7 days of curing in CO₂CC improved by 13.53% the compressive strength obtained for 28 days of curing in CCC. These results highlight the difficulty of making comparisons between CO₂CC and CCC curing for compressive strength purposes, as it depends on the nature of each mix. Several authors have already indicated that the mechanisms of natural and accelerated carbonation are different [23,90]. It indicates that the effect of curing in CO₂ after 14 days was insignificant, it being possible that from this age the samples were fully carbonated (perhaps it could be indicated for 7 days, especially for the control and M-100-MCA mixture). With the mixtures studied, it is not necessary to cure for up to 28 days in CO₂CC. Rather, 7 or 14 days are sufficient. The results obtained also indicated that CO₂CC can be used in a non-reinforced precast plant, increasing productivity and decreasing the curing time.

3.3. Dry Bulk Density and Accessible Porosity for Water

Figure 11 shows dry bulk density and accessible porosity for water at 28 days of curing for CCC and CO₂CC. The substitution of CG and FG for RCA and MCA decreased dry bulk density and increased accessible porosity for water for all the mixtures and under both curing environments. This was due to the lower dry bulk density and higher water absorption of RCA and MCA versus CG and FG (Table 1), mainly due to the cementitious mortar adhered to RCA and MCA surface having greater porosity than natural aggregates [91,92].

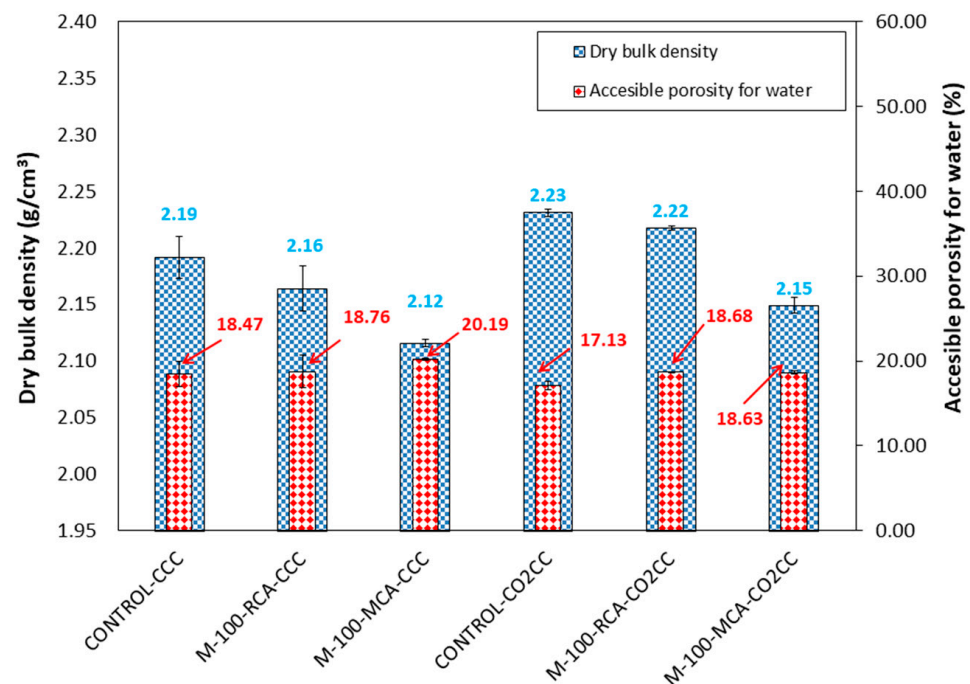
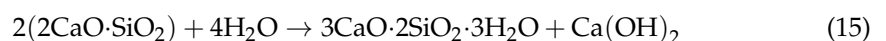
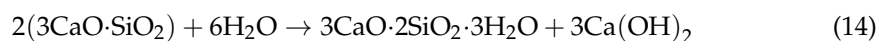


Figure 11. Dry bulk density and accessible porosity for water of control, M-100-RCA and M-100-MCA under CCC and CO₂CC at 28 days.

Curing in CO₂ (CO₂CC) increased the dry bulk density and decreased accessible porosity for water, which is in accordance with the improvement in compressive strength found (Figure 10) and was due to the pore-filling effect produced by the carbonation, in accordance with other studies [12,38,93,94].

3.4. X-ray Diffraction Analysis

Figure 12 shows the XRD results for the CONTROL mixture under the two curing environments (CCC and CO₂CC) at ages 7, 14 and 28 days. For the mixture, the control mixture at 7 days under CCC for the main phases was calcite (CaCO₃) (05-0586) [60] and dolomite (CaMg(CO₃)₂) (36-0426) [60]. These phases came from the aggregates used (CG, FG, S1 and S2). The portlandite (Ca(OH)₂) (44-1481) [60], ettringite (Ca₆Al₂(SO₄)₃(OH)₁₂·26 H₂O) (00-0059) [60] and calcium silicate hydrate (2CaSiO₃·3H₂O) (03-0556), also named C-S-H [60] phases, were the main hydration reactions [95–97]. The same phases were found at 14 and 28 days with no significant changes. Note that at 7 days, neither the alite phase (also sometimes called hatrurite) nor the belite phase were not found. In other investigations with similar mixtures [5,12,30], they were found, especially at 1 and 3 days of curing. This was the result of the alite having reacted almost completely at 7 days, together with gypsum, which was also not found, to form ettringite, as shown in Equation (13). It is also the result of the hydration of Portland cement, according to Equations (14) and (15) [98].



