# PETROGRAPHIC CHARACTERIZATION *VS.* LABORATORY TEST METHODS APPLIED TO GRANITIC AGGREGATES

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**Abstract**

A large number of Portuguese concrete structures were built in the 1960-70's with granitic aggregates, one of the main rock types used in the construction industry. Some of the structures have recently started to exhibit signs of deterioration due to alkali-silica reactions.

It has been verified that the classification of granitic aggregates according to the national standards regarding the petrographic analysis and also to the ultra-accelerated mortar-bar test (ASTM 1260 or RILEM AAR-2) does not always agree with the performance of this type of aggregates in other laboratory tests and in field structures.

A comparison of the results obtained for the first three samples of granitic aggregates according the different methods is presented. An attempt is made to point out the main advantages and limitations of each test method in what concerns granitic rocks.

## 1 INTRODUCTION

It is of upmost importance to define the laboratory tests, following the petrographic analyses, that best express the field performance of aggregates in concrete. A lot of work has been done to achieve this goal with different types of aggregates, but at least in what concerns granitic rocks the results obtained are not totally clarifying. The next paragraphs summarize the studies developed so far.

In the middle of the last century, the first statements that alkali-aggregate reactions in quartz bearing rocks were a consequence of this mineral's defective crystal lattice were made [e.g. 1]. Till then, little importance was given to textural features of rocks used as aggregates. Taking into account the work of DeHills and Corvalán [2] in Chilean granitic rocks, who found it was possible to relate the value of the undulatory extinction angle of quartz crystals, the degree of deformation in their lattices and the intensity of deformation processes, Gogte [3] showed the importance of the petrographic examination of aggregates by concluding that the potential alkali-reactivity of some crystalline rocks was related to the content and straining effects in quartz. Further researches using the measurement of the undulatory extinction angle as a parameter for evaluating the potential alkali-reactivity of aggregates have followed [4]. Meanwhile, the usefulness of the undulatory extinction angle was questioned [e.g. 5-7] and it was pointed out as being a possible indicator rather than a diagnostic feature of alkali-silica reaction (ASR) [8].

Further studies showed that there is a positive correlation between the amount of microcrystalline quartz and expansion tests [e.g. 5-7,9,10]. Kerrick and Hooton [9] and Shayan [10] attributed ASR to the presence of microcrystalline quartz in replacement of deformed quartz and along foliation layers and grain boundaries. A new approach made by Wigum [11], which included the quantification of the average surface

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area of quartz through grain size measurement in cataclastic rocks, made the estimation of the amount of microcrystalline quartz present in the rock possible. Therefore, the alkali-reactivity of those kind of rocks was related to the total grain boundary area of quartz which is strongly influenced by subgrain development [11,12].Taking those facts into account, the recrystallisation of the quartz can explain the decrease in the reactivity of strongly mylonitised rocks [13], since the amalgamation of smaller grains into larger ones leads to a smaller total grain boundary area of quartz [12]. Wenk et al. [7] demonstrated a positive correlation between ASR and dislocation density, playing dislocations a major role in the process. In addition, the comparison of two granitic aggregates from Spanish dams permitted to conclude that granite can develop a rapid or slow alkali-silica reaction and that the main mechanism of formation and storage of gel was associated to microcracks rather than to subgrain boundaries [14].

Throughout time, various attempts to define which expansion tests best express the performance of slowly-reactive aggregates have been made. Batic and Sota [15] demonstrated the effectiveness of the ASTM C 1293 concrete prism test method [16] and its results consistency with field performance in granite aggregates from Argentina. The ASTM C 1260 [17] limits failed to detect slowly-reactive aggregates of granitic nature from Australia which exhibited gneissic features [18]. Also, it was confirmed that RILEM concrete prism test at 60ºC (RILEM AAR-4 [19]) overcomes the deficiencies of RILEM concrete prism test at 38ºC (RILEM AAR-3 [20]) for Australian slowly-reactive aggregates, such as deformed granite, which contains microcrystalline quartz as the reactive component [21]. This fact was confirmed in the European PARTNER Project where the AAR-4 [19] and the RILEM accelerated mortar bar test at 80ºC (RILEM AAR-2 [22]) successfully identified slowly reactive aggregates, although they did not necessarily show that the aggregates would expand slowly [23].

