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18TH - 23RD OF JUNE 2023

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CLUSTER PORTUGAL MINERAL RESOURCES
UNIVERSIDADE DE ÉVORA

GLOBAL STONE CONGRESS 2023 | BATALHA, JUNE 18 – 23

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NEW CHALLENGES ON DIMENSION STONES, FROM PORTUGAL TO THE WORLD

Responsibility for the information and views set out in this publication lies entirely with the authors

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GLOBAL STONE CONGRESS 2023 | BATALHA, JUNE 18 - 23

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CLOISTERS OF THE MONASTERY OF BATALHA

INVESTIGATION OF THE EFFECTS OF FIRE ON STONE MATERIALS: THE RIO DE JANEIRO CASE

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Summary:

This paper discusses the need for in-depth studies to understand the damage caused by fires on heritage stones in Brazil, particularly on stony monuments. The article highlights two common types of heritage stones found in Rio de Janeiro, Facoidal gneiss and Leptinito gneiss, and describes the changes in some properties when exposed to high temperatures.

For the Facoidal gneiss, the article compares the analysis results of actual samples exposed to fire to those obtained in fresh samples. For the Leptinito gneiss, cubic samples were subjected to high temperatures using oven-based techniques at varying temperatures, residence times, and cooling methods.

The results showed that the fired Facoidal gneiss' colour changed by superficial deposits and oil and a reddening zone close to the surface. Cracks, fractures, and surface hardness reduction were other results of this gneiss. Meanwhile, the Leptinito gneiss exposure to high temperatures in a muffle furnace resulted in various modifications, such as the red staining and whitening of the samples, biotite and garnet colour changes, cracking, and fracturing, with consequent p-waves velocity and surface hardness drop and porosity increase. Non-penetrative fractures at 800ºC became penetrative at higher temperatures, and the minerals underwent transformations leading to a volumetric increase and activation of foliation planes.

Overall, the study highlights the importance of in-depth studies to guide restoration actions after fire damage to heritage stones.

Keywords: *fire damage, heritage stones, high temperature, Facoidal gneiss, Leptinito gneiss.*

1. Introduction

Temperature variation is responsible for rocks' structural changes, the development of pre-existing microcracks and new cracks formation (Freire-Lista et al.,2016). As the density and geometry of cracks and pores are parameters that control the rocks' physicomechanical properties, the damage generated by fire on building stones can affect the construction stability, which is especially relevant for built heritage (Dionísio, 2007; Gomez-Heras et al., 2009; Vázquez et al., 2022). In Rio de Janeiro, events such as the accidental one that heavily damaged the National Museum in 2018 and the vandalic fire that affected the Monument to Pedro Álvares Cabral in 2021 put on evidence the lack of studies regarding the effects of fire on stones in Brazil. To understand the final situation of the stones after the fires and to guide restoration actions, in-depth studies are necessary (Martinho & Dionísio, 2020).

The National Museum, established on June 6, 1818, by D. João VI, is a university museum with an academic and scientific profile that focuses on producing and disseminating knowledge in natural and anthropological sciences. The National Museum headquarters occupies the former residence of Emperor D. Pedro II, the Quinta da Boa Vista Palace, which became a 'hollow' bicentennial building and lost more than 80% of its collections after six hours fire. Beyond the impressiveness of its collections, including the oldest human fossil ever found in Brazil, called "Luzia", among 22 million items, the institution poses a tremendous national significance and responsibility as the oldest museum of the country and witness of its political, historical and scientifical evolution (Duarte, 2022). The most representative building heritage stones of Rio de Janeiro (Castro et al., 2020; 2022), known as Facoidal gneiss and Leptinito gneiss, were used at National Museum. The first serves as basement blocks and dressing of the main façade and columns; both gneisses as doors and window frames (SAMN, 2021).

Figure 1 shows the aspect of some of those elements after the fire.

Figure 1: National Museum rocks after fire.

The Monument to Pedro Álvares Cabral (Figure 2) was fired during a protest against the modification of the indigenous law. Flammable material (rubber tyres and oil) was ignited on the Monument's pedestal base. The Monument was commissioned as part of the fourth centenary of the Discovery of Brazil commemorations, celebrated in 1900. The work by Rodolfo Bernardelli has a 10-meter pedestal in Facoidal gneiss over which bronze sculptures of three important historical figures stand: Pedro Álvares Cabral, commander of the fleet that arrived in Brazil in 1500; Pero Vaz de Caminha, clerk of the fleet; and Friar Henrique de Coimbra, chaplain and celebrant of the first mass (Tabet & Puma, 1983).