Following the above, this indicates that applying accelerated carbonation after 7 days of curing to the control mix will be less “efficient”, as Equations (1)–(5) would be reduced to Equations (3)–(5). This was in accordance with the slight improvements found after 7 days in the compressive strength for the control mix in Figure 10, under CO₂CC.

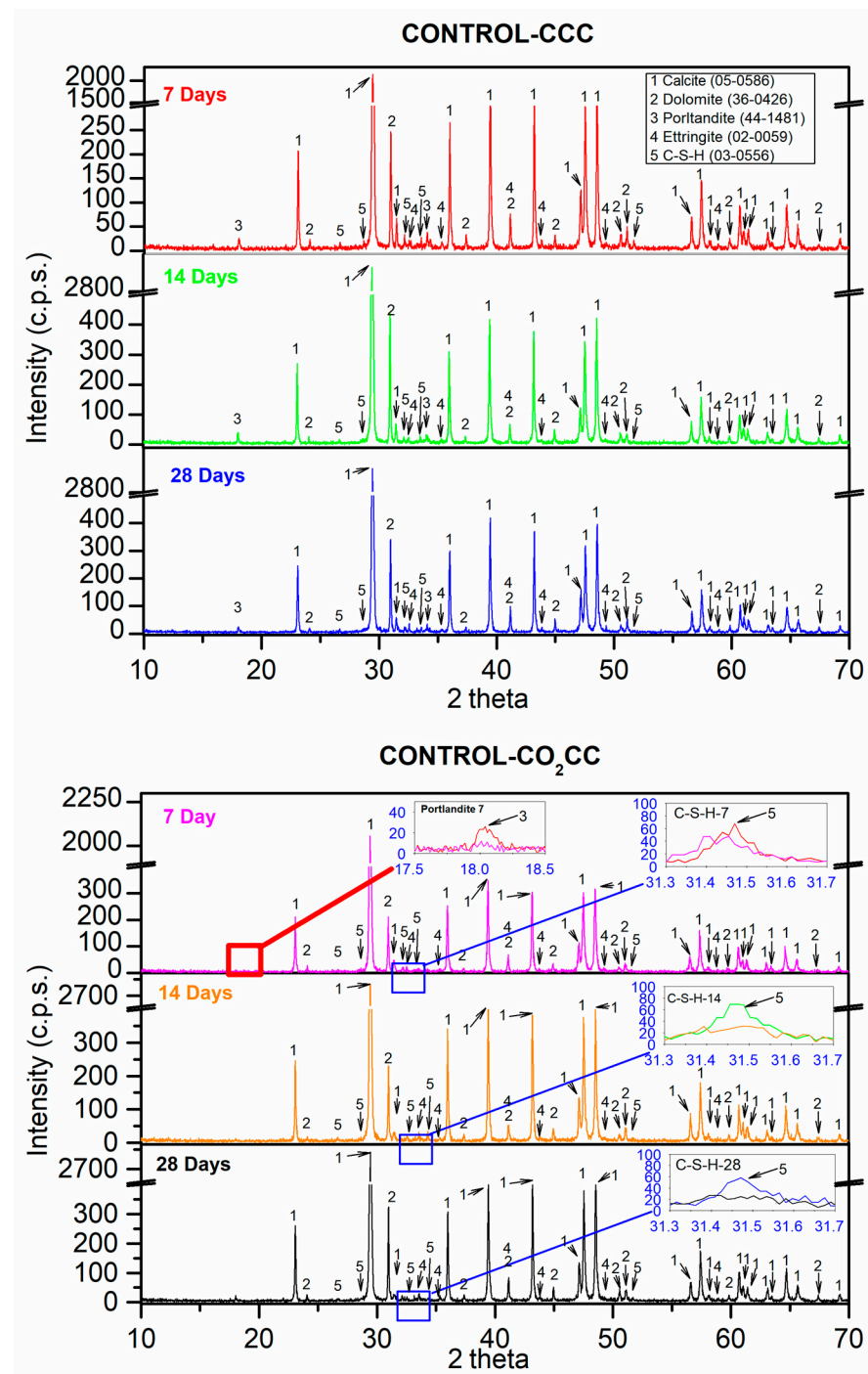


Figure 12. X-ray diffraction obtained for control under CCC and CO₂CC at 7, 14 and 28 days.

Basically, carbonation at ambient pressure primarily includes three steps: (1) diffusion of CO₂ producing CO₃²⁻; (2) dissolution of calcium-based phases, generating Ca²⁺ and (3) nucleation and precipitation of CaCO₃ according to Equations (1)–(5) [99,100]. Applied to the phases found, carbonation applied with CO₂CC should “consume” phases such as portlandite and C-S-H Equations (3)–(5). In fact, at 7 days, under CO₂CC it can be observed in the inset labelled “Portlandite 7” that the portlandite phase has practically disappeared (red line vs pink line). Additionally, a slight decrease in the C-S-H phase was observed when CO₂CC was used. This is indicated in the inset labelled “C-S-H-7” (red line vs pink line). The rest of the phases found were the same as those found under CCC, although perhaps with a little more intensity in the calcite phase, which is in accordance

with Equations (3)–(5). At 14 and 28 days, under CO₂CC, the portlandite phase was logically still absent. As for the C-S-H phase, the decrease produced by the contact of this phase with CO₂ is fulfilled, which can be observed in the inset “C-S-H 14” and “C-S-H 28” respectively for the age of 14 and 28 days.

