THE STUDY OF THE AZOREAN VOLCANIC AGGREGATES FROM THE POINT OF VIEW OF ALKALI SILICA REACTION

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Abstract

Volcanic rocks are the main source of concrete aggregates in Azores archipelago. In order to understand the reactivity of these rocks in this Portuguese territory, a study was implemented in the past three years to evaluate the potential alkali-reactivity of thirteen aggregates collected in these islands. For this study, a petrographic characterization and a chemical analysis were made on the rock samples and expansion tests were performed on the aggregates.

The petrographic and the chemical analyses of the rocks confirmed that one of the samples contains free silica and three samples present interstitial volcanic glass. Microcrystalline quartz was confirmed by SEM/EDS as a secondary product filling the interstices of trachyte sample. The expansion tests show that the trachyte is the only aggregate with potential alkali-reactivity in the accelerated mortar-bar test.

This paper presents the main results of eleven aggregates regarding the potential alkali-reactivity of the volcanic rocks from this archipelago.

Keywords: ASR, volcanic rocks, petrography, expansion tests, Azores archipelago

1 INTRODUCTION

The alkali-silica reaction (ASR) was discover in 1940 and since then has been a source of problems in many concrete structures worldwide. All over the years, much research has been done to understand: (1) the chemical mechanism of the reaction, (2) which types of rocks are considered reactive, (3) what are the best methods to detect the potential reactivity of the aggregates, and (4) which preventive measures can be adopted when alkali-reactive aggregates have to be used. There is still not a consensus regarding the types of rocks that are considered reactive. Volcanic rocks of intermediate to basic composition are one of the rock types for which there is still a lot uncertainty concerning ASR, especially regarding the basaltic rocks. Many studies have been done on volcanic rocks by several authors from the point of view of their reactivity [1-7].

In Portugal, the reactivity of volcanic rocks has been unknown for several years. There are only two studies related to volcanic rocks, one is concerning some rocks of Azores archipelago [8] and the other is about Madeira archipelago [9]. There is also a study made by the National Laboratory for Civil Engineering (LNEC) regarding a deteriorated concrete pavement with ASR in Azores, where volcanic rocks were used in the manufacture of concrete for the construction of this structure [10]. More recently, a research project called ReAVA (*Characterization of potential reactivity of the volcanic aggregates from the Azores Archipelago: implications on the durability of concrete structures*) was implemented in the Azores archipelago in order to evaluate the potential alkali-reactivity of the volcanic rocks in this region. For the development of this project, there was a joint cooperation between the University of Azores, the University of Porto, the LNEC and the Regional Laboratory of Civil Engineering of Azores (LREC). In the scope of the project, a set of methods were used that included: (1) petrographic analysis of all

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the aggregates selected for the study, (2) assessment of their performance in expansion tests, and (3) site inspection of existing large concrete structures.

The ReAVA project also integrated a national project – IMPROVE (*Improvement of performance of aggregates in the inhibition of alkali-aggregate reactions in concrete*) with the objective of establishing the most precise method for the evaluation of the main types of aggregates in the Portuguese territory, including the volcanic aggregates from Azores and Madeira archipelagos. This evaluation also included the petrographic examination and the performance of mortar and concrete expansion tests, namely ASTM C 1260 [11], RILEM AAR-3 [12], RILEM AAR-4.1 [13] as well as field performance surveys.

This paper presents the results regarding the petrography analysis of all the rock samples and the performance of the aggregates in the expansion tests. The petrographic characterization started with the methodology proposed in the LNEC Specification E 415 [14], followed by the bulk chemical analysis of rock and the laboratory expansion tests.

