Statistical analysis of a database collecting properties of recycled then carbonated concrete aggregates based on parametric studies

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Abstract

In the context of carbon capture by accelerated carbonation of recycled concrete aggregate (RCA), the FastCarb project proposed a data collection from the literature and experimental campaigns which can be processed for statistical analysis depending on desired parameters such as RCA properties. Parametric statistical analysis of absorption, $CaCO_3$ content, and mass gain measurements are presented here. Considering a specific RCA carbonation protocol established during FastCarb project, an experimental campaign was carried out to different gradings RCA. Compared to RCA properties, CRCA absorption and its range value was reduced, $CaCO_3$ content increased, its range value was reduced. A mass gain was systematically obtained. It was more difficult to establish correlations between properties that are independent of the parameters.

1 Introduction

In the current context of global limits, a key objective is to optimise the reuse of rubble from demolishing old structures in new structural elements. Due to the specific properties of recycled concrete aggregates (RCA), a greater number of tests need to be carried out before they can be considered for reuse [1-2]. In addition, the variability of RCA properties may act as a barrier to their widespread use, as each RCA sample requires a suitable formulation [3].

Accelerated carbonation can improve their properties, which also enables the capture of carbon dioxide (CO_2) from the atmosphere or from a particular industrial process [4]. Most studies on accelerated carbonation of RCA aim at optimising the carbonation process parameters [5]. The aim is to enhance CO_2 capture while improving recycled aggregates properties [6-7].

However, the performance of different accelerated carbonation treatments depends on many parameters [8-9] : process parameters such as the time required for treatment, the total pressure and relative humidity in the chamber, the CO_2 partial pressure, the gas flow rate through the chamber, and the chamber rotation speed [10], *etc*. In addition, the effectiveness of carbonation methods is strongly influenced by the inherent properties of the recycled aggregates to be treated and their conditioning prior to carbonation [11-12]. These inherent properties are the state of initial carbonation, the degree of saturation, the grain size, the characteristics of the parent material, including the nature of the cement and the nature of the aggregates, *etc*.

Given the large number of factors involved, there is no consensus in the literature regarding the effectiveness of the various techniques, which then leads to a multifactorial influence of accelerated carbonation on the properties of carbonated RCA.

Thus, in this context of carbon capture by accelerated carbonation of recycled concrete aggregate (RCA), the FastCarb French national project proposed a data collection with the aim of comparing the properties of RCA and carbonated RCA (CRCA). Sourcing of results came from literature and from specific experimental campaigns carried out by FastCarb partners [13].

The analysis of these data, with a view to the wider use of RCA in structural concrete, aims to answer the following three questions: (i) Can trends in the influence of accelerated carbonation on

RCA properties be identified independent of material and protocol parameters? (ii) Can correlations between RCA and CRCA properties and accelerated carbonation be quantified? (iii) Does accelerated carbonation reduce variability in RCA properties? Thus, in this study, we do not intend to analyze the database to identify effects related to carbonation parameters, nor to identify effects related to initial material parameters (RCA).

The collected data presented in this study focused on absorption, $CaCO_3$ content and mass gain measurements. For one of the experimental campaigns conducted in laboratory, an analysis of RCA and CRCA properties with a focus on the evolution of their variability (properties standard deviation) during the carbonation process was carried out.

2 Database development and content

The database was developed using Excel© and the associated programming language Visual Basic for Applications (VBA©). The database was completed following scheme described Fig. 1. It also contains information on crush method and parent concrete, (initial material).

All the results from the literature (82 experiments) and experimental campaigns (69 experiments) have been compiled in this database which can be processed for statistical analysis depending on the desired parameters.

A pivot table in Excel© can display the contents of the experimental and bibliographical input data (before carbonation) and output data (after carbonation).