Figure 2: a) Monument aspect the day after fire and b) Detail of the damage. Photos by Pedro Dias.

The monument to Pedro Alvares Cabral is located at Largo da Glória, next to the Nossa Senhora da Glória do Outeiro Church, as shown in figure 3.

Figure 3: Location of the monument in Largo da Glória. Source: Google Earth, 2023.

Facoidal Gneiss (here identified as FAC) is a Neoproterozoic orthogneiss, composed of K-feldspar mega crystals usually deformed, looking like eyes (augen), surrounded by biotite lamellae arranged in a quartz-feldspar and biotite-rich matrix. Its colour varies from pink to greyish. It is essentially composed of K-feldspar (+microcline) (35%), quartz (30%), plagioclase (20%), and biotite (10%), and may contain garnet, muscovite, and other accessory minerals. Microcline can constitute more than 50% of samples with larger crystals (Castro et al., 2020).

Leptinito gneiss (LEP) is a Neoproterozoic orthoderivated garnet leucogneiss, of fine-to-medium grain size, with foliation defined by biotite lamellae and elongated quartz/feldspar grains. It is composed of quartz (30–50%), microcline (15–30%), plagioclase (15–20%), biotite (2–5%) and garnet (1–8%). Colour and textural aspects of this gneiss present variations, but the white tone (yellow when oxidised) facies, with uniform and parallel foliation, is the most common (Castro et al., 2022).

There are no studies dealing with the impacts of fire on these stones, which are the most representative on the built heritage of Rio de Janeiro.

2. Objectives

The main objective is to observe how the fire and the temperature decrease after the extinction affect two common heritage stones of Rio de Janeiro, Facoidal gneiss and Leptinito gneiss, describing changes in their physicomechanical properties.

3. Methodology

For built heritage is not possible to use samples of the specific rock, but in the case of the monument to Pedro
For built heritage is not possible to use samples of the

specific rock, but in the case of the monument to Pedro Alvares Cabral, the City Hall of Rio de Janeiro provided two scales of FAC that came loose due to the fire: one external, directly exposed to the fire, and one from the immediate inner layer (Fig. 4).

The external piece was cut in three parts for observation in a stereoscopic magnifying glass (*Carl Zeiss*), and then the soot, fumes and ash were scraped off for future analysis. X-ray diffraction analysis (XRD), scanning electron microscopy (SEM, *Hitachi TM3030Plus*) with an EDS detector (*Bruker Quantax 70*), bulk density, apparent porosity, and water absorption (ABNT NBR 15845-2), Leeb surface hardness - HLD (*Equotip 3, Proceq*), p-waves ultrasonic velocity - UPV (*PUNDIT PL-200, Proceq*), and colour (CieLab, *Spectroguide Sphere Gloss, BYK Gardner*) were determined and compared to those obtained in fresh samples from reference data from CETEM (Fig. 4a).

Twenty-four cubic samples of LEP were extracted from a block donated for academic studies by the São Bento Monastery (Fig. 4b). They were heated in a muffle furnace at three different temperatures (800, 1,000, 1,200°C), four residence times (1, 2, 3, and 4 hours), and two different cooling methods, air and cold water.

Figure 4: Samples in this study. a) Monument pieces of FAC and b) Cubic samples of LEP.

The laboratory analyses of some petrophysical properties of the LEP (stereoscopic magnifying glass, bulk density, porosity, water absorption, hardness, p-waves ultrasonic velocity, and colour) were conducted before and after exposure to high temperatures.

The UPV and HLD results were compared to the reference data from CETEM. Those references were obtained from samples extracted from the cores of demolition blocks, as there are no active quarries to sample fresh rock. Notwithstanding, the reference materials' petrographic analysis and technological properties were compatible with those of similar fresh rocks.

4. Results and Discussion

Cracking and colour changes were found in this study as related by Chakrabarti et al. (1996), Sippel et al. (2007), Vazquez et al. (2016), Nemeth et al. (2021), and other studies summarized in the reviews of Martinho and Dionisio (2020), Leroy et al. (2021), and Sciarretta et al. (2021). The main effects of high temperatures exposition of LEP and FAC are colour changes, cracks and fractures development and mineral loosing.

Stereoscopic magnifying glass

The visible effects of fire in the FAC of the monument are related to cracking (fractures), exfoliation, loss of material (rounding off the edges), black deposits (fumes and ashes), and staining (oil), as seen in Figure 2.

