

THE ROLE OF AGGREGATES IN THE DURABILITY COMPARATIVE STUDY OF CARBONATED AND NON-CARBONATED CONCRETE SAMPLES WITH ADMIXTURES***Jacques Herve Koung à Bediang**

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Abstract

The purpose of this study is to investigate the effect of aggregates on the carbonation of quarry sand-based concrete. The concrete is made of 100% quarry sand 0/6.3, gravel 8/15, and 15/25 from the Arab Contractor quarry in Nomayos, Cameroon, with CEM II B-P 42.5 R from CIMENCAM (Cimenteries du Cameroun). The study employed two admixtures: Sikamen and Sika liquid. Carbonation was performed on concrete samples at several dates until 545 days. Carbonated and non-carbonated concrete samples are compared in terms of durability aspect like Thermo Gravimetric Analysis (TGA) and capillary absorption. The findings of study indicate that the compressive strength is satisfactory. The microstructure becomes firmer and more resistant to hostile treatments. A reduction in water and pore space causes mass loss, which leads to an increase in elastic deformation. Sikament additive gives concrete very high plasticity and prolonged rheology, which improves compactness and durability. Sika water repellent reacts with the cement's lime to generate comparable crystallizations that block the mortar's capillaries, making it hermetic with the position of granules during chemical reactions. Accelerated.

Keywords: Carbonation, Concrete, Aggregates, Quarry sand, Sikament, Water-repellent

INTRODUCTION

The cement industry is one of the biggest emitters of greenhouse gases. In France, it accounts for 2.9% of CO₂ (carbon dioxide) emissions, while on a global scale this figure rises to 6% [1]. Aware of the need to act, cement manufacturers have committed to reducing their emissions by 24% by 2030 and 80% by 2050, compared with 2015 [2]. To meet these targets, the cement industry's roadmap [3] identifies several action levers, one of the main ones being accelerated carbonation of aggregates. Concrete carbonation, which is the reaction between cement hydrates (portlandite Ca(OH)₂ and C-S-H gel) and carbon dioxide CO₂ (the main greenhouse gas and one of the main causes of global warming), and which leads to the formation of calcium carbonates CaCO₃, is an effective treatment for recycled concrete aggregates (GBR) as it reduces their high porosity and high water absorption capacity) for their reuse [4]. Accelerated carbonation of concrete aggregates is therefore one of the solutions envisaged by the cement industry to reduce its CO₂ emissions and move towards carbon neutrality by 2050. This type of aggregate can play an important role in the storage of CO₂ following their carbonation. In order to limit the environmental impact of concrete and promote recycling and the circular economy in the construction industry, a great deal of research has been carried out in this field, including the carbonation of recycled concrete aggregates. Studies have shown that this process can significantly improve the properties of recycled aggregates, potentially facilitating their use in concrete [5], [6], [7]. What about the carbonation of simple aggregates from quarries, particularly those in Cameroon?

In Cameroon, the use of quarried aggregates in civil engineering structures has increased considerably in recent years, with the production of crushed sands. These sands are used in the formulation of mortars and concretes, but scientific requirements for their use are rare. This shows the renewed interest in the contribution of crushed sands to mortar properties. Research on quarry sand has shown that: B. Menadi et al. found that using a maximum of 15% fines [8]; O. A. Cabreara et al. demonstrate the link between void content and paste volume [9]; and A. Al-Ameeri et al. rely on the mineralogical source of sand, which has an impact on concrete behavior [10]. As for B. Benabed et al. found that the granular nature of quarry sand had an additional impact on mechanical and rheological properties. This finding refers to the granite sand grain configuration [11]. This led to a host of research studies: Benachour et al [12], M.L.K Khouadia et al [13], Lawrence, [14], Michel, [15] ... etc. on the use of quarry sand. Analysis of published research has led to the conjecture that several directions can be targeted. and that quarry sand is an adequate solution to limit the over-exploitation of alluvial sand, unless the mechanical properties and durability of concrete are not diminished [16]. Other researchers have focused on the effects of admixtures such as SIKA liquid water repellent and/or SIKAMENT superplasticizer. [17], [18], [19]. As carbonation is a natural phenomenon occurring with CO₂, accelerated carbonation requires hard environment with a high concentration of CO₂ [20], [21], [22]. In cementitious materials, CO₂ hardening thickens the microscopic structure, limits the required hardening time, ameliorates air quality and some properties [23], [24]. In this paper, our aim is to assess the role of aggregates on the carbonation of quarry sand concrete, by carrying out a comparative study between carbonated and non-carbonated concrete. The aim is to provide estimated criteria for optimal life-cycle inspection of quarry sand concrete structures as an alternative to river sand.