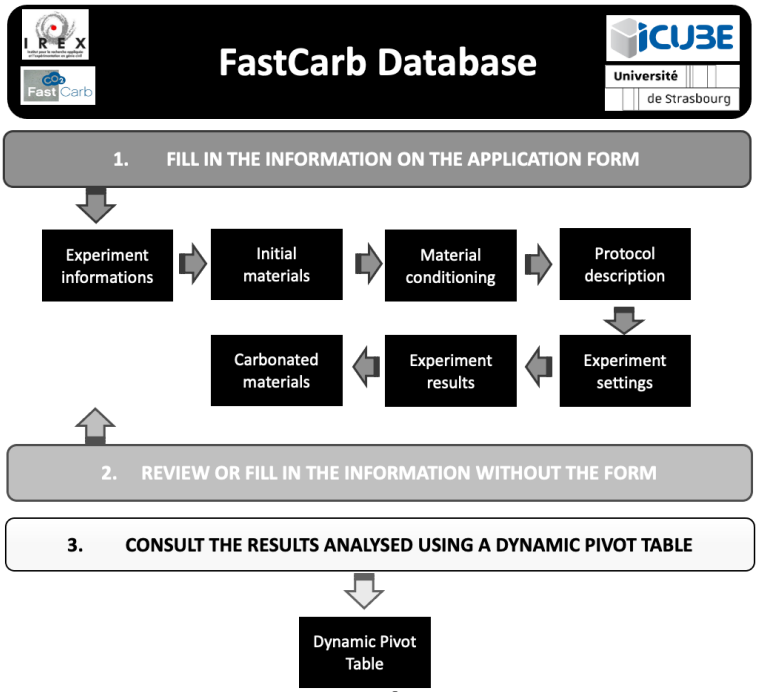

Fig. 1 Operation of the database created in Excel©.

2.1 Experimental and bibliographical input and output data content TCD

2.1.1 RCA parameters and tests

The FastCarb database built for this study includes 10 sources (studies and literature) *i.e.*151 lines of data, each corresponding to one RCA coupled with one carbonation condition. 5 studies from the FastCarb National Project and 5 studies from published sources represent 25 concretes and 40 RCA:16 concretes and 28 RCA from FastCarb, 9 concretes and 12 RCA from the bibliography. Of the 40 RCA: 33 are from laboratory concretes, 4 from recycling platforms 3 from worksites and worksite returns; 31 are from 19 known parent concretes (but 2 whose composition is not detailed), 9

from 6 unknown parent concretes. For known concretes, strength classes range from C20/25 to C80/95, with water/cement mass ratios varying from 0.32 to 0.68. The aggregates used in the parent concretes are either siliceous (4 concretes), calcareous (2 concretes) or silico-calcareous (6 concretes).

The RCA (including those in the FastCarb database), for which the composition of the parent concrete is known (manufactured in the laboratory), are formulated with different types of cement: 6 with CEM I cement (NF or EN – 280 to 485 kg/m^3); 4 CEM II cement (NF or EN – 280 to 350 kg/m^3); 3 with CEM III (NF or EN – 280 to 350 kg/m^3); 4 with Type I cement (ASTM – 300 to 485 kg/m^3).

The concretes studied were crushed into RCA covering 17 granular classes with grain sizes ranging from 0mm to 20mm.

The RCA obtained were characterized prior to carbonation. These tests mainly concerned properties likely to be modified by the carbonation stage, such as absorption, true density, porosity and calcium carbonate content. However, it should be noted that not all the works cited include all these characterizations.

The absorption of all 40 RCA in the database was characterized, covering an absorption range from 3.6% to 16.44%. Fig. 2 presents the distribution of the absorption for the RCA of which absorptions were measured before and after carbonation. Each vertical line of dots corresponds to just one initial RCA submitted to several carbonation protocols.

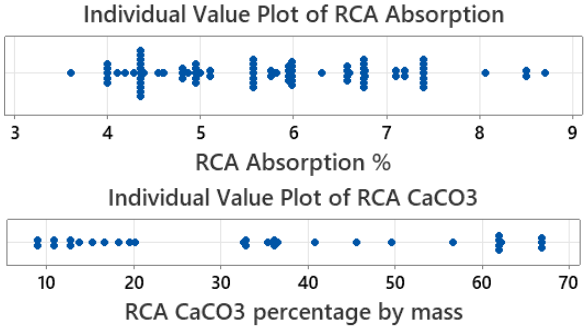

Fig. 2 Absorption distribution and $CaCO_3$ content of the RCA form the data base

True density was measured for only 20 RCA from 2.15 kg/m^3 to 2.653 kg/m^3. Water porosity was measured on 16 RCA (40%) and ranged from 8.6% to 15.9%. The initial CaCO3 content of the RCA was measured for 25 (63%) of them, ranging from 8.64% to 66.8%. One laboratory carried out dry attrition resistance tests on RCA on 4 granular classes of the same parent laboratory concrete with MDE coefficients ranging from 5.75% to 11.88%.

2.1.2 Carbonation protocol settings

All these studies represent 45 different carbonation protocols. The parameters that differentiate the protocols are mainly their static or dynamic nature, the duration and temperature of the treatment, the rate of CO_2 and the relative humidity of the carbonation environment, and the degree of water saturation of the materials treated.

The mass of samples treated is only specified for 11 protocols for values ranging from 100 g to 2550 g per sample and for a total mass of samples treated simultaneously of up to 12 kg.

The protocols referenced differ in terms of their static or dynamic nature: 2 protocols are carried out with a dynamic gas flow, while 32 are carried out in rotating drums with speeds of 20rpm to 100rpm.

Treatment temperatures were specified for 37 protocols and varied from 20°C to 50°C, with 20°C (10 protocols) and 22°C (16 protocols) being the most common.

