

# Study of the alkali-silica reaction rate of Spanish aggregates. Proposal of a classification based in accelerated mortar bars tests and petrographic parameters

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**ABSTRACT:** The alkali-silica reaction has been studied in depth due to the evolution in the knowledge of the expansive phenomenon. One of its most important aspects is the reaction rate of the aggregates. In Spain, at the early 90s of the 20th century, aggregates were considered almost non-reactive. However, the use of accelerated curing and other environmental factors revealed that there were potentially reactive siliceous aggregates. Nevertheless, there are several siliceous and limestone aggregates with siliceous inclusions that show reactivity over long period. In the present work, open porosity, expansion and petrography with quartz reactivity index have been determined, in 68 siliceous, limestone and dolomitic aggregates, from quarries located in areas with diagnostic reactivity. Based on these parameters and their interrelation, a classification method is proposed to detect slow-reacting aggregates.

**KEYWORDS:** Alkali-silica reaction; Spanish aggregates; Reaction rate; Mortar bar test; Petrography; Quartz reactivity index.

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**RESUMEN:** *Estudio de la velocidad de reacción álcali-silíce de áridos españoles. Propuesta de clasificación basada en el ensayo acelerado de barras de mortero y en parámetros petrográficos.* La reacción árido-álcali es un fenómeno cada vez más estudiado en profundidad debido a la evolución en el conocimiento del fenómeno expansivo. Uno de los aspectos que tiene mayor importancia es la velocidad de reacción de los áridos. En España, a principio de los años 90 del siglo XX se consideraba que los áridos eran prácticamente no reactivos. No obstante, la utilización de curados acelerados y otros factores ambientales pusieron de manifiesto que había áridos silíceos potencialmente reactivos. Sin embargo, hay distintos áridos silíceos y calizos con inclusiones silíceas que muestran reactividad en largos periodos de tiempo. En el presente trabajo se ha determinado porosidad abierta, expansión y petrografía con índice de reactividad de cuarzos, en 68 áridos silíceos, calizos y dolomíticos, de canteras situadas en zonas con reactividad diagnóstica. A partir de estos parámetros y la interrelación de los mismos se propone un método de clasificación para detectar áridos de reacción lenta.

**PALABRAS CLAVE:** Reacción álcali-silíce; Áridos españoles; Velocidad de reacción; Ensayo acelerado de barras de mortero; Petrografía; Índice de reactividad del cuarzo.

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## 1. INTRODUCTION

Concrete is considered a durable material. However, environmental exposure, loads and the passage of time can cause material deterioration. According to the ACI Durability Guide (1), one of the main causes of deterioration of concrete is the alkali-aggregate reaction. Until the 1990s it was considered that in Spain these aggregates were not potentially reactive and that there was no deterioration due to this expansive process, except for some dams with granitic aggregate (2, 3). However, from the 90s of the 20th century, and mainly linked to accelerated curing in precast pieces, cases of significant deterioration in different concretes began to be identified in Spain (4, 5). The late recognition of concrete with an alkali-aggregate reaction is due to the fact that Spanish aggregates are mostly slow reacting, a concept that had not been developed until then. The concept of reaction rate is introduced later, although it must be taken into account that aggregates have a strong local component (6).

The petrographic analysis of thin sections, with polarized light with crossed Nicols, allows characterizing the reactivity of the aggregates. The quartz grains are analyzed to observe those that show undulatory extinction (reactive due to their high density of dislocations) or straight extinction of light or dark color (non-reactive). Bragg (7, 8), developed a petrographic method to define the reaction rate of aggregates as a function of the quantity of quartz particles with undulatory extinction: < 1% non-reactive (very slow), 1-10% slight ratio (tendency to non-reactive), > 10% and < 20% moderate ratio (tendency to reactive) and > 20% fast ratio (reactive). For its part, B. Wigum (9), considers that the volume of highly reactive particles required to produce the expansion is very small, although the required amount of reactive particles to produce expansion in slow reacting aggregates cannot be established. According to Lagerblad and Trägård, and Wigum (10, 11) there is no pessimum effect for slow-reacting rocks, and 100% reactive particles may be required.

Petrographic analysis has been mainly used to analyze the potential reactivity of aggregates, based on the number of reactive particles. However, in general, petrography are usually only descriptive if they are not performed by petrologists with enough experience in ASR (alkali-silica reaction). For this reason, most of the ASR regulations require the analysis of the potential expansion of specimens in mortars or concrete. In addition, petrographic analysis is the most widely used method based on the ASTM C1260 (12) standard (UNE 146508 (13) in Spain). A comparative summary between different petrographic analysis standards and expansion-based test methods is described in (14, 15).

On the other hand, Bragg and Foster (16) investigated the relationship between the petrographic

examination and the results of accelerated test of mortar bars (12). Aggregates with alkali-reactive minerals between 15% and 40% were rated as good, tending to be reactive, while those with over 40% alkali-reactive rocks were rated as highly reactive. The agreement between petrography and accelerated mortar bar testing was found to be 83%, although this correlation is questionable for slow reactive aggregates.

Jensen (17) suggests a classification of reactive minerals and rock constituents, divided into three groups according to their reactivity (very fast, fast and slow), based mainly on the lists in table A.1.2 and table A.1.3 of the recommendation of RILEM AAR-1.1 (18). For its part, Jensen, 2012 (17), proposes the inclusion of an additional category of highly reactive aggregates, which would include fine dolomite crystals and expansive clay minerals, while granite aggregates are included in the slow reactions group, where structural effects may appear 10 years after the completion of a construction project.

G. Neto *et al.* (19) have carried out several studies trying to link the potential reactivity of aggregates with the dissolution of the silica of different aggregates (20, 21). He classified them into two groups, according to the reaction kinetics. The first group consists of vitreous or amorphous minerals such as volcanic glass and opal, in which the reaction develops very quickly. In the second group, which includes deformed crystalline minerals such as quartz deformed by tectonic processes, the reactions and expansions are slow (20, 21).

Granitic aggregates are widely used in concrete structures around the world and they can show different responses in terms of alkali-aggregate reaction, although they are generally considered slow-reacting (22, 23).

According to Jensen, Alaejos and Lanza (17, 24), the deformation of quartz with the development of subgrains and the presence of microcrystalline and cryptocrystalline phases, provide important characteristics for the evaluation in petrographic analysis. For its part, Wigum (9, 11) studied deformed granite rocks, observing that the variables having the greatest influence on expansion are the presence of quartz subgrains, the total surface of the grain boundary and the size of the grains.

Tiecher *et al.* (25) studied the dissolution of three Brazilian aggregates (granite, mylonite and quartzite) with different degrees of deformation. Grains with higher deformation (quartzite and mylonites), with marked deformation bands and undulatory extinction, dissolve more easily and produce greater expansions in the mortar bar test, compared to granite, which had a higher content of recrystallized quartz subgrains. Based on these results, it is considered that there is no conclusive proof regarding the influence of the aggregate size on

the reactivity of quartz and quartz subgrains with smaller dimensions. Dolar-Mantuani, Sims *et al.* and Grattan-Bellew (26, 27, 28) also analyzed petrographically different species of SiO<sub>2</sub> and they related, qualitatively, the state of the quartz lattice with its reactivity, taking into account the undulatory extinction angles of quartz.

Prendes *et al.*, and Menéndez *et al.* (29, 30), quantitatively analyzed the external perimeter of grain and the internal perimeters of contact between subgrains with deformation, defining an index of the reactivity of the quartzs ( $I_{QR}$ ). This is a dimensionless index with values between 0 and 1, being the value to start considering reactivity  $< 0.39$ , with  $I_{QR}$  values closer to 0 being more reactive.

Most of the European and international regulations base the classification of the aggregates, with respect to the alkali-aggregate reactivity, in standards and procedures of petrographic characterization and in accelerated tests of mortar bars and concrete prisms. The standards used in Spain are: UNE-EN 932-3, UNE-EN 932-3/A1, UNE 146508, UNE 146509, UNE 83967, UNE 83968 and UNE 83969 (13, 31-35). On the other hand, the prevention strategies for ASR applied in France, North America and Australia are collected in the Monograph N° 430 of the CSIC (36), in addition to the recommendations of the Spanish EHE (37) and a proposal for a comprehensive prevention strategy for AAR. This proposal is based on a specific characterization of the concrete components and the test of real concrete mixtures, using semi-accelerated expansion methods (36).

Velasco-Torres *et al.* (38) carried out a comparative study of two granitic rocks extracted from two dams affected by alkali-silica reaction, with slow and fast reactions, respectively, classifying the reactivity of the rocks according to their geological term. They concluded that the reaction can be slow or fast for any given type of rock depending on its components and/or its microstructural characteristics. They also concluded that, for the time being, there are no sufficiently reliable methods to evaluate the reactivity of slow reacting aggregates, which are the most frequent in Spain.

In fast-reacting aggregates, the attack by the concrete pore solution begins to dissolve the microcrystalline quartz zones by contact with the cement paste. This generates a large amount of gel, which accumulates in the surrounding paste in a short period. In addition, there is also a slow reaction, caused by deformed or microcracked quartz. On the other hand, in slow-reacting aggregates, the concrete pore solution slowly enters the aggregate, mainly through microcracks and, to a lesser extent, through subgrain boundaries. The expansive gel fills the fissure system, giving rise to a silica solution.

Rocker *et al.* (39), indicate that the term slow reacting aggregates has been introduced since the 90s and it is widely used throughout the literature

(40-49). According to Shayan (43), it is important to apply a reliable alkali-silica reaction test method, to provide the expansion limit for the classification of aggregates as “non-reactive”, “slowly reactive” or “reactive”. This classification was considered necessary due to the large number of cases of AAR observed over time, due to have been great damages in significant structures in Australia, because of use of aggregates with slow reaction rate by the presence of meta-basalts or granite gneisses (49).