2 MATERIALS AND METHODS

2.1 Aggregates

Thirteen crushed aggregates were selected from a total of eight islands. Only one out of the nine Azorean Islands was not selected due to the inexistence of local aggregate production. The aggregates were taken from eleven quarries, one excavation and one crushing plant. The crushed aggregates were collected from stockpiles of different size fractions, i.e. between stone dust (< 4 or 5 mm) and crushed stone (from > 4 to < 32 mm), for the laboratory tests. The sampling of the aggregates was carried out according to the Portuguese standard NP EN 932-1 [15], as being representative of the thirteen different aggregates collected in each exploitation area at the time of the visits.

The area of the excavation is inside a volcanic neck of benmoreite composition with an age between 0.67 Ma and 0.54 Ma B.P. [16]. The rocks from the crushing plant were taken from a stream nearby and crushed for concrete purposes and, therefore, the age of the rocks is uncertain. The quarries are located in basaltic or trachytic lava flows with the exception of one quarry that is located in a submarine eruptive centre in Santa Maria Island. The ages of the quarries range from 4.13 Ma in Santa Maria island [17] to less than 40 ka in Pico island [18].

Hand samples were also taken from the different exploitation areas in order to produce thin sections and perform bulk chemical analysis. A code was given to the rock samples according to the name of the island and number of areas exploited/island. The rock samples came from the islands of Santa Maria (SMA-SM1 and SMA-SM2), São Miguel (SMG-SM1, SMG-SM2 and SMG-SM3), Terceira (TER-SM1 and TER-SM2), Graciosa (GRA-SM1), São Jorge (SJO-SM1), Pico (PIC-SM1), Faial (FAI-SM1) and Flores (FLO-SM1 and FLO-SM2).

2.2 Methods

Polarizing microscopy

Mineralogical and textural characteristics of the samples were examined in conventional thin-sections under Olympus CX31 polarizing microscope and images acquired with Olympus SC100 camera.

The main goal was to identify volcanic glass, opal, tridymite, cristobalite, chalcedony, microcrystalline and cryptocrystalline quartz and altered minerals (e.g. clay minerals). No point counting was carried out due the small size of the minerals.

Scanning Electron Microscopy - Energy Dispersive Spectroscopy (SEM-EDS)

Some polished thin-sections were carbon-coated and examined by SEM equipped with an EDS (JEOL JSM-6301F, NORAN-VOYAGER: 15 kV, working distance 15 mm, collection time 60 seconds, dead-time 30%). SEM/EDS were used only in the SMA-SM1, SMG-SM2 and TER-SM1 samples to examine or confirm some minerals that were not easily identified by optical microscopy.

Bulk rock chemical analyses

Geochemical analyses were made at Activations Labs, Canada. The major oxides were determined by fusion-inductively coupled plasma (FUS-ICP: Thermo Jarrell-Ash ENVIRO II ICP). The results were plotted on a TAS diagram to classify the rock samples.

Expansion tests

The aggregates were first sieved and weighed at the LREC after being collected in the different areas of exploitation and then sent to LNEC. In LNEC, the different expansion tests were performed according to the requirements in ASTM C 1260 (80ºC) accelerated mortar-bar test (AMBT) [11], RILEM AAR-3 (38ºC) [12] and RILEM 4.1 (60ºC) [13] concrete prism tests (CPTs).

Mortar mixes were made using a cement type CEM I 42.5 R with 0.89% of Na_2O_{equiv}, graded aggregates (0.15-4.75 mm), cement/aggregate (0.44) and water/cement ratio (0.47). The mortar bars, 25 x 25 x 285 mm in size, were immersed in a 1N NaOH solution at 80ºC and length variation measurements extended up to 28 days to confirm the results obtained at 14 days.

Concrete mixes were made with the same cement as above, graded aggregates (<22.4 mm) (i.e. using together the coarse and fine aggregates from the same source), cement/aggregate (0.25) and water/cement (0.45). The concrete prisms, 75 x 75 x 285 mm in size, were stored at 38 or 60ºC and R.H. > 95%, with length and mass change measurements extended until 36 weeks for RILEM AAR 4.1 test [13] and to 2 years for RILEM AAR-3 test [12].