These carbonation protocols all used CO_2. However, its concentrations in the carbonation environment, specified for 43 protocols, varied from one trial to another, ranging from 0.03% to 100%, the two most frequently used concentrations being 100% (12 protocols) and 15% (10 protocols).

Relative humidity was only specified for 19 of the 45 protocols cited: 6 protocols used a relative humidity of 50%, 11 of 60%, 1 of 70% and for one protocol, it was only specified that the relative humidity was less than 5%.

Of all the experiments carried out, 144 specified the duration of carbonation, which ranged from 0.5 h to 24 h. The most frequently used durations were 7 h (36 trials - 17 protocols) and 24 h (90 trials - 15 protocols).

The preparation of the materials to be carbonated corresponds to a known or imposed initial water content. For the 36 protocols for which it is specified, the initial water content of the materials to be treated varies from 0% to 12%.

2.1.3 Carbonated RCA parameters and tests

At the end of the carbonation stage, the treated materials were again characterised to quantify changes in their properties following treatment.

Only one study carried out attrition tests (Micro-Deval test - MDE) to assess the retention of the granular skeleton of RCA potentially usable as concrete aggregate (8 values). The results presented show that the range of MDE coefficients for treated materials varies from 4.62% to 20%.

The absorption of carbonated RCA was measured for 92 test lines (87 of which were validated), some of which were carried out before separation of the granular classes, thus giving rise to repetitions on the statistical graphs characterized by the vertical lines in Fig. 3.

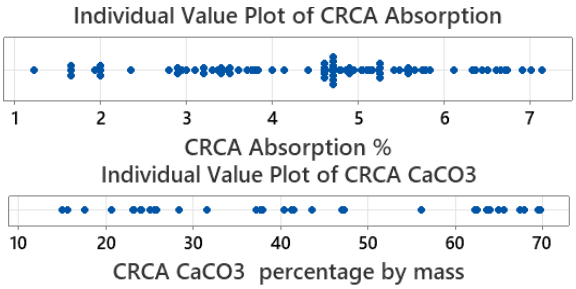

Fig. 3 Absorption distribution and $CaCO_3$ content of the CRCA form the data base

These tests cover an absorption range of 1.23% to 7.17%. The true density was measured for 25 lines, with values ranging from 2.2kg/m^3 to 2.67kg/m^3. Water porosity was measured for 34 lines and ranged from 3.66% to 14.7%. The $CaCO_3$ content of treated RCA, measured for 34 test lines by TGA (17 tests) and calcimetry (17 tests), ranged from 7.86% to 69.7%.

2.2 Database statistical analysis

Statistical data analysis was performed using Minitab©. The following statistical variables and their graphical representations are compared between the RCA and the CRCA on the basis of the same sampling (depending of test): mean, median, Q1, Q3, minimum, maximum, standard error of the mean, standard deviation represented by histograms with normal curve and Boxplots. This statistical comparison is shown for absorption (w) and calcite mass content ($CaCO_3$). The existence of correlations between variations (as a result of accelerated carbonation) in calcite content, mass (Δm) and absorption (w) is studied. The same variations are also correlated with the initial characteristics of RCA.

3 Accelerated carbonation material and method - one laboratory campaign

Considering the carbonation protocol established during the FastCarb project, an experimental campaign was carried out to different gradings RCA produced from one concrete of the same composition: 4 RCA from 1 concrete in the previous database

3.1 Materials

The concrete used in this study was produced in 2019 using a standard concrete mix design. The cement used is CEM II/B-M(S-LL) 32.5R CE CP1. The aggregates are 0/4 mm sand and 4/16 mm

rolled siliceous gravel from the Moselle region. No admixture was used. Table 1 presents the formulation of this concrete. The specimens made from these concretes are 16x32cm cylinders. Until they were crushed in March 2020, the samples were stored at ambient temperature and humidity, protected from carbonation, in carton moulds with covers. Crushing the samples produced 5/8, 8/10, 10/14 mm and 14/16 mm grading. After crushing, the aggregates were kept dry in big bags in the laboratory, then dried in an oven at 60°C to avoid mineralogical degradation of the material until taken for the various tests. Absorption and density were measured following EN 1097-6 expected that immersion time was 48h.

Table 1 Concrete formulation

	[kg/m^3]
Cement C	340
Efficient water WEff	205
0/4 mm sand S	750
4/16 mm gravel G	973

Table 2 RCA properties

class (mm)	water absorption w_{48} (%)	SD Pearson (%)	Oven-Dried particle density ρ_{rd} (%)	SD Pearson (%)
5/8	7.40	0.26	2.22	0.02
8/10	6.75	0.34	2.25	0.01
10/14	5.57	0.26	2.28	0.01
14/16	4.95	0.97	2.29	0.05

3.2 Carbonation protocol

A water content of 75% of absorption was adopted for the conditioning of the RCA. This water content was applied after the RCA had been completely dried out.

