

Carbonation of recycled concrete aggregates affected by alkali-silica reaction

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ABSTRACT Concrete recycling has become a major challenge for the construction industry. The use of recycled concrete aggregates (RCA) in structural applications is limited due to their often lesser mechanical and durability performances. Alkali-silica reaction (RAS), in particular, is a pathology which could affect recycled concretes to an even larger extent than in the original concrete, due to the higher alkalis content of the RCA. The present work attempts to study the effects of RCA carbonation in order to reduce the quantity of alkali hydroxides and the mobility of the alkali ions in the cement paste to prevent water-swelling gel formation. Concrete samples were produced from reactive natural aggregates and crushed at different expansion levels. The obtained RCA were submitted to CO₂. Their alkali reactivity was assessed through an autoclave mortar bar test and comparison with not carbonated RCA and original natural aggregates was performed. Reactivity of carbonated RCA is less important than the one of original natural reactive aggregates, but however higher than the acceptable limit. Carbonation still enables a reduction by 20 to 50% of the reactivity.

Key words recycled concrete aggregates, alkali-silica reaction, carbonation, expansion, reactivity

I. INTRODUCTION

Concrete, the most widely used material in the world, constitutes the largest part of construction and demolition waste [1]. Waste management has become more and more challenging, in particular in large cities [2]. In addition, awareness of the need to preserve natural resources and promote circular economy increasingly favor the use of recycled concrete aggregates (RCA) [3]. Due to the high porosity of the cement paste, the characteristics of RCA are significantly different from those of natural aggregates, particularly water absorption and density. Their mechanical performance is therefore generally lower, limiting their use in structural applications [1; 4]. But the durability of recycled concrete can also be compromised, more particularly if the original concrete is affected by a specific pathology. Specifically, a secondary induced expansion may happen when using RCA from an ASR affected structure, but its amplitude and the mechanisms taking place are still not well understood [6]. As ASR is one of the leading causes of premature concrete deterioration, it requires further investigation to ensure RCA can be used in a safe way.

Alkali-silica reaction is a deleterious swelling reaction in hardened concrete between alkali hydroxides of the pore solution and reactive silica presents in coarse or fine aggregates, forming a hygroscopic alkali-silica gel. In the presence of water, this gel expands and exerts swelling pressures inside the reactive aggregate particles, which can induce cracks within the aggregates that can extend to the cement paste.

As RCA coming from ASR-affected structures contain both reactive silica and additional alkalis (in their cement paste or in the ASR gel already present), they may potentially induce a secondary ASR in the new concrete [7]. But as RCA is a two-phase material, many factors have an effect on the ASR in RCA concrete and make it more complex than in conventional concrete: original aggregates reactivity, extent of the primary ASR-induced expansion, production procedure influencing the size, density, residual mortar content and exposing new faces [1; 6].

Supplementary cementing materials (SCMs) are often used to mitigate ASR expansion in conventional concrete. However, it has been observed that this preventive measure will require higher amounts of SCMs when using ASR-affected RCA than for the original virgin aggregate. [6; 7]. Various methods exist to enhance RCA performances. It has been shown that their treatment through carbonation can improve their quality as well as the mechanical and durability properties of the RCA concrete [2; 3]. When RCA are exposed to CO_2 , cement hydration products will react with it to form calcium carbonate, which precipitates gradually and fills the pore of the mortar paste. Reduction of porosity reduces RCA water absorption and decreases concrete transport properties [7]. As a consequence, RCA carbonation could be beneficial regarding ASR, as it can lower the pH and reduce the quantity of alkali hydroxides within the particles available to fuel ASR, but also by reducing the transport properties of the RCA concrete. Thomas et al. [7] reported that in some conditions, carbonation of RAC produced from mortar containing a highly-reactive sand can prevent ASR expansion when this measure is combined with a low-alkali binder whereas ASR expansion occurs when untreated RAC are used.