The FAC sample exposed to the fire (Fig. 5) presents colour changes and cracking. Regarding colour, it exhibits the three distinct zones described by Gomez-Heras (2006), from the surface to the inner part: superficial deposits of fumes and ashes (Fig. 5a), a thermal oxidation reddish zone (Fig. 5b) and the standard rock colour. Large feldspar crystals show cracks (Fig. 5c). The inner part shows some staining on the front but no apparent alterations on the back.

Figure 5. Normal cut of the FAC fired surface: fumes and ash external deposits (a); thermal oxidation zone (b); and feldspar cracking (c). B: biotite, Q: quartz and F: feldspar.

In the samples of LEP subjected to 800 °C, the rock dilated, but when it cooled, it was contracted, and it was possible to see some loosing at the contacts. The rocks that stayed for four hours at this temperature show increased cracks development. The colour of the biotite changed to golden. The specimens' colour changed to reddish, by thermal oxidation of iron-bearing minerals and whiter, probably due to the intense quartz fracturing (Sippel et al., 2007; Vazquez et al., 2016; Sciarreta et al., 2021). The water-cooled samples developed more cracks than the air-cooled ones, and all the specimens' surfaces were rough.

The crack density and extension increased largely in the LEP samples subjected to 1,000 °C. Biotite expanded and changed colour to golden, and garnets became grey-coloured. The red colour is more intense in the air-cooled specimens than in those cooled with water; the latter are whither. Cracks are visible on the specimen's surface, mainly following the foliation planes, but some fractures prone to material detachment also appeared, with more intensity on the air-cooled samples. Surface roughening and small crystals loosening could be observed.

After cooling the samples heated at 1,200 °C, the damage is evident in all the specimens, although the apparent state of the air-cooled ones is the worse. Fractures are enlarged following the foliation planes or not, with areas showing material losses. Several fractures and minerals loosing were also observed in the water-cooled samples, but their fractures were not as wide as those developed in the air-cooled specimens showing a higher apparent stiffness reduction. Air-cooled samples were also redder than the water-cooled ones that looked white.

Figure 6 illustrates the transformation of the LEP specimen heated to 1,200 °C for two hours and cooled with water, and Figure 7 illustrates the LEP sample after three hours of heating and air cooling.

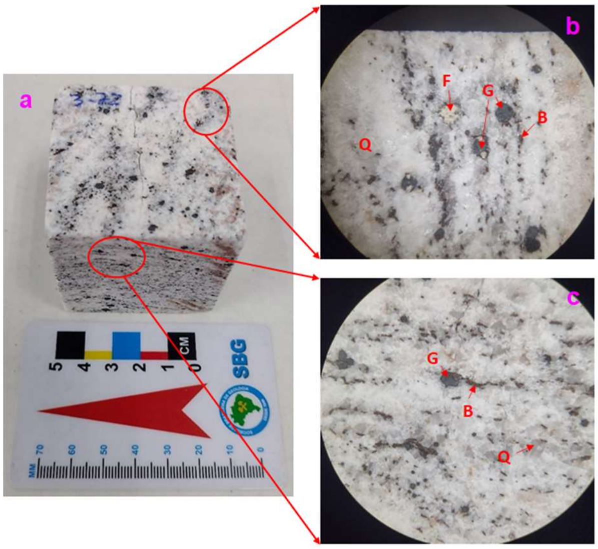

Figure 6: Change in mineral aspects in LEP subjected to 1200°C and cooled with water: a) Cracking, b) and c) garnets became grey and biotite golden. G: garnet, B: biotite, Q: quartz and F: feldspar.

The cracking process seemed to slow down in the samples cooled with water, and the fractures were less wide than in the air-cooled samples. Although quenching should be more damaging due to thermal shock (Chakrabarti et al., 1996; Pires et al., 2014; Shao et al., 2014), this was not observed in this experiment. In this specific case, the reason could be mineralogical variations within the samples of the same block, as some destructive fractures developed in the more reddish specimens, which is also an indication of the suffered damage (Vazquez et al., 2016; Nemeth et al., 2021). Further investigations on the mineral structure and texture of the samples will be needed. The water-cooled specimens' colour was almost white, which following Sippel et al. (2007), could be due to the intense fracturing of quartz.

Figure 7 illustrates the damages observed on the LEP specimen cooled naturally after three hours in the muffle furnace at 1,200 ^{o}C: destructive fractures developed, material losses, individual mineral expansion and intense reddish colour.

