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Evolution of surface properties of ornamental granitoids exposed to high temperatures

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HIGHLIGHTS

- Heated granitoids varied their properties of surface, colour and roughness.
- Grain size and mica content influence roughness variation.
- Colour changes are conditioned by mineralogy.
- Granitoids with clays from alteration did not experiment catastrophic failure.
- Surface properties are good indicators of thermal decay in granitoids.

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ABSTRACT

Granite submitted to high temperatures may lead to the loss of aesthetic values even before structural damage is caused. Thirteen granitoids were exposed to target temperatures, 200 °C, 400 °C, 600 °C, 800 °C and 1000 °C. Damage characterisation, including roughness, colour and oxidation of chromogen elements by means of X-ray photoelectron spectroscopy (XPS) was assessed. Altered granitoids are more resistant to structural failure but redden rapidly. Black mica-rich granitoids turn into yellow with a maximum at 800 °C. Alkali feldspar-rich granitoids redden progressively due to iron oxidation. Roughness varies progressively in mica-rich granitoids, while in mica-poor granitoids, an increase in roughness precedes catastrophic failure.

1. Introduction

Standards usually consider stone as a material resistant to fire since they are no combustible materials (classification A1 in accordance with the standard UNE 13501:2007 [1]). Fire cannot propagate through stone, so it is perceived among the most resistant material used in construction. Nevertheless, fire may cause irreversible damage to buildings both structurally and aesthetically, being the most noticeable decay reddening, which is also called rubefaction [2]. There are two main factors which affect building materials during a fire. Firstly, the heat produced by the flames. Heat affects materials mainly by radiation, but also by incandescent particles in direct contact to the material [3]. In most cases, the high temperature reached is enough to produce short-term irreversible physical–chemical changes on the stone. Secondly, ashes and fumes emitted by fires may have a longer-lasting effect on building materials. Because of this, fire became a relevant topic in the study of stone decay from early times [e.g. 3–6].

Before the microscopic studies made by Tarr [4] on samples affected by fire, only qualitative observations existed on the effects reddening of fire on rocks, with comments on the lower resistance of granitoids to fire in relation to some sedimentary rocks, and the higher decay shown by coarse-grain rocks compared to fine-grain

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ones [7]. Two main groups of rocks, in relation to their mechanical behaviour after a fire, could be distinguished from Tarr's research: crystalline competent rocks, showing cracking after a fire and sedimentary rocks that would not show structural damage.

The effect of fire on building stone has been widely researched in the last few decades. The first studies of the effect of high temperatures on building stone focused on the variation of bulk mechanical properties in crystalline stones and colour changes due to iron oxidation in sedimentary stones [3,8,9]. From this initial focus on macroscopic variations, research in the last years drifted to assessing mineralogical and textural changes and their effect in building stones [10–23]. Most of these studies focused on the effects of temperature increase, without taking into account ashes and fumes, and therefore were carried out in furnaces on relatively large samples [e.g. 8–25]. Commonly, samples were heated with different rates, kept at constant temperatures during a certain time interval and then cooled down freely. Sometimes, the focus was on thermal shock produced by spraying or by immersion in cold water [11,25] or the consequences of localized heat, such as the effect of fire jet to induce flamed artificial finishing of building stone [26] or lasers as a way of reproducing heating effects in small samples [27]. Although these laboratory studies were sometimes complemented with studies of samples burnt in real fires of historical monuments [9,12,13,17,18] there is still few laboratory research on the effects of "real fires", i.e. considering flame heterogeneity, ashes and fumes [24,25]. The results obtained were characterized by a heterogeneity on the damage pattern, due to the random movement of fire, with blackening, reddening and fracture as principal damage forms.

Building stone composition and texture condition its behaviour against fire [9]. All rocks show a general increase in porosity and cracking with temperature, especially those with lower porosity [15]. Limestone and marbles often change colour when iron minerals are present [17,22]. Sandstone strength increases when heating is moderate, but it is reduced substantially when temperature increase is more intense. The overall degree of decay will depend on the amount and composition of matrix and cement and the subsequent changes in mineralogy, colour and cracking [10,12,14,15,21,23]. Granitoids show different degree of fracturation depending of the temperature [28], showing generally variations in roughness [19,20,26].

Granitoids are widely used as building stone due to their appearance and comparative resistance to weathering agents. Ornamental granitoids are characterized by a low porosity and, therefore, decay less in terms of overall strength in comparison to other stone types when subject to weathering agents such as salt crystallization or freezing [29–31]. However, granitoids may show intense changes of surface properties, such as gloss, colour or roughness, which may lead to inadmissible aesthetic damage when these are used as ornamental stone [29,32–34]. Stone surface properties depend on mineralogy, texture and artificial finishing. Colour, for example, is related to mineralogy and texture and to a lesser extent to surface finishing [32,35]. Roughness is an usual parameter to evaluate decay since it is a non-destructive technique and gives information about dissolution or fracturation as consequence of weathering [19,20,23,27,35,36]. In the case of granitoids, the variation of parameters related to peaks and valleys variations provides information about the decay such as dissolution, crack opening or mica detachment [26,29,34].