Portuguese granitic rocks have different geological histories which generated different degrees of strain which are directly responsible for some granites' main characteristics (e.g. texture, mineralogy). The granitic rocks referred in this study outcrop in the Central Iberian Zone (CIZ), the internal domain of the Iberian Massif according to the zonal division of Lotze [24], modified by Julivert et al. [25]. The CIZ granitic rocks represent about 60-70% in volume of the outcropping rocks. In this zone, three main ductile Hercynian deformation phases were recognized: D1, D2 and D3 [e.g. 26,27]. The D1 (380-360 Ma) and D2 phases (360-330 Ma) correspond to the collisional stage of the Hercynian orogeny. The last ductile deformation phase (D3) (310-300 Ma, intra-Westephalian in age) is followed by fragile deformation phases. Based on the emplacement ages relatively to the main Hercyniam events, namely D3, the CIZ granitoid magmatism has been classified as: syn-tectonic pre-D3; syn-tectonic syn- to late-D3; late- to post-tectonic [28]. The main characteristics of the different granites were described by several authors [e.g. 28,29].

The awareness of the problems caused by ASR, in Portugal, only began in the early 1990's and, since then, a variety of cases have been investigated [30-33]. At the moment, a national project – IMPROVE (Improvement of performance of aggregates in the inhibition of alkali-aggregate reactions in concrete) is being carried out with the objective to establish the most accurate method for the evaluation of the main types of aggregates in the country, including the granitic ones. This evaluation includes the petrographic examination and the performance of mortar and concrete expansion tests, such as ASTM C 1260 [17], RILEM AAR-3 [20], RILEM AAR-4 [19] as well field performance testing.

In the present work, the first results of this research, concerning granitic aggregates, are presented aiming to contribute to the reformulation of the national recommendation and to establish a possible correlation among the results from different methods.

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## 2    MATERIALS AND METHODS

### 2.1    General

The rich geological and tectonic history of the Iberian Peninsula, were responsible for the wide variety of lithologies in the Portuguese territory. For this reason, several rocks are available for exploitation as aggregates for concrete purposes. Among them, the most used are: granites, limestones and alluvial deposits.

In this study, three types of crushed granitic aggregates, from distinct Portuguese quarries, were studied, aiming their petrographic characterization and their laboratory performance, when applied as aggregates in mortar and concrete.
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### 2.2    Materials and mixture proportions

All the granitic rocks used in this study are Hercynian [29]. To keep the producers' request for confidentiality of the quarries locations, aggregates have been denoted as A, B and C.

Aggregate A is composed of medium to coarse-grained, porphyritic, monzonitic granite. It is a late-tectonic. Aggregate B is composed of late to post-tectonic medium-grained, porphyritic, monzonitic granite. Aggregate C is a syn-tectonic medium to coarse-grained granite.

Hand samples of the three different granites were field sampled to produce thin-sections. Crushed aggregates of various grain sizes from each quarry were collected. Mortar mixes were prepared according to ASTM C 1260 [17] and concrete mixes were prepared according to RILEM AAR-3 [20] and AAR-4.1 [19].

### 2.3    Methods for assessment and analysis

*General*

In order to predict the field performance of the granitic aggregates and to compare results, petrographic characterization and expansion tests, namely ASTM C 1260 [17] accelerated mortar bar test (AMBT), RILEM AAR-3 [20] (CPT 38ºC) and RILEM AAR-4.1 [19] (CPT 60ºC) concrete prism tests were carried out. These two groups of methods are presented next in more detail.
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*Petrographic characterization*

