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Colour and Roughness Measurements as NDT to Evaluate Ornamental Granite Decay

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Abstract

Granitoids are selected for ornamental purposes due to their texture and colour. The determination of granitoid durability has been slightly neglected since most of standard weathering tests have been developed for stones with more than 5 % porosity. In addition, the evaluation of the decay is commonly focused on loss of mass or fracturation. In this study, the decay of granitoids was assessed with NDT such as colour and roughness. For this research, four granitoids commonly used as ornamental stones have been studied. They show differences in colour and texture, thought porosity is similar. Four weathering tests were carried out: salt crystallization, Freeze-Thaw cycles, SO_2 exposure and heating to different temperatures. Standard test were adapted to focus the damage on the surface. Colour and roughness measurements were carried out before and after the tests. We concluded that, in the decay tests in which chemical reactions were not involved, roughness measurements gave accurate information about the decay. In the decay tests in which chemical reactions are produced, such as SO_2 interaction or mineral transformation due to high temperatures, both colour and roughness could evidence the decay, even if colour change is faster and easier to observe and measure.

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Keywords: Granitoids; colour; roughness; NDT; weathering test.

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1. Introduction

When granitoids are placed in a building, their surface is exposed to a wide range of weathering agents, which produce changes in their aspect (Esbert, 2007). Most of the researches on granitoid decay are focused on fracturation

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due mainly to temperature changes (Ruiz de Argandoña *et al.,* 1986; Menéndez *et al.,* 1999; Gomez-Heras *et al.,* 2006; Vázquez *et al.,* 2015a). However, weathering forms of new panels in existing buildings are less identified. When a stone panel is used as façade in buildings and monuments with decorative purposes, surface aesthetics are the main criteria to evaluate the decay. Roughness, gloss and colour are key properties to assess surface weathering (Alonso *et al.,* 2008; Vázquez *et al.,* 2013). In urban and industrial areas, the deposition of pollutants leads to the formation of soot patinas and black crusts. The consequence is a total change on the visual appearance of buildings stones, such as blackening (Grossi *et al.*, 2003; Grossi et al 2007; Török et al., 2011). Changes on the surface finish, mainly related with roughness, may also cause colour variations (Benavente et al., 2003). The increase in surface roughness of stones is one of the most important consequences and indicators of weathering (Alonso et al., 2008; Lopez-Arce et al., 2010; Vázquez et al., 2013; Vázquez et al., 2015b). Dissolution, fracturation and deposits can be quantified by roughness measurements. In granitoids, each mineral shows a different response to weathering. For example mica may detach and plagioclase may be altered to sericite. Cracks are formed if submitted to temperature changes due to mineral different thermal expansion (Gómez-Heras et al., 2006; Gómez-Heras et al., 2008; Vázquez et al., 2015a) or to the pressure induced by ice and salt crystallizations (Alonso et al., 2008; López-Arce et al., 2010; Vázquez et al., 2013). Four granitoids commonly used as ornamental stones have been studied. They showed differences in colour and grain size, thought porosity was similar and very low. Four weathering tests were carried out: salt crystallization, freeze-thaw cycles, SO_2 exposure and heating to different temperatures. Due to their low porosity, standardized tests are not suited to evaluate the damage of these stones since porosity must be at least 5%. Standard test were adapted to focus the damage on the surface. The decay was assessed by non-destructive techniques such as visual observation, colour and roughness.

2. Materials

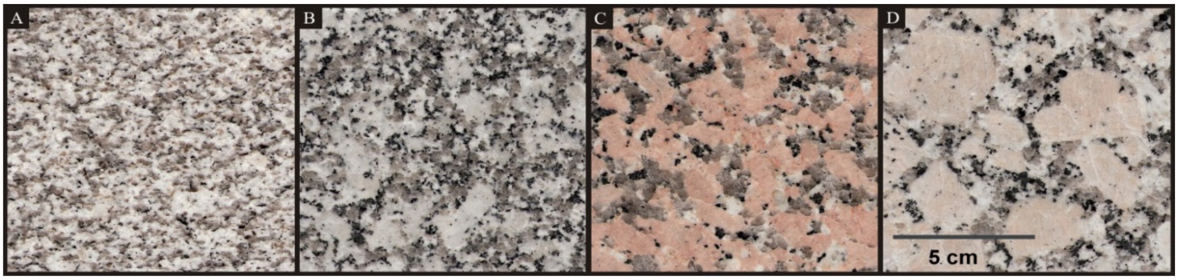

Fig. 1: Selected granitoids. A) Gris Alba; B) Grissal; C) Rosa Porriño; D) Rosavel.