Another suggested method is RILEM AAR-4 (50), which allows identifying the reactivity of slow-reacting aggregates (51).

The Australian standard AS 1141.60.1 (52) applies new limits to detect slow reacting aggregates, replacing the classification of “uncertain reactivity” with that of “slow reaction” in the accelerated expansion test of mortar bars. The results of the tests showed that the proposed limits could distinguish between harmless aggregates and slow reaction. This standard classifies aggregates as non-reactive ( $< 0.10\%$  at 21 days), slow reactive ( $< 0.10\%$  at 10 days and  $< 0.30\%$  at 21 days) and reactive ( $\geq 0.10\%$  at 10 days and  $\geq 0.30\%$  at 21 days), using the accelerated mortar bar method. On the other hand, the UNE 146508 (13) has the following limits: at 14 days ( $\leq 0.10\%$  non-reactive and  $\geq 0.20\%$  reactive) and 28 days ( $\leq 0.20\%$  non-reactive and  $\geq 0.20\%$  reactive); and between 14 and 28 days is doubtful with expansion between  $> 0.10\%$  and  $< 0.20\%$ .

In the present work, 68 Spanish aggregates and sands, mainly siliceous, have been analyzed. These aggregates come from quarries in operation and have mainly caused problems due to alkali-silica reactivity, this damage appearing as early as one year after the concrete manufacturing and up to 50 years. This is not only due to the reaction rate of the aggregates, but also to the type of curing, the environmental conditions of exposure and the geometry and volume of the concrete structure.

A petrographic characterization has been carried out and the reactivity index of the quartz has been determined. The open porosity has also been determined and accelerated expansion tests of mortar bars, extended in time up to one year, have been carried out.

With these parameters, the reaction speed of the aggregates has been analyzed and a reaction speed classification for Spanish aggregates has been proposed, based on the accelerated mortar bar test.

## 2. MATERIALS AND METHODS

### 2.1 Materials and localization

As it is mentioned before, the materials tested are 68 Spanish aggregates, mainly from quarries sited

in areas of siliceous soils. In addition, most of the aggregates are siliceous gravels and sands. The denomination and properties of the samples are collected in Table 1.

These aggregates are mainly located in the siliceous part of the soil of Spain. Their location is represented in the lithological map of the Peninsula Ibérica (Iberian Peninsula) (Figure 1).

A Portland cement CEM I-42.5 R was used to prepare mortars, following UNE-EN 197-1 and UNE-EN 196-2 standards (54, 55). The chemical composition of this cement, expressed as the most stable oxide, is shown in Table 2.

Mineralogical characterization was performed by X-ray diffraction (XRD). Thus, the crystalline compounds present in the cement were determined

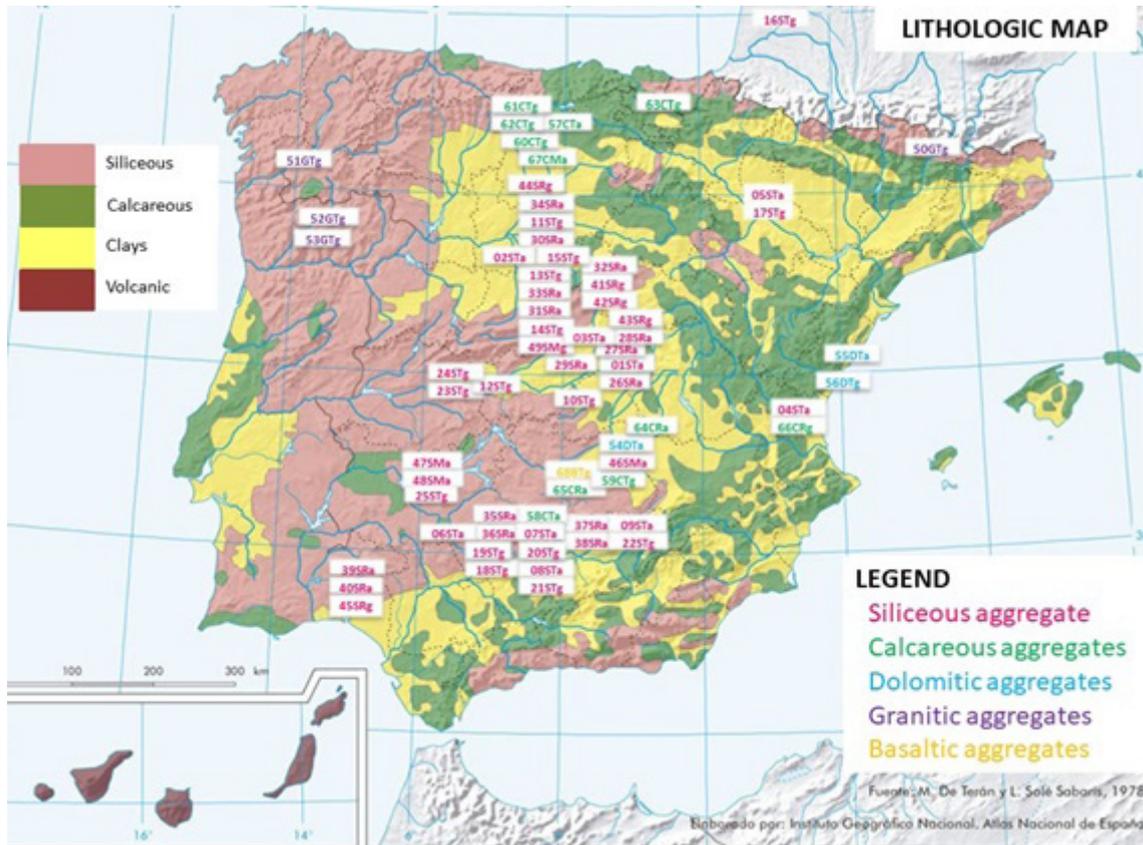


FIGURE 1. Location of aggregates in the lithological map of the Atlas Nacional de España (Spanish National Atlas) (53).

TABLE 1. Denomination of aggregates and their properties.

Denomination	Nº of order	Nature of aggregate (*)	Preparation type	Size of aggregate
00-X-Y-z	1 to 49	Siliceous (red)	S Cracking	T Sand
	50 to 53	Granitic (blue)	G Rolling	R Gravel
	54 to 56	Dolomitic (cyan)	D Mix	M
	57 to 67	Limestone (green)	C	
	68	Basaltic (orange)	B	

(\*) Code of colors in Figures, except in Figure 1

TABLE 2. Chemical composition of Portland cement CEM-I 42.5R.

Component	LOI <sup>1</sup>	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	SO <sub>3</sub>	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	Na <sub>2</sub> O <sub>eq</sub> <sup>1</sup>
Mass (%)	3.18	18.81	5.15	3.18	63.70	2.69	1.50	1.02	0.19	0.86

<sup>1</sup> LOI: Loss of ignition; Na<sub>2</sub>O<sub>eq</sub> = Na<sub>2</sub>O + 0.658 K<sub>2</sub>O

by XRD. Diffraction data of the cement were recorded using a D8 Advance powder crystal X-ray diffractometer (Bruker) with 2.2 kV Cu anode ceramic X-ray tube. Crystalline compounds were identified with the DIFFRAC.EVA v4.2.1 software, which supports a reference pattern database, derived from the Crystallography Open Database (COD) for phase identification. The semi quantitative analysis of cement showed Alite ( $C_3S$ ), Belite ( $C_2S$ ) and Brownmillerite ( $C_4AF$ ) as major crystal compounds and Gypsum ( $CaSO_4 \cdot 2H_2O$ ) as a minor compound. All of them are characteristic components of a Portland cement.

## 2.2. Test methods

### 2.2.1. Open porosity

The open porosity (accessible to water) and the density were determined following the standard process UNE 83980:2014 (56) and calculated according to Equation [1]:

$$\text{Open porosity, \%} = \frac{m_2 - m_1}{m_2 - m_3} \cdot 100 \quad [1]$$

where  $m_1$  is the weight of the sample after drying ( $110^\circ\text{C} \pm 5^\circ\text{C}$  for 24h),  $m_2$  is the weight of the sample after vacuum conditions and  $m_3$  is the apparent weight of the mortar sample (hydrostatic weight, i.e. underwater weighing).

This analysis allows measuring the relative quantity of aggressive entry in the different aggregates analyzed.

### 2.2.2. Petrography

The aggregates were observed by optical microscopy using 25  $\mu\text{m}$  thick sections. These sections were prepared in the Instituto Geominero of Spain. The analysis was done by means of polarized microscopy in transmission mode using an OLYMPUS BX51 microscope, with objectives 4x, 10x, 20x and 40x. The treatment of the photographs was done using the software Analysis docu (Olympus Soft Imaging Solutions GmbH).

The petrographic analysis is used mainly to identify the mineral phases and their morphology. In the case of the alkali-silica reaction it is possible to analyze the presence characteristics of reactive minerals, for instance, the undulatory extinction of the quartz and the degree of it. The quartz, depending on its crystallographic characteristics, may contribute with siloxane groups to the interstitial solution, favoring the nucleation of neo-formed phases and the process of alkali-silica reaction.

With respect to the classification of reactivity of the aggregates the criteria of the RILEM Recommendation AAR-1.1 (18) is used. The potential reactivity of the aggregate is classified in terms of alkaline reactivity due to the presence of mineralogical phases potentially reactive. The classification is based in three classes:

- Class I: Unlikely reactivity.
- Class II: Uncertain reactivity. The aggregate cannot be classified directly in class I or III.
- Class III: Reactivity very likely.