RCA were carbonated for 24 h in controlled chambers at 15% CO_2, atmospheric pressure, initial relative humidity of 60% and temperature of 20°C and 50°C respectively. The total amount of material per carbonation is 3kg divided into batches of 500-750g. The chamber volume was 260l.

The water absorption before and after carbonation was measured on dried aggregates with 4 or 5 repetitions for each granular class.

4 Statistical processing of RCA and CRAC characteristics

4.1 Analysis of the database

4.1.1 Statistical variables evolution

Accelerated carbonation of RCA reduces their absorption coefficient by 1.19%, representing an average relative reduction of 22% compared to the initial value of RCA. The data shown in Table 3 is calculated for all 87 experiments (lines of data) for which the absorption of RCA and CRCA is given. Analysis of this table, illustrated in Fig. 4, also provides an indication of how the distribution of data is evolving: the data are more uniformly distributed around the mean value: shape of the curve, value of ‘Q3-Q1’ and values of the mean and median closer together. The standard deviation remains similar. The global range of values is shifted towards lower values but increased, in part because of a significant reduction in the absorption value for some specimens.

Table 3 RCA and CRCA statistical values

Variable	Unit	N	Mean	SE Mean	St Dev	Min.	Q1	Median	Q3	Max.
w_{RCA}	[%]	87	5.70	0.13	1.25	3.60	4.39	5.57	6.75	8.70
w_{CRCA}	[%]	87	4.51	0.15	1.42	1.23	3.41	4.7	5.41	7.13
Δw	[%]	87	1.19	0.09	0.80	0.04	0.52	1.17	1.60	3.50
$\Delta w/w_{RCA}$	[%]	87	21.74	1.74	16.27	0.81	9.34	18.81	27.5	71.72
$CaCO_3$ RCA	[%] p. mass	34	35.33	3.62	21.08	8.94	13.47	34.1	61.9	66.8
$CaCO_3$ CRCA	[%] p. mass	34	41.38	3.19	18.60	14.94	24.01	39.15	62.78	69.7
$\Delta CaCO_3$	[%]	34	6.05	1.01	5.88	-9.46	1.83	4.9	10.93	16.56

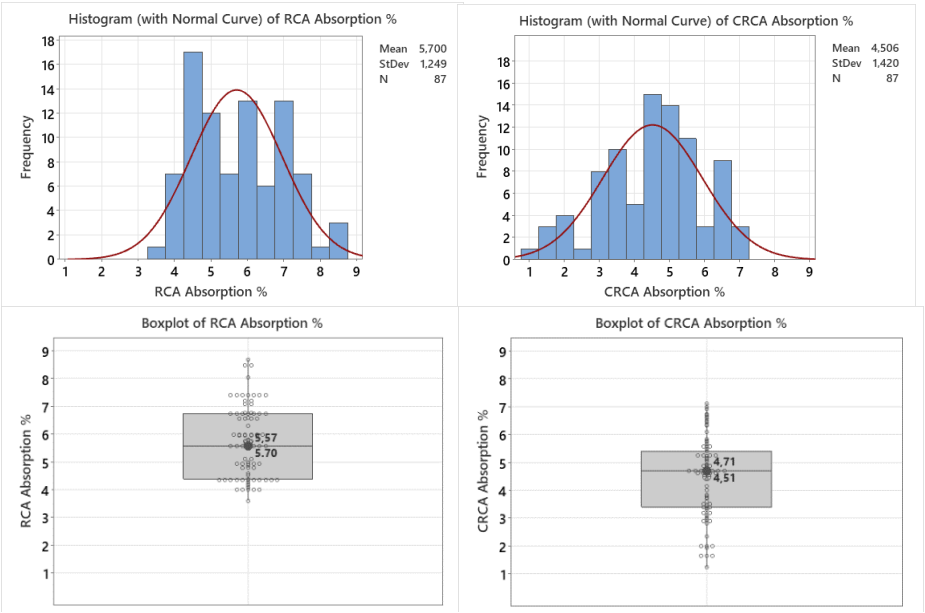

Fig. 4 Water absorption (w) statistical variables for RCA and CRCA

Accelerated carbonation of RCA leads to capture CO_2 represented by a $CaCO_3$ content per mass increased by 6.05% in average (see Table 3), *i.e.* 21.18 g of CO_2 captured by kg of RCA. As previously, the data shown in Table 3 is calculated for all 34 experiments (lines of data) for which the $CaCO_3$ content of RCA and CRCA is given. Concerning the evolution of the distribution of the data, Fig. 5 and Table 3 show not only that the range of data has been reduced, but also that the values are more centered around the mean. However, the range of variation remains large and not well distributed as normal plot, this is due to the different nature of the parent aggregates (calcareous, silico-calcareous).