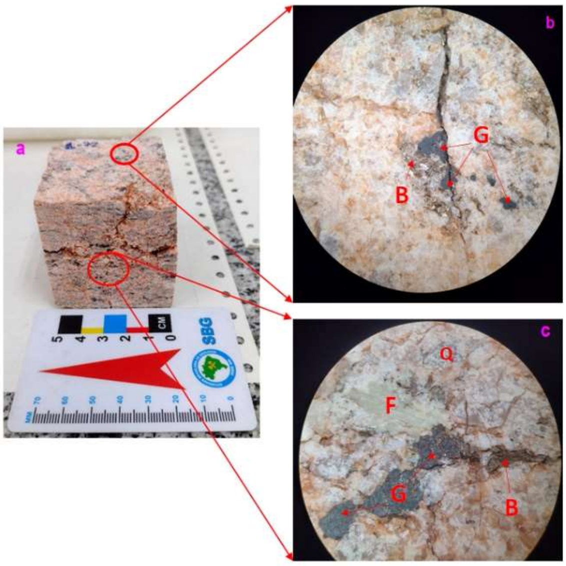

Figure 7: Changes in LEP aspects subjected to 1200 °C and cooled naturally: a) Rupture of the sample, b) and c) garnets became gray and biotite golden and enhance the cracking.

Complementing the petrography observations of FAC, the results of the X-ray diffraction mineralogical composition analyses identified quartz, k-feldspar (sodic albite), microcline, k-feldspar (calcic albite), sanidine, sodium aluminium silicate hydrate, anorthite and biotite. The presence of two kinds of albite, sanidine and anorthite, indicates a very high temperature in the fire (Haldar, 2020). Sanidine is typical of igneous rocks and forms between 650-800 °C.

Figure 8 shows micro-fissures propagation in FAC: intragranular fissures (quartz-quartz and feldspar-feldspar grain) and intergranular cracks (quartz-feldspar contacts).

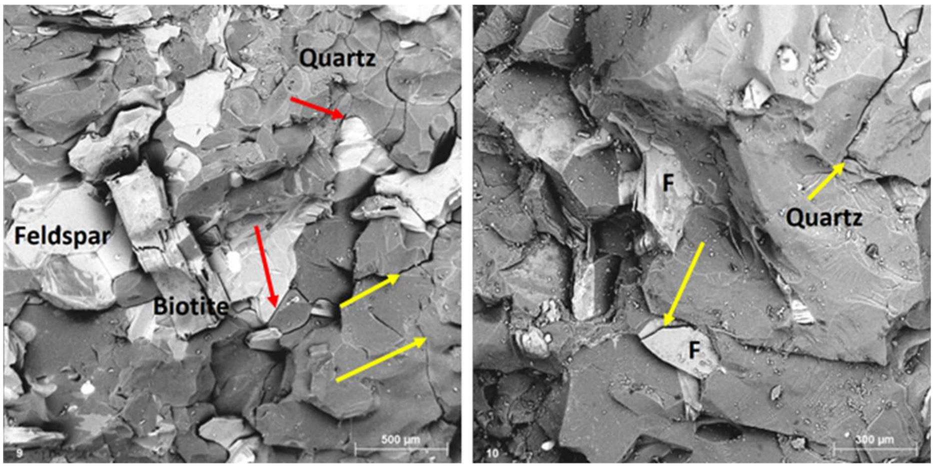

Figure 8: SEM images of intragranular (yellow arrows) and intergranular (red arrows) microfractures of FAC. F: feldspar.

Colour

Figure 9 shows the differences in colourimetric data of LEP samples in the CIEL*a*b* system. All the samples, after heating, had a positive shift of a* (red to green), and most of them also of b* (blue to yellow). This displacement of the a* and b* parameters is visible by the reddish staining of the samples by the thermal oxidation of iron-bearing minerals (Dionisio et al., 2021, Németh et al., 2021). The higher shift for the 1,000 and 1,200 ºC groups after air cooling, as explained before, is clearly illustrated in Figure 9, as it is the observed whitening of the samples. The figure also shows no apparent differences in colour variation between the cooling methods at 800 °C. There was a high increase of the L* parameter (black to white) from 1,000 ºC and above, which, together with the a* and b* variations, lead to total colour variations (ΔE*) of 4,9 to 11,1, clearly perceivable by the human eye.