### 2.2.3. Quartz reactivity index

The quartz reactivity index is a dimensionless parameter that is defined as ratio between the quartz perimeter and the total perimeter of quartz sub-grains. It is usually calculated in quartz with undulatory extinction, Equation [2]:

$$I_{Qr} = \frac{P_{ext}}{\Sigma P_{int}} \quad [2]$$

where,  $I_{Qr}$  is the Index of reactivity of quartz,  $P_{ext}$  is the external perimeter of the quartz grain in  $\mu\text{m}$  and  $\Sigma P_{int}$  is the summation of internal perimeters of the sub-grains of quartz, in  $\mu\text{m}$ .

The petrographic classification was done according with the standard ASTM C294 (57), with a classification more detailed than the standard UNE-EN 932-3 (31). The grains of quartz were analyzed in angles of  $11^\circ$  with crossing Nicols and the images were combined to obtain an image with the borders of sub-grains. Later, the perimeter and the length borders were measured using the program ImageJ (Wayne Rasband, National Institutes of Health,

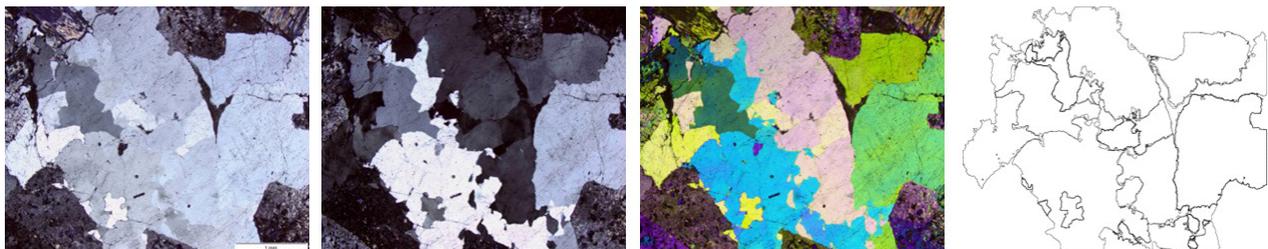


FIGURE 2. Numerical calculation of the IQr, measured with the ImageJ (29, 30).

USA), this software was used to carry out the picture treatment, the extraction of information and the count. Treatment of information was done using Microsoft Excel.

A scheme of the calculation process of  $I_{Qr}$  is shown in Figure 2.

The values of the  $I_{Qr}$  vary between 0 and 1. The qualification of the grains of each grain of quartz could be:

- $I_{Qr} \geq 0,39$ : Non-reactive particles
- $I_{Qr} < 0,39$ : Potential reactive particles. The lowest values are the most reactive.

A representative number of particles of quartz, between 10 and 15, must be analyzed in each aggregate. The average value is the result for  $I_{Qr}$  of an aggregate (29, 30).

#### 2.2.4. Expansion by UNE 146508 (13) similar to ASTM 1260 (12)

The expansion is, along with the petrography, the most frequent parameter to analyse the potential reactivity of the aggregates. In addition, the accelerated mortar bar test is the most used due to the short duration and flexibility.

The alkali-aggregate test method used in this research work is detailed in the Spanish standard UNE 146508 (13), equivalent to ASTM C1260 (12). In preparing the mortar bar specimens, the coarse aggregates were washed, dried ( $105 \text{ }^\circ\text{C} \pm 5 \text{ }^\circ\text{C}$ ), crushed, and sieved into the five fractions (from 0.160 mm to 5 mm), as per the requirement of UNE 146508 (13). Then, the potential reactivity of the aggregates in the mortars was evaluated in three mortar prisms ( $2.5 \times 2.5 \times 28.5 \text{ cm}^3$ ) for each aggregate. They were prepared mixing 400 g of cement (CEM-I 42.5R) and 900 g of aggregate with a water-to-cement ratio (by weight) of 0.47, and the graded aggregates to total cement ratio (by weight) of 2.25. Special moulds were used with a stainless-steel gauge stud into both ends of the longitudinal section of the prism. The effective gauge length was  $254 \pm 2.5 \text{ mm}$ . The mortar test specimens were demoulded after 24 hours, and then, stored immersed in water in closed containers which were placed in an oven maintaining the temperature of  $80 \text{ }^\circ\text{C} \pm 1 \text{ }^\circ\text{C}$  for 24 h. Thereafter, they were removed from the containers for which the zero readings were recorded. Afterward, the prisms were submerged in the 1 N NaOH soak solution at  $80 \text{ }^\circ\text{C} \pm 1 \text{ }^\circ\text{C}$  in plastic containers held in an oven at  $80 \text{ }^\circ\text{C} \pm 1 \text{ }^\circ\text{C}$  for a further fourteen days. Subsequent expansion readings were made from 2 to 14 days in accordance with UNE 146508 (13). However, additional readings were measured over 90-days and 365-days periods instead of the conventional 14-days period prescribed by the standard. Mortar expansion was calculated according to Equation [3], and the average expansion of the

three prisms for each exposure time is given as the mortar expansion result:

$$\text{Mortar bar expansion (\%)} = \frac{(L_n - L_0)}{L_c} \times 100 \quad [3]$$

where  $L_n$  is the length at the testing time,  $L_0$  is the initial length after 24 h of water immersion at  $80 \text{ }^\circ\text{C} \pm 1 \text{ }^\circ\text{C}$ ,  $L_c$  is the calibration length ( $L_c = 254 \text{ mm}$  according to UNE 80113 (58))

The ASR classifications of the aggregates were evaluated based on the 14-day expansion upper limit of mortar bars of 0.10% (non-reactive aggregate), prescribed by the Spanish standard UNE 146508 (13), similar to the ASTM C1260 (12). Expansions over 0.20% indicate a potentially reactive aggregate. Nevertheless, additional 28-day expansion evaluation is made when the 14-day expansion results are between 0.10% and 0.20%. If it is beyond 0.20% at 28 days, the aggregate is considered potentially reactive.

This test methodology is used as a reference to do a classification of the potential reactivity of the aggregates. However, it has the limitation of not detecting the potential reactivity of aggregates with pessimum effect, even for aggregates known to be slowly reactive (e.g. granites). The ASR classifications of the aggregates were evaluated based on the 14-day expansion upper limit of mortar bars of 0.10% (non-reactive aggregate), prescribed by the UNE 146508 standard (13). Expansions over 0.20% indicate a potentially reactive aggregate. Nevertheless, additional 28-day expansion evaluation is made when the 14-day expansion results are between 0.10% and 0.20%. If it is beyond 0.20% at 28 days, the aggregate is considered potentially reactive.

##### 2.2.4.1. Expansion AS 1141.60.1 (52)

The methodology of this test is the same as the previous one, but with a different test time, in this case the duration of the test is 21 days. This standard classifies aggregates as non-reactive ( $< 0.10\%$  at 21 days), slowly reaction ( $< 0.10\%$  at 10 days and  $< 0.30\%$  at 21 days) and reactive ( $\geq 0.10\%$  at 10 days and  $\geq 0.30\%$  at 21 days).

## 3. RESULTS AND DISCUSSION

### 3.1. Open porosity

The open porosity is determined in each of the 68 aggregates, using a representative sample of them. The results are grouped by type of aggregate. Figure 3 presents the dispersion graphic, with the mean value of porosity indicated by a solid point, for each type of aggregate.

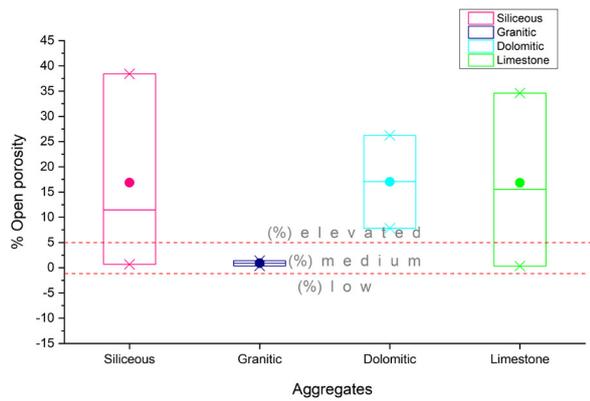


FIGURE 3. Open porosity of each type of aggregates, (basalt isn't included, because there is a single result).

The values of open porosity can be classified as follows: low  $\leq 5\%$ , medium  $> 5\%$  and  $\leq 10\%$  and elevated when a value is  $> 10\%$ , according to the standard UNE 83980:2014 (56).

It should be noted the low porosity of the granites that, although there are few samples, have very little dispersion. This low porosity makes it difficult for aggressive ions to enter the aggregate grains, which justifies the low reaction rate. The basalt isn't repre-

sented in the Figure due to the fact that there is just a single sample, its porosity being 7.9%.

For its part, dolomitic and limestone aggregates have high porosity and, consequently, if they have reactive siliceous particles inside, they will react relatively quickly. With regard to siliceous aggregates, they show a lot of dispersion, although the average value is similar to limestone and dolomites. However, their dispersion can be attributed to the large number of samples of different types and origins.

The relationship between crushed and pebble gravel and silica sand has also been analysed. In principle, crushed gravel and sand should be more porous than pebble sand. However, a clear relationship has not been found in either gravel or sand. This behaviour is attributed to the homogeneity of the crushed aggregates and the different origins of the aggregates.

### 3.2. Petrography and Quartz Reactivity Index

In the petrographic analysis, the typology of the aggregates is characterized and its potentially reactive phases are quantified. In addition, the  $I_{QR}$  of the quartz particles is calculated, classifying these in terms of their reactivity.