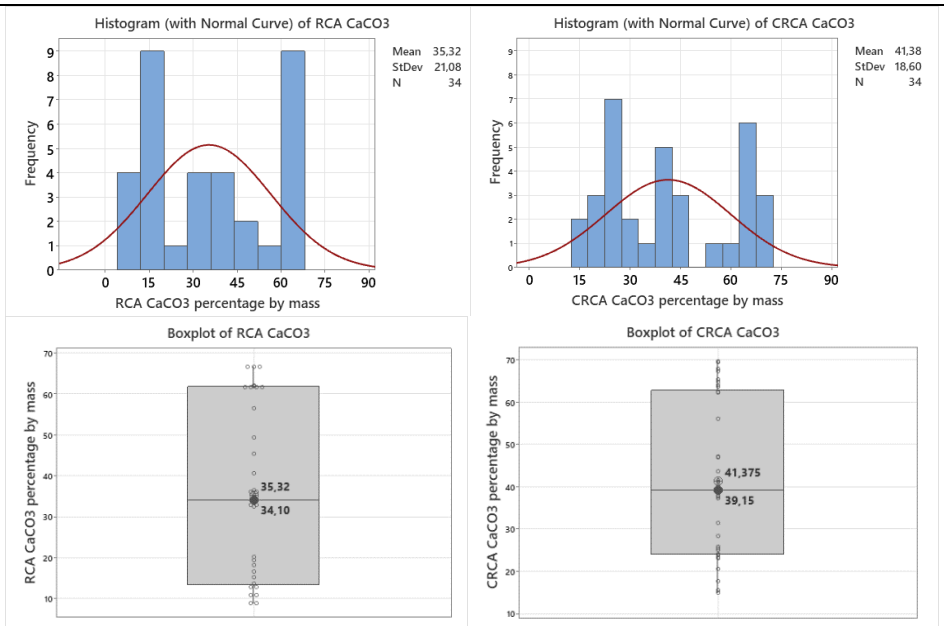

Fig. 5 $CaCO_3$ content per mass (%) statistical variables for RCA and CRCA

4.1.2 Correlations between variations of properties due to accelerated carbonation

In the aim to answer to the question "Can correlations between RCA and CRCA properties and accelerated carbonation be quantified independent of material and protocol parameters?", potential correlations were investigated between the following results:

- $\Delta w/w_{RCA}$ versus w_{RCA} and RCA $CaCO_3$ content
- $\Delta CaCO_3$ content versus w_{RCA} and RCA $CaCO_3$ content
- relative Δ_m versus w_{RCA} and $CaCO_3$ content
- $\Delta w/w_{RCA}$ versus $\Delta CaCO_3$ content
- $\Delta w/w_{RCA}$ versus relative Δ_m
- $\Delta CaCO_3$ versus relative Δ_m

Only some of these comparisons are presented Fig. 6 and Fig. 7. Fig. 6 presents not only the correlations between RCA and CRCA absorption or $CaCO_3$ content but also the correlations with the evolution of absorption or the evolution of $CaCO_3$ content respectively. In the figures on the right, the size of the bubbles represents the variation in the property being studied: the larger the bubble, the greater the variation. The line indicates no change in the property studied by carbonation. As the size of the bubbles is not correlated with any range of the x-axis, this indicates that there is no significant correlation independent of the other parameters. Scatter plot graphs on the left of Fig. 6 confirm that.

Due to the wide variation in input parameters (composition of the parent concrete, granularity of the RCA, initial absorption and initial carbonation of the RCA, etc.) and accelerated carbonation protocols, no significant independent correlation of these parameters can be reliably established. Only the comparison between the mass gain (Δm) during the test (relative to the initial dry mass) and the variation in $CaCO_3$ content seems to indicate a correlation, but this is not clear as the R^2 regression fit line is equal to 36.2% (see Fig. 7 at the top left). R^2 are equal to 55.6% and 41% for mass gain vs delta absorption (see Fig. 7 at the top right) and delta absorption versus delta $CaCO_3$ content (see Fig. 7 at the bottom left) respectively.

