Ageing tests for dimension stone - experimental studies of granitic rocks from Brazil

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Abstract: Dimension stones are products of the extraction of block-type rocky materials and their subsequent sawing, polishing and cutting into plates and tiles. They are largely used as building stone either as structural elements (columns and pillars) or, especially, as vertical (walls and façades) and horizontal (pavements and floors) coverings of interiors and exteriors of buildings. Granitic rocks, for their durability and great variety of colours and textural and structural patterns, have been the most used.

They are, like other stone and construction materials, naturally weathered over time. However, this process can be speeded up in aggressive climatic conditions, polluted environments or by inadequate construction or maintenance procedures, resulting in stone decay.

The processes of deterioration have been empirically related to the interaction of the petrographic, physical and mechanical characteristics of the rocks with environmental agents, pollution, human interventions and others. Characteristically, most of these processes, if started, are practically irreversible, and may cause alteration of the exposed surface of the rock, which can vary from aesthetic modification (loss of brightness, staining) to damage (swelling, scaling, mineral detachment) or even loss of mechanical strength.

The knowledge of possible deterioration in use situations has fundamental importance to its prevention and it is the main objective of the experimental studies presented. They were carried by means of the simulation of climate variation (thermal shock), action of atmospheric pollutants (SO_2), salt mist (both in climatic chambers), and salt crystallization (efflorescence), by partial immersion in acidic solutions. The tested materials were granitic rocks, representative of the Brazilian commercial types.

These ageing tests aimed at the establishment of laboratory methods that would permit the anticipation of deterioration of stone and, alternatively, to introduce durability concepts among the selection criteria of dimension stone. Also, they are important techniques for conservation or restoration of stone monuments or historical buildings.

Résumé: Les pierres de la dimension sont les produits de l'extraction de bloc type matières branlantes et leur sciage subséquent, en polissant et coupant dans plaques et carreaux. Ils sont utilisés comme construire la pierre comme éléments structurels non plus pour une grande part (colonnes et piliers) ou, surtout, comme vertical (murs et façades) et horizontal (chaussées et sols) revêtements d'intérieurs et extérieurs de bâtiments. Les rocs graniteux, pour leur durabilité et grande variété de colours et textural et modèles structurels, ont été les plus usagés.

Ils sont, comme autre pierre et matières de la construction, a tanné naturellement avec le temps. Mais, ce processus peut être des speeded dans conditions climatiques agressives, environnements pollués ou par construction inadéquate ou procédures de l'entretien, résulter en déchéance de la pierre.

Les processus de détérioration ont été en rapport avec l'interaction du petrographic, caractéristiques physiques et mécaniques des rocs avec les agents de l'environnement, empiriquement pollution, interventions humaines et autres. D'une manière caractéristique, la plupart de ces processus, si a commencé, est pratiquement irrévocable, et peut causer modification de la surface exposé du roc qui peut varier de modification esthétique (perte de luminosité, tacher), endommager (enfler, peser, indifférence minérale) ou perte égale de force mécanique.

La connaissance de détérioration possible en usage les situations ont l'importance fondamentale à sa prévention et c'est le but principal des études expérimentales présenté. Ils ont été portés au moyen de la simulation de variation du climat (choc thermique), action de polluants atmosphériques (SO2), brouillard du sel (les deux dans les chambres climatiques), et cristallisation du sel (floraison), par immersion partielle dans les solutions acides. Les matières testées étaient rocs graniteux, représentant des types commerciaux brésiliens.

Ces épreuves vieillissantes ont visé l'établissement de méthodes de laboratoire qui autoriseraient l'anticipation de détérioration de pierre et, ou bien, introduire des concepts de la durabilité parmi les critères de la sélection de pierre de la dimension. Aussi, ils sont des techniques importantes pour conservation ou restauration de monuments de pierre ou bâtiments historiques.

Keywords: weathering; laboratory studies; igneous rock; metamorphic rocks; revetments; durability.