Since each of the studied rocks has homogeneous characteristics in the area exploited in each quarry, representative thin-sections of the aggregate from the three quarries were produced and studied under a Nikon Eclipse E 400 POL polarizing microscope. This procedure is named "whole rock petro" [34]. To complement the petrographic examination with quantitative values, a PELCON automatic point-counter was attached to the polarizing microscope. The point-counting procedure was carried out by traverses in regular increments in two directions to form a virtual orthogonal grid, covering the whole thin-section, in order to identify the mineral directly under the crosshairs. The evaluation was made according to RILEM AAR-1 [35] and to Portuguese specification LNEC E 461 [36]. Similar categories of aggregates are defined by both RILEM AAR-1 [35] and LNEC E 461 [36]: Class I – innocuous (reactive silica < 2%); Class II – ambiguous; Class III – potentially alkali-reactive. The assessment of reactivity to alkalis was based on local experience with granitic aggregates namely with the deformation evidences of the quartz crystals (recrystallized quartz) and on their dimensions (identification of microcrystalline quartz < 100µm [5]). Goticular and myrmekitic quartz were also included in the potentially reactive forms of silica. Following the literature about the subject, the occurrence of undulatory extinction was not considered for the assessment of the potential reactivity.
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*Expansion tests*

For aggregates classified in Class II or III, the assessment is followed by expansion tests, starting with the accelerated mortar-bar test [36].

The aggregates were crushed, sieved and weighed according to the requirements of ASTM C 1260 (80ºC) AMBT [17], RILEM AAR-3 (38ºC) [20] and RILEM 4.1 (60ºC) [19] CPTs. The preparation of the mortar and concrete mixes was made according to the recommendations mentioned above. For mortar mixes, a cement type CEM I 42.5 R with 0.86% of $Na_2O_{equiv}$, an aggregate/cement (a/c) ratio of 0.44 and a water/cement (w/c) ratio of 0.47 were used. For concrete mixes, a cement type CEM I 42.5 R with 0.89% of $Na_2O_{equiv}$, an a/c ratio of 0.25 and a w/c ratio of 0.45 were used for all aggregates.

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## 3 RESULTS

The results of the point-counting are displayed in Table 1.

Aggregate A shows an inequigranular texture with well-developed K-feldspar megacrysts. Plagioclase, quartz, K-feldspar, muscovite and biotite are the main minerals. The accessory minerals are represented by zircon, apatite, andalusite and opaque minerals. As secondary phases, chlorite, rutile, clay minerals, muscovite and epidote were identified. Plagioclase is the most common mineral. It presents euhedral to anhedral shapes and, sometimes, compositional zoning. Alteration of its crystals to microcrystalline muscovite is very common, especially in cores. Regarding the characteristics of the quartz crystals, the petrographic analysis revealed that aggregate A contains strained anhedral shaped quartz crystals showing undulatory extinction (~18º) (Figure 1A1). Quartz is also present, in smaller dimensions, as myrmekite in plagioclase and in goticular forms in K-feldspar. Quartz, along with K-feldspar and plagioclase crystals, is frequently microcracked (Figure 1A2) and the cracks are filled with oxides. K-feldspar presents itself as euhedral to subhedral microcline megacrysts. Inside its well developed euhedral crystals there are goticular quartz, biotite, muscovite and plagioclase. Microcline is usually altered to clay minerals. Muscovite appears in well-developed, tabular, subhedrical crystals containing inclusions of biotite, quartz and K-feldspar. Sometimes it is possible to observe deformation of its cleavage plains. Biotite appears as subhedral to anhedral, well-developed crystals. This mineral shows usually, innumerous zircon, apatite, muscovite and opaque minerals inclusions. The chloritization of biotite is also evident along with the existence of rutile and epidote.

Aggregate B shows a similar composition to aggregate A with plagioclase, K-feldspar, quartz, biotite and muscovite as the main minerals. Apatite, zircon, fibrolite, opaque minerals and fluorite are the accessory minerals. Chlorite, rutile, clay minerals, microcrystalline muscovite and epidote were identified as secondary phases. Plagioclase, as in aggregate A, is the most common mineral in this granitic aggregate and presents euhedral to subhedral shaped crystals. It is possible to observe in some crystals a compositional zoning. This mineral is frequently altered to microcrystalline muscovite and to epidote. This alteration is, usually, stronger in plagioclase's core. Subhedral to anhedral megacrysts of pertitic microcline are also common. These crystals present inclusions of plagioclase, biotite and, sometimes, goticular quartz. K-feldspar is altered to clay minerals. The examination of aggregate B also showed the existence of anhedral quartz crystals with undulatory extinction with angles of about 16º. Goticular quartz and, particularly, myrmekite are common (Figure 1B1 and 1B2). Well-developed biotite crystals exhibit euhedral to subhedral shapes and is more abundant than in aggregate A. Its zircon inclusions are associated with apatite, fibrolite, fluorite and opaque minerals. The chloritization of biotite is frequent along with the occurrence of rutile crystals. Muscovite occurs as well-developed crystals that can be associated to biotite can result from plagioclase alteration.