Four granitoids with different texture and mineral composition, have been selected (Fig. 1). Gris Alba (GA) is a two mica, grey, medium and homogeneous grain sized monzogranite. The colour of this stone is light grey. Grissal (G) is a coarse grain sized and, heterogeneous monzogranite. The colour of this stone is pale grey. Rosa Porriño (RP) is a coarse grain sized and heterogeneous syenogranite. The general colour of this granitoid is orange-red. Rosavel (R) is a porphyritic quartzsyenite. The general colour of the stone is pale pink. More details can be found in Vázquez et al., 2013.

3. Methodology

2.1. Decay tests

The stone durability was assessed by means of artificial ageing tests, such as salt crystallisation, freeze-thaw cycles, SO_2 and high temperature exposure. Some of the tests were modified in order to focus on the damage on the surface. This test was based on the standard UNE-EN 12370:1999. The drying stage was carried out during 20 hours in each cycle at 25 ºC to simulate real environments. This temperature avoided thermal shock and produced a low evaporation rate that allows the salt to migrate to the surface and crystallize as efflorescence. The duration of the test was increased to 60 cycles. The specimens tested were four slabs of each type of stone with dimensions 10 × 10 × 2 cm (Vazquez et al., 2013).

Freeze-Thaw test was carried out based on the standard UNE-EN 12371:2011. In this study, the specimens were immersed in water during the whole test. The reason was to force the damage onto the surface as a result of ice formation and melting. The test was performed for 200 cycles. Specimens were cubes of 5 x 5 x 5 cm.

This test was carried out following the standard UNE-EN 13919:2003. The test consisted of placing the specimens in a closed recipient with a film of sulfurous acid solution for a period of three weeks that creates an acid atmosphere. The specimens tested were 12 x 6 x 1 cm slabs. High temperature tests were carried out heating the samples in a furnace at target temperature of 200 ºC, 400 ºC, 600 ºC, 800 ºC and 1000 ºC respectively. The temperature was risen up with a heating rate of 6 ºC/min in order to produce irreversible thermal expansion in granitoids. Once the temperature was reached, the specimens were maintained for a period of three hours, in order to assure that the core sample gets the same temperature as the surface (Argandoña et al., 1986; Chakrabarti et al.,1996). After this time, the temperature decreased freely with a cooling rate about 0.3 ºC/min so that the damage was produced only by heating. The specimens were tablets of 7 x 4 x 2 cm.

2.2 Damage evaluation after the tests

Colour was measured and quantified with a MINOLTA CR-200 colourimeter. Due to the heterogeneous texture of the studied granitoids 100 data points were the minimum required in each slab. Measurements were expressed following the CIE L* a* b* and CIE L* C* h* systems (EN ISO 105-J03:2009). ΔE* was introduced as the total colour change, in order to compare the variations before and after the tests:

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$$

Because of the quartz transparency, surface roughness was measured using a contact profilometer Mitutoyo Surftest SV-2000N2, with a 2 μm diamond tip that moves on the surface of measure with constant speed. The number of profiles measured was 25 with a spacing of 0.5 mm and 50 mm length. Some granitoid deformation as the produced by heating may exceed this threshold. In this case, an optical stereomicroscope Leica MZ-16A with the aid of the software "Leica Stereo Explorer 2.1" was used. Fifteen profiles in two areas of 2.5 x 1.8 cm were measured in each slab (Acuña, 2011). Three parameters were selected to define roughness (EN 4287, 1997): Ra Arithmetical mean deviation of the profile, Rp Maximum profile peak height; Rv Maximum profile valley depth.

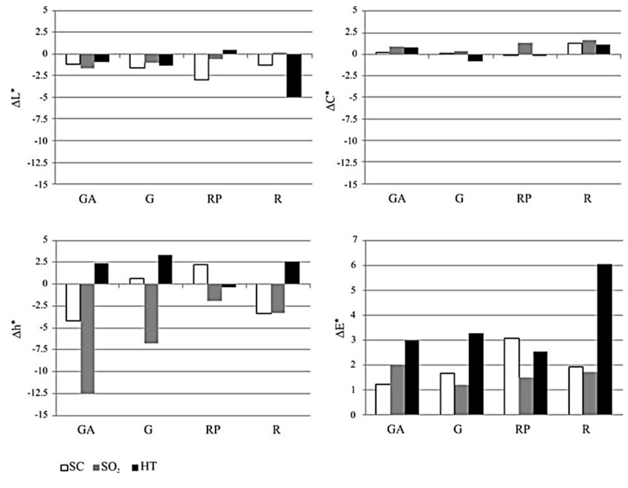

Fig. 2: Absolute variation of colour parameters before and after each ageing test.