INTRODUCTION

Dimension stones are products of the extraction of block-type rocky materials and their subsequent sawing, polishing and cutting into slabs, plates and tiles. They are largely used as building stone either as structural elements (columns and pillars) or, especially, as vertical (walls and façades) and horizontal (pavements and floors) coverings of interiors and exteriors of buildings.

Granitic rocks, for their durability and great variety of colours, and textural and structural patterns, have been the most used. They are also used as ornamental elements (statuary art, mortuary art) and in the making of sinks, table tops and other decorative pieces.

Most building materials, among them dimension stone, tend to undergo alteration with the time due to weathering; a process which is accelerated when they are subjected to aggressive climatic conditions, polluted atmospheres or inadequate construction or maintenance procedures. These processes, named as stone degradation or simply deterioration, refer to an altered state to worst or, in other words, to the decomposition, damage of the rocky material.

Deterioration processes are usually empirically related to the interaction of the various petrographic, physical and mechanical characteristics of rocks with environmental agents, and construction, cleaning and maintenance procedures. In general, these interactions result in the alteration of the exposed stone surface, be it by the modification of its aesthetic aspects (loss of brightness, staining and others) up to its damaging or loss of mechanical strength.

Diagnoses studies on dimension stone deterioration (Frascá 2004) indicated that most of the problems are related to the end user's lack of understanding of the role played by the modifications resulting from stone processing with the natural and/or polluted atmosphere and its in-use conditions. Considering that any deterioration process, if started, is practically irreversible, a previous knowledge of the possible deterioration mechanisms under in-use conditions is of fundamental importance.

Experimental studies presented here were carried out by simulating climatic variation (thermal variations), the action of atmospheric pollutant and of salt crystallization (efflorescence) with a major focus on granitic rocks. They aimed at, on one hand, to establish a laboratory tests methodology that could anticipate stone deterioration under in-use situations and, on the other hand, to include durability concerns between the criteria of selection of dimension stone for flooring and cladding.

STONE DETERIORATION

The weathering of rocks begins as soon as they get in contact with atmospheric conditions on the Earth's surface. When these rocks are used for civil construction, their degradation over time occurs through changes in their properties under direct contact with the natural environment (Viles 1997). These modifications may be physical and/or chemical, resulting in variations in aesthetic appearance, as well as in a decrease in the strength of the rock.

Changes in aesthetic appearance result in numerous effects such as fading, formation of crusts, stains, surface exfoliation, fragmentation and detachment of minerals, cavities and many others, all of which ultimately resulting in financial costs associated to the need of repairing, cleaning and restoration

The rate and the type of degradation are determined by the nature of the material used and by the environment to which it is exposed. According to Viles (1997), deterioration may be considered a problem when one or more of the following three conditions are in play:

- the natural deterioration process is accelerated if: the environment is naturally corrosive like in coastal areas and desert areas with abundant saline aerosols; and /or the material is relatively weak, ready to undergoing degradation and disintegration;
- the human influence accelerates the deterioration process due to atmospheric pollution, poor maintenance etc.;
- buildings and monuments of great value or significance are affected.

Petrographic characteristics of the rock – mineralogy, alteration, texture and structure, presence of microcracks and/or fissures (porosity) – and climate (temperature and rain intensity, among others) have great influence on the susceptibility and rate of physical and chemical weathering of dimension stone.

Physical modifications of dimension stone attributed to extraction and processing techniques may increase microcracking, porosity, to mention two (Frascá, 2003) which, in the long run, may contribute in accentuating the deleterious effects of weathering agents or of anthropic action.

Chromatic alterations and staining of stone tiles for flooring are frequently caused by reactions with chemical reagents contained in cleaning products. Degradations of the exposed surface of the rock, such as swelling, scaling and detachment of mineral fragments, often result from the action of crystallization of salts (efflorescence and sub-efflorescence) usually derived from mortar (Frascá, 2004).