3 RESULTS

The petrographic analysis was performed in all thirteen samples. The rocks were classified according to their mineral composition and textural features (Figure 1). Table 1 presents the main characteristics of the different rocks samples studied.

3.1 Petrographic characterization

The basanites (SMA-SM1 and SMA-SM2) from Santa Maria Island are composed of olivine with iddingsitized rims, clinopyroxene and less frequent plagioclase phenocrysts and opaque minerals (ilmenite and magnetite) in a matrix formed by the same minerals but less amount of olivine. In SMA-SM1 sample, there are carbonates filling the cracks and some minerals filling some of the voids in the rock. These minerals were identified by SEM/EDS analysis as anhedral intergranular analcite (zeolite). In SMA-SM2, the iddingsitized rims in the olivine are thicker and there is interstitial plagioclase in several areas of the thin section.

The samples from São Miguel Island (SMG-SM1 - trachybasalt, SMG-SM2 – transition between trachybasalt and basalt and SMG-SM3 - basalt) are formed by olivine, clinopyroxene, plagioclase and opaque minerals in a matrix composed of the same phases plus apatite and biotite. Under optical microscope, the following components were identified in the trachybasalt sample (SMG-SM1): (1) brownish and isotropic volcanic glass in the intergranular groundmass and (2) a xenocryst of quartz surrounded by pyroxenes and some opaque minerals. The volcanic glass contains 58% of SiO_2 according to EDS analysis performed in Japan [19].

The samples from Terceira Island are a trachyte (TER-SM1) and a basalt (TER-SM2). The trachyte is formed by large crystals mainly of sanidine with smaller minerals of nepheline, biotite and opaque. The trachytic groundmass consists of feldspars (plagioclase, anorthoclase), pyroxenes (aegirine, augite), amphibole (aenigmatite), apatite and opaque minerals (mainly magnetite). The analysis performed with SEM/EDS confirmed the presence of interstitial pure silica as microcrystalline quartz (Figure 2). This rock shows evidence of alteration containing interstitial iron silicate gel (SiO_2 ~50%, Fe_2O_3 ~26%), which is pseudomorphic after aegirine and more siliceous than hisingerite but of unknown alkali-reactivity [19]. The basalt (TER-SM2) has phenocrysts of olivine, clinopyroxene and plagioclase set in fine-grained groundmass composed of the same minerals and opaque minerals dominated by ilmenite. By SEM/EDS performed in Japan, andesitic glass inclusions within the plagioclase were also identified with a composition of 55% to 58% of SiO_2 [19].

The trachybasalt (GRA-SM1) from Graciosa Island is composed of plagioclase, olivine, rare clinopyroxene and opaque minerals, set in intergranular groundmass of the same minerals but with lower olivine and higher pyroxenes contents.

The basalts from São Jorge (SJO-SM1) and Pico (PIC-SM1) Islands have phenocrysts of olivine and clinopyroxene and also plagioclase in PIC-SM1 sample and opaque minerals. Both samples are set in a matrix formed by the same minerals.

Trachybasalt (FAI-SM1) from Faial Island has an assemblage of large crystals of olivine and smaller and less frequent crystals of plagioclase and clinopyroxene. The matrix is formed by the same minerals and opaque minerals, predominantly magnetite.

The samples from Flores Island (FLO-SM1 and FLO-SM2) show a microporphyritic texture. In both cases, the larger crystals are predominantly plagioclase and the matrix is formed by biotite, opaque minerals, clinopyroxene, alkali feldspar and plagioclase. No olivine was found on both samples. In FLO-SM1 sample, the presence of carbonates is observed either in the matrix or filling the cracks inside the plagioclase feldspar. In some cases, the carbonates seem to be replacing the feldspars.