Several parameters influence RCA carbonation. Water content, linked to relative humidity, is a key parameter governing carbonation. A low relative humidity will limit CO_2 dissolution while the high water content of concrete at high relative humidity will restrict CO_2 diffusion. As a result, carbonation is maximal for an intermediate value, evaluated to be between 40 and 80 % in different researches [3; 8]. Temperature is also an important parameter known to be related to carbonation rate. The reaction is enhanced until 60 °C and begins declining after that point. [9]

The present work attempts to study the effects of coarse RCA carbonation in reducing alkali hydroxides quantity and alkali ions mobility in the cement paste, to prevent the water-swelling gel formation. Concrete samples were produced from reactive natural aggregates and crushed at different levels of expansion into RCA that were submitted to CO_2 . Alkali reactivity was assessed through an autoclave mortar bar test and compared to the one of not carbonated RCA and original natural aggregates. RCA coming from returned concrete were also tested to evaluate the potential reactivity of RCA coming from concrete not likely to be affected by ASR.

II. MATERIALS AND METHODS

A. Materials

A conventional Portland cement (CEM I 52.5 N) with an alkali content of 0.68% $\text{Na}_2\text{O}_{\text{eq}}$ was used to prepare mortar samples for all reactivity tests as well as to manufacture RCA in the laboratory. Three different types of aggregates were used in the mixes: natural reactive aggregates, recycled aggregates from returned concrete and potentially reactive RCA produced in the laboratory.

Natural reactive aggregates

Fine and coarse natural reactive aggregates used in this research are Tournaisis siliceous limestone coming from a Belgian quarry and well known for their alkali-reactivity [10; 11]. Fine and coarse aggregates show a density of 2677 kg/m³ and 2660 kg/m³ and a water absorption coefficient of 0.3% and 0.5%, respectively.

RCA from returned concrete

RCA reclaimed from returned concrete (RCA-RC) were obtained from a Belgian building company. These aggregates are a mix of demolition waste and leftover concrete coming from their construction sites. Both are processed (i.e., crushed and sieved) to get RCA that are reused by the company in their own projects. They contain a maximum of 5% of impurities. These aggregates were dry sieved in the laboratory to isolate the 4/20 mm fraction, which is the studied fraction, presenting a density and a water absorption coefficient of 2320 kg/m³ and 5.37% respectively.

Manufactured potentially reactive RCA

Laboratory concretes were manufactured with fine and coarse Tournaisis aggregates in order to produce potentially reactive RCA by crushing. Concretes were made following the requirements of the NF P18-594 standard for long term tests. A series of samples was boosted according to this standard, i.e. raising total alkali content to 1.25% Na₂O_{eq} by the use of NaOH, while another series was not boosted. Mix proportions followed the standard with a 0.52 water-to-cement ratio, even if cement content was lower than in the Standard (395kg/m³). Proportions of the different aggregates fractions were determined based on Dreux-Gorisse method. A water-reducing plasticizer (RHEOBUILD 1100 Con.30%) was necessary to achieve the S2 consistency class required by the standard.

The series without addition of NaOH (named NBOC for Non-Boosted Original Concrete) is used to produce, by crushing, RCA with alkalis content similar to ordinary concrete. Boosting the other series (named BOC for Boosted Original Concrete) accelerates the alkali-silicate reaction and then produces RCA with an advanced damage level within a short period of time. Prismatic samples of dimensions 7 x 7 x 28 cm³ were cast, demolded after 24h, measured and finally stored in climatic chambers in a water saturated atmosphere at two different temperatures: 20°C and 38°C. Their expansions were monitored for 6 months and are reported on the Figure 1. Table 1 summarizes the original concretes characteristics.