Causes of stone deterioration

One important property of rocks for flooring and cladding is their durability which is largely reflected in a more effective protection of the building structure thus prolonging the service life. However, the contact of rocks with external phenomena compromises the durability due to temperature variations, acid attack by pollutant and others. Climate, in all its aspects, is one of the fundamental causes of degradation of buildings, through the failure of their constituent materials which, on their part, affect the structure (Feilden, 1994). Physical and chemical actions of water and chemical actions of the atmosphere components are important, above all, when they act together.

The atmosphere and its solid, liquids and gaseous components act on dimension stone through several mechanisms: physical (wetting, salt crystallization and other phenomena caused by variations in the hygrometric state of the atmosphere), chemical (sulfatation of carbonaceous rocks, hydrolysis phenomena in granitic rocks and others) and biological (chemical and/or physical actions caused by various microorganisms: bacteria, algae, fungi and likens, moss and even bushes).

Temperature

The changes in air temperature are almost entirely due to solar heat during the day and the loss of this heat during the night. All construction materials, including dimension stones, expand when heated and contract as they cool down, the so-called thermal movement, which is the major degradation agent of buildings.

The colour and the reflectivity of the material have a direct influence on their heat absorption capacity, which is responsible for the increase in temperature and hence the overall thermal movement. Normally, dark materials (low albedo) absorb more heat than light colour materials (high albedo).

Action of pollutants

The knowledge of the rate and mechanisms of pollutant action could be an important tool for establishing preventive measures and for the protection of rocky material thus increasing their useful life. An inevitable partner of any chemical attack by pollutants is water, because: (a) it acts as a solvent of aggressive agents; (b) it acts as a mean of transport of these agents and of any other products of reaction; (c) in some cases, it is one of the components of the products of reaction (ex. gypsum) (Zivica & Bajza 2001).

In practice, the consequence of the chemical effects due to acidic attack is the gradual degradation of the engineering properties of the rocky material. As already commented, this process begins with the deterioration of the exposed surface in the form of swelling, scaling and mineral detachment, and as deterioration progresses from the exterior to the interior of the rock, there is the gradual loss of strength caused by, among others, an increase in porosity.

Action of the crystallization of salts

The crystallization of salts is one of the most powerful agents of weathering since it is the main cause of rock weathering in marine environments, humid climates and polluted environments. The action of salt crystallization in porous media, more especially in sedimentary rocks, results in the loss of grain cohesion. The main mechanism of degradation is the pressure resulting from the crystallization of salts and depends on the degree of saturation and the pore size (Winkler & Singer 1972).

In general, in the lower parts of a building, closest to the soil, a saline solution can rise through the rock by capillary action until it reaches a potential height determined by its capillarity. In this capillary zone, evaporation and consequent crystallization of salts takes place. The immersion zone, based on this concept, refers to the portion of the rock that is closer to the soil.

The capillary zone depends on the pore system of the rock and is characterized by efflorescence (when salt crystallizes on the surface of the rock), sub-efflorescence (when salt crystallizes underneath the exposed surface of the rock) and by the loss of material (Uchida *et al.*, 1999). If sub-efflorescence takes place, the mechanical action accompanying this phenomenon initiates the destructive action.

THE ALTERABILITY AND DURABILITY OF ROCKS

Aires-Barros (1991) defined the alterability of rocks (M) as a dynamic concept which refers to the ability of a rock to alter with time. Under weathering, this time is said to be geologic while when referring to alterability, it is said to be human, in other words, referring to all phenomena that take place simultaneously while the rock is in use, at the human scale and his engineering works.

$$M = f(i, e, t) \qquad (1)$$

where M = alterability; i = intrinsic factors which are dependent on rock type (nature of the material, microcracking or porosity, which means, the surface exposed to alteration); e = extrinsic factors which are a function of the environment in which alteration is taking place (temperature, pH, Eh, amount of water, biotic forces); t = time.