3.2 Rock chemistry

The results of the geochemical analysis plotted in a TAS diagram showed that most of the rocks are classified as basalts and trachybasalts. One of the samples is in the transition between basalt and trachybasalt. There are also two basanites, one basaltic trachyandesite, one trachybasalt and one trachyte. All the rocks were classified according to Le Maitre *et al.* [20].

The samples of SMA-SM1, SMA-SM2, TER-SM1 and FLO-SM1 show a value of loss on ignition (LOI) higher than 1%.

The chemical analysis also shows that the samples TER-SM1, FLO-SM1 and FLO-SM2 present a content of silica higher than 50%. The majority of the rocks are considered silica undersaturated.

3.3 Expansion tests

Only the aggregate TER-SM1 showed an expansion greater than 0.10% at 14 days in the AMBT (Figure 3A). Expansions of less than 0.10% after 14 days in NaOH distinguish a non-reactive from a potentially reactive aggregate [11]. The other aggregates do not show any significant expansion. The tests were carried out for another 14 days to confirm the results and eventually for 140 days. The aggregate TER-SM1 is considered potentially reactive and requires further testing through concrete prisms.

In the CPT at 60ºC, all the aggregates are considered non-reactive according to the [13] (Figure 3B). The limits are still an issue in this test-method. However, in the Portuguese specification, the limit of 0.02% is accepted [14], while expansions of less than 0.03% after 15 weeks in the CPT at 60ºC [13] indicate that the aggregate can be regarded as non-reactive [21].

In the CPT at 38ºC, most of the aggregates show a steadily increasing expansion trend over the full testing period carried out in this study (Figure 3C). At the end of 2 years of testing, the aggregates SMG-SM2, TER-SM2, GRA-SM1, SJO-SM1 and PIC-SM1 crossed the boundary limit of 0.05%. However, expansions of less than 0.05% after the "standard" 1 year testing period used in the CPT at 38ºC classify the aggregates as non-reactive [13].

4 DISCUSSION

According to Lorenzi *et al.* [22], volcanic rocks are considered reactive when they contain microcrystalline quartz, volcanic glass and quartz polymorphs. The petrographic study showed that most of the samples do not contain potentially reactive forms of silica. According to the petrographic analysis, volcanic glass was identified in small amounts on three samples (SMG-SM1, TER-SM1 and TER-SM2). SMG-SM1 and TER-SM2 samples show a volcanic glass composition of SiO_2 58% and 55%, respectively [19], both being considered of andesitic composition. In general, andesitic glass is considered not deleterious but according to Katayama *et al.* [1], a basalt was considered reactive with a composition of 20% of andesitic volcanic glass. Volcanic glass is considered reactive when it contains more than 65% of silica even if it is present in basalt, andesite, dacite and rhyolite [1]. This type of volcanic glass has been considered reactive and is many times associated to ASR [1,5,23].

TER-SM1 sample contains an interstitial iron silicate gel containing SiO_2 ~50% and Fe_2O_3 26%; the presence of microcrystalline quartz was confirmed by SEM/EDS [19].

All of the three samples are considered potentially reactive, based on the petrographic assessment.

Petrography also allowed the identification of some alteration products and secondary minerals which are common in many volcanic rocks. The presence of iddingsite is highly noticed in the basanites (SMA-SM1 and SMA-SM2), especially in the sample of SMA-SM2 where it is more evident. Both SMA-SM1 and FLO-SM1 samples show carbonates either filling cracks and voids in the rocks (SMA-SM1) or replacing plagioclase crystals (FLO-SM1). Zeolites were identified by polarizing microscope and by SEM/EDS in SMA-SM1 sample. Zeolites are secondary minerals that are usually found in cavities of basaltic rocks.

The relatively high values of LOI for SMA-SM1, SMA-SM2 and FLO-SM1 suggest that the rocks are moderately altered.

Based on the bulk chemical analysis of the rock, on TAS diagram and on petrography, the basaltic rocks are considered: (1) silica undersaturated, (2) belonging to the alkaline series, and (3) in rare cases the groundmass contains volcanic glass. According to Mackenzie *et al.* [24], alkali olivine basalts contain very rare glass in the groundmass unlike the tholeiitic basalts that contain varying amounts of interstitial brown glass or devitrified glass. This is in agreement with the fact that volcanic glass was not found in the majority of the samples. On the other hand, basaltic quarries have a high heterogeneity. This makes the rocks present variable behaviors that should warrant special attention when used as aggregates.

The AMBT showed that almost all the samples are considered non-reactive (Figure 3). The only exception is the trachyte that is considered in a "grey zone" in terms of potential alkali-reactivity after 14 days of testing according to ASTM C 1260 [11], confirming the results of the petrographic and chemical analysis. This is one of the few tested volcanic rocks that have silica content higher than 50%, small amounts of interstitial volcanic glass and microcrystalline quartz. However, the basalts that were classified as potentially reactive in the petrography showed no reactivity in this test. Wigum *et al.* [25] studied several Icelandic basaltic aggregates and showed that most of them were considered deleterious at the end of 14 days of expansion in AMBT.

The aggregates tested in the accelerated CPT (60ºC) did not show any reactivity by the end of 15 weeks. All the aggregates show oscillatory curves and the expansion curves never reached the threshold of reactivity (Figure 3).

The CPT at 38ºC, which is considered a more realistic expansion method, showed that most of the aggregates have an increasing expansion trend over time (Figure 3). It should be taken into consideration that the aggregates SMG-SM2, TER-SM2, GRA-SM1, SJO-SM1 and PIC-SM1 reached the boundary limit of 0.05% but only after 2 years of testing; petrographic examination of the concrete prisms at the end of testing is in progress and is expected provide further insights on the reason for the long-term expansive behaviour of the above aggregates. The expansion of trachyte sample (TER-SM1) did not reach the limit boundary after 2 years of testing, meaning that it was considered non-reactive, while it was classified as potentially reactive on the AMBT. The testing for SMG-SM1 is still in progress.

Despite the fact that the RILEM recommendation states that the CPT at 38ºC should be performed during a period of 1 year – and all tested aggregates did not reached the boundary limit of 0.05% at the end of that period, thus being considered non-reactive – it is known that some aggregates require more time since they exhibit late expansion reactions, which may be the case of these aggregates. Therefore, it is suggested that these aggregates are not to be used in the construction of dams and bridges (concrete structures with more complexity, that are more structurally demanding and for which a long service life is expected) without using a certain percentage of fly ash or pozzolan in the concrete mixture.

5 CONCLUSIONS

The different methods performed on volcanic aggregates from Azores archipelago lead to the following conclusions:

- Petrography identified small amounts of volcanic glass in three samples (SMG-SM1-Trachybasalt, TER-SM1-Trachyte and TER-SM2-Basalt). SEM/EDS showed that the volcanic glass on: (1) SMG-SM1 sample has a silica content of 58% and (2) TER-SM2 sample has a silica content of 55%, below the content of 65% considered to be related to potential alkali-reactivity.
- In the sample of trachyte (TER-SM1), the analysis made by SEM/EDS confirmed the presence of interstitial silica as microcrystalline quartz. The geochemical analysis shows silica content higher than 50%, which is defended by some researchers to be one of the conditions for the rock to be reactive. Also, the AMBT performed on this aggregate showed that it is somewhat potentially reactive after 14 days of testing. The CPT does not indicate, however, that this sample is potentially reactive.
- The samples from Flores Island (FLO-SM1 and FLO-SM2) have silica content slightly higher than 50%, which is one of the conditions of potential reactivity. However, the petrography and the expansion tests indicate that both samples are non-reactive.
- The other samples are considered non-reactive by the petrography, geochemical analysis and by the expansion tests.
- The inexistence of interstitial volcanic glass in most samples and the silica undersaturated character could justify the non-reactivity results in the majority of the methods.
- The CPT showed that after 2 years of testing some samples crossed the limit boundary between non-reactive and reactive recommended by RILEM recommendations. These results should be taken into consideration if these aggregates should be used in concrete structures with more complexity and long service life demands, like dams or bridges. The trend of the curves indicates that these reactive aggregates start to expand late but the expansion curves do not flat out, suggesting that the reaction is maintained after 2 years for some of the samples.

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TABLE 1: Main petrographic characteristics of the samples.

	SMA-SM1	SMA-SM2	SMG-SM1	SMG-SM2	SMG-SM3	TER-SM1	TER-SM2
Islands	Santa Maria		São Miguel			Terceira	
Crystallinity	Holocrystalline						
Crystal shapes	Euhedral	Euhedral Subhedral	Euhedral Subhedral	Euhedral Subhedral	Euhedral	Euhedral	Euhedral
Granularity	Porphyritic Aphanitic						
Texture	Porphiritic Intergranular Trachytic	Porphiritic Ophitic	Porphiritic Intergranular Trachytic	Porphiritic Intergranular Trachytic	Porphiritic Intergranular Trachytic	Intergranular Trachytic	Porphiritic Intergranular Trachytic
Secondary minerals Alteration products	Olivine with iddingsite rims Carbonates Zeolites	Olivine with thick iddingsite rims	-	-	-	-	-
Reactive silica	-	-	Volcanic glass	-	-	Volcanic glass Mycrocristalli-ne quartz	Volcanic glass
	GRA-SM1	SJO-SM1	PIC-SM1	FAI-SM1	FLO-SM1	FLO-SM2	
Islands	Graciosa	São Jorge	Pico	Faial	Flores		
Crystallinity	Holocrystalline						

Crystal shape	Euhedral	Euhedral	Euhedral	Subhedral	Euhedral	Euhedral	
Granularity	Porphyritic Aphanitic				Microporphyritic Aphanitic		
Texture	Porphiritic Intergranular Trachytic	Porphiritic Intergranular Trachytic Vesicular-	Porphiritic Intergranular Trachytic	Porphiritic Intergranular Trachytic	Micoporphi-ritic Intergranular Trachytic	Micoporphi-ritic Intergranular Trachytic	
Secondary minerals Alteration products	-	-	-	-	-	Carbonates	
Reactive silica	-	-	-	-	-	-	

TABLE 2: Geochemical composition of the samples.

Major Oxides (wt %)	SMA-SM1	SMA-SM2	SMG-SM1	SMG-SM2	SMG-SM3	TER-SM1	TER-SM2
SiO_2	43.31	43.55	47.25	46.45	45.69	64.79	48.54
Al_2O_3	12.98	13.28	14.09	14.08	13.36	13.92	14.65
Fe_2O_3-total	11.88	12.16	12.2	12.87	12.26	6.1	12.12
MgO	11.05	9.96	8.06	8.19	8.00	0.65	6.82
CaO	11.52	11.75	9.16	9.39	10.75	0.71	10.88
Na_2O	3.62	2.80	3.25	3.11	2.90	6.35	3.09
K_2O	0.76	0.57	2.04	1.89	1.68	4.97	0.97
TiO_2	2.29	2.40	3.48	3.59	3.41	0.61	3.11
P_2O_5	0.42	0.62	0.61	0.65	0.67	0.07	0.59
MnO	0.17	0.19	0.16	0.18	0.173	0.22	0.17
LOI	2.15	3.25	-0.20	-0.49	-0.63	1.13	-0.1
SUM total	100.2	100.5	100.1	99.92	98.26	99.51	100.8
Major Oxides (wt %)	GRA-SM1	SJO-SM1	PIC-SM1	FAI-SM1	FLO-SM1	FLO-SM2	
SiO_2	47.7	45.73	48.00	48.53	53.36	51.66	
Al_2O_3	16.76	15.39	14.93	16.35	16.4	16.58	
Fe_2O_3-total	11.11	12.82	11.29	11.39	8.37	9.06	
MgO	7.15	8.58	8.04	6.26	2.15	3.29	
CaO	9.58	10.07	10.51	9.36	5.65	7.08	
Na_2O	3.82	3.22	3.32	3.91	5.65	4.66	
K_2O	1.36	1.07	1.21	1.69	2.73	2.55	
TiO_2	2.74	3.51	2.69	2.92	1.84	2.43	
P_2O_5	0.56	0.53	0.44	0.62	0.67	0.93	
MnO	0.17	0.17	0.17	0.17	0.24	0.22	
LOI	-0.73	-0.47	-0.64	-0.55	3.16	0.64	
SUM total	100.2	100.6	99.94	100.6	100.2	99.1	

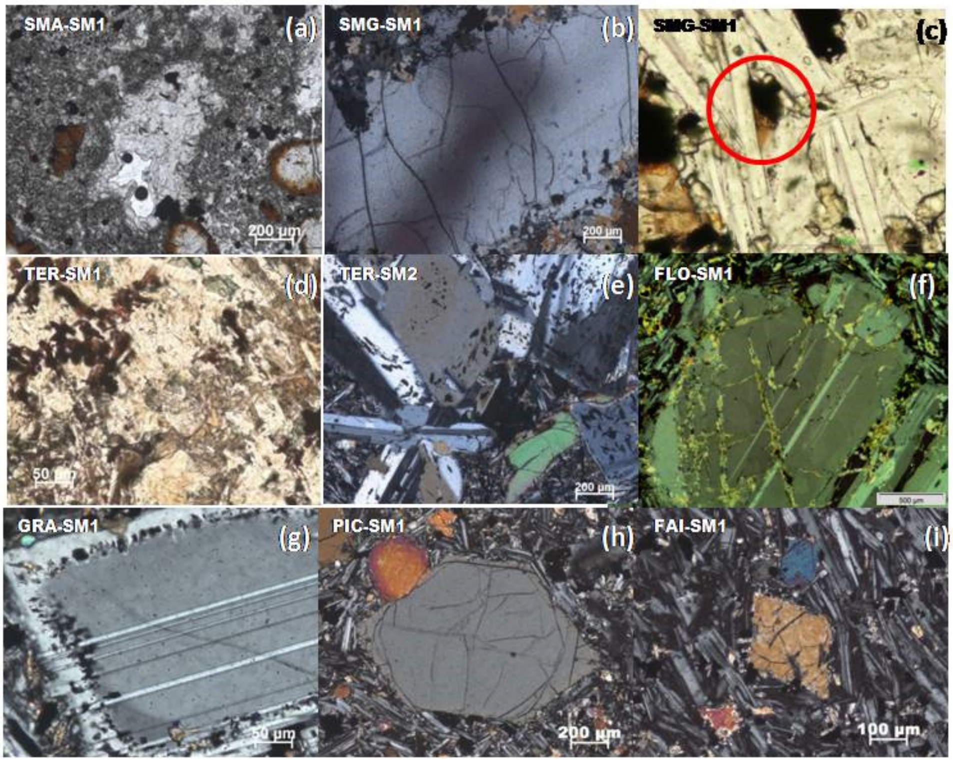

FIGURE 1: Photomicrographs of the volcanic aggregates analysed: (a) Zeolite and olivine with iddingsitized rims in SMA-SM1 sample (PPL); (b) Xenocryst of quartz in SMG-SM1 sample (XPL) (c) Volcanic glass as an intergranular component in SMG-SM1 sample (PPL); (d) Biotite and amphibole in TER-SM1 sample (PPL); (e) Glomeroporphyritic plagioclase feldspar in TER-SM2 sample; (f) Plagioclase with carbonates filling the cracks in FLO-SM1 (XPL) (g) Plagioclase in GRA-SM1 sample (XPL); (h and i) Euhedral olivine in PIC-SM1 and FAI-SM1 samples (XPL).

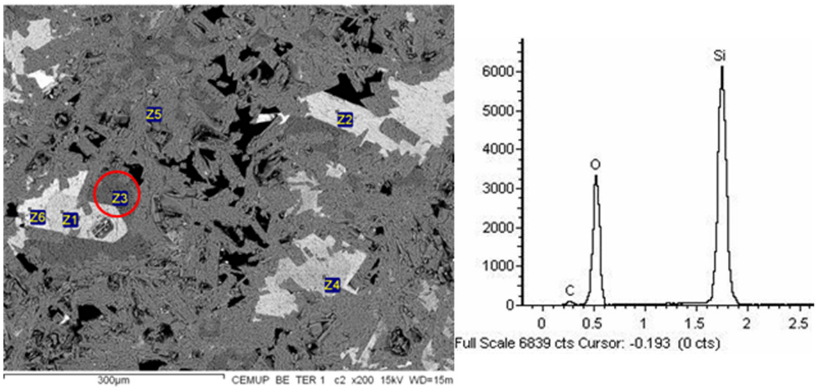

FIGURE 2: SEM image and EDS spectrum of silica in trachyte (microcrystalline quartz) TER-SM1 sample. (analysis Z3)

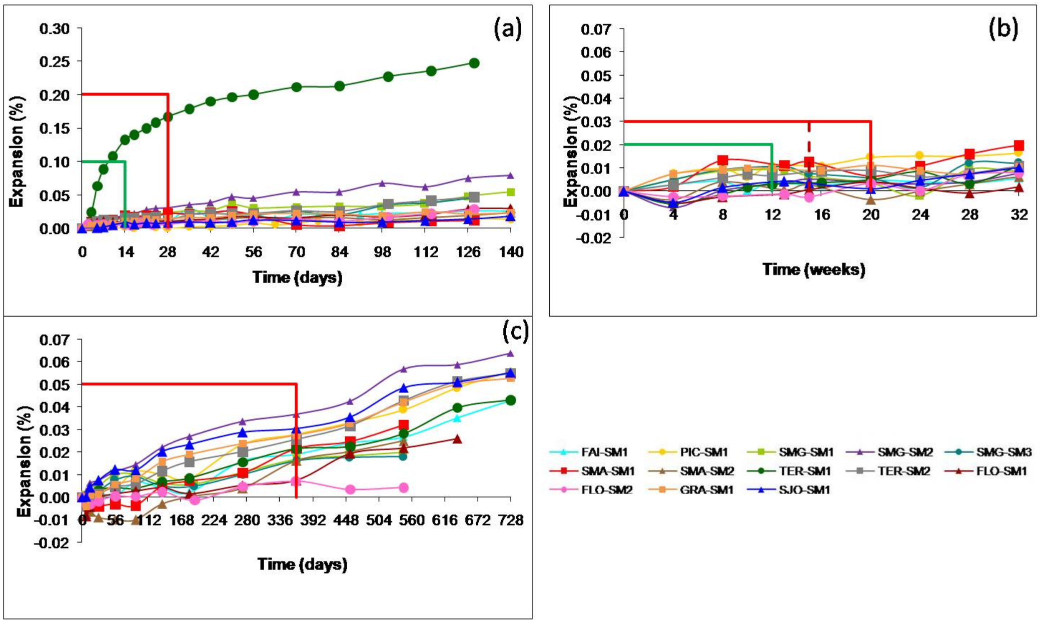

FIGURE 3: Expansion test results of all samples studied: a) Accelerated mortar bar test (ASTM C 1260); b) Concrete prism test at 60ºC (AAR-4.1); c) Concrete prism test at 38ºC (AAR-3).