Aggregate C, besides an inequigranular texture with K-feldspar megacrysts, exhibits a preferential orientation of the minerals. The main minerals are represented by K-feldspar, plagioclase, quartz, muscovite and biotite. The accessory minerals are tourmaline, apatite, zircon and opaque minerals. The secondary phases are represented by chlorite, rutile, clay minerals and muscovite. Subhedral to anhedral pertitic microcline megacrysts are rich in goticular quartz, plagioclase and muscovite inclusions. The alteration of this K-feldspar is more effective along cleavage plains. Plagioclase presents compositional zoning and is extremely

altered to microcrystalline muscovite. Alteration is more intense in the core. Aggregate C, besides featuring quartz with irregular grain boundaries along with a more evident undulatory extinction (~20º) and deformation lamellae, exhibits a quite substantial amount of elongated, microcrystalline and sub-granulated quartz (Figure 1C1). Goticular forms of quartz are present in K-feldspars and plagioclases in myrmekites. Deformation of the granite is also confirmed by phyllosilicates (Figure 1C2) and deformed twins in plagioclases (e.g. tapering edges) and by preferential orientation of the minerals, namely quartz, muscovite and biotite. The occurrence of muscovite is similar to the ones of aggregates A and B, but, in this case, the evidences of deformation are much stronger. The anhedral crystals of biotite present inclusions of zircon, apatite and opaque minerals. It is frequent to find completely chloritized biotites associated to rutile.

When submitted to AMBT [17], aggregates A, B and C showed 14 days expansion values of 0.02%, 0.01% and 0.02%, respectively (Figure 2a). Expansions of less than 0.10% after 14 days distinguish a non-reactive from a reactive aggregate [23]. With the aim to confirm the results, the tests were carried on for another 14 days, but the behavior was maintained since stabilization of all the expansion values was verified at 0.03%.

Expansions of less than 0.05% after the 1 year-long CPT 38ºC [20] classify the aggregate as non-reactive [23]. Final expansion values of about 0.02% were obtained for aggregates A and B and of 0.47% for aggregate C (Figure 2b). In accordance to the shape of the expansion curves, aggregates A and B show, during the whole test, quite constant expansion values with no significant increases. Aggregate C concrete prism shows a substantial boost of its expansion value, around day 168, reaching the limit of 0.05% around day 224. Another strong increment in the expansion value took place around day 252. The galloping increase continued till the end of the test, the shape of the curve suggesting that its stabilization is far from being reached.

Although in the Portuguese specification, the limit of 0.02% is still accepted [36], expansions of less than 0.03% after 20 weeks in CPT 60ºC [19] indicate that the aggregate can be regarded as non-reactive [25]. After the completion of this test, final expansions of 0.035, 0.027 and 0.070% were obtained for aggregates A, B and C, respectively (Figure 2c). The behavior of aggregates' A and B curves showed to be very similar during all the 20 weeks. The slight decreases of expansion which took place from week 8 till week 10 enable to distinguish aggregate A as being more expansive than aggregate B, although this difference is rather small. From week 10 till the completion of the test, both expansions are continuously increasing, but for aggregate A the limit of 0.03% is scarcely crossed while for aggregate B this limit is almost reached. Regarding aggregate C, the expansion of its specimens increased during the whole test. From week 6, the limit of 0.03% was reached and the expansion rapidly increased towards the end of the test.