Based on the fundamental characteristic of the concept of alterability, that is the dynamics of time, several techniques can be used in its evaluation. All of these techniques measure the variation of a certain intrinsic parameter or of a parameter that is intimately correlated to alterability over time.

ASTM (2005) defines durability as a measure of the ability of dimension stone to endure and to maintain its essential and distinctive characteristics of strength, resistance to decay and appearance. Durability is based on the length of time that a stone can maintain its innate characteristics in use. This time will vary depending on the environment, the use and the finish of the stone in question (for example, outdoor versus indoor use). Durability is also conditioned to physical properties, among them the porosity and the pore system, the specific surface and the hydric properties associated with the movement of fluids within the stone material.

ROCKY MATERIALS

Samples were gathered from a large diversity of granite-type rocks mined as dimension stone in Brazil. These rocks were chosen taking into account their previous history on the use for flooring or building cladding as well as their commercial importance in domestic and foreign markets. They are representative of granites (*stricto sensu*) and gneiss, in equivalent quantities. Their mineralogical composition and macroscopic characteristics (Table 1) indicate that they are mainly composed of quartz, feldspar, biotite and/or garnet (Frascá & Yamamoto 2004).

Table 1. Colour and petrographic classification of selected granitic rocks.

SAMPLE	COLOR	PETROGRAPHIC CLASSIFICATION
VCB-1	medium to dark red	(biotite) syenogranite
VCB-2	light red	(biotite) syenogranite
VCB-3	light brownish red	biotite syenogranite
AZF	light bluish gray	biotite monzogranite
PIT	light yellowish gray	biotite monzogranite
LBD	dark brownish green	charnockite (hypersthene syenogranite), garnet bearing
ITN-1	greenish white	monzogranite gneiss, garnet bearing
ITN-2	greenish white	monzogranite gneiss, garnet bearing
CRV	greenish white	"cordierite"-garnet microcline granite gneiss
RIC	pink	(biotite) monzogranite
BCR	greenish pink	(biotite) monzogranite
BCE	white	albite granite
CSB	pinkish white and greenish black	quartz syenite gneiss bands irregularly alternated with muscovite tonalite gneiss bands (migmatite)
GVN	yellowish pink	syenogranite gneiss, garnet bearing
ARB	greenish gray	biotite monzogranite gneiss, garnet and sillimanite bearing
SCC	grayish orange	biotite monzogranite gneiss, garnet and sillimanite bearing

ACCELERATED ALTERATION TESTS

Accelerated alteration tests simulate situations of deterioration under laboratory condition. They are carried out with the objective of providing information on the alterability of rocks in relation to the weathering agents and hence investigate the mechanisms responsible for each type of degradation process.

Besides, these investigations also intended to include the durability concepts among the parameters to be considered when choosing and selecting dimension stone. The durability of building materials is now fundamental, because of the more pronounced and increasing aggressiveness of the environment.

Several causes and mechanisms of stone deterioration may be listed. Due to their importance, the exposure to marine and polluted environments, thermal variations and the action of the salt crystallization (Table 2) were selected as the subject of the presented investigation.

Table 2. Situations of potential degradation of dimension stone.

SITUATION	TEST	OBJECTIVE
Thermal Variations	Exposure of polished tiles to thermal shock	Verify eventual decay of the rock strength after cycles of alternate heating and immediate cooling in water.
Action of Pollutants	Exposure of polished tiles to acid and saline atmospheres, in climatic chambers.	Simulation of polluted urban (humidity and H_2SO_4) and marines (salt mist) atmospheres, potentially harmful to stone materials.
Action of Salt Crystallization	Partial immersion of polished tiles in acid solution.	Simulation of salt crystallization (efflorescence and sub-efflorescence) on the polished surface of rock tiles in order to observe possible deleterious effects.