Figure 13 shows the XRD results for the M-100-RCA mixture under the two curing environments (CCC and CO₂CC) at 7, 14 and 28 days. For the mixture M-100-RCA at 7 days under CCC, the main phases were calcite (CaCO₃) (05-0586) [60] and dolomite (CaMg(CO₃)₂) (36-0426) [60]. These phases came from S1 and S2 (Figure 6). The quartz (SiO₂) (05-0490) [60], coming from the use of RCA, also appeared as a main phase (Figure 7). As with the control mixture, the phases of portlandite (Ca(OH)₂) (44-1481) [60], ettringite (Ca₆Al₂(SO₄)₃(OH)₁₂·26 H₂O) (00-0059) [60] and calcium silicate hydrate (2CaSiO₃·3H₂O) (03-0556) [60] were found. No changes were found at 14 and 28 days cured in CCC. The absence of alite and belite that was found in other research of similar blends [5,12,30] is indicative that “it makes no sense” to apply carbonation at 7 days of curing.

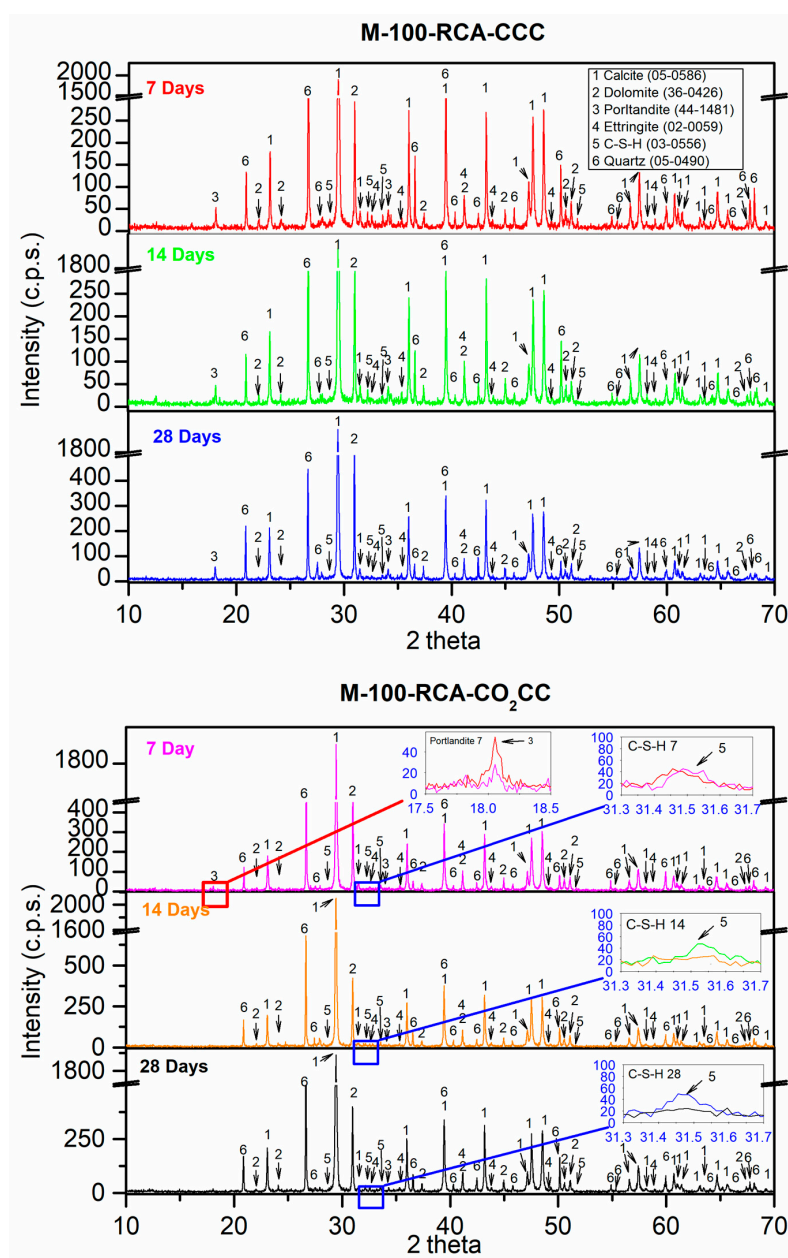


Figure 13. X-ray diffraction obtained for M-100-RCA under CCC and CO₂CC at 7, 14 and 28 days.

Under CO₂CC, at 7 days, it can be observed in the inset labelled “Portlandite 7” that this phase still exists, although with little intensity. For the control mixture, this phase did not exist. This may be due to the portlandite already present in the RCA itself (Figure 7). Therefore, this may be indicative that the use of RCA with the portlandite phase is a “CO₂ sink” according to Equation (3). Again, a small decrease in the C-S-H phase was also observed, indicated by the inset labelled “C-S-H 7” (red line vs. pink line). At 14 days, the disappearance of the portlandite phase was observed, which is in accordance with the fact that after 14 days the compressive strength remained approximately constant, and there was no significant increase (Figure 9). Logically, the same is true at 28 days of curing. The insets labelled “C-S-H 14” and “C-S-H 28” show the same as those obtained for the control mix.