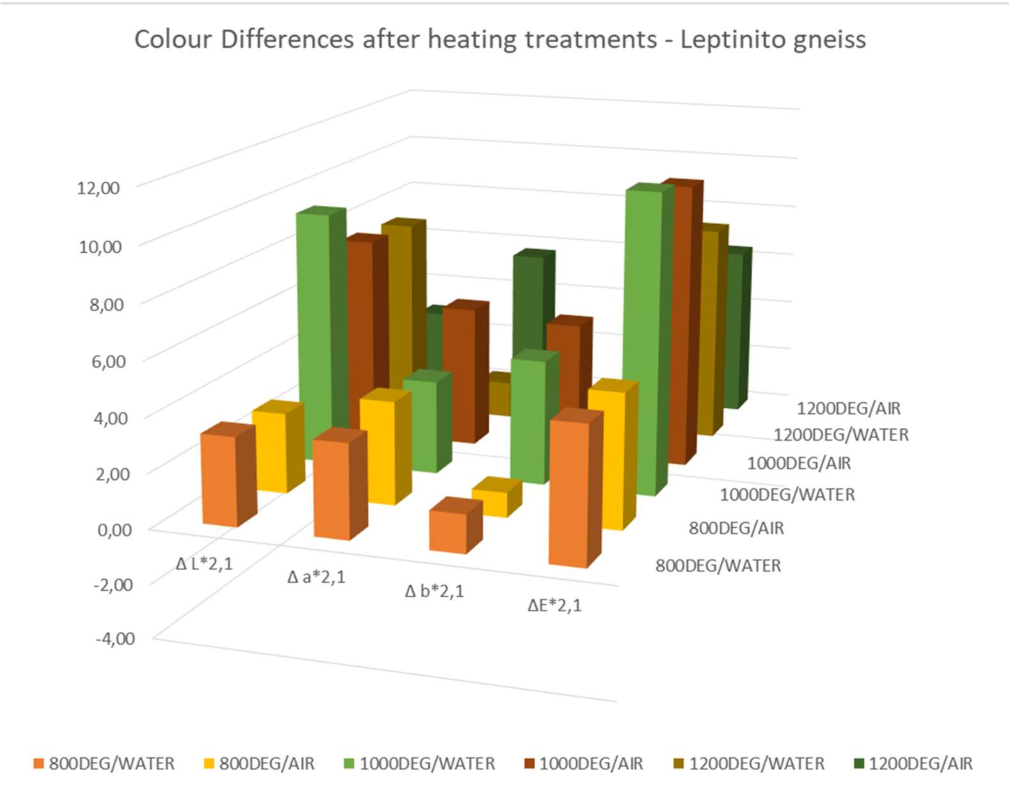

*Figure 9: Averaged colourimetric variations of LEP samples after heating, CIE L*a*b* system.*

Bulk density, apparent porosity and water absorption

As expected, the apparent porosity of all the samples increased (Leroy et al., 2021). This increase was higher for the LEP than for the FAC, which could be due to the unlike heating conditions (real fire and oven heating) and mineralogical composition and structure.

LEP regular and parallel foliation offers weakness planes where some fractures developed with temperature. It has higher quartz content than FAC. Quartz content is a critical parameter for the fire decay on silicate stones, having volumetric expansion much higher than other rock-forming minerals and anisotropic behaviour regarding linear thermal expansion (Winkler, 1997). Quartz thermal expansion grows with increasing temperatures and is twice as high perpendicular to the c axis, producing ever-growing thermal strains inside the stone, up to 573 °C, when thermal strains stabilize (Sciarretta et al., 2021). At temperatures beyond 573 °C, such as the ones in this experiment, the transition from low-to-high quartz changing the crystalline arrangement entails a huge expansion producing eye-naked visible cracks, as observed by Nemeth et al. (2021). Silicate rocks' residual strains (after cooling) are high if heated at more than 700 °C when the stones expand through the crack's growth and intergranular cracks formation (Sippel et al., 2007). LEP mineral composition and arrangement may have affected the observed thermal decay. It has high quartz content and, probably, higher thermal expansion perpendicular to foliation than parallel, as the mylonitic orthogneiss studied by Sippel et al. (2007). Cracks were observed in grain boundaries, as shown in Figure 10.

The FAC samples did not show as much cracking as the laboratory-heated LEP samples. More study is needed to assess the damage on those samples properly, but its distinct structure and texture may help it to be more resistant to thermal damage than the Leptinito, as it is to natural weathering. With lower quartz content than the LEP, quartz crystals in FAC are distributed in the biotite-rich matrix that may accommodate the thermal expansion of those crystals (Vazquez et al., 2022). Also, Lima et al. (2002) measured the thermal linear expansion coefficient of 61 Brazilian granitic building stones and found that the thermal linear expansion coefficient of rocks with 5% more quartz content was 16% higher, and that coefficient was also 2% to 6% higher for medium grain size than for coarse grain size rocks.