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Table 1. Physical characteristics, Chemical and Mineralogical Composition of cement (manufacturer's data) and Paste composition

Physical characteristics											
Status		On Powder				On paste					
Nature		Refusal to 0.08 mm (%)	SSB (cm ² /g)	DA (T/m ³)	PS (T/m ³)	Expansion (mm)		Setting (mn)			
						Cold	Hot	Start	End		
CEMII		1.01	3396	0.86	3.08	0	0	2h58	3h46		
Chemical Composition of cement (manufacturer's data)											
Elts (%)	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MnO	MgO	CaO	Na ₂ O	K ₂ O	TiO ₂	P ₂ O ₅	LOI
	25.5	7	6	0.13	5	52	5	0.91	0.97	0.26	2.67
Mineralogical composition of cement (manufacturer's data)											
Constituents	C ₃ S			C ₂ S	C ₃ A	C ₄ AF	CaSO ₄	Natural pozzolan			
Proportions (%)	65-79			65-79	65-79	65-79	2.5-3.5	21 - 35			
Paste composition											
Paste composition	Cement			Normal sand		Water	Adjuvent	Start-up			
Proportions (%)	500 g			0 g		162 g	0.0 %	8 : 12			

SSB : Bladine Specific Surface ; - DA : Apparent Density, - PS : Specific Weight ; - LOI : Loss On Ignition at 1000°C, - C3S : Tricalcium Silicate ; - C2S : Dicalcium Silicate ; - C3A : Tricalcium Aluminate ; - CaSO4 : Calcium Sulfate.

Table 2. Sand particle size analysis, Physical properties of sand and Chemical composition of crushed sand (%) from XRF (Argile, Géologique et Environnement Sédimentaires Laboratory, University of Liège, Belgium (AGES))

Sand particle size analysis											
Nature		Particle size analysis (% passing sieve)									
Screen		0.08	0.16	0.315	0.63	1.25	2.5	5	6.3	8	
Sand (% passing)		8.0	12.9	20.2	27.1	36.5	52.9	85.5	99	100	
Physical properties of sand											
Sand	TF	MF	DA(T/m ³)	PS(T/m ³)	ES (%)	Visual	ES (%) Piston				
0/6.3	6.3	3.65	1.68	2.98	76		74				
Chemical composition of crushed sand (%) from XRF (AGES).											
Elts	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MnO	MgO	CaO	Na ₂ O	K ₂ O	TiO ₂	P ₂ O ₅	LOI
0/6.3	65.5	15.69	7.76	0.16	2.74	1.37	1.76	2.68	0.95	0.14	0.57

- TF : Fine Content ; - MF : Fineness Module, - ES : Sand Equivalent. [NF EN 933-8-Août 1999] [25]

Table 3. Gravel particle size analysis, Physical properties of gravel

Gravel particle size analysis										
Nature		Particle size analysis (% passing sieve)								
Screen (mm)		5	6.3	8	10	12.5	16	20	25	31.5
Gravel 8/16		2.6	3.2	6.5	16.7	42.7	86.3	100		
Gravel 15/25				3.5	4.8	6.6	15.9	50.8	92	100
Physical properties of gravel										
Nature	LA (%)	MDE (%)	DA(T/m ³)	PS(T/m ³)	AP (%)			PR (%)		
Gravel 8/16	/	/	1.46	2.94	18< 30%			0.99< 3%		
Gravel 15/25	10/14	10/14	1.45	2.92	14<30%			0.82< 3%		
	47.7	20.04								

- LA : Los Angeles ; - MDE : Micro Deval; - AP: Flattening Coefficient.