Figure 14 shows the XRD results for the M-100-RCA mixture under the two curing environments (CCC and CO₂CC) at 7, 14 and 28 days. For the mixture M-100-MCA at 7 days under CCC, the main phases were calcite (CaCO₃) (05-0586) [60], dolomite (CaMg(CO₃)₂) (36-0426) [60] and quartz (SiO₂) (05-0490) [60]. Other minority phases were also found: portlandite (Ca(OH)₂) (44-1481) [60], ettringite (Ca₆Al₂(SO₄)₃(OH)₁₂·26 H₂O) (00-0059) [60] and calcium silicate hydrate (2CaSiO₃·3H₂O) (03-0556) [60]. These phases were also found in the control and M-100-MCA mixtures. Again, belite and alite phases were not found, which is again indicative that it is not necessary to carbonate this type of sample at over 7 days of curing.

Under CO₂CC, at 7 days it can be observed in the inset labelled “Portlandite 7” that the portlandite phase has practically disappeared (red line vs pink line). Also, a slight decrease in the C-S-H phase was observed when CO₂CC was used. This is indicated in the inset labelled “C-S-H-7” (red line vs pink line). At 14 and 28 days, under CO₂CC, the portlandite phase is logically still absent. As for the C-S-H phase, the decrease produced by the contact of this phase with CO₂ is fulfilled, which can be observed in the inset “C-S-H 14” and “C-S-H 28” respectively at 14 and 28 days.

3.5. Thermogravimetric Analysis and Differential Thermal Analysis

Figures 15–17 show thermogravimetric analysis (TGA) and differential thermal analysis (DTA) of all samples studied under CCC and CO₂CC. Table 5 shows the weight losses for the different stretches. This analysis can determine the CO₂ absorption produced through CO₂ curing [101,102] by just comparing the amount of calcium carbonate in the same mixture before and after curing in CO₂, as indicated in Equation (16). This can be done because the main product of carbonation is CaCO₃, as indicated in Equations (1)–(5).

$$\text{CO}_2 \text{ sequestered (wt.\%)} = \text{CaCO}_3 \text{ in CO}_2\text{CC} - \text{CaCO}_3 \text{ in CCC} \quad (16)$$

Several common stretches were found in all the samples studied [5,12,30]: (i) From room temperature to 105 °C, ambient humidity was lost. It was observed that this loss was higher in the M-100 RCA and M-100-MCA mix than in the control mix, both for CCC and CO₂CC, due to the higher water absorption of both RCA and MCA compared to CG and FG, as indicated in Table 1; (ii) From 105 to 400 °C, loss of ettringite and C-S-H detected by XRD occurred [103–105]. A slight decrease in weight loss was observed when comparing CCC versus CO₂CC, which was already detected by XRD (see insets named “CSH 7”, “CSH 14” and “CSH 28”) for all samples; (iii) From 400 to 460 °C, decomposition of portlandite, if present, occurred. Its existence was identified with an endothermic peak in DTA. Under CCC, portlandite existed in all samples, as demonstrated by XRD. However, under CO₂CC, it only appears in sample M-100-RCA at 7 days, which is in accordance with the XRD findings. The weight loss (Table 5) was lower in CO₂CC than in CCC, which is in accordance with the above; (iv) From 460 to 650 °C, the initial carbonates formed in the hardening process were lost [106] and from 650 to 1000 °C, the calcium carbonate was lost [82], which is in accordance with the remarkable weight loss found. In this study, we will consider the last two sections together.