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## 4 DISCUSSION

According, strictly, to the percentage of potentially reactive features, it can be said that only aggregate A (1.0%) should be classified as innocuous to alkalis by petrographic characterization [36]. On the contrary, aggregate B and, especially, aggregate C present values of potentially reactive features above 2%, which classify them as ambiguous. The potential-reactivity of aggregate C was already expected since several authors [e.g. 7-13] had mentioned the presence of microcrystalline quartz with irregular boundaries and deformed micas and feldspars as responsible for ASR. The value of 2.4% obtained for the reactive features in aggregate B is very close to the limiting value of 2%. It should be kept in mind that the accuracy and precision of the point-counting method depend, among other parameters, on the number of points counted and the proportion of constituents of interest within the sample [35]. This can help to justify the classification of aggregate B as non-reactive by all expansive tests. Also, these observations contribute to support the fact that syn-tectonic granites are more likely potentially reactive to alkalis than the younger granites [33]. In order to

explain this behavior of the granitic samples studied, the undulatory extinction of quartz was observed. The difference in intensity of the undulatory extinction in quartz crystals can be interpreted as the result of the degree of deformation of the granitic rocks of different ages present in a single folded belt affected by successive orogenic events [2]. Quartz undulatory extinction is more intensely developed in older rocks, decreasing in intensity in more recent rocks. Actually, aggregate C is older than aggregates A and B, and comes from a quarry placed close to a major fault, facts responsible for a more evident deformation.

According to the expansion results achieved by the ASTM C 1260 AMBT [17], all aggregates were considered as being non-reactive. By comparing these results with the ones obtained by Shayan [18] for gneissic granitic aggregates with strained and microcrystalline quartz, aggregates A, B and C show much lower expansion values, since even at 28 days, 0.20% of expansion is far from being reached. It should be highlighted that the aggregates referred in [18] are different from the ones of the present study, showing similarities just with aggregate C. The slowly reactive aggregates studied by Shayan [18] show, at 28 days, values very close to 0.20%. Although ASTM C 1260 [19] is widely used for the assessment of aggregates for concrete purposes, it is still not well recognized that its limits correspond to the reactivity of some of the slowly reactive aggregates, leading to their incorrect classification as non-reactive or uncertain [18]. This method has not been successful when performed on Portuguese granitic aggregates [32] and therefore it has been considered as inappropriate to evaluate the alkali-reactivity of this type of slow reactive rock. The explanation for the results obtained might remain in the findings reported in [37] in which it is stated that the crushing of rocks to obtain very fine aggregates can destroy the original microstructure and thus under-estimate the alkali reactivity of the rocks in accelerated mortar bar tests. In fact, the influence of the size of the aggregate particles used in laboratory tests has been discussed and results published [37-40].

Aggregates A and B were classified by the AAR-3 CPT 38°C [20] as non-reactive. On the other hand, aggregate C was classified as being reactive, showing a high final expansion value (0.47%). However, according to several authors [18,21,23], the RILEM AAR-3 method [20] was classified as not being suited to identifying slowly reactive aggregates, since it was not able to detect some of the aggregates of gneissic origin containing microcrystalline quartz as the reactive component. Also, this concrete prism method was pointed out as not being conclusive at the age of one year and a longer test period may be needed for some slowly reactive aggregates to produce expansion over the critical limit in the conditions of this test [18,21]. This assumption was taken from the continuing expansion at the end of the test of the expansion curves. Nonetheless, in the present study, this phenomenon only occurs for aggregate C, but there is no doubt that this aggregate is potentially reactive. Also, aggregate A and B expansion curves leave no uncertainties towards their non-reactive behavior.

The aggregates performance during the 20 weeks long RILEM AAR-4.1 CPT 60°C [19] classified aggregates A and C as being reactive, with aggregate C displaying the highest final expansion (0.07%). Expansion values reached by Shayan [21] for gneissic granites show, at 20 weeks, values close to 0.08% which are similar to the final value obtained by aggregate C. The same author obtained for a granite, at 20 weeks, a value close to 0.04% which is consistent with the values found for aggregate A. However, the final expansion results obtained for this aggregate (0.035%) and for aggregate B (0.027%) are included in the band of uncertainty for the limiting value of 0.03% for differentiating reactive and non-reactive aggregates considered for this test (<0.01%) [23], presenting a borderline behavior. Regarding the aggregate A, the potential reactivity detected could be attributed to the fact that there is a large number of microcracks in the aggregate [14].