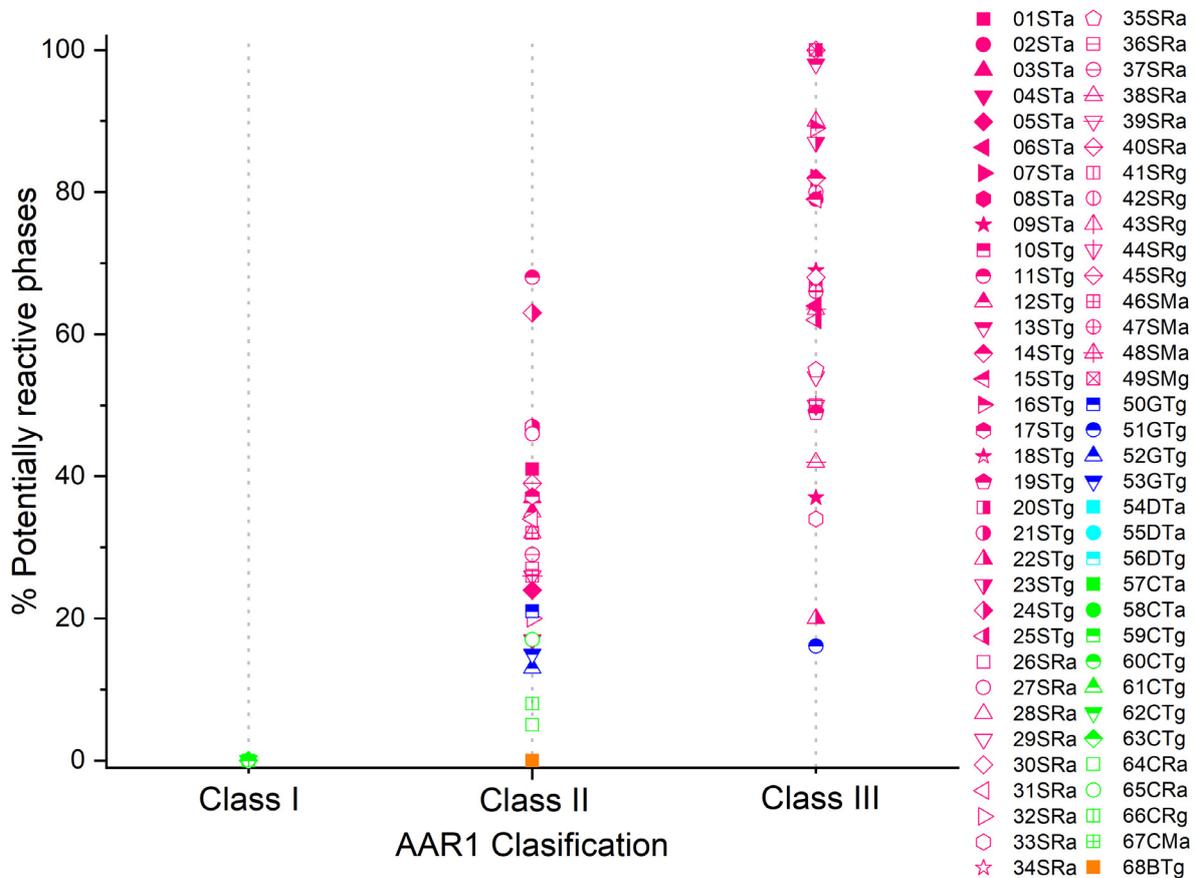


FIGURE 4. % potentially reactive phases versus AAR1.1 classification.

TABLE 3. Petrographic classification of aggregates, according ASTM C295 (59) and RILEM AAR-1.1 (18). Index of reactivity of quartz (Q) and reticular state of them.

Sample	Petrographic classification ASTM C295	Petrographic classification RILEM AAR-1.1	$I_{Qr}$	Reticular state of the Q
01STa	Siliceous sand	Class II-S	0.55	Q with undulatory extinction, Q microcrystalline and cryptocrystalline in chert, clays and others
02STa	Siliceous sand	Class III-S	0.23	Q with undulatory extinction, Q microcrystalline and cryptocrystalline in matrix
03STa	Siliceous sand	Class II-S	0.65	Q stable monocrystalline, Q deformed and chert
04STa	Siliceous sand	Class II-S	0.78	Q with undulatory extinction in low proportion
05STa	Siliceous sand	Class II-S	0.67	Predominant Q stable monocrystalline, Q polycrystalline, Q slightly deformed
06STa	Siliceous sand	Class III-S	0.37	Predominant Q stable monocrystalline, Q polycrystalline, Q slightly deformed
07STa	Siliceous sand	Class III-S	0.31	Predominant Q monocrystalline deformed, Q microcrystalline, cherts and clays
08STa	Siliceous sand	Class II-S	0.46	Predominant Q monocrystalline, some limestone and Q microcrystalline and cherts
09STa	Siliceous sand	Class III-S	0.52	Undulatory extinction Q polycrystalline
10STg	Gravel milonite	Class III-S	0.11	Q with undulatory extinction, Q microcrystalline and cryptocrystalline
11STg	Siliceous gravell	Class III-S	0.21	Undulatory extinction, Q polycrystalline
12STg	Siliceous gravell	Class II-S	0.67	Q deformed in sedimentary rocks
13STg	Opal	Class III-S	0.001	Q without crystalline structure defined and Q cryptocrystalline filling partially holes
14STg	Siliceous gravel	Class III-S	0.36	Undulatory extinction, Q polycrystalline
15STg	Siliceous gravel	Class III-S	0.23	Q with undulatory extinction, Q microcrystalline and cryptocrystalline in matrix
16STg	Calcedony	Class III-S	0.001	Q microcrystalline and cryptocrystalline
17STg	Siliceous gravel	Class II-S	0.57	Q with undulatory extinction in quartzite, y Q microcrystalline y saccharoids
18STg	Siliceous gravel	Class III-S	0.31	Predominant Q polycrystalline microcrystalline and deformed
19STg	Siliceous gravel	Class III-S	0.30	Predominant Q monocrystalline and Q polycrystalline microcrystalline
20STg	Siliceous gravel	Class III-S	0.20	Predominant Q deformed and Q
21STg	Siliceous gravel	Class II-S	0.53	Predominant Q monocrystalline , some limestone, Q microcrystalline, cherts
22STg	Siliceous gravel	Class III-S	0.55	Q with undulatory extinction in quartzite, fragments of chalcedony high reactive
23STg	Siliceous gravel	Class III-S	0.30	Undulatory extinction Q and Q microcrystalline
24STg	Siliceous gravel	Class II-S	0.47	Undulatory extinction Q with Q microcrystalline
25STg	Siliceous gravel	Class III-S	0.39	Undulatory extinction Q with Q microcrystalline and cryptocrystalline in cherts, clays and others
26SRa	Siliceous sand	Class II-S	0.69	Q with strength extinction and some with undulatory extinction
27SRa	Siliceous sand	Class II-S	0.56	Predominant Q stable monocrystalline, and some Q deformed and chert
28SRa	Siliceous sand	Class II-S	0.61	Predominant Q stable monocrystalline, and some Q deformed and chert
29SRa	Siliceous sand	Class III-S	0.47	Q stable monocrystalline, with frequent Q deformed
30SRa	Siliceous sand	Class III-S	0.21	Undulatory extinction Q in polycrystalline aggregates
31SRa	Siliceous sand	Class II-S	0.63	Predominant Q stable monocrystalline, some Q deformed and chert
32SRa	Siliceous sand	Class II-S	0.73	Predominant Q stable monocrystalline, and Q deformed in polycrystalline particles and some chert

Sample	Petrographic classification ASTM C295	Petrographic classification RILEM AAR-1.1	$I_{Or}$	Reticular state of the Q
33SRa	Siliceous sand	Class III-S	0.65	Predominant Q stable monocrystalline, and deformed Q and chert
34SRa	Siliceous sand	Class III-S	0.41	Q with undulatory extinction, Q cryptocrystalline in cherts, clays and others
35SRa	Siliceous sand	Class III-S	0.34	Q with undulatory extinction, Q microcrystalline and saccharoids in cherts, clays and others
36SRa	Siliceous sand	Class III-S	0.38	Predominant Q monocrystalline and Q polycrystalline, with particles chert
37SRa	Siliceous sand	Class II-S	0.62	Q stable and undulatory extinction in polycrystalline Q
38SRa	Siliceous sand	Class III-S	0.49	Q stables, undulatory extinction Q in polycrystalline
39SRa	Siliceous sand	Class II-S	0.71	Predominant Q stable monocrystalline, and some Q deformed in polycrystalline
40SRa	Siliceous sand	Class II-S	0.61	Predominant Q stable monocrystalline, with some Q deformed in polycrystalline and chert
41SRg	Siliceous gravel	Class II-S	0.64	Q polycrystalline, Q stable, others polycrystalline with undulatory extinction and Q microcrystalline
42SRg	Siliceous gravel	Class III-S	0.36	Undulatory extinction Q, Q microcrystalline and cryptocrystalline in cherts, clays and others
43SRg	Siliceous gravel	Class III-S	0.34	Undulatory extinction Q, Q microcrystalline and cryptocrystalline in cherts, clays and others
44SRg	Siliceous gravel	Class III-S	0.46	Undulatory extinction Q, Q cryptocrystalline in cherts, clays and others
45SRg	Siliceous gravel	Class III-S	0.51	Q polycrystalline with stable Q stables, others polycrystalline with undulatory extinction and Q microcrystalline
46SMa	Siliceous sand	Class II-S	0.62	Q with straight extinction, and some with undulatory extinction
47SMa	Siliceous sand	Class III-S	0.39	Undulatory extinction Q, Q microcrystalline and cryptocrystalline in cherts, clays and others
48SMa	Siliceous sand	Class III-S	0.37	Undulatory extinction Q, Q microcrystalline and cryptocrystalline in cherts, clays and others
49SMg	Siliceous gravel	Class III-S	0.36	Undulatory extinction Q in polycrystalline
50GTg	Granodiorite	Class II-S	0.41	Q deformed with ramified cracks and borders with ramified cracks and with sutured edges
51GTg	Granite	Class III-S	0.37	Crystals micro granular, cracking saccharoids
52GTg	Granodiorite	Class II-S	0.52	Micro cracking and cracks intergranular
53GTg	Granodiorite	Class II-S	0.46	Micro cracking and cracks intergranular
54DTa	Calcite dolomite	Class I	0.90	Low proportion of Q with straight extinction
55DTa	Dolomitic sand	Class I	0.90	Low proportion of Q with straight extinction
56DTg	Dolomite gravel	Class I	0.90	Low proportion of Q with straight extinction
57CTa	Calcite sand	Class I	0.90	Q with straight extinction in low proportion
58CTa	Calcite sand	Class I	1.00	Q it isn't observed
59CTg	Limestone	Class I	0.90	Q with straight extinction in low proportion
60CTg	Calcite gravel	Class I	1.00	Don't have Q
61CTg	Calcite gravel	Class I	1.00	It isn't observed Q deformed
62CTg	Calcite sand	Class I	1.00	It isn't observed Q deformed
63CTg	Calcite sand	Class I	0.89	Microgranular crystals with straight extinction
64CRa	Calcite sand	Class II-S	0.79	Undulatory extinction Q, Q microcrystalline in very low proportion
65CRa	Calcite sand	Class II-S	0.64	Undulatory extinction Q in very low proportion
66CRg	Calcite gravel	Class II-S	0.54	Undulatory extinction Q in very low proportion
67CMA	Calcite sand	Class I	0.90	Q with straight extinction in low proportion
68BTg	Basalt	Class II-S (*)	0.90	Q with straight extinction in low proportion