4. Results and discussions

3.1 Colour Changes

Colour changes are related to the nature of the decay agent, the chemical reactions that may take place and the mineralogy and texture of the granitoids. If the decay agent produced only mechanical damage, no strong colour changes were expected. If chemical reactions took place, a colour variation more or less pronounced could be attained. Figure 2 shows the colour change for the different ageing test. The freeze-thaw test did not show any variation since only mechanical decay was produced. Therefore, this test is not represented in the graphs.

Salt crystallized as efflorescence on the stone surface, producing a slight mechanical damage (Alonso et al., 2008; Vázquez et al., 2013). Even if little chemical reaction occurred between the sodium sulfate and the granitoid minerals, some colour variations were observed at the end of the test. The most remarkable variation in lightness was observed in RP, while R was the only granitoid that increased its chroma. The plagioclases from these granitoids have an anorthite proportion between 10-25%. The calcium of the plagioclases may react with the sodium sulfate and produce sericitisation. Thus, RP and R exhibited at the end of the test a strong deterioration in plagioclases with greenish colour due to chemical alteration that explains the decrease in lightness. If total colour change is evaluated, only RP after 60 cycles may reach 3 units, considered as the visible threshold (Fig. 2).SO_2 exposure produced both mechanical and chemical decay. SO_2 reacts with the calcium from the plagioclases and gypsum crystals precipitate on the surface, preferably in the grain boundaries and between mica sheets. Besides, the acid attack produces the iron oxidation and the colour change in gypsum crystals and minerals to yellow and orange (Fig. 3a, b) (Grossi et al., 2007; Vázquez and Alonso, 2010; Török et al., 2011). Colour changes were remarkable in grey granitoids, showing a decrease in lightness, increase in chroma and an evident and homogeneous yellowing measured as a decrease in hue. Pink-red granitoids did not exhibit any variation perceptible to human eye, therefore they are considered optimal to be utilized in polluted areas (Fig. 2). The total colour change did not exceed the visible threshold. However, colour variations were clearly perceptible due to their heterogeneous distribution. (Fig.3b).If temperatures are high enough, chemical changes such as clay dehydration and iron oxidation may take place, and consequently a variation in the stone colour (Chakrabarti et al., 1996; Beck et al., 2015). When the grey granitoids were heated up to target temperature of 200ºC, yellow colour exhibited a strong increase. L* and b* augmented very slightly during the first heating stages at 200 ºC, 400ºC and 600ºC and rose evidently at high temperature ranges. That gave hue that turned into orange tones. For the grey granites, the variation of ΔE* is almost imperceptible through these temperatures, with values around 3-4 at 600ºC (Fig. 2). From 800 ºC to 1000 ºC all the parameters continue to increase in those granitoids that were not destroyed. When the pink-red granitoids were heated, the colour changed progressively with temperature. ΔE* was evident in R, due to the change of colour of pale pink alkali feldspars to yellow tones. In relation to all the weathering tests, colour changes were especially evident in SO2 exposure and heating tests. Colour hue tended to yellowing in the case of SO2 exposure, and reddening in the case of granitoid heating. In both cases grey granitoids exhibited a high colour change, evident with human eye. In red granitoids, the mineral inherent colour masked the reddening or yellowing, making more difficult its detection with naked eye.

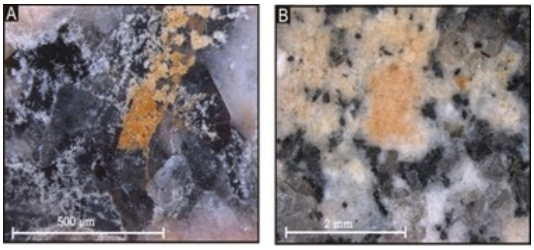

Fig. 3: SO_2 attack on granitoids. A) Gypsum crystals contaminated by iron and growing preferentially in the grain boundaries and mica sheets. B) After cleaning, oxidation remained as orange colours in white minerals.

3.2. Roughness Changes

Roughness changes are mainly related to mechanical damage as cracks or fissures measured as an increase in valleys, or the salt deposit that vary the roughness equally in valleys and peaks. Fig. 4 shows average roughness (Ra) variation in each test in all the studied stones, Rp that means mica changes over the surface and Rv that can be explained as crack growing and mica detachment. In salt crystallization, roughness increased with the cycles. R showed the highest variations in most of the parameters followed by GA (two micas granitoid). RP, with the biggest and more cracked quartz crystals, showed the deepest valleys after the test. In general, minerals exhibited different behavior: there was an increase of mica peaks while feldspars remained intact (Alonso et al., 2008, López-Arce et al., 2010; Vázquez et al., 2013) (Fig. 5).