We obtained granulometric analysis of the aggregates on the curve in figure 1.

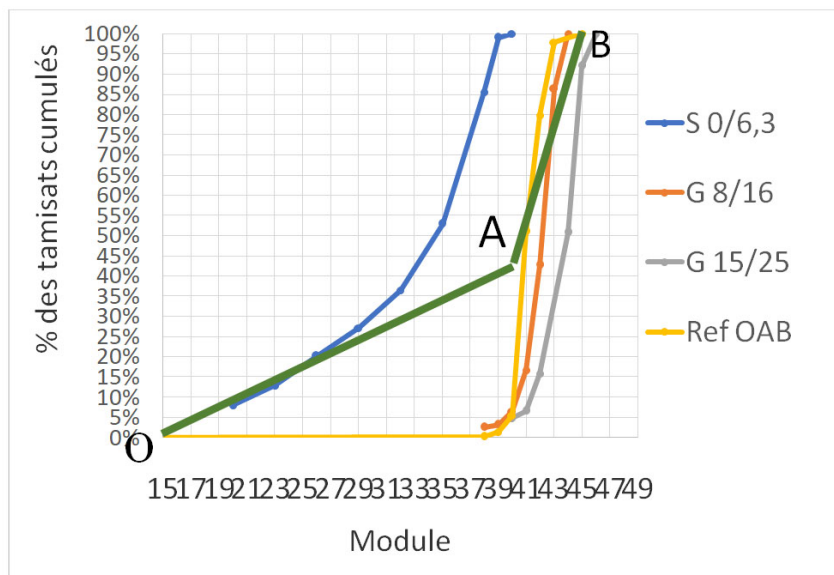


Figure 1. Size curve for the aggregates utilized

MATERIALS AND METHODS

Materials

Cement

We are used Portland NC 234: 2017 CEM II 42.5 R. Table 1 shows the results for the physical attributes, the chemical and mineralogical compositions of cement and paste.

Sand

The sand come from the Arab Contractor quarry in Nomayos, Cameroon. Tables 2, summarize results of sand from LABOGENIE (Laboratoire National de Génie Civil).

Aggregates

We use: 8/16 and 15/25 mm, both from the Arab Contractor quarry. Tables 3 describe the results of LABOGENIE's identification tests, which are of gneissic origin.

Admixtures

Sikament and water repellent are used, in standard of ASTM C 494 type G [26] and NF EN 934-2 [27].

Methodology

The 16 x 32 cm test specimens were created at LABOGENIE for tests at Laboratoire de Mécanique et Matériaux de Génie Civil (L2MGC), CY Cergy Paris University. The quantities of the ingredients are defined in Table 4 for 1m³ (W/C ratio = 0.471).

Carbonation

An accelerated carbonation test under the following conditions: 3% of CO₂, 65% of RH, 20°C of regulated temperature was done. The environment laboratory conditions have a temperature of 20 ± 1°C. The samples have 14 days in an oven at 45°C and 50% RH. Measurements dates of carbonate thickness are 0 days, 180 days, 360 days and 545 days.

The carbonation depth was determined by a phenolphthalein color indicator with the AFPC-AFREM guide [28], [29]. It's obtained by the color of the indicator, which varies from non-carbonated to carbonated zones, taking the form dark pink to colorless. A témoin specimen was soaked in phenolphthalein before being placed in a room heated to 20°C and 23% HR in order to compare the changes in carbonation depth with the specimens on display in the room. We use for the test mortar specimens of 150 x 100 mm² [NF XP 18 458, 12/2022] [30]. The photographs in Figure 2 show the apparatus employed.

Thermogravimetric analysis (TGA)

Thermogravimetric analysis was carried out on 100 to 110 g of powdered material from the non-carbonated and carbonated concrete samples. The tool used was the STA 449 F1 Jupiter developed by NETZSCH. Thermal analysis is carried out according to the following temperature program: Start at room temperature (close to 25°C); - Linear heating from room temperature up to 1100°C at a rate of 10°C/min under air sweep (80 ml/min).