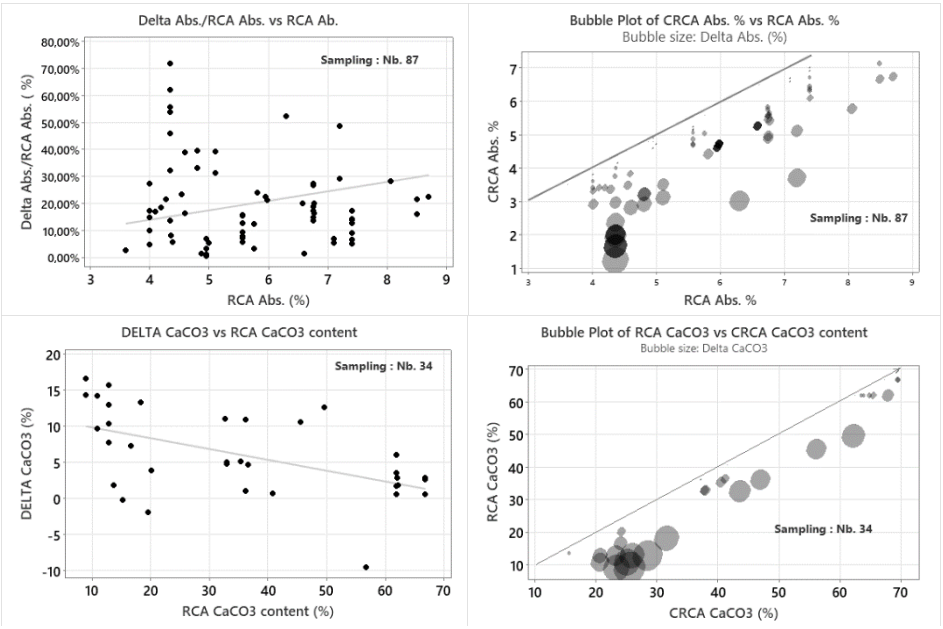

Fig. 6 Comparisons of RCA, CRCA properties and their variations

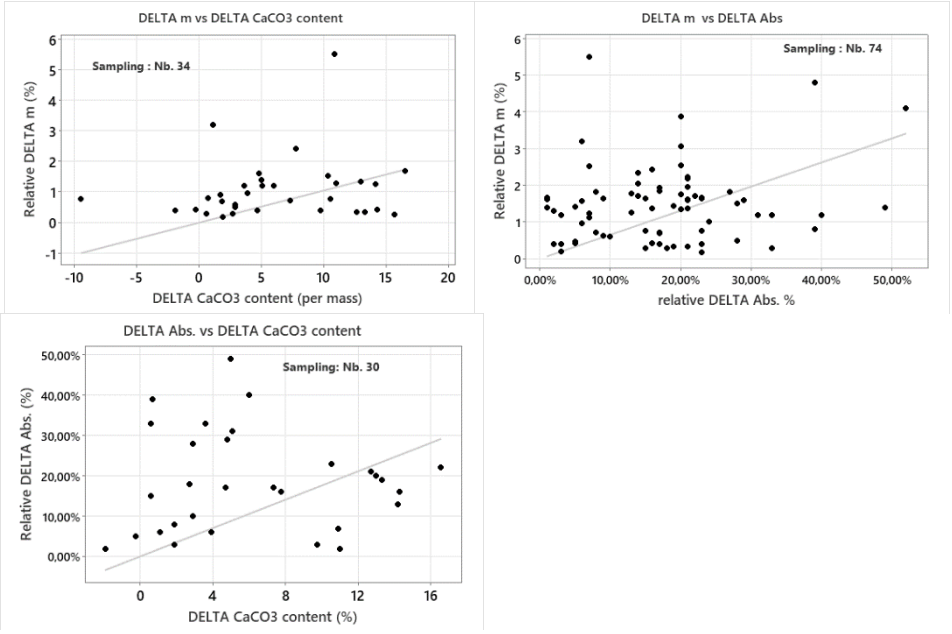

Fig. 7 Comparisons of RCA to CRCA properties variations

To see whether the correlations would be better if the results were filtered, similar representations were made on samplings reduced to the following carbonation parameters: 15% CO_2 for 24h. Comparison between the mass gain during the test (relative to the initial dry mass) and the variation in $CaCO_3$ content presented for CEMI cement and CEMII/CEMIII cement respectively on Fig. 8 indicate that with CEMI correlations is less significant (R^2 = 35%) than correlation with CEMII & III (R^2 = 58.4%). This confirms the strong influence of the nature of the cement of the parent concrete

and the difficulty of establishing a universal protocol when the RCA received come from an unknown source.

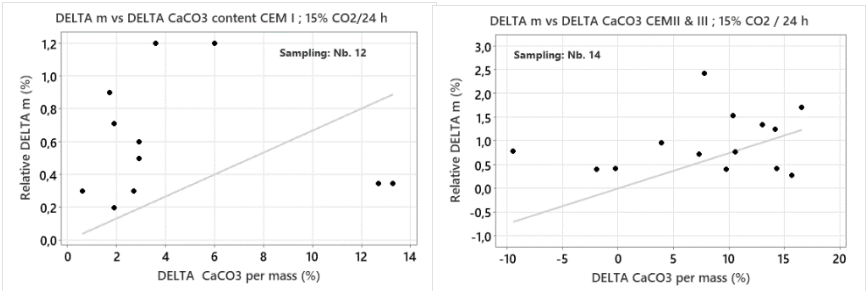

Fig. 8 Comparisons of RCA to CRCA properties variations - 15% CO_2 for 24h.