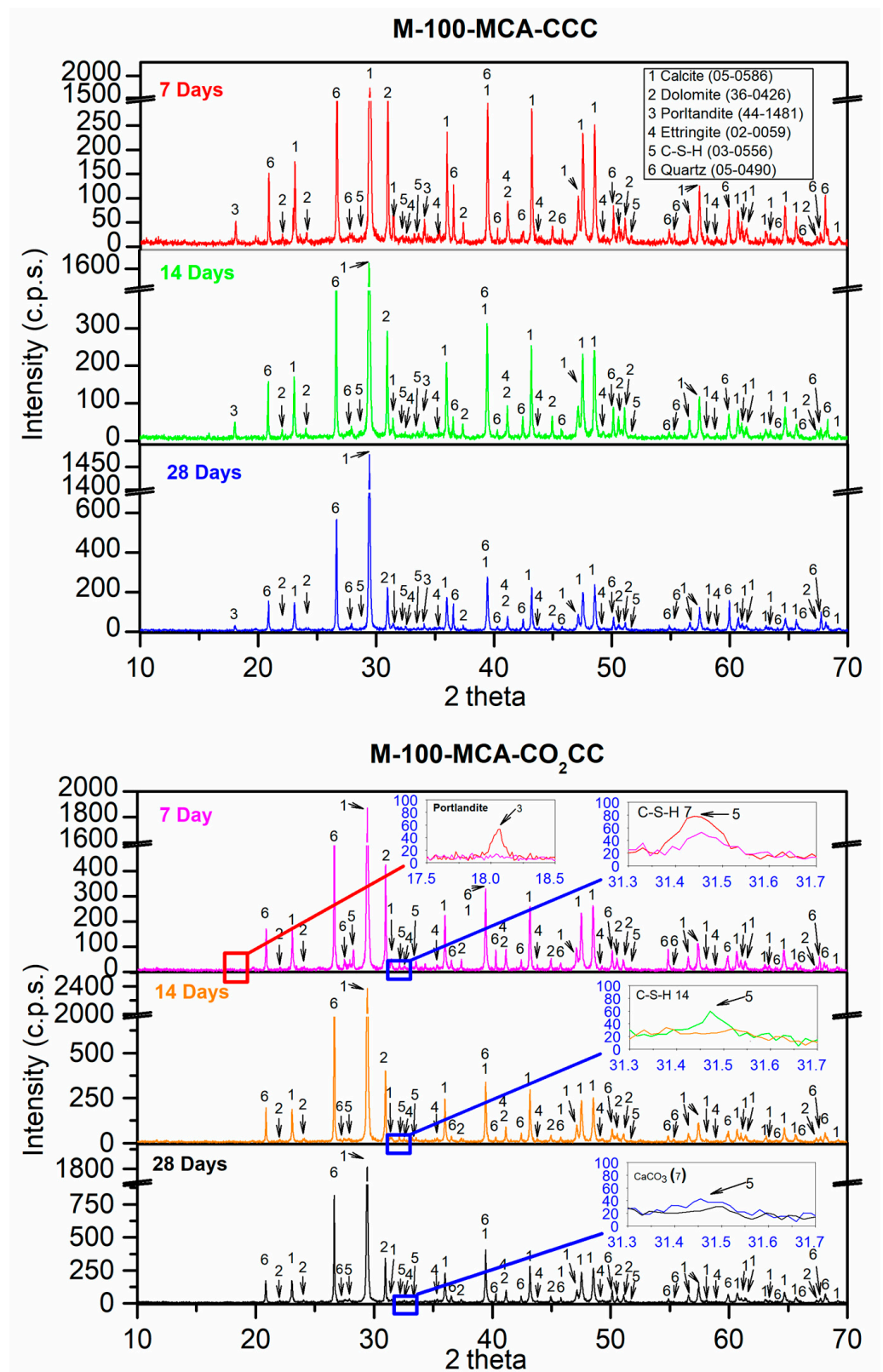


Figure 14. X-ray diffraction obtained for M-100-MCA under CCC and CO₂CC at 7, 14 and 28 days.

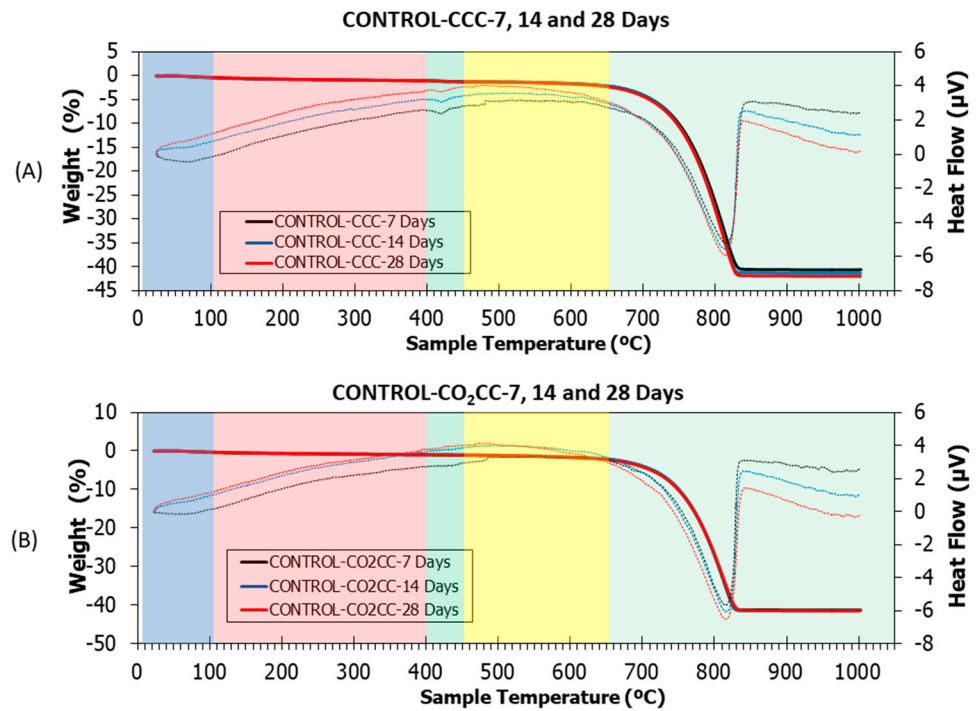


Figure 15. TGA (solid lines) and DTA (dotted lines) of control under (A) CCC and (B) CO₂CC at 7, 14 and 28 days.