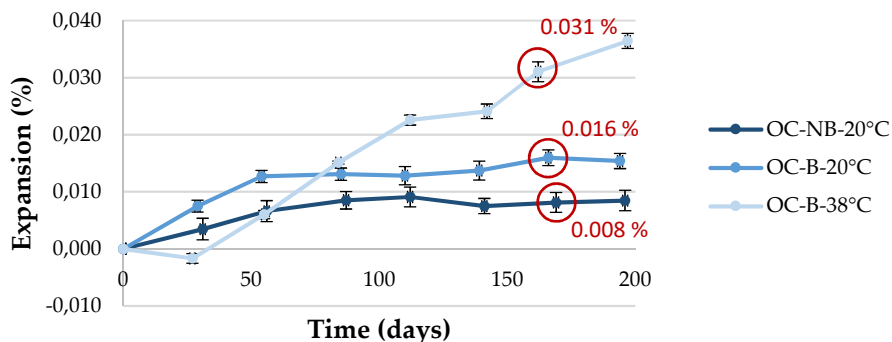


FIGURE 1. Original concrete samples expansion

The samples were broken into 4-5 cm maximum height pieces using a hydraulic press and then crushed using a laboratory jaw crusher. The resulting particles were sieved to recover coarse RCA (4 to 20 mm) and named after the original concrete as shown in Table 1.

TABLE 1 : Aggregates characteristics

	Alkali content (% Na ₂ O _{eq})	Storage temperature (°C)	Expansion after 6 months (%)	Obtained RCA
OC-NB-20°C	0.68	20	0.08	RCA-T-NB-20°C
OC-B-20°C	1.25	20	0.016	RCA-T-B-20°C
OC-B-38°C	1.25	38	0.031	RCA-T-B-38°C

B. Carbonation

Two air-tight incubators were used as curing chambers. Test was performed at atmospheric pressure. Temperature and CO₂ concentration can be regulated directly by the equipment. They were set at 39°C and 20%, which corresponds to the maximum values allowed by the incubators. A saline solution was placed in the chambers to maintain a relative humidity close to 60%.

Carbonation pretreatment was applied to the 4/20 mm fraction of all RCA. Removable trays were filled up to 25 mm layer thickness. A few days before starting carbonation, RCA were placed in the chambers with the saline solution, until constant mass was reached at 60% RH. All RCA samples were then carbonated at the same time, due to laboratory constraints, during 11 days.

To follow RCA carbonation, several tests were used to give indications about the reaction level. First, carbonation can be evaluated by the mass gain due to the formation of calcium carbonates, thus the mass difference before ($m_{initial}$) and after (m_{final}) carbonation. However mass gain during carbonation is not only due to CaCO₃ formation but also to water released from carbonatable phases (Ca(OH)₂ and C-S-H) [3; 12]. This carbonation-induced water loss (m_{water}) was collected by the saline solutions placed in the incubators and thus added to the final mass. Carbonation mass gain (Δm) was estimated according to Equation (1).

$$\Delta m = \frac{m_{final} + m_{water} - m_{initial}}{m_{final}} \quad (1)$$

Thermal analysis is another quantitative method which is often used for evaluating carbonation. The sample is submitted to temperatures that can reach 1250°C. A thermobalance records mass losses associated to evaporable water, deshydroxylation of cement hydrates (C-S-H and portlandite) and decarbonation of calcium carbonates, corresponding to specific temperatures ranges [3]. In this research, sample were ground and tested using a thermal-gravimetric analyzer with temperature ranging from 20 to 1000°C at a 10°C/min rate in a nitrogen atmosphere.

As it is quite difficult to distinguish peaks associated to C-S-H and evaporable water due to overlap and as decarbonation of calcium carbonates comes partially from limestone aggregates, the amount of portlandite was the only one considered in absolute terms and calculated from TG and DTA, based on its decomposition reaction:

$$\% \text{Ca(OH)}_2 = \Delta \text{Ca(OH)}_2_{(ATG)} \times \frac{M_{\text{Ca(OH)}_2}}{M_{\text{H}_2\text{O}}} \quad (2)$$

where $\Delta\text{Ca}(\text{OH})_2_{(ATG)}$ corresponds to the mass variation due portlandite deshydroxylation (identified by DTA), $M_{\text{Ca}(\text{OH})_2}$ and $M_{\text{H}_2\text{O}}$ to molar masses of portlandite and water respectively [3].

D. Characterization of RCA

Water absorption and density evaluation followed the EN 1097-6 standard [13]. Aggregates are saturated for 24 h in water. The water absorption coefficient is determined based on the water content at saturated surface dry (SSD) state. 0,063/5 mm aggregates were exposed to a gentle current of warm air and SSD was detected using a slump test with a truncated cone

Cement paste content was evaluated using the method developed by Zhao et al. [14] based on salicylic acid dissolution. It allows the dissolution of most phases contained in cement paste but not the main phases contained in limestone aggregates [14], unlike hydrochloric acid e.g.

E. Reactivity tests on mortars

Samples for accelerated tests on mortar by autoclave were prepared and tested according to NF P18-594 standard [15]. Mortars were manufactured with recomposed sand having a grain size of 0.16/5 mm prepared by crushing the original aggregate with a jaw crusher in order to follow the particle size distribution required by the standard. The crushing procedure was adopted based on a trial-and-error approach aiming at minimizing material losses in order to preserve chemical and mineralogical properties of the RCA. This mass loss was limited to 6 to 12% of the initial mass. For the test on the natural aggregates, the original sand was simply sieved to match the requirements.

As water absorption of RCA is higher than the one of natural aggregate, which is usually neglected, it can modify the effective water-to-cement ratio of the mortars and then have an influence on the measured expansion. In their study, Delobel et al [16] concluded that water absorption has a strong influence on the expansion of mortars made with RCA. It is therefore necessary to adapt the test to take it into consideration in the composition of the mixes. Dried recycled sands obtained from RCA crushing were here pre-saturated with 85% of their water absorption coefficient by adding the supplementary water in the sand 30 minutes before mixing.

To avoid lixiviation of RCA alkalis, the washing procedure imposed by the standard was also adapted to minimize this loss. The procedure suggested by Adams et al [17] was followed. The washing consisted in spraying water on each fraction retained on a sieve during a specific time varying depending on particle sizes. In addition, one mix was prepared with 4/20 RCA that were soaked 12 days in water with water renewal after 3 days before crushing.

Mortars were alkalinized to 4% $\text{Na}_2\text{O}_{\text{eq}}$ according to the cement mass by addition of NaOH in mixing water. For RCA coming from returned concrete and concrete manufactured in the laboratory without addition of NaOH, alkalis of RCA were not taken into account in the calculation, allowing to consider the effect of additional alkalis present in the RCA cement paste. On the contrary, for RCA coming from alkali-boosted concrete manufactured in the laboratory, the artificial alkalinization was deducted to the quantity of NaOH needed to reach 4% $\text{Na}_2\text{O}_{\text{eq}}$.

Prismatic samples of dimensions $4 \times 4 \times 16 \text{ cm}^3$ were cast. Mixes were manufactured with two different cement/aggregate ratios (C/A): 0.5 and 1.25. For each C/A ratio, three prisms were tested. The test consists in measuring the dimensional variations of lengths before (L_{0i}) and after (L_{1i}) a

cure in autoclave at a temperature of 127 °C and under a pressure of 1.5 bar during five hours. Prisms expansions can be calculated from Equation (4), with a reference length (L_0) of 140 mm.

$$\varepsilon_i (\%) = \frac{L_{1i} - L_{0i}}{L_0} \quad (4)$$

The aggregate is considered as non-reactive if the average expansion of these three test prisms is smaller than 0.15% for each C/A ratio and potentially reactive if the average expansion is higher for at least one C/A ratio. In addition to that, potentially reactive aggregates are considered to present a pessimum effect when expansion does not decrease when C/A ratio increases.

III. RESULTS

A. Sands characterization – influence of carbonation

Results of the characterization of sands obtained from not carbonated (NC) and carbonated RCA (C) are given in Table (2). It should be noticed that WA_{24} and density tests were performed on the complete sand fraction used for testing RCA reactivity (0.16/5 mm), while cement paste content, mass gain and Portlandite content were obtained from a part of the sand fraction (2.36/5 mm), due to material restraint.

TABLE 2 : Sands characteristics – influence of carbonation

Type of crushed aggregate	RCA pre-treatment	WA_{24} (%)	% WA_{24} variation (%)	SSD density (kg/m^3)	% SSD density variation (%)	Cement paste content (%)	% cement paste cont. variation (%)	Mass gain (%)	Portlandite content (%)
RCA-T-NB-20°C	NC	5.28		2500		9.03		-	1.80
	C	5.61	-6.25	2455	-1.8	8.81	-2.4	0.91	0.98
RCA-T-B-20°C	NC	5.47		2470		8.78		-	2.05
	C	5.06	7.5	2503	1.3	5.35	-39	1.18	*
RCA-T-B-38°C	NC	6.91		2415		13.70		-	2.65
	C	5.21	20.3	2478	2.6	6.99	-49.0	1.66	*
RCA-RC	NC	5.11		2489		6.98		-	0.41
	C	4.95	3.1	2485	0.2	6.09	-12.7	0.32	*

* peak associated to portlandite deshydroxylation could not be distinguished

They highlight the influence of the adherent mortar porosity of recycled aggregates. WA_{24} and SSD density are around 5% to 6% and 2400-2500 kg/m^3 respectively, which is significantly different from natural aggregates characteristics. Except for RCA-T-BN-20°C, WA_{24} is reduced and density is increased after carbonation even if these variations are not substantial. Cement paste contents is also lower in all cases after carbonation, which indicate a higher content of $CaCO_3$ in carbonated pastes that is not solved by salicylic acid. A mass gain due to carbonation was recorded for all samples. Thermal analysis confirms a decrease in portlandite content: except for RCA-T-NB-20°C,

peak associated to portlandite deshydroxylation cannot be distinguished anymore in none of the samples after carbonation.

All these results tend to demonstrate that the carbonation procedure implemented in this study is effective and impacts RCA properties. However, its influence is not similar for all samples. RCA-T-B-38°C seem to be the most affected aggregates, followed by RCA-T-B-20°C, RCA-RC and finally RCA-T-NB-20°C. It was expected that RCA-RC would already be at least partially naturally carbonated. It is indeed confirmed by thermal analysis showing a low portlandite content compare to manufactured RCA. The difference between the other samples could simply be explained by their position in the incubator: the lower they were placed, the higher carbonation effects could be noticed. The ventilation system might not have been efficient enough to ensure homogeneous CO₂ concentration within the chamber.

B. Reactivity tests on mortar

As expected, results of reactivity tests on mortar classifies the Tournaisis natural reactive aggregate as potentially reactive, with expansions of 0.52% and 0.48% for 0.5 and 1.25 C/A ratios respectively (orange spots on Figure 2 (a), (b) and (c)). It is consistent with results of Guédon-Dubied et al. [11] who worked on similar aggregates coming from the same quarry. When compared to that, expansions of RCA made from this natural aggregate (dark blue triangles on Figure 2 (a), (b) and (c)) are quite lower, but still higher than the 0.15% limit. It can be noticed that all RCA show a pessimum effect, unlike original aggregates. This pessimum effect for RCA is also observed in the literature [18]. But this particular behavior could be specific to the autoclave test as other tests like silica soluble dosage or micro-mortar could not give the same conclusions, as it was the case in the RECYBETON research [18]. It should therefore be confirmed by other tests.

Surprisingly, RCA coming from return concrete can also be classified as potentially reactive aggregates with pessimum effect, even if expansions are just above the limit (dark blue triangles on Figure 2 (d)). It shows that usual RCA used in the construction industry might induced ASR reaction and potentially create damage to a structure.

When comparing the three different RCA manufactured in the laboratory without carbonation (dark blue triangles on Figure 2 (a), (b) and (c)), it can be observed that expansions are the highest for RCA-T-NB-20°C (C/A=0.5: 0.32 – C/A=1.25: 0.38), then for RCA-T-B-20°C (C/A=0.5: 0.28 – C/A=1.25: 0.36) and the lowest for RCA-T-NB-38°C (C/A=0.5: 0.24 – C/A=1.25: 0.30). The difference between RCA produced from non-boosted and boosted concrete could probably be explained by the fact that this is the theoretical Na₂O_{eq} content of the boosted concrete that was deducted when calculating the quantity of NaOH to be added to mortar mix. However this content might have been lowered by lixiviation during storage. We can also notice that the higher the original concrete expansion the lower the RCA mortar expansion. It is particularly visible for RCA-T-NB-38°C, which is the one for which the alkali-silica reaction in the original concrete was the most advanced. It implies that less reagents might be available for the secondary ASR. But these conclusions should be nuanced by the fact that RCA-T-NB-38°C is presenting higher cement paste content compared to RCA-T-NB-20°C and RCA-T-B-20°C, meaning that less natural reactive aggregates are present.

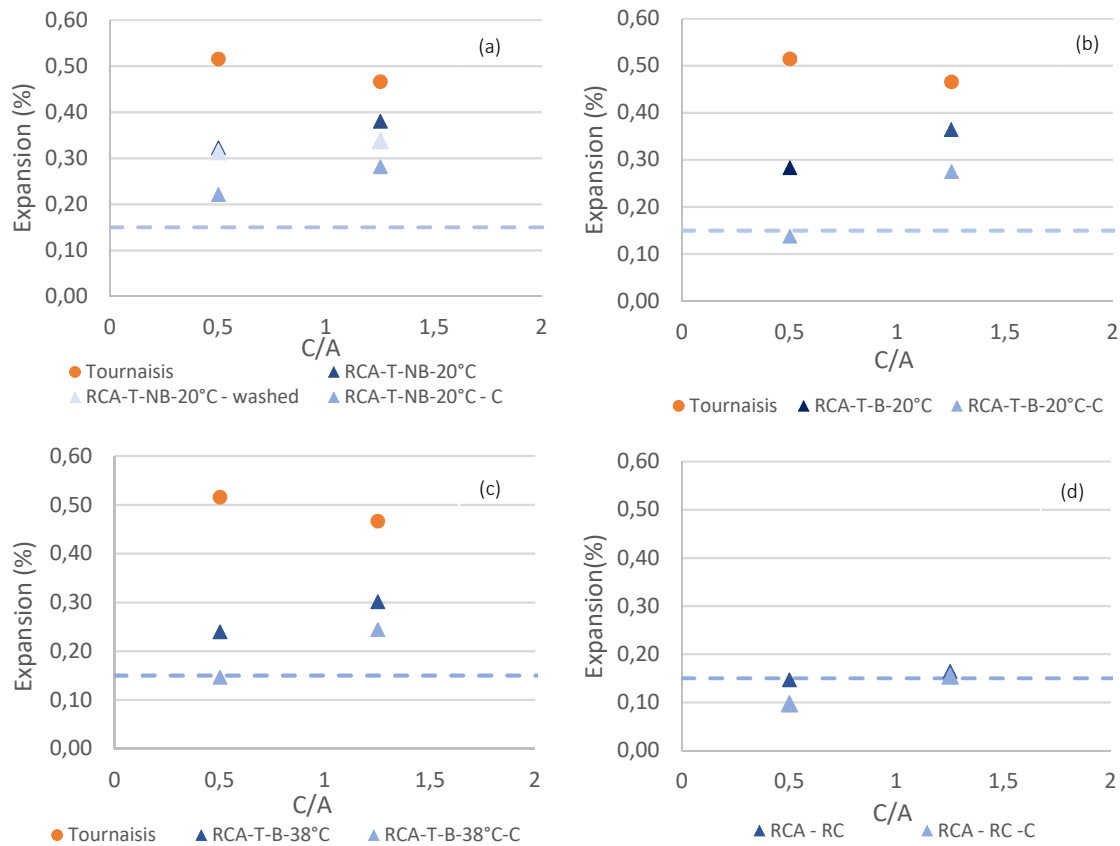


FIGURE 2. Expansions on mortar reactivity test : (a) Laboratory manufactured RCA from non-boosted concrete stored at 20°C, (b) Laboratory manufactured RCA from boosted concrete stored at 20°C, (c) Laboratory manufactured RCA from boosted concrete stored at 38°C; (d) RCA from returned concrete. Error bars were not indicated as they could not be distinguished from the spots, data variability being very low.

Carbonation effect on mortar expansions is visible on Figure 2 (light blue triangles). Same trends as for non-carbonated RCA can be observed. However, all carbonated RCA were subjected to expansion reduction compared non-carbonated RCA. They are still classified as potentially reactive aggregates with pessimum effect, even if expansions for $C/A=0.5$ are below the limit for RCA-T-B-20°C, RCA-T-B-38°C and RCA-RC-C. On Figure 2(a), (b), (c) and (d), expansions are reduced by 31%, 50%, 38% and 33% for $C/A=0.5$ and 26%, 22%, 20% and 0% for $C/A=1.25$ respectively. It can be noticed that the lower the C/A ratio, the higher the carbonation influence.

Figure 2 (a) shows that the test performed with RCA that were soaked in water before use (pale blue triangles) gives slightly lower expansions than non-washed RCA, which can probably be explained by alkalis lixiviation during washing. However, this effect is not clearly pronounced and indicates that RCA washing by itself is not an efficient method to mitigate RSA.

These results are promising as they show that carbonation has a discriminant effect on the reactivity decrease of potentially reactive RCA (up to 50%), even if carbonation of these aggregates is not complete. Portlandite, which is consumed during carbonation reaction, is indeed necessary to alkali-silica reaction. For high C/A ratio, this reduction of Portlandite within the RCA is mitigated by Portlandite available in larger quantities in the new cement paste of the mortar.

Tests on concrete should be performed to confirm these results. They should be more suitable to evaluate the effect of RCA carbonation. Beside the fact that new faces of the original reactive natural aggregates are exposed and that a part of cement paste is eliminated through fine particles when RCA are crushed to be tested on mortar, crushing might also reduce the effect of carbonation which is more important at the surface of the aggregate than inside.

IV. CONCLUSIONS

The main objective of the test program carried out in this study was to evaluate the effects of coarse RCA carbonation in order to mitigate ASR. RCA were produced from concrete containing reactive natural aggregates. RCA coming from returned concrete were also tested. The main conclusions are summarized below:

- The carbonation procedure implemented in this study is effective and impacts RCA properties: slight reduction of water absorption (up to 20%), reduction of cement paste contents based on salicylic acid (up to 49%) indicating a higher content of CaCO_3 in carbonated pastes, mass gain due to carbonation (up to 1.6%) and decrease in Portlandite content.
- Mortar expansions of RCA made from natural reactive aggregate are lower than the expansion produced by the original aggregate, but still higher than the 0.15% limit. They show a pessimum effect, unlike original aggregate, which should be confirmed by other tests.
- The higher the original concrete expansion, the lower the RCA mortar expansion. But this conclusion should be nuanced by the fact that cement paste and alkali contents were not exactly the same in the compared samples.
- RCA coming from return concrete used in this study can also be classified as potentially reactive aggregates with pessimum effect, even if expansions are just above the limit.
- Carbonation, even uncomplete, can significantly decrease potentially reactive RCA expansion. The reduction is more important for low C/A ratio. But carbonatation alone does not seem to be sufficient to guarantee expansions below the 0.15% limit. Reasonable quantities of SMCs combined with carbonation could mitigate ASR expansion.
- RCA washing is not an efficient method to mitigate RSA as reduction of expansions expected from the lixiviation of alkalis during washing is not significative.

Tests on concrete should be performed to confirm these results. They should be more suitable to evaluate the real effect of RCA carbonation.

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