The aim of this first analysis of a large database was to find out if trends in the effect of accelerated carbonation on RCA properties could be seen independently of material and protocol parameters, if there were correlations between RCA and CRCA properties and accelerated carbonation that could be measured, and if accelerated carbonation leads to a reduction in the variability in RCA properties. For this reason, in this analysis, effects related to carbonation parameters or to initial material parameters (RCA) are not highlighted. However, the results of the experimental study that follows allow us to partially decouple the interdependent effects of materials and carbonation protocol, since the same parent concrete was used to produce the RCAs and the same carbonation protocol was used at two different temperatures.

4.2 Evolution of the properties and variability of laboratory RCA

To go further in the study of the influence of accelerated carbonation on RCA properties and on their variability, the study now focuses on RCA of different grades produced in the laboratory from a single concrete.

Results presented confirm the influence of accelerated carbonation on water absorption decreasing and also provide information on the influence of the granular class: the absorption reduction is more pronounced for smaller classes. Temperature does not seem to influence the improvement of this characteristic.

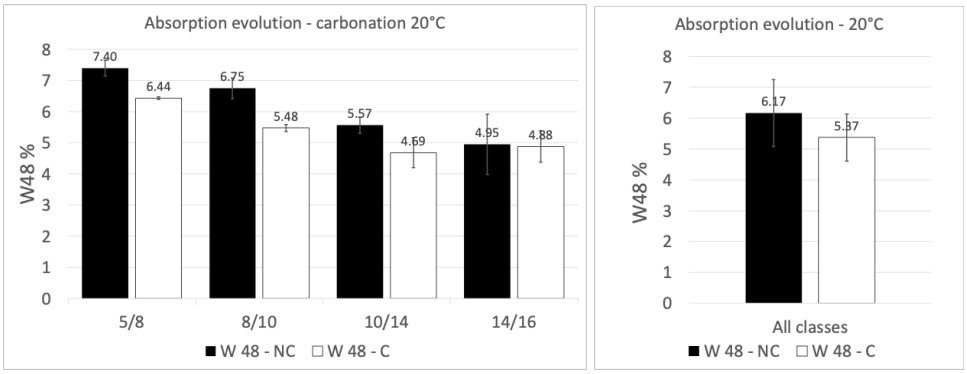

Fig. 9 RCA and CRAC water absorption for 20°C carbonation

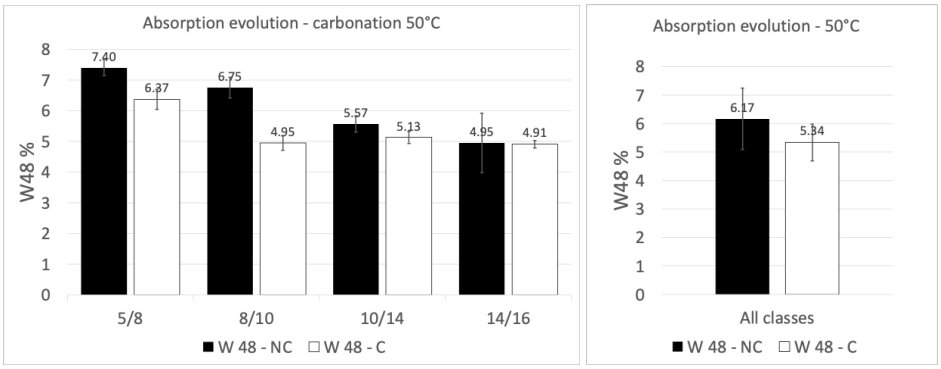

Fig. 10 RCA and CRAC water absorption for 50°C carbonation

The evolution of the absorption variability during carbonation is very interesting to study. A clear decrease is observed, especially for the 10/14 mm grain size class. It can therefore be assumed that even if the absorption value is only slightly reduced by the accelerated carbonation process, the distribution of the porosity in the aggregate will be homogenized.