The simulation of the above mentioned alteration tests aimed at verifying any modifications with respect to the mineralogical composition of the rock based on the response of their intrinsic characteristics when exposed to the same potentially degrading environments. These tests were carried out within a group of homogeneous granitic rocks relative to their mineralogy, but showing a heterogeneous behaviour relative to the geological environment.

Tests in climatic chambers

Due to the need to evaluate the resistance of the materials to weathering much more rapidly than under outdoor exposure, several equipments usually used in accelerating the degradation, designated generically as "climatic chambers", were conceived and built. Among them, those that simulate the exposure to sulphur dioxide and salt mist were selected.

Exposure to sulphur dioxide

In the degradation tests under exposure to SO_2, the sample was exposed to a certain number of 24 hours-cycles. Firstly, the chamber was maintained heated to a temperature of 40°C and relative humidity of 100% for 8 hours. It was then switched off and left open to ventilate for 16 hours. After that, the water in the chamber was renewed to start a new cycle. The operation of the equipment was based on DIN (1997) standard. The concentration of SO_2 employed in this test according to the aforementioned standard was of 0.67%, resulting in a pH of approximately 2.

Three test specimens per sample were tested. From each sample, a standard specimen was set aside for control. Inspections were carried out periodically by registering the observed modifications. A detailed description of this method and major results were presented by Frascá & Yamamoto (2004). The main degradations observed are shown in Figures 1 and 2.

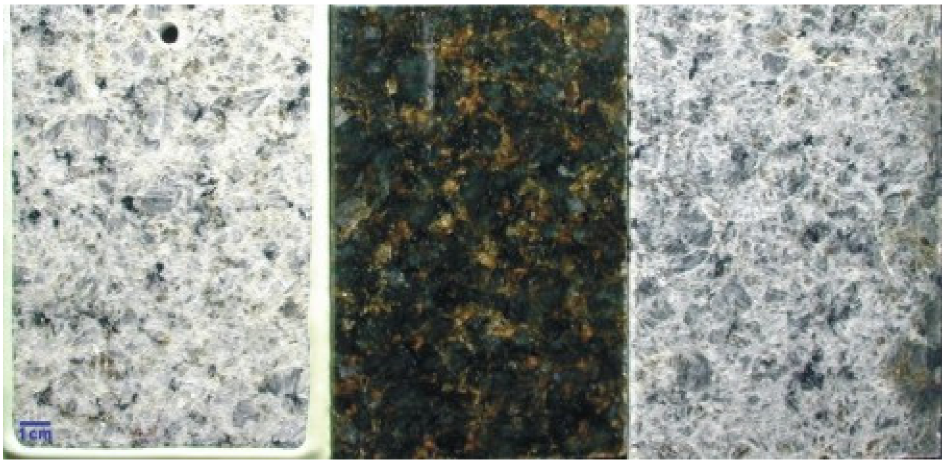


Figure 1. Almost total bleaching of the rock (control specimen in the centre) after exposure to sulphur dioxide.

Figure 2. Formation of salt (in white) in surface microcracks and locally on biotite after exposure to sulphur dioxide (control specimen on the right).

Exposure to salt mist

The salt solution used for this artificial weathering tests was prepared by dissolving sodium chloride in water in such a quantity that the pH of the resulting solution is between 6.5 and 7.2. The test specimens were then placed on supports in a testing chamber at an angle of 15° to 30° relative to vertical that allowed free exposure to the salt spray. The operation of the equipment was based on ASTM (1997) standard codes.

Three specimens per sample were tested and one per sample was set aside for control. They were maintained permanently inside the chamber and removed only for periodic inspections. During each inspection, which did not last more than 30 minutes, the specimens were washed to remove the salt on the surface. Any changes were then registered. The main results observed are illustrated in Figures 3 and 4.