In the [23] it was referred that RILEM AAR-4.1 [19] seems to be more effective than RILEM AAR-3 [20] in the assessment of slowly reactive aggregates. However, the slowly reactive aggregates tested in the scope of the Partner Project are not of granitic rocks, which makes it difficult to establish a comparison with

the present samples. On the other hand, Shayan [21], also confirmed the effectiveness of CPT 60ºC [19] in comparison to CPT 38ºC [20] in the identification of slowly reactive aggregates in which deformed granites were included. The CPT 60ºC [19] eliminated a dormant period of about 3 to 4 months, which was usually seen in CPT 38ºC [20], i.e., the expansion of specimens under the RILEM AAR-4.1 [19] occurred more rapidly than in RILEM AAR-3 [20].

# 5    CONCLUSIONS

The tests performed in the granitic samples lead to the following conclusions:

- ASTM C-1260 AMBT [17] shows a poor correlation with the petrographic characterization of aggregate C, confirming previous results with Portuguese granitic rocks, and, therefore, should not be used for assessing the potential-reactivity of these aggregates.

- It should be kept in mind that crushing certain types of aggregates for laboratory tests may change some of their characteristics (microstructure) and therefore the grading actually used in the concrete structures should preferably also be used as such in the laboratory tests. Concrete prism tests should be privileged to reproduce field conditions.

- RILEM AAR-3 (38ºC) and RILEM AAR-4.1 (60ºC) concrete prism tests [20,19] are showing to better mirror the expected reactivity assessed by petrographic methods.

- The expansion limit of 0.02% for the RILEM AAR-4.1 (60ºC) test [19], considered by the Portuguese specification [36], should be revised.

- Aggregate C is, undoubtedly, potentially reactive to alkalis, fact which was confirmed by all methods with exception of the ASTM C 1260 AMBT [17].

- There are slight discrepancies concerning aggregates A and B concrete prism tests results towards the petrographic characterization. Further experiments need to be done to study the accuracy of the RILEM AAR-3 [20] and of RILEM AAR-4.1[19] tests for granitic aggregates.