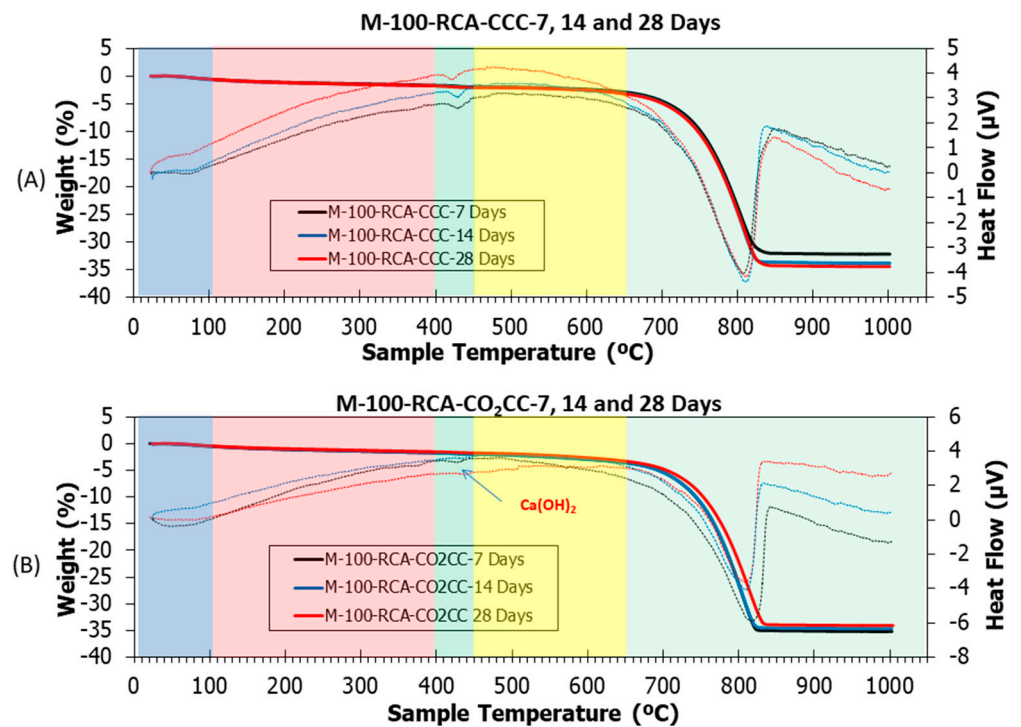


Figure 16. TGA (solid lines) and DTA (dotted lines) of M-100-RCA under (A) CCC and (B) CO₂CC at 7, 14 and 28 days.

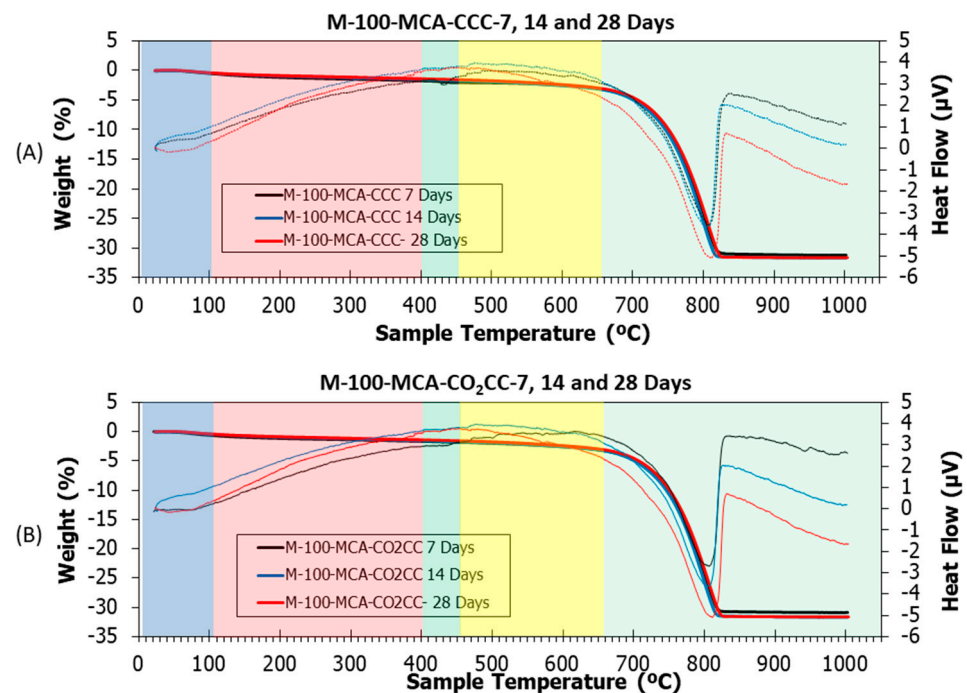


Figure 17. TGA (solid lines) and DTA (dotted lines) of M-100-MCA under (A) CCC and (B) CO₂CC at 7, 14 and 28 days.

In order to observe the effect of CO₂ curing, a comparison was made between CCC and CO₂CC for the same samples and curing ages (Table 5). Generically, it was observed that the capture capacity increases under the CO₂CC curing environment, which is indicative that CO₂ curing of these materials not only improves compressive strength, but also absorbs CO₂.

The increase for the control mix was 2.9, 3.6 and 3.6 kg/CO₂ t sample under CO₂CC. Two aspects are derived from the above: Firstly, it can be seen that using CO₂CC longer than 14 days does not make sense from the point of view of increasing CO₂ absorption. Secondly, it may appear as a low CO₂ uptake. However, considering that the average CO₂ level in the atmosphere is 400 ppm and, considering that the density of CO₂ is 1.8 mg/cm³ under normal conditions, the amount of CO₂ in 1 m³ of air is only 721.6 mg [107]. To reach the CO₂ level of the pre-industrial era (280 ppm), only absorbing 120 ppm would be sufficient, i.e., 216.48 mg per m³ of air. With 1 tonne of the control mixture, 13,604 m³ of air could be decarbonised after only 7 days of curing.

For the M-100-RCA mixture, again it can be seen that it does not make sense to cure in CO₂CC for more than 14 days. In this case, the CO₂ absorption is higher than for the control mix, and this is due to the portlandite phase found in the RCA aggregate (Figure 7). Therefore, with only 7 days of curing, the 1 tonne of M-100-RCA mix could decarbonise under a CO₂CC environment of about 36,077 m³ of air. It also does not make sense from a CO₂ absorption point of view to cure the M-100-MCA mix for more than 14 days. In this case, 7 days of curing of 1 tonne of M-100-MCA mix would decarbonise 24,685 m³ of air. Considering that a conventional cobblestone has dimensions of 20 × 10 × 6, and using the dry densities obtained in Figure 11 as an estimate, the following was obtained: 1 single paving stone could decarbonise 3.61, 9.59 and 6.56 m³ of air at pre-industrial levels with the control, M-100-RCA and M-100-MCA mixtures, respectively.