(\*) Basaltic rocks have small crystals of quartz, and this affect to the classification of RILEM AAR-1.1

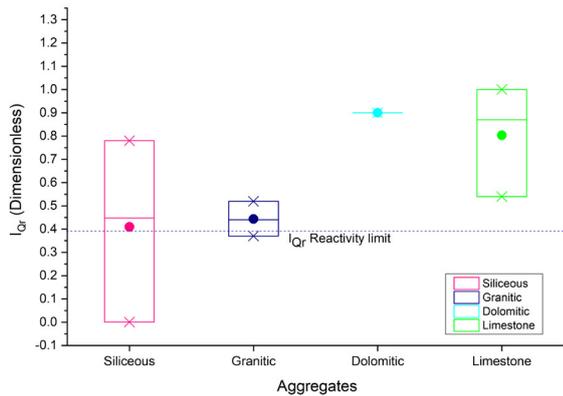


FIGURE 5. Reactive quartz index (IQR) of the aggregates (Basalt isn't included because there is a single result).

Table 3 shows the petrographic classification of aggregates, according to ASTM C295 (59) and RILEM AAR-1.1 (18). In addition, the  $I_{QR}$  values and the reticular state of the quartz are collected.

Most of the gravels and siliceous sands have quartz particles with undulatory extinction and many of them show  $I_{QR}$  values lower than 0.4%. This implies a high reactivity. The granites show a moderate presence of reactive phases and  $I_{QR}$  values at the limit of reactivity. This corroborates the slow reaction ki-

netics of this type of aggregate and the large volume of concrete necessary for the alteration evidence to be significant.

Regarding the classification of RILEM AAR-1.1 recommendation, all siliceous aggregates are classified as potentially reactive (Class III) or as uncertain reactivity (Class II). However, limestone, dolomite and several granites that have a low percentage of potentially reactive phases are also classified as uncertain reactivity. These results indicate that this classification method is not very precise since it only detects one of the limestone aggregates as non-reactive. Although, in practice, the limestone aggregates have not been found to have expansion, while all the granites tested presented alkali-silica reaction in field concrete structures (mainly dams and bridges).

Figure 4 shows the classification of aggregates according to the classification of the RILEM AAR1.1 recommendation, the percentage of reactive phases analyzed by petrography and the relationship between both methods.

It is observed that the classification of the RILEM recommendation AAR1.1 is capable of detecting potentially reactive aggregates (Class III) quite rigorously. However, many of the aggregates that have a high percentage of potentially reactive particles are classified as doubtful, although, in

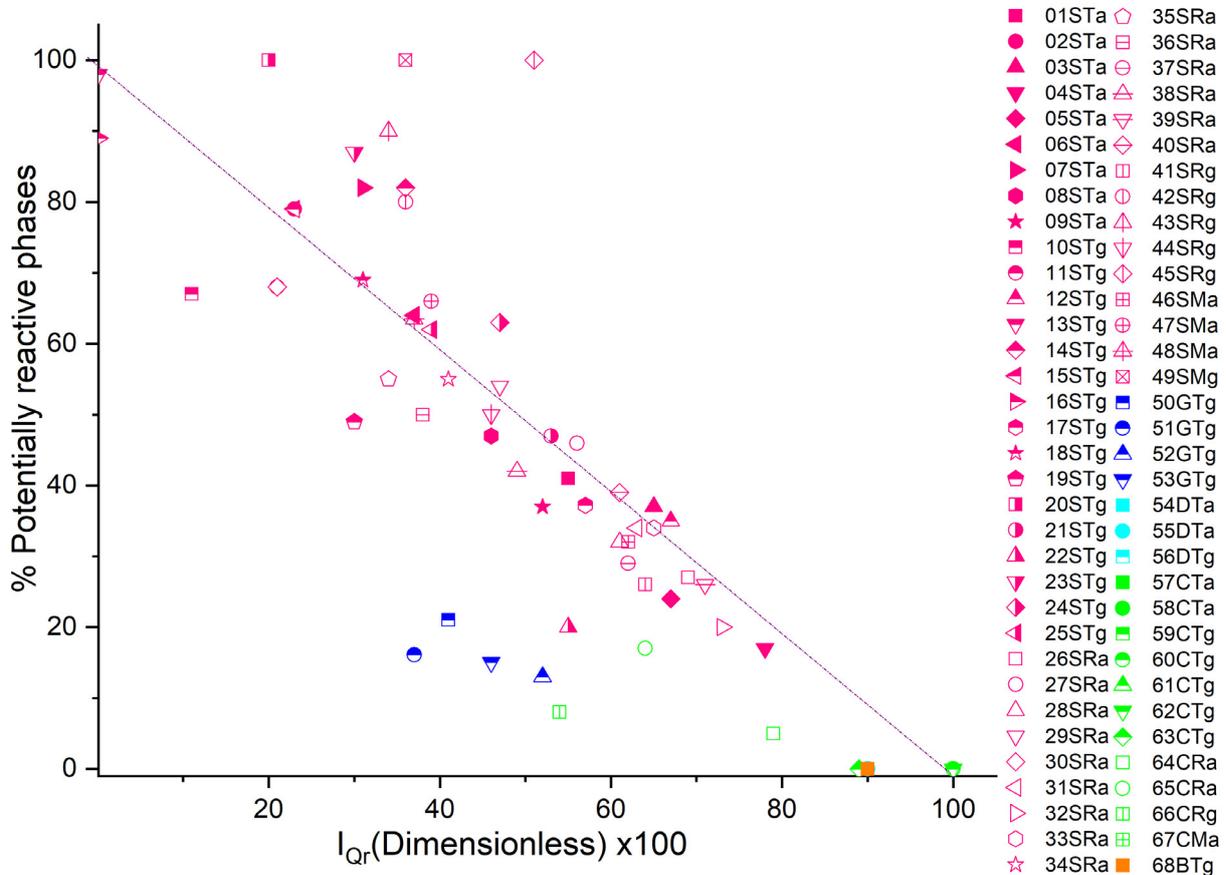


FIGURE 6. % potentially reactive phases versus reactivity index of aggregates.

practice, these aggregates show reactivity in construction sites (such as granites and many of the siliceous aggregates).

Regarding the  $I_{Or}$ , it can be seen in Figure 5 that most of the siliceous aggregates would be classified as potentially reactive. However, the dispersion of the results is high, so they could be classified as non-reactive, although they mostly have a slow or very slow reaction rate. For its part, granites have little dispersion, mainly due to the low number of samples and low porosity. In the  $I_{Or}$  classification, the granites would be very close to the reactivity limit, so this parameter ( $I_{Or}$ ) is better for classifying granites than other test methods, although the reactivity range should be extended to 0.55. This would also allow the inclusion of reactive siliceous aggregates that are not classified as such with the limit of 0.39 of the  $I_{Or}$  value. Figure 5 shows the average results and the dispersion of the different types of aggregates analyzed.

If the percentage of reactive phases is represented versus the  $I_{Or}$  parameter (Figure 6), it is verified that, in general, there exists a very good correlation for siliceous, granitic and limestone aggregates, although each type of aggregate shows different slopes. The largest dispersions are produced for siliceous aggregates with a high percentage of potentially reactive phases, since the  $I_{Or}$  will underestimate them.

### 3.3. Expansion

The expansion has been analyzed under the test conditions indicated in the ASTM C1260 (12) standard (similar to the UNE 146508 (13)), although the measurements have been prolonged up to 1 year, in order to analyze the long-term behavior and observe the trend changes in the expansion of aggregates over time.