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| TABLE 1: Results of the point-counting method (values in %). | | | |
|---|---|---|---|
| *Mineral* | *Aggregate A* | *Aggregate B* | *Aggregate C* |
| Plagioclase | 36.0 | 30.5 | 26.1 |
| Quartz | 28.8 | 25.1 | 12.5 |
| Potassium Feldspar | 20.4 | 26.8 | 26.3 |
| Muscovite | 9.5 | 3.8 | 9.7 |
| Biotite + Chlorite | 3.8 | 10.2 | 3.1 |
| **Microcrystalline with irregular boundaries and/or goticular, and/or myrmekitic quartz** | 1.0 | 2.4 | 21.7 |
| Accessory minerals | 0.5 | 1.2 | 0.66 |
| Total | 100.0 | 100.0 | 100.0 |
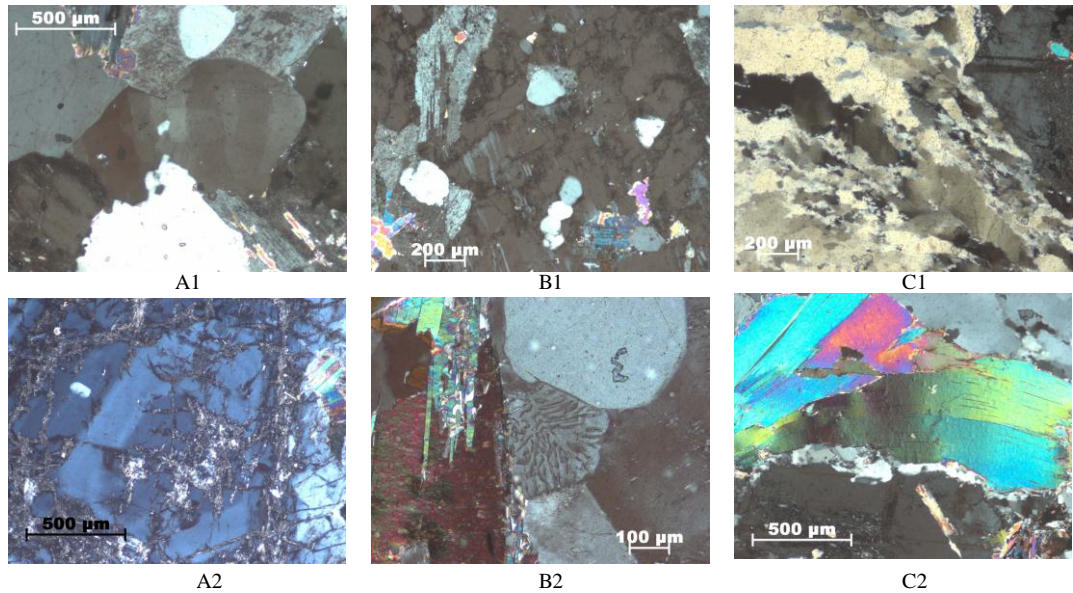
FIGURE 1: Aggregate A: undulatory extinction in quartz crystals (A1); presence of microcracks in a zoned plagioclase crystal (A2). Aggregate B: goticular quartz (B1); myrmekites (B2). Aggregate C: existence of preferential orientation of deformed quartz (C1); strained crystals of muscovite (C2). All images are taken in crossed-polarized light.
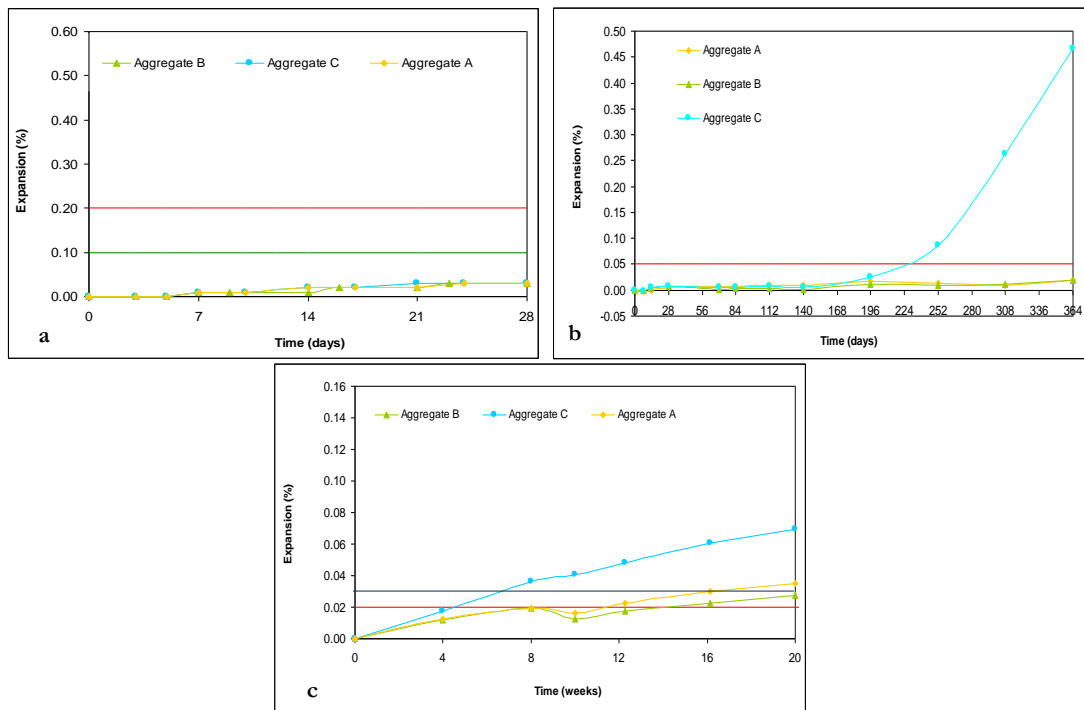


FIGURE 2: a)Expansion behavior for aggregates A, B and C in the ASTM C 1260 AMBT [17]; b) Expansion behavior for aggregates A, B and C in the AAR-3 CPT 38ºC [20]; c) Expansion behavior for aggregates A, B and C in the AAR-4.1 CPT 60ºC [19].