Furthermore, the increase in CO₂ sequestered per m³ has been calculated using the dry densities obtained in Figure 11. This will be used to calculate the carbon emission evaluation.

Table 5. Different weight losses for all the mixtures studied and increase in CO₂ sequestrated using CO₂CC.

Mixes	Δ Mass (%)					Δ Mass (450–1000 °C)	CO ₂ Sequestrated (wt.%) According to Equation (16)	Increase in CO ₂ Sequestrated (g/t)	Increase in CO ₂ Sequestrated (g/m ³)
	RT-105 °C	105–400 °C	400–450 °C	450–650 °C	650–1000 °C				
CONTROL-CCC- 7 Days	−0.465	−0.585	−0.230	−0.879	−38.615	−39.494			
CONTROL-CCC- 14 Days	−0.414	−0.609	−0.186	−0.927	−39.351	−40.278			
CONTROL-CCC- 28 Days	−0.366	−0.734	−0.186	−1.028	−39.775	−40.804			
CONTROL-CO ₂ CC- 7 Days	−0.412	−0.526	−0.155	−1.058	−38.730	−39.789	0.2945	2945.21	6567.83
CONTROL-CO ₂ CC- 14 Days	−0.320	−0.588	−0.096	−1.087	−39.559	−40.647	0.3693	3693.83	8237.24
CONTROL-CO ₂ CC- 28 Days	−0.326	−0.647	−0.009	−1.175	−39.995	−41.170	0.3663	3663.32	8169.12
M-100-RCA-CCC- 7 Days	−0.617	−1.086	−0.261	−0.955	−29.467	−30.422			
M-100-RCA-CCC- 14 Days	−0.698	−1.244	−0.282	−1.083	−30.912	−31.996			
M-100-RCA-CCC- 28 Days	−0.639	−1.164	−0.260	−1.144	−31.416	−32.561			
M-100-RCA-CO ₂ CC- 7 Days	−0.584	−1.081	−0.205	−1.665	−29.538	−31.203	0.7810	7810.94	17,340.28
M-100-RCA-CO ₂ CC- 14 Days	−0.526	−1.097	−0.198	−1.644	−31.197	−32.842	0.8457	8457.52	18,775.57
M-100-RCA-CO ₂ CC- 28 Days	−0.516	−1.051	−0.175	−1.593	−31.817	−33.410	0.8492	8492.99	18,854.44
M-100-MCA-CCC- 7 Days	−0.563	−1.186	−0.289	−1.007	−28.325	−29.332			
M-100-MCA-CCC- 14 Days	−0.444	−1.116	−0.229	−1.388	−28.663	−30.051			
M-100-MCA-CCC- 28 Days	−0.611	−1.022	−0.188	−1.120	−28.306	−29.427			
M-100-MCA-CO ₂ CC- 7 Days	−0.669	−0.987	−0.209	−1.317	−28.550	−29.867	0.5344	5344.98	11,491.71
M-100-MCA-CO ₂ CC- 14 Days	−0.490	−0.953	−0.160	−1.530	−29.296	−30.826	0.7705	7702.25	16,652.22
M-100-MCA-CO ₂ CC- 28 Days	−0.467	−0.945	−0.148	−1.440	−28.757	−30.197	0.7745	7745.27	16,560.97

Based on the authors' knowledge, no studies have been found that study the CO₂ capture capacity of concrete mixes such as the one presented. They were found for very porous mortars, using similar levels of carbonation, but only using 7 days of curing [12], using carbonated water as curing and/or mixing water [5,20]. In other research, although CO₂ is calculated, it is only estimated qualitatively [108]. This research fills this information gap.

3.6. Carbon Emission Evaluation

According to Tables 2, 3 and 5, as well as Equations (8) and (9), the carbon emissions of each of the mixtures with different materials and curing were assessed and are presented in Table 6. Table 7 shows the results obtained for the incremental capture per m³ calculated through TGA/DTA (Table 5) with the respective carbon emissions calculated in Table 6.

Table 6. CO₂ emissions for materials and Curing.

Materials	CO ₂ Emission Control (kg CO ₂ eq/m ³)	CO ₂ Emission M-100-RCA (kg CO ₂ eq/m ³)	CO ₂ Emission M-100-MCA (kg CO ₂ eq/m ³)
Cement	210.420	210.420	210.42
Superplasticizer	0.575	0.575	0.575
Water	0.046	0.059	0.063
Coarse gravel	0.820	-	-
Fine gravel	6.909	-	-
Sand 1	0.558	0.558	0.558
Sand 2	3.852	3.852	3.852
Recycled concrete aggregate	-	1.223	-
Mixed concrete aggregate	-	-	1.038
CO ₂ emitted materials	223.180	216.687	216.506
Curing	7 Days (kg CO ₂ eq/m ³)	14 Days (kg CO ₂ eq/m ³)	28 Days (kg CO ₂ eq/m ³)
Conventional Chamber (0.15 kW/h)	5.04	10.08	20.16
CO ₂ Chamber (0.15 kW/h)	5.04	10.08	20.16

Table 7. Summary of total CO₂ emissions by mixtures and CO₂ sequestration.