Figure 7 shows the expansion up to 28 days of testing, as well as the limits to consider aggregates as reactive according to ASTM C1260 (12) and AS 1141.60.1 (52). For its part, Figure 8 shows the expansion results of the aggregates up to 365 days. In the first case, it is observed that only non-reactive aggregates stabilize their expansion before 28 days, while in the 365 days test most of the siliceous and granitic aggregates and some of the limestone continue to expand, with different reaction rates. It is found that granites and limestone with silicon inclusions expand much more slowly than siliceous aggregates.

Observing in detail the expansion up to 365 days, it can be seen that, before the first 150 days of testing, there is a change in trend in the expansion curves of the reactive aggregates. Figure 9 shows the mean value and the dispersion of the number of days elapsed until the change in trend in the expansion curve by type of aggregate. If this change in trend is analyzed for each type of aggregate, it is observed that it occurs around 40 days for siliceous aggregates, around

60 days for granites and over 65 days for limestone aggregates. This is associated with the reaction rate in aggregates, the fastest being siliceous, followed by granites and limestone with quartz inclusions.

If we take a limit of 0.25% expansion at 365 days, to consider the aggregates as potentially reactive, 7 siliceous aggregates, 7 limestone aggregates and all the dolomites and the basalt are qualified as non-reactive. Moreover, 42 siliceous, 4 limestone and the 4 granites aggregates are considered as potential reactive.

Definition of Expansion Rate as a Function of the Porosity, Petrographic Analysis and Quartz Reactive Index ( $I_{Or}$ ) and Accelerate Mortar Bar Test.

The open porosity of each aggregate has been plotted versus the reactivity index of quartz in order to analyse the possible relationship between high porosity and high reactivity, but no significant relationship is observed due to the different nature of the aggregates (Figure 10). It should be noted the high porosity of some siliceous, limestone and dolomitic sands, which is associated with the retention of water between small particles.

In Figure 11, the porosity versus the expansion of each aggregate at 14 days, 28 days and 365 days is represented. In each bar, the data on the left corresponds to the expansion at 14 days; the one in the middle shows the expansion at 28 days and the one on the right the expansion at 365 days.

Although the hydroxyl ions have to be in contact with the aggregate to start the reaction, in the case of siliceous aggregates there is no clear relationship between open porosity and reaction rate over time. However, in granite aggregates, with very low porosity, a slowdown of the reaction is observed due to this characteristic. The limestone aggregates are mostly non-expansive and those that show expansion are due to the inclusions of deformed quartz.

On the other hand, in the case of siliceous aggregates with very low porosity, the alteration of the aggregates occurs from the homogeneous surface, so they initially show low expansion (6, 60); although at 365 days they show a high expansion, like the rest of siliceous aggregates.

Monograph No. 230 of the IETcc-CSIC (36) contains a proposal on the qualification of the reaction speed, defining five groups:

- Non-reactive aggregates (<0.10% at 14 days or > 0.20% at 28 days, according to the limits of ASTM C 1260 and UNE 146528 (12,13)).
- Slow reaction rate aggregates (0.10 to 0.20% at 14 days or between 0.20% and 0.25% at 28 days).
- Moderate reaction aggregates (0.25% to 0.35% between 14 days 28 days).
- Fast reaction aggregates (0.35% to 0.45% between 14 days 28 days)
- Very fast reaction aggregates (>0.45% between 14 days 28 days).

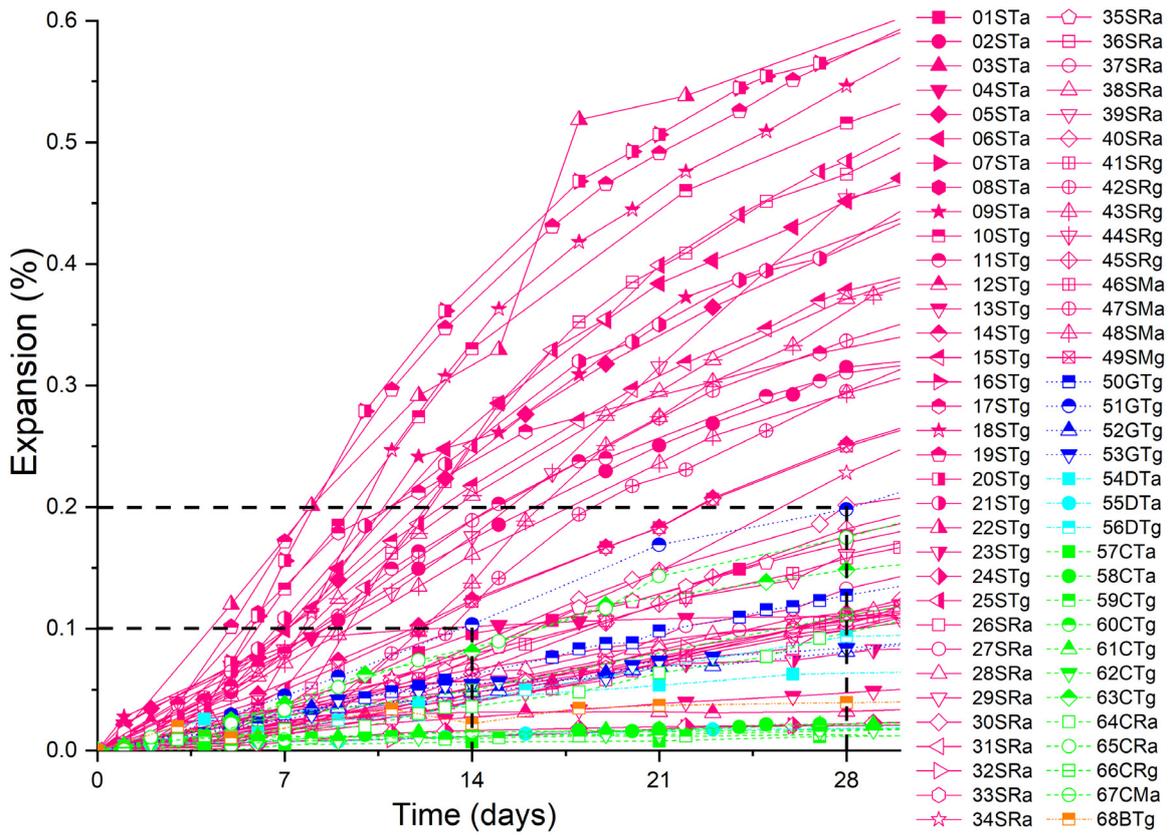


FIGURE 7. Expansion at 28 days.

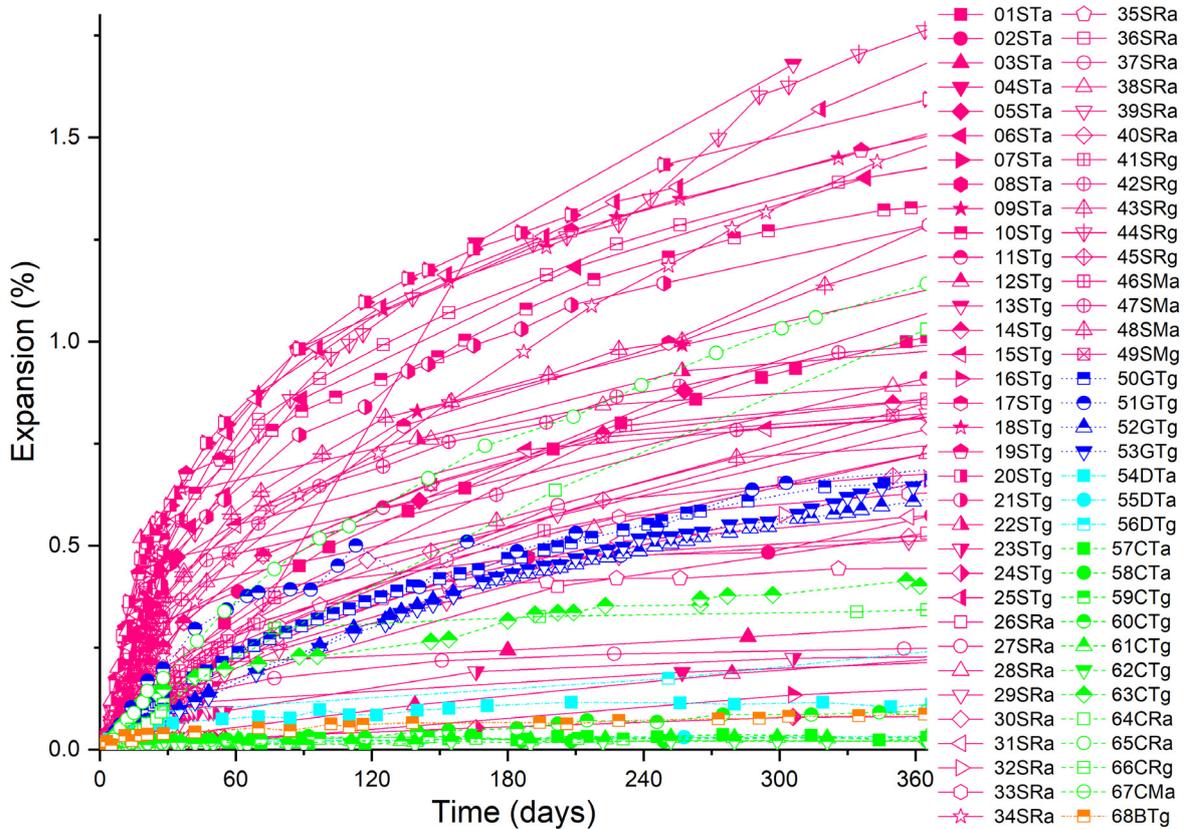


FIGURE 8. Expansion at 365 days.