Surface properties of granitoids are affected substantially by fire. Annerel and Taerwe [37] explained how colour may indicate the maximum heating temperature. Studies on the variation of surface properties of granitoids exposed to high temperatures are scarce and references on roughness variations are always found in relation to structural decay [19,26]. Under the light of these considerations, the aim of this paper is to study the variation of colour and roughness through different temperature heating with a focus on the aesthetic value of granitoids as ornamental stone. To reach this aim, thirteen granitoids with different mineralogy, colour, grain size, and initial weathering degree have been tested. All tested granitoids had the same surface finishing for roughness variations to be compared. In addition, X-ray photoelectron spectroscopy (XPS) analyses were carried out in order to explain the chemical variations leading to colour changes.

2. Materials

Thirteen granitoids used internationally as dimension and ornamental stone were studied. All of them differ in their mineralogy, texture and physical properties. Their commercial names are Albero (A), Azul Platino (AP), Grissal (G), Gris Alba (GA), Gris Mondariz (GM), Negro Galicia (NG), Rosavel (R), Rosa Porriño (RP), Silvestre Moreno (SM), Tezal (T) all from Spain; Golden SKI (GS) from Portugal, Eagle Red (ER) from Finland and Rojo Multicolor (RM) from India (Fig. 1). All of them have saw finish.

Mineral proportion and grain size were studied using optical polarising microscopy (MOP) and digital image processing (DIP). The mineralogical composition of samples was corroborated by powder X-ray diffraction (XRD) on a PANalytical X'Pert Pro. XRD patterns were collected and interpreted using the XPowder software package. The qualitative search-matching procedure was based on the ICDD-PDF2 database. Results are shown in Table 1. All granitoid types were also analysed with a Thermo Scientific NITON energy-dispersive X-ray fluorescence (EDXRF) analyzer, model XL3t GOLDD+ with a beam diameter of 5 mm. Measurements were made in different areas of the intact stones and the maximum and minimum are shown in Table 2. Only the composition in major elements that can influence chemical changes on the stone are represented in Table 2 such as iron and titanium.

- Albero (A) (Fig. 1a) is a homogeneous fine-medium grained (≈5 mm) granodiorite. Alkali feldspar and plagioclase are whitish. Mica content (biotite group minerals and muscovite) is very high (≈25%), with similar proportion of both mica types. A shows a notable initial weathering with presence of clays that gives the stone a yellowish hue. A has elongated xenomorphic minerals oriented following the foliation. The proportion of alkali feldspar is one of the lowest among the studied granitoids. This granitoid is characterised also by open transgranular cracks, and consequently the highest porosity among the studied stones.
- Azul Platino (AP) (Fig. 1b) is a heterogeneous coarse grained (≈9 mm) granodiorite. Feldspars show tabular habit and grey-blue tonalities which give to the stone a characteristic blue colour. AP exhibit higher content in muscovite (11%) than biotite group minerals (2%). AP shows subhedral crystals. Cracks are mainly intergranular although also transgranular in quartz can be found.
- Eagle Red (ER) (Fig. 1c) is a homogeneous coarse-grained (≈6 mm) alkaline feldspar granite. ER shows euhedral red alkali feldspar and homogeneous sized and shaped quartz, giving to the stone a general red colour. Quartz and alkali feldspar content are one of the highest within the studied granitoids with scarce and fine biotite group minerals and plagioclase. Cracks are very thin and with intergranular distribution. The porosity is one of the lowest among the selected granitoids.
- Grissal (G) (Fig. 1d) is a heterogeneous coarse grained (≈10 mm) monzogranite. Feldspars are pale grey and idiomorphic. Plagioclases are bigger and more calcitic in relation to the majority of granitoids and show microcracking. The overall colour of the stone is greyish.
- Gris Alba (GA) (Fig. 1e) is a homogeneous fine grained (≈4 mm) monzogranitoid. GA has subhedral to anhedral minerals with irregular boundaries in quartz. The proportion of muscovite:biotite group minerals is approximately of 2:1 and they exhibit mineral shape orientation. Cracks are mainly intergranular following mica boundaries. Feldspars are white, so that the general colour of the stone is grey.
- Gris Mondariz (GM) (Fig. 1f) is a highly heterogeneous coarse-grained (≈14 mm) monzogranite. Alkali feldspars show brownish colour and tabular habit, with higher dimensions than the rest of minerals. All the minerals have euhedral to subhedral shapes. Cracks are mainly intergranular taking advance of the mica boundaries. Due to the colour of the alkali feldspars, the stone exhibit a brownish colour.
- Golden SKI (GS) (Fig. 1g) is a homogeneous fine grained (≈4 mm) monzogranite. Quartz and feldspars are white and subhedral, muscovite exhibit euhedral shape and bigger size in relation to the rest of minerals and within in the rest of granitoids. Plagioclase is much smaller in this stone. GS exhibits mica orientation (biotite group minerals and muscovite), higher content in quartz than in feldspars and similar proportions of alkali and plagioclase feldspars. GS is characterised by an initial weathering, evidenced by the presence of clays, intragranular cracks mainly in plagioclase and open transgranular cracks. This granitoid shows a yellow colour and the second highest porosity.