Absorption by capillarity

Capillary absorption was determined on 150 x 50 mm sample slices from 1500 x 300 mm specimens previously dried in a ventilated oven at T = 80°C for 7 days, followed by drying in a ventilated oven at T = 105°C until mass stabilization (weighed to the nearest 0.01 g) to within 0.05%. The samples were then filmed on their face to ensure absorption on the cross-section, while avoiding drying on the lateral face (see figure 3).

RESULTS AND DISCUSSION

Carbonation

Figure 4. depicts the progression of carbonation depths. In these photos, the discolored zone's measurement were pH decreases from 13 to roughly 8 or 9 depending of indicator. The images in Figure 4 show: The carbonate zone, in which portlandite has completely disappeared; The mixed zone, in which the amount of calcium carbonate decreases and the amount of portlandite increases; The healthy zone, in which no mineralogical changes are observed.

Table 4. Concrete formulation using quarry crushed sand

Concrete constituents C30/37	SA 0/6.3	Gravel 8/16	Gravel 15/25	CEM II BP 42.5R	Water	Sikament	Water repellent	Slump test (cm)	TD (kg/m ³)
Dosage for 1 m ³	889.0 kg	398.6 kg	712.7 kg	400 kg	188.4 L	1.4 L	2.8 L	7.5 cm	2589
Dosage for a 50 kg	111.3	49.82	89.09	50	23.55	0.35	0.98	/	/

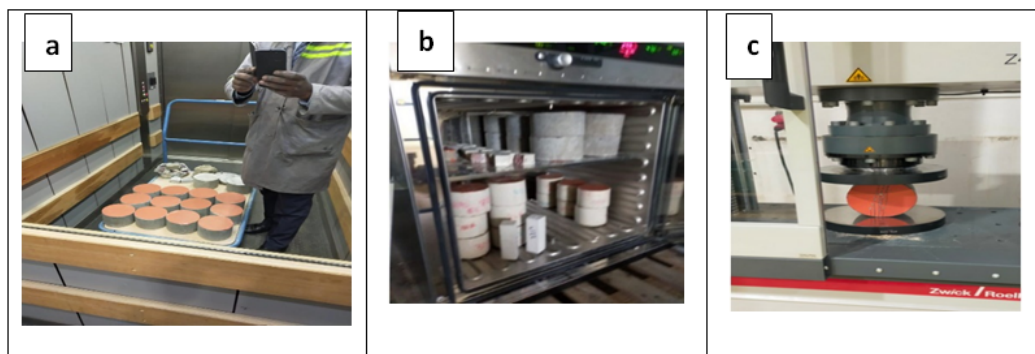


Figure 2. Demonstrates: a) preparation of specimens filmed on both cross-sectional faces to force CO₂ diffusion in one direction only: b) depositing specimens in the carbonation chamber: c) Splitting the specimens to spray phenolphthalein

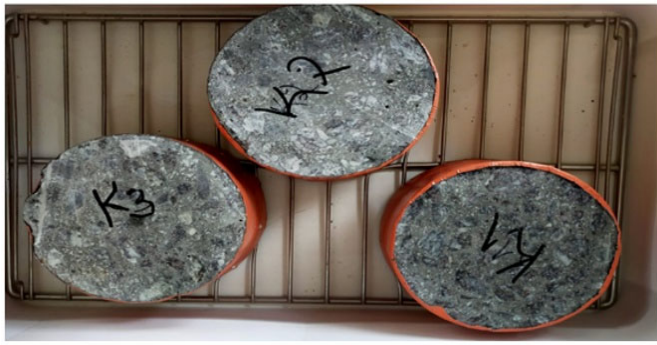


Figure 3. Sample preparation for capillary absorption test

Exposure time	Image of the carbonation front	Carbonation depth
0 day (indicator)		0 mm
180 days		25 mm
360 days		31 mm
545 days		36 mm

Figure 4. Carbonation depth changes throughout different study dates

Thermogravimetric analysis (TGA)

Thermogravimetric analysis (TGA) is the process of recording mass variations during a thermal cycle that are caused by chemical reactions or the departure of volatile constituents adsorbed or combined in a material. The temperatures at which these mass losses occur provide additional information to that obtained by ATD for identifying the physicochemical phenomena involved, and the two characterizations are frequently performed concurrently in the same instrument. These mass variations are due, among other things, to hydrate dehydroxylation and calcite decomposition. The mass vs. temperature curves are used to quantify hydrates, primarily portlandite, in the temperature range where they dehydroxylate. Similarly, calcite, particularly that formed by carbonation, is quantified according to its decomposition temperature range. This technique allows for the quantification of the quality of portlandite and calcium.