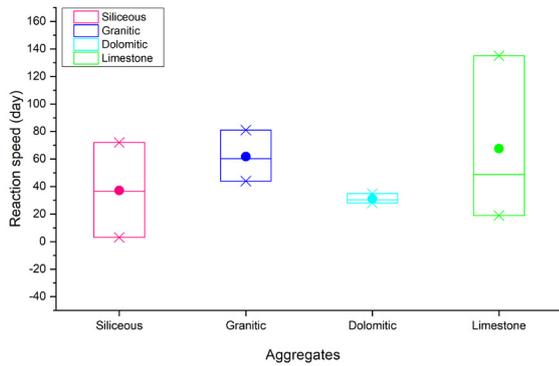


FIGURE 9. Change in reaction speed of each aggregate (basalt isn't included because there is a single result).

The expansion up to 28 days is represented in the scheme with the reaction rate rating described above (Figure 12), and it is found that most of the slow-reacting aggregates cannot be detected with the usual classification of the Standard Test Method for Potential Alkali Reactivity of Aggregates (ASTM C 1260 and UNE 146528 (12,13)). This method would classify as non-reactive all granites, limestones with siliceous inclusions, and more than a quarter of siliceous

aggregates, all of which have shown reactivity on site. The potential reactivity of these aggregates appears with the extension of the test time.

Representing the expansion of each aggregate at 14 days and 28 days versus the amount of quartz multiplied by the reactivity index of this quartz, we can divide the aggregates by their reaction rate into three groups: non-reactive, slowly reaction and reaction fast. This classification is carried out based on the total percentage of reactive quartz ( $I_{Qr} \cdot Q_i$ ) and the expansion value at 14 days and 28 days. The fast reaction would have a value greater than 30% of the total percentage of reactive aggregates and a percentage of expansion at 28 days  $> 0.20\%$ . The slow reaction ones between 5% and 30% and a percentage of expansion at 14 days  $> 0.10\%$ , the non-reactive ones would be those with a total percentage of reactive quartz  $\leq 5\%$  and a 28-day expansion percentage  $< 0.20\%$ .

In Figure 13, the total percentage of reactive quartz ( $I_{Qr} \cdot Q_i$ ) versus the expansion value at 14 days and 28 days is shown. Moreover, in Figure 14 the total percentage of reactive quartz ( $I_{Qr} \cdot Q_i$ ) versus the expansion value at 14 days, 28 days and 365 days is shown. Most of the siliceous aggregates are classified as fast reacting, while granites, some siliceous and limestone with quartz inclusions are classified as slow reacting. For its part, dolomites and most limestone are rated as non-reactive.

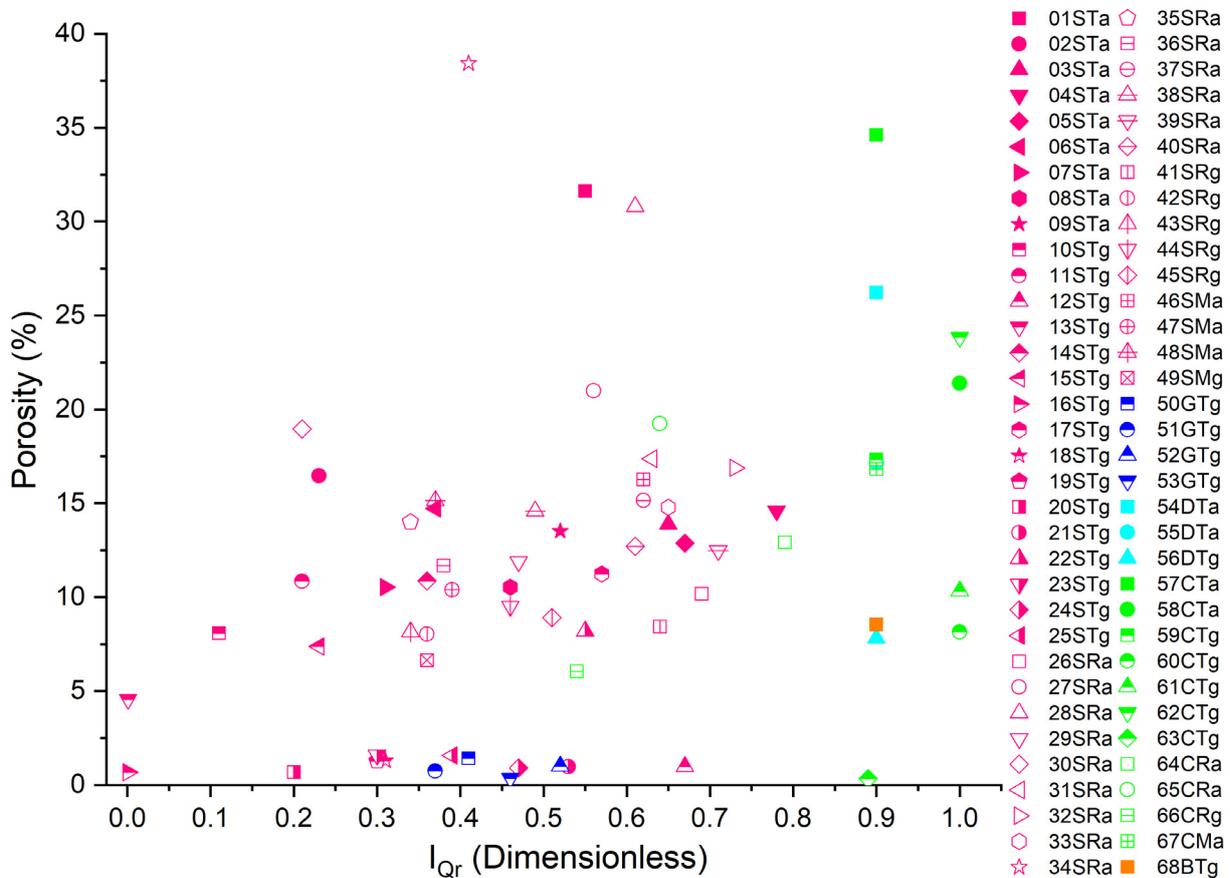


FIGURE 10. Open porosity versus reactivity index of quartz of each aggregate.

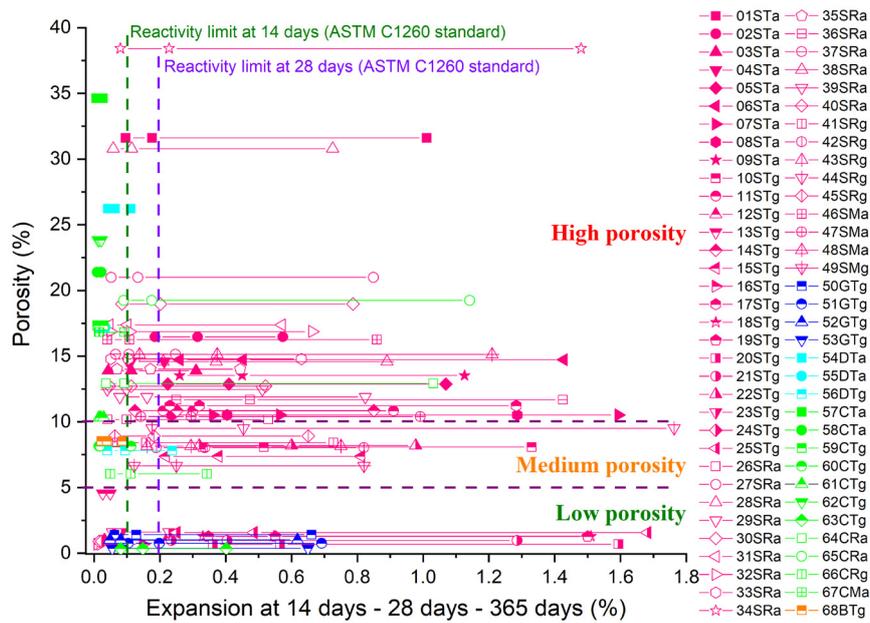


Figura 11. Expansion at 14 days, 28 days and 365 days vs porosity.

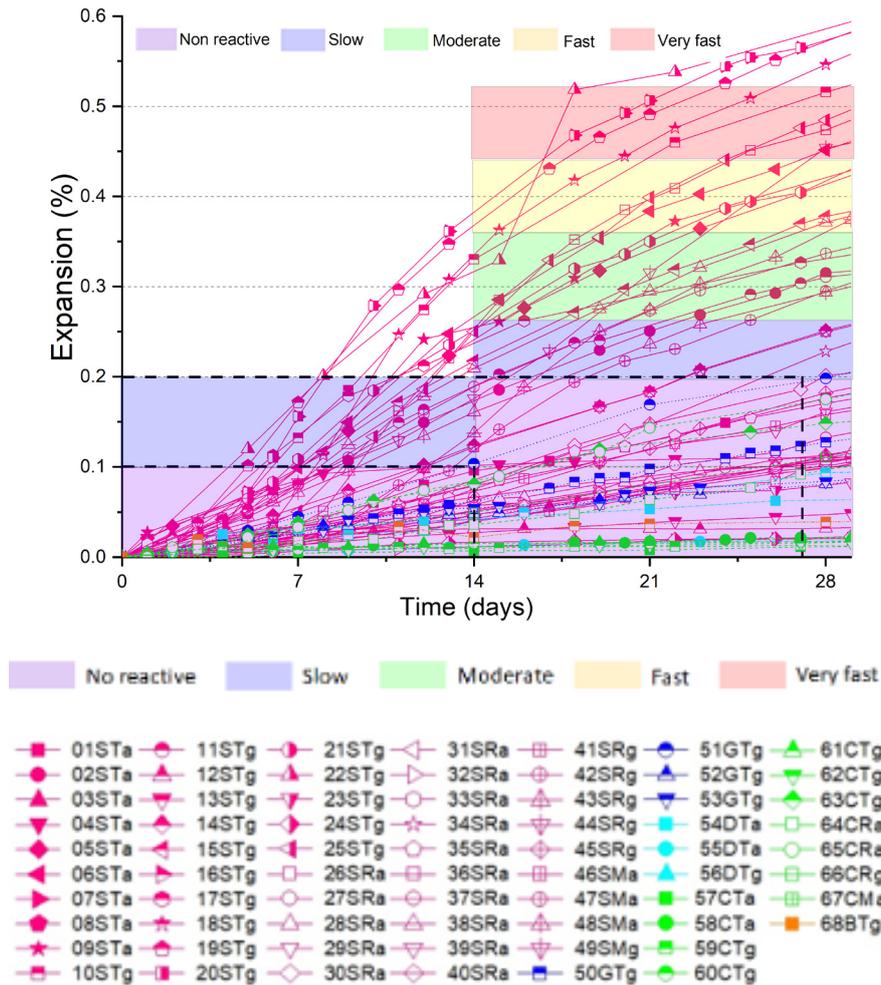


FIGURE 12. Classification of the aggregates according to the proposal of qualification at 14 days and at 28 days.