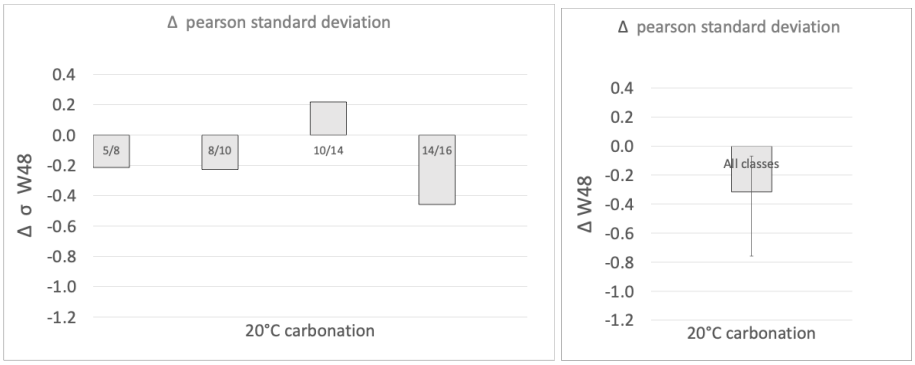

Fig. 11 RCA and CRAC water absorption variability for 20°C carbonation

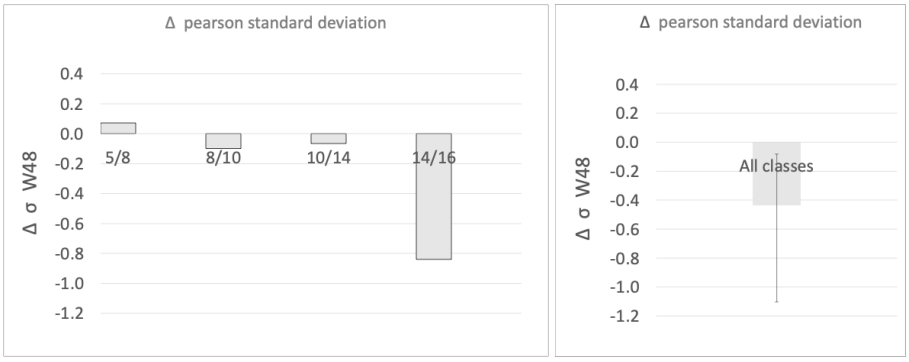

Fig. 12 RCA and CRAC water absorption variability for 50°C carbonation

Previous study carried out on RCA issued from different initial compositions and properties of concrete led to the conclusion that the variability of absorption measurement varies according to the composition of the parent concrete and can therefore decrease or increase after carbonation [14].

4.3 Limits of this study

Several hypotheses may explain the lack of correlations observed of the study presented here.

First of all, there may be no correlation because the parameters are truly independent. For example, the $CaCO_3$ initial content in RCA does not influence the CO_2 storage during the accelerated carbonation process because $CaCO_3$ is an already carbonated mineral phase that cannot fix CO_2. However, Fastcarb previous studies [1] showed that $CaCO_3$ initial content of RCA influences the accuracy of experimental results.

Secondly, there may be a connection between the input parameters (the composition and physical properties of the RCA). For example, a low-porosity RCA will limit the diffusion of CO_2 within the material and therefore limit CO_2 storage. However, if the material has a high cement and clinker content, it will have an increased potential for carbonation. As the database contains few lines with perfectly identical input parameters, this possible interdependence could not be analyzed.

Finally, another hypothesis to explain the lack of correlation is linked to methodological limitations such as partial knowledge of the input parameters, of the accuracy of the measurements or of the experimental methods/conditions recorded in the database. This was observed in round robin tests carried out by different laboratories during the Fascarb project. Indeed, for these round robin tests, the repeatability of tests within the same laboratory was very satisfactory, whereas the inter-laboratory reproducibility of the same tests was not as reliable [1].

5 Conclusion

Based on processing for statistical analysis 151 results from the literature and experimental campaigns compiled in a database, it was noted, among other things that:

Absorption is reduced for all CRCA compared to RCA (relative reduction of 22%). Accelerated carbonation results in an evolution of the data distribution of absorption values in the line with a data distribution more uniform around the mean value. The standard deviation remains similar, and the range of values is wider. This is due to the different absorption reduction according to the RCA.

An average value of 21.18 g of CO_2 captured by kg of RCA is obtained for the 34 lines of data analyzed. The range of data of $CaCO_3$ (CO_2) content has been reduced by accelerated carbonation and the values are more centered around the mean but stay wide. This is due to the different nature of the parent aggregates.

Due to the wide variation in input parameters (composition of the parent concrete, granularity of the RCA, initial absorption and initial carbonation of the RCA, etc.) and accelerated carbonation protocols, no significant independent correlation of parameters (mass gain, absorption reduction, CO_2 captured) can be reliably established. The reduction of the sampling size in order to limit the number of variables (input parameters) leads to analysis based on studies examined from few campaign/laboratory. In this case, certain correlations can be improved. Trends can be studied.

The statistical analysis of a large number of experimental data is interesting. However, to go further in identifying correlations between material parameters, their evolution and accelerated carbonation protocols, it would be necessary to carry out a round robin test with perfect control of the input data, even if they are variable.

Thus, as part of the experimental campaign carried out, it was noted, that the absorption of CRCA is systematically reduced, and more significantly in the case of small granular classes. The evolution of the absorption variability during carbonation is significantly decreased and the distribution of the porosity in the aggregate seems to be homogenized by accelerated carbonation.

As a perspective, a further statistical study dealing with this database could be conducted to discern the impact of input parameters (materials and their condition) and/or of carbonation conditions. This should help explain the non-correlations observed at this point.

Acknowledgements

Authors wishing to acknowledge the French Ministry of Ecology and Solidarity for funding this project, as well as all the FastCarb project partners.

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