Thermal shock

The thermal shock test was based on the of BSI (2003) standard procedures. This test was carried out with the objective of verifying the decay of the rock after cycles of heating and cooling, what means, after abrupt thermal variations due to constant expansion and contraction, one of the major causes of the degradation of rocky materials for building construction purposes. The test is also recommended for the verification of any possible oxidation in igneous rocks and any disaggregation in marbles and limestone.

Figure 3. Increasing of pre-existent oxidation after exposition to salt mist (above: control specimen).

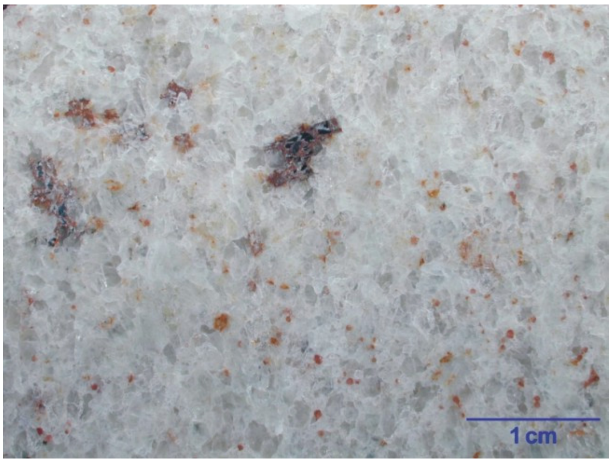


Figure 4. Increasing of pre-existent yellowing (oxidation?) after exposition to salt mist.

The test itself consisted of 20 cycles of alternate heating and cooling. For each cycle, the specimen was placed in an oven for 18h at 105°C and then immediately submerged in water at room temperature for 6h. After the 20th cycle, the specimen was dried again and then subjected to flexural strength test, in accordance to ASTM (1998).

The decay in strength due to thermal shock is given by the expression:

$$\Delta_{RF} = \frac{RF_f - RF_i}{RF_i} x100 \qquad (2)$$

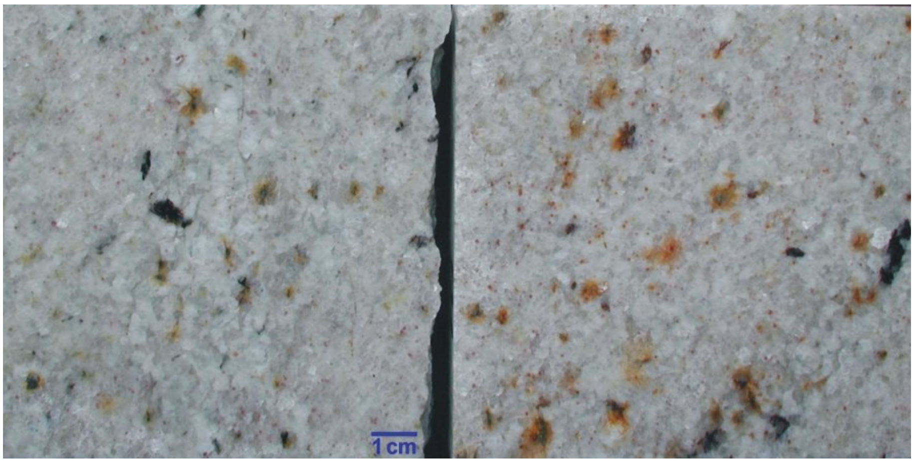
where Δ_{RF} = variation in flexural strength (%); RF_f = final flexural strength (MPa); RF_i = initial flexural strength (MPa).

Besides the variable and sometimes strong strength decay (up to 50% in some tested rock), another important result obtained from the thermal shock tests carried out in granitic rocks was the change in colour due to the oxidation of some minerals. It usually induced yellowing, with greenish tonalities, as well as the appearance of ferruginous spots (Figure 5).

Partial immersion in acid solution

In order to reproducing the deterioration of rock due to the salt crystallization, test specimens were put in contact with a solution of sulphuric acid (H_2SO_4 - 1%), with pH ranging from 1 to 1.5. The choice of sulphuric acid was based on earlier observation that showed that the attack by SO_2 is one of the major atmospheric pollutant rock degrading agents.