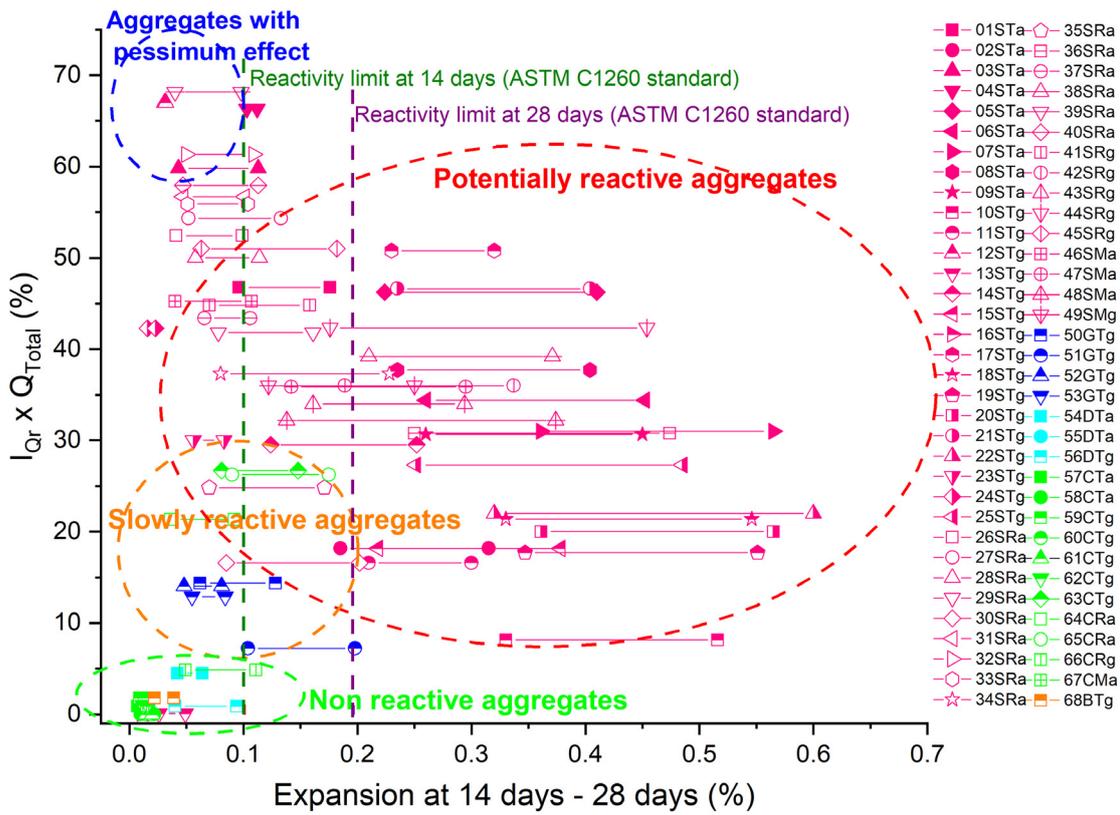


FIGURE 13. Expansion at 14 and 28 days versus IQR of aggregates.

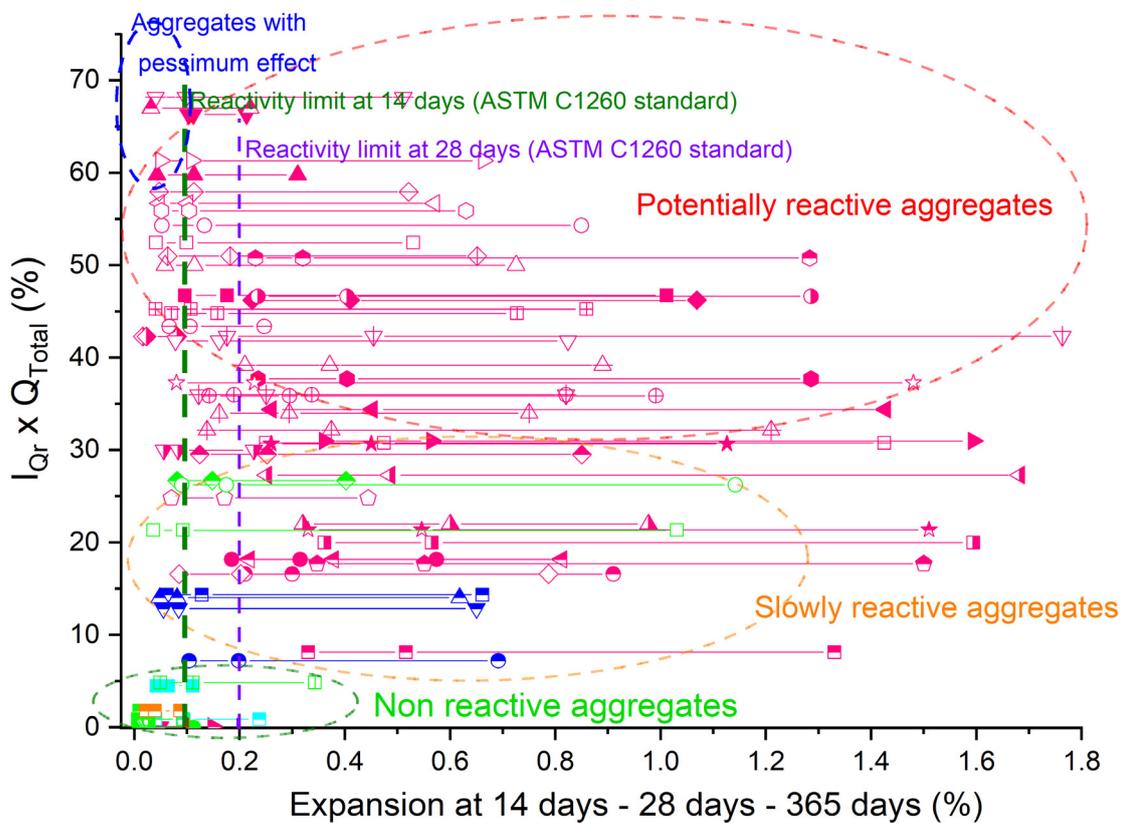


FIGURE 14. Expansion at 14, 28 and 365 days versus IQR of aggregates.

In the case of the Spanish aggregates studied, just 1 of the siliceous aggregates can be considered as non-expansive, while 13 are slow reactive, 31 are considered as potential reactive aggregates and 4 are considered as potential reactive aggregates with pessimum effect. For its part, the 4 granites and 3 of the 11 limestone aggregates are considered as slow reactive aggregates. The limestone aggregates considered as slowly reactive have some reactive particles of quartz in their composition. Finally, neither dolomites nor the basalt can be considered as potential reactive aggregates.

## CONCLUSIONS

The open porosity should have a direct relationship with the potential reactivity of the aggregates. However, as these are aggregates of a different nature, this correlation cannot be observed. However, the low open porosity, as in the case of granites and quite a few siliceous aggregates, show the difficulty of entry of the OH<sup>-</sup> groups that causes the breaking of the siloxane bridges of the deformed quartz.

On the other hand, the expansion extended in time up to 1 year, makes it possible to verify the evolution of this expansiveness, especially for slow-reacting aggregates. However, the extension of these tests to a normative level is not considered, since it would not be feasible to wait this time in the vast majority of concrete works.

The relationship between the percentage of total reactive quartz in an aggregate and the expansion extended to 1 year, allows classifying reactive aggregates, both slow reacting and those classified with the accelerated mortar bar method as potentially reactive. The total reactive quartz of an aggregate ( $I_{Qr} \cdot Q_t$ ) is calculated by multiplying the reactivity index of the quartz by the total quantity of quartz in the concrete aggregate. According to the results obtained in this work, the following classification is proposed:

- Non-reactive:  $I_{Qr} \cdot Q_t \leq 5\%$  and a 28-day expansion  $< 0.20\%$
- Slow reactive:  $I_{Qr} \cdot Q_t$  between 5% and 30% and a expansion at 14-day  $> 0.10\%$
- Fast reactive:  $I_{Qr} \cdot Q_t > 30\%$  and a 28-day expansion  $> 0.20\%$
- Pessimum effect:  $I_{Qr} \cdot Q_t > 60\%$  and a 28-day expansion  $< 0.20\%$

If one of the two conditions is not fulfilled, the total percentage of reactive aggregates ( $I_{Qr} \cdot Q_t$ ) prevails.

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