	Total CO ₂ Emissions (kg CO ₂ eq/m ³)	Total CO ₂ Emissions—CO ₂ Sequestered (kg CO ₂ eq/m ³)
CONTROL-CCC- 7 Days	228.22	228.22
CONTROL-CCC- 14 Days	233.26	233.26
CONTROL-CCC- 28 Days	243.34	243.34
CONTROL-CO ₂ CC- 7 Days	228.22	221.66
CONTROL- CO ₂ CC- 14 Days	233.26	225.02
CONTROL- CO ₂ CC- 28 Days	243.34	235.17
M-100-RCA-CCC- 7 Days	221.72	221.72
M-100-RCA-CCC- 14 Days	226.76	226.76
M-100-RCA-CCC- 28 Days	236.84	236.84
M-100-RCA-CO ₂ CC- 7 Days	221.72	204.38
M-100-RCA-CO ₂ CC- 14 Days	226.76	207.99
M-100-RCA-CO ₂ CC- 28 Days	236.84	217.99
M-100-MCA-CCC- 7 Days	221.54	221.54
M-100-MCA-CCC- 14 Days	226.58	226.58
M-100-MCA-CCC- 28 Days	236.66	236.66
M-100-MCA-CO ₂ CC- 7 Days	221.54	210.05
M-100-MCA-CO ₂ CC- 14 Days	226.58	209.93
M-100-MCA-CO ₂ CC- 28 Days	236.66	220.10

CO₂ emitted materials amounted to 223.18, 216.68 and 216.50 kg CO₂ eq/m³ respectively for the control, M-100-RCA and M-100-MCA mixtures, respectively. This result is slightly below the range shown by W. Xing et al. [49] (278.35–524.44 kg CO₂ eq/m³) in a review where they studied the environmental impact of 57 concrete products. These differences are caused by the effect of substituting fine or coarse aggregate [109,110], the recycling process [111,112] and the source and quality of primary material [113], among others. Furthermore, this result showed the feasibility of replacing CG and FG (natural aggregates) with RCA and MCA (recycled aggregates from construction and demolition waste).

However, it was observed that CO₂ emissions for curing for 14 and 28 days were quite high (at least with the equipment used at the laboratory scale). In fact, for the control mix, CO₂ emissions for curing at 14 and 28 days (10.08 and 20.16 kg CO₂ eq/m³ shown in Table 6), are higher than the increase in CO₂ sequestered (8.23 and 8.16 kg CO₂ sequestered by m³ shown in Table 5). This again shows that, for the control mix it, does not make sense to cure more than 7 days in CO₂CC. Using the same comparison, for the M-100-RCA and M-100-MCA mixtures, the CO₂ emissions for curing would allow curing in CO₂CC for up to 14 days. It is therefore not feasible to cure for 28 days in CO₂CC for any of the mixtures studied.

Therefore, a CO₂CC cure of up to 7 days (or even less) was sufficient to use CO₂ curing as a tool to increase productivity, improve compressive strength and decrease CO₂ emission in an unreinforced precast plant. It is not recommended to use more than 7 days in CO₂ curing, as compressive strengths were maintained and only CO₂ emissions were increased. No studies were found that relate the CO₂ sequestered by similar mixtures and that also make an assessment of CO₂ emissions, relating both results.

The mixture with the lowest total CO₂ emissions was “M-100-RCA-CO₂CC- 7 Days”. According to the strengths obtained in Figure 10, it could be classified as C16/20 “ordinary concrete” only at 7 days [57]. Therefore, “M-100-RCA-CO₂CC- 7 Days” presents a very promising result to be tested on a real scale, such as paving stones, kerbs or any non-structural precast.

4. Conclusions

CO₂ curing in vibro-compacted precast concrete with recycled/mixed concrete aggregates (RCA or MCA) is a promising technology. The CO₂ curing enabled carbon sequestration, improved the compressive strength, increased the dry bulk density and decreased the accessible porosity for water. The findings are detailed in the following points:

- The portlandite phase found in RCA and MCA by XRD is a “potential” CO₂ sink;
- The method of replacing natural aggregate with RCA and MCA should be carried out with very similar particle sizes. This even improves the compressive strengths obtained;
- Curing in CO₂ improved the compressive strength in all samples (CONTROL, M-100-RCA and M-100-MCA). It does not make sense to apply CO₂ curing longer than 7 days on the mixes with natural aggregate and MCA, as the strengths remained constant. A CO₂ curing of 14 days can be applied to the RCA mixes;
- XRD and TGA/DTA showed that it does not make sense to apply CO₂ curing beyond 7 days, since from that age all the blends were practically carbonated (except the blend with RCA, which did not carbonate until 14 days);
- The mixtures of 1 tonne of control, M-100-RCA and M-100-MCA using CO₂ curing could be decarbonised after only 7 days of curing 13,604, 36,077 and 24,635 m³ of air, respectively;
- According to the carbon emission evaluation and the TGA/DTA results, curing longer than 7 days in CO₂ for the reference mix (CONTROL) had higher CO₂ emissions than the sequestered CO₂. The mix with RCA and MCA would allow up to 14 days, but according to the compressive strength obtained; XRD and TGA/DTA results, only up to 7 days is recommended;
- The total CO₂ emissions by mixture using CO₂ curing at 7 days were 221.26, 204.38 and 210.05 kg CO₂ eq/m³ for CONTROL, M-100-RCA and M-100-MCA, respectively.

This was calculated with the carbon footprint assessment and the CO₂ sequestered obtained with TGA/DTA.

In conclusion, the findings of this study provide a valuable contribution to carbon emission evaluation of CO₂ curing in vibro-compacted precast concrete with recycled/mixed concrete aggregates (RCA or MCA). The new approach facilitates carbon capture and use and guarantees enhanced compressive strength of the concrete samples.

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