The test consisted on the partial submerging (up to 1 cm from the bottom) of test specimens in flat bottom plastic containers, with the mentioned solution. The polished faces were exposed to the environment so that any eventual modifications would be registered on them. It is important to note that this test should be carried out in a dry room with a temperature of about 25ºC.



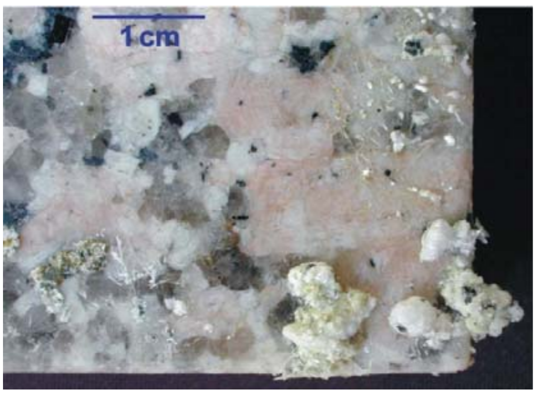
natural after thermal shock

Figure 5. Yellowing and appearance of ferruginous spots after thermal shock.

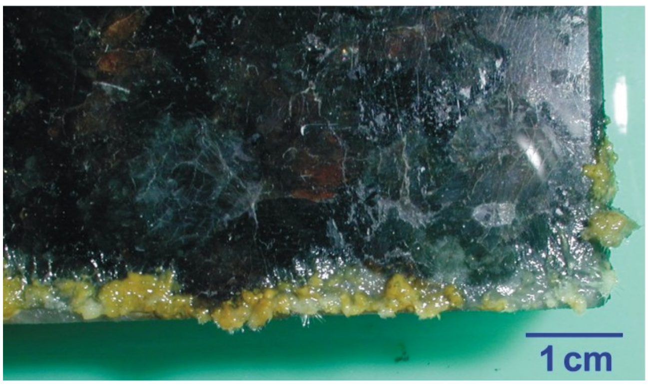
The specimens were subjected to testing for a period of 30 consecutive days with daily monitoring and changing of the solutions in alternate days and annotation of any observed modifications. After this period, the solution was removed and the specimens left in the same environment for another period of 30 days, monitoring until they were fully dry and efflorescence and sub-efflorescence formed. Only after this period the specimens were washed with deionised water, dried at room temperature and then, ready for investigation of any deterioration that might have taken place by comparing with their counterpart.

The major aspects of efflorescence formed by the partial immersion in sulphuric acid are illustrated in Figures 6 and 7 (a, b). The most typical degradations observed in the studied granitic rocks are shown in Figure 8 (a, b).

Figure 6. Crystallization of salts at the borders of test specimen, preferentially on biotite sites (control specimen on the right).