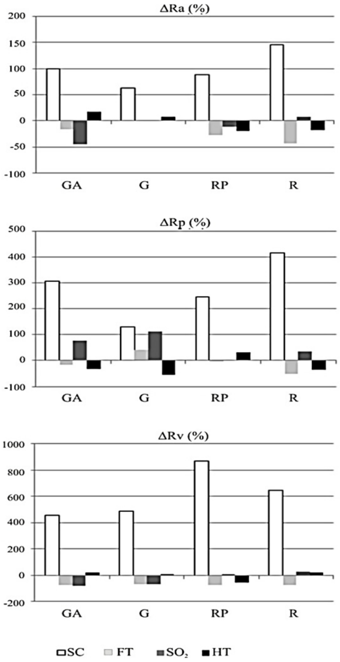

Fig. 4: Percentage variations of roughness parameters before and after each ageing test.

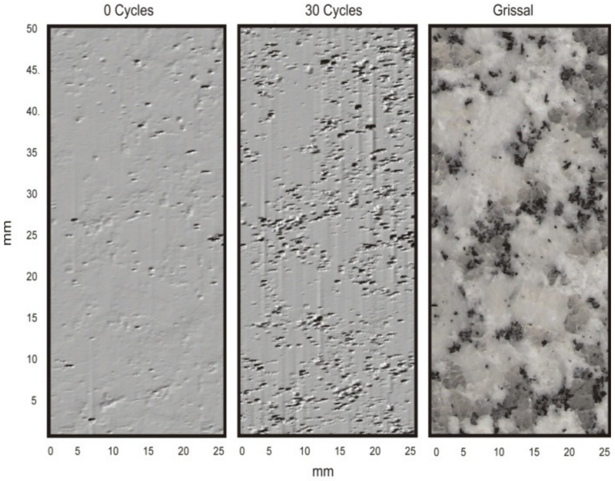

Fig. 5. Roughness variation before and after 60 cycles of salt crystallization (Grissal granitoid, G). Mica detachment can be observed, whereas feldspar remained intact.

After Freeze-Thaw test the average roughness variation was similar for all granitoids. In general, roughness decreased slightly. That can be explained by an erosion of the surface due to the ice layer. This caused a diminution of Rp, and by equilibration of the roughness profiles, also a decrease of Rv. SO_2 exposure test demonstrated a roughness variation similar to the salt crystallisation test, but with less intensity (Vázquez et al., 2015b). The main changes corresponded to an increase of peaks due to the mica detachment and a decrease of valleys due to cracks filling with gypsum crystals. This effect was more evident in grey granitoids GA and G due to higher plagioclase content. An increase in temperature produces mechanical decay due to the differential thermal expansion between minerals. These differences produce stresses that lead to microcracking (Menéndez et al., 1999; Gomez-Heras et al., 2006; Gomez-Heras et al., 2008; Vázquez et al., 2015a). When exposed to high temperatures, the coarse grain sized granitoids G, R and RP showed no variation of roughness even after heated up to 600ºC. At 800ºC there are increases of all the parameters, and again at 1000ºC a diminution that leads to the recovering of the initial values. GA, with fine grain size and high mica content showed an evident variation of roughness, although patterns were not easy to recognize. Values rose and decreased randomly through the heating stages, although Ra and Rv showed a general trend to increase throughout temperatures. That implies the crack opening, mainly intergranular, and also grain detachment (Gómez-Heras et al., 2010; Vázquez et al., 2015a). The sample fracturation is clearly visible at 600ºC and high temperatures. Sodium sulfate, ice and gypsum crystallize on the surface, mainly in the highest relief areas, which in granitoids belong to mica exfoliation planes. The main changes were the consequence of physical damage related to mica (Lopez-Arce et al., 2010) and physico-chemical decay of Ca-rich plagioclases (Vázquez et al., 2013). This phenomenon correlated with the high increase in roughness after the sodium sulfate crystallization, medium increase in roughness after SO_2 exposure and gypsum crystal formation, and almost no change after the ice damage. Heating produced a differential mineral expansion that produced microcracking. Differences between coarse and fine grain sized granitoids were detected, with a higher increase of roughness in fine grain sized and mica-rich granitoids.

5. Conclusions

Surface properties exhibited evident changes and with variable intensity through the different tests. Colour and roughness measurement resulted in optimal non-destructive techniques to evaluate the decay. Colour variations are related to chemical attack. Thus, SO_2 exposure and heating were the tests in which the colour changes were the most evident. SO_2 exposure produced not only a particle deposition but also a reaction with the Ca present in plagioclases, leading to gypsum formation and a yellowing of the surface. The acid atmospheres reacted also with iron oxides that produced orange spots on the stone. A temperature increase produced as well colour changes due to the oxidation of iron mainly from mica. Roughness experimented high variation through mechanical processes. Salt and ice exert a pressure when crystallize in the mica planes and grain boundaries. The increase of temperature leads to a mineral expansion, producing micro cracking and mineral detachment. GA the fine grained size and mica rich granite showed the highest variation.

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