The difficulty with TGA, however, remains in determining these temperature ranges, which vary according to the authors Mounanga et al [31]. Furthermore, in concrete with limestone aggregates, it is difficult to distinguish between the calcium carbonate formed by carbonation and that contributed by the aggregates. According to Thiery [32], TGA can be combined with chemical analysis to determine the cement fraction actually present in the sample, and thus quantify the various elements, particularly hydrates, for a representative elementary volume of material, independent of cementitious phase segregation, hydric and carbonation state. Several authors have described the reactions that occur in cementitious materials at high temperatures. In this framework, it is possible

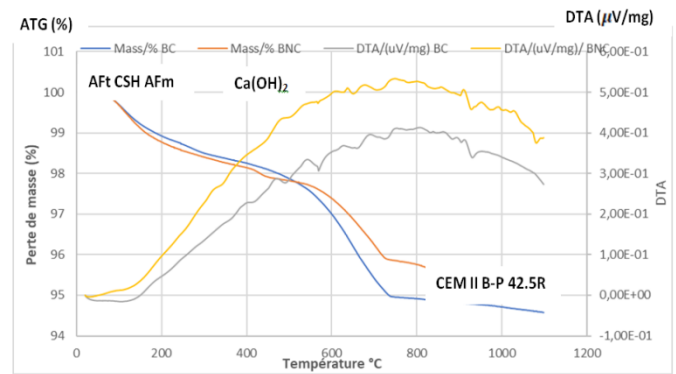


Figure 5. TGA and DTA curves for carbonation concrete (CC) and non-carbonation concrete (NCC) samples

- The curves can be used to identify and quantify the mineral phases present in concrete. Their presence or absence correlates with temperature thresholds.
- The mass losses associated with each inflection point are used to calculate portlandite, C-S-H, and calcite concentrations. In terms of TGA, non-carbonated and carbonated concrete show four peaks (4 inflection points) and three peaks (3 inflection points), respectively.
- Between 20°C and 400°C: decomposition of AFt phases and C-S-H. However, several authors argue that the bound water of C-S-H is evacuated over a broader range, such as 25 to 550°C, according to Taylor [33]. In this context, we rely on recent data from Borges et al [34], who conclude that the amount of water released by C-S-H is negligible after 300°C. Decomposition of AFm phases. Gypsum decomposition. Ramachandran et al [35] observed the latter over a temperature range of 140-170°C. Rivas-Mercury et al. [36] discovered katoite dehydroxylation at temperatures ranging from 300 to 450°C. Decomposition of brucite. Collier et al. [37] expand the temperature range to 350-450°C.- The fourth between 800°C and 1000°C: decomposition of the magnesian part of dolomite. It is identified over a wider range, from 710°C to 910°C, according to Maitra et al [39]. TGA/DTG tests can be used to estimate the amount of portlandite directly. However, the technique is not suitable for estimating C-S-H content, due to the superposition of DTG peaks from the AFt and AFm phases. The C-S-H content is therefore estimated using water sorption tests and the Olson & Jennings method [40].