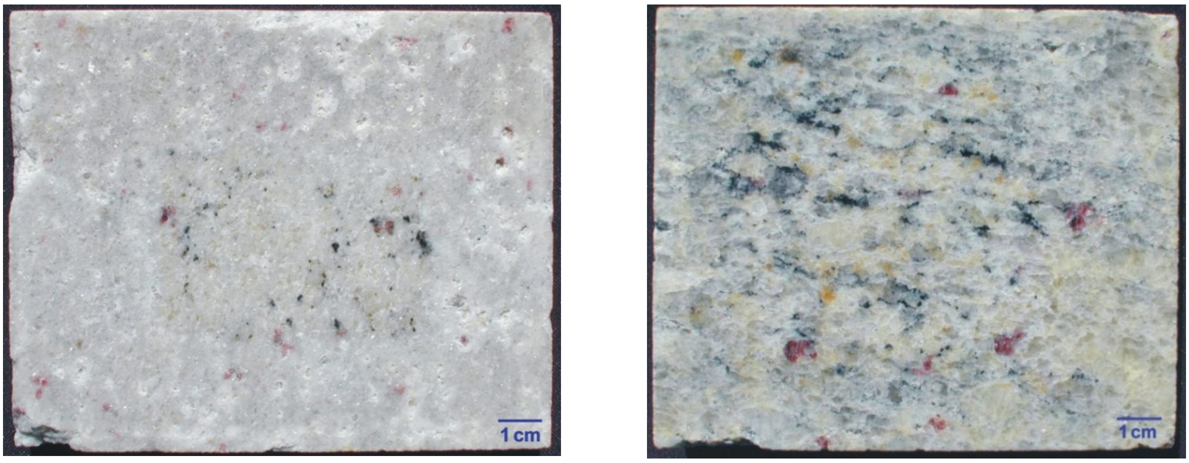


(a)



(b)

Figure 7 a, b. Different aspects of efflorescence formed after partial immersion in sulphuric acid of granite (a) and charnockite (b) rocks. Note the separation of sheets of biotite (in black), which are held to the surface of salt grains (a).



(a) (b)

Figure 8 a, b. Aspects resulting from the crystallization of salts (efflorescence e sub-efflorescence) after continuous partial immersion in sulphuric acid solution. Note the formation of a cavity and the breaking on the corners.

CONCLUSIONS

The visual observation of the effects of the accelerated weathering tests on the studied rocks evidenced the following mechanisms of deterioration:

- Oxidation of the rock and minerals: this mechanism was observed in practically all simulated situations and is related the behaviour of iron as consequence of its oxidation potential in an exogenous environment. In this study, it was given by the acid pH environment (exposure to sulphur dioxide and the partial immersion in sulphuric acid) or by the of temperature and humidity variations (thermal shock test);
- Discoloration (bleaching) of the rock: observed when some specimens were exposed to sulphur dioxide and partial immersion in sulphuric acid. This degradation is attributed to iron leaching of dark minerals in acid environment, especially biotite;
- Crystallization of salts (efflorescence and sub-efflorescence): observed in the exposition to sulphur dioxide and partial immersion in sulphuric acid. It was marked by swelling, scaling and even exfoliation. Locally, the presence of salts in the fissures promoted detachment of fragments of garnet and feldspars. However, biotite was the most susceptible mineral.

The characteristics of the alteration tests carried out can be synthesized as follows:

- Exposure to salt mist: this test was accompanied by unnoticeable visual effects. Nonetheless, under the microscope the effects due to degradation caused by salts (NaCl) dissolved in the atmosphere may be seen, as: increasing in the alteration of previously weathered plagioclase crystals; enlargement of pre-existent microcracks; intensification of oxidation and/or appearance of ferruginous spots etc.;
- Exposure to sulphur dioxide: demonstrated the role played by sulphur dioxide as a potential degrading agent. The resulting deteriorations affected, in different ways, depending on the intrinsic characteristics of the rock, practically all granitic rocks tested and ranged from simple staining up to scaling;
- Thermal shock: quite an aggressive test that provided substantial information on possible aesthetic changes due to the mineral oxidation and modifications in mechanical strength of the material due to weathering action and cyclic variations in temperature and humidity. The decay in flexural strength, after this test, was shown to be directly proportional to the strength of the rock *in natura* (before tests). However, this relationship was not noticed for the case of the susceptibility to oxidation which, a priori, is related to the intrinsic characteristics of the rock (mineralogy, alteration, microcracking, etc.);
- Partial immersion in sulphuric acid: also a very aggressive test. It made it possible to verify the potentiality of any deterioration resulting from the crystallization of salts (efflorescence and sub-efflorescence). The mechanism of degradation consisted in the migration of the solution through the rock, by capillary action, from the immersed zone to the exposed zone (polished faces), where salt crystallization occurred after evaporation and supersaturation.

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