The heat flux peaks are primarily related to the phase change temperatures of the various hydrates in the cement paste, as the majority of limestone aggregates remain stable up to 700°C. The first two endothermic peaks in the TGA curves of BNC and BC primarily indicate the separation of water from the constituents of certain hydrates, CSH and ettringite [41]. The degradation of ettringite and CSH gel is then linked to these two peaks, as chemically bound water begins to evaporate from concrete at these temperatures. Castellotea et al [42] confirm that ettringite dehydration occurs before 90°C. In contrast, a small endothermic peak is observed. The peak at temperatures between 600°C and 800°C on the BNC TGA curve indicates the dehydration of hydrated calcium monocarboaluminate. $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaCO}_3 \cdot 11\text{H}_2\text{O}$ [43]. The BNC TGA curve shows a significant endothermic peak at 755°C, indicating the decomposition of portlandite to free lime via the following reaction [44], [45]: $\text{Ca}(\text{OH})_2 \rightarrow \text{CaO} + \text{H}_2\text{O}$. It should be noted that portlandite is a key component in cement paste. It is derived from the hydration of cement's main

minerals, C_2S and C_3S . Several researchers, such as Noumowé [46], have set a temperature range for portlandite degradation between 450°C to 550°C. The decomposition of CSH and portlandite reflects the degradation of concrete's compressive strength when exposed to temperatures above 400°C. We conclude that the mass loss is primarily caused by the dehydration of the hydrates in cement paste, CSH, ettringite, hydrated calcium monocarboaluminate, and portlandite. This loss represents approximately 12% of the sample's initial mass. Several authors, including H. Fares [45], A. Noumowé [47], and T. Belkacem [48], have demonstrated the impact of temperatures above 600°C on concrete behavior. They demonstrated that CSH decomposes and is transformed into a new form of dicalcium silicate ($-C_2S$) at temperatures ranging from 600 to 700°C, while aggregates and calcareous fillers decompose between 700 and 900°C. This is accompanied by the emission of CO_2 .

Absorption by capillarity

The role of water in the deterioration mechanism of porous building materials has long been recognized. Its penetration into building materials creates a mechanism and pathway for aggressive materials to penetrate and cause corrosion of steel reinforcement in concrete [49]. Capillary absorption (depth of water penetration) is a water property that describes a stone's ability to absorb a specific amount of water per unit time and surface area when only one face is in direct contact with water. The capillary water absorption coefficient, $C(w,s)$, is expressed as $g/m^2.s^{1/2}$. Water absorption in dry concrete is known to be dependent on two major parameters: the concrete's effective porosity and the rate of absorption via capillary rise (absorptivity) [50]. Concrete has tiny, communicating capillaries ranging from 10 μm to 0.01 μm that can absorb, retain, and release water through slow evaporation. Figure 6 depicts the water absorption kinetics for carbonated and non-carbonated concrete samples.

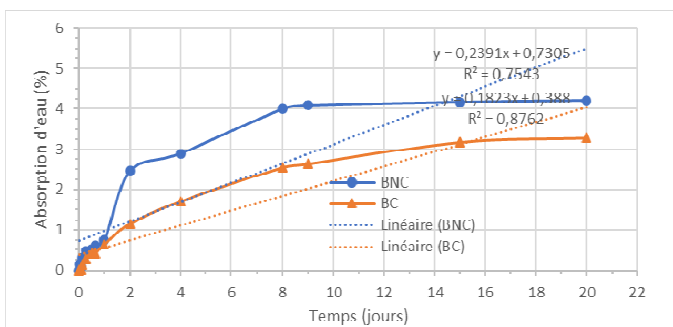


Figure 6. Water absorption kinetics for carbonated and non-carbonated concrete

Figure 6 shows that all concretes' capillary absorption kinetics rapidly increase after the first hour of testing, particularly the non-carbonated concrete. The increase in concrete absorption is similar for up to one day. After one day, however, the kinetics of the two concretes diverge. The kinetics of carbonated and non-carbonated concrete differ by more than 1.5%. This is undoubtedly due to porosity. This is because carbonation of the hydrated phases that comprise the cementitious material causes an increase in the volume of the solid phase. The reaction relative to the transformation of one mole of portlandite into one mole of calcium carbonate causes an increase in the volume of the solid phase and, as a result, a decrease in the pore space (by 2 $cm^3.mol^{-1}$ for calcite), which

is consistent with the work of certain authors [51]. Above all, quarry sand contains a significant proportion of the fines generated by jaw crushers. Other authors attribute this fall to a high portlandite content, which also tends to maximize the porosity decrease [52]. Absorption kinetics is defined as variations in the amount of water absorbed per unit area as a function of time. Figure 8 shows a decreasing slope, indicating that as the test progresses, finer and finer capillaries participate in the water absorption process. Two parameters can be obtained:

The first part of the curve, between 0 and 2 days, represents the filling of the largest pores. These larger pores can be identified based on their initial absorption, which is the amount of water absorbed between 0 and 2 days.

The second, more linear part of the curve represents the finest capillary filling. For example, the slope of a straight line between 1h and 24h can represent the second part. This slope is referred to as "absorptivity". When it comes to determining durability, the most important factor is obviously the initial absorption of the largest capillaries. The water absorption test was chosen to determine the porosity of the embedding concrete and the size of its largest capillaries, both of which are important factors in carbonation durability. They used an existing model to accomplish this, with the criterion being the amount of water absorbed after one hour, which, according to the proposed model, is sufficiently representative of the average radius of the largest capillaries. The results show that the test demonstrates how increasing cement dosage affects the average radius of the largest capillaries in the concrete mix. The same results show that curing has a beneficial effect on reducing the average radius of the largest capillaries. Correlations between carbonated depth and amount of water absorbed after one day demonstrate the test's validity for characterizing concrete's resistance to carbonization. When concrete or mortar is wetted with water, the early absorption rate is proportional to the square root of time. In general, the more porous the material, the better the absorption.

Conclusion

- The findings indicate that the carbonation of quarry sand causes significant changes in its physical and mineral properties over time. The findings indicate that carbonation causes mineral changes such as portlandite dissolution, CSH decalcification, and calcium carbonate precipitation. Three types of calcium carbonate have been identified. The results show that carbonation of the portlandite is the dominant process, with calcite precipitating primarily. As carbonation progresses, portlandite disappears and CSH carbonation takes over, resulting in the precipitation of less stable $CaCO_3$ polymorphs (aragonite, vaterite). Carbonation of CSH polymerizes their structure and is the primary cause of carbonation removal, promoting fissuration.
- Carbonation has the effect of decreasing the water absorption coefficient. Carbonation reduces water absorption in granules, improves mechanical properties, and increases resistance to diffusion of external substances. A decrease in water and pore surface area leads to mass loss. This demonstrates the extensive chemical and mechanical changes that occur in concrete during carbonation. When river sand is replaced by quarry sand in the concrete, the physical and microstructural properties

remain nearly identical. Despite their high fineness module, quarry sand base concrete has good compression resistance because intergranular gaps are filled. At times, the carbonation depth is quite high and exceeds the minimum coverage value of the concrete structure. This is due to the accelerated carbonation and hardening conditions, the cumulative effect of the granules, and, most importantly, the use of two adjuvants. The microstructure becomes rigid and resistant to aggressive substances.

- At the material level, the carbonation process is divided into two major stages. The first stage marks the start of carbonation, when the carbonated and non-carbonated zones coexist in the échantillon. During this stage, two opposing mechanisms emerge: pore clogging and fissuration. The experimental data show that these two mechanisms compensate for macroscopical elasticity at the start of carbonation while increasing permeability. The second phase begins once the majority of the segment has been carbonized. At this point, pore clogging and microfissure closure take priority. This is largely due to the position adopted by the aggregates after reconfiguration following testing.
- Thermogravimetric analysis (TGA), the primary method for determining the composition of granules and, in particular, fixed CO₂, enables the laborious verification of a representative sample. It is recommended to use isothermal sequences and low heating speeds to avoid or reduce mass losses caused by successive dissociations and granulat. Aside from granule formation, properties such as water absorption and porosity play an important role in concrete production. However, a number of important properties for the formation of new concretes, such as elastic modulus and diffusion coefficient, cannot be determined directly from granular material.
- Sikament gives the concrete a high plasticity and significant rheology, which improves compaction and durability. The Sika hydrofluoride reacts with the ciment chaux to form complementary crystallizations that obstruct the mortar's capillaries, resulting in hermeticity with the granules' positions during chemical reactions accelerated.

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Declaration of Competing Interest

The authors report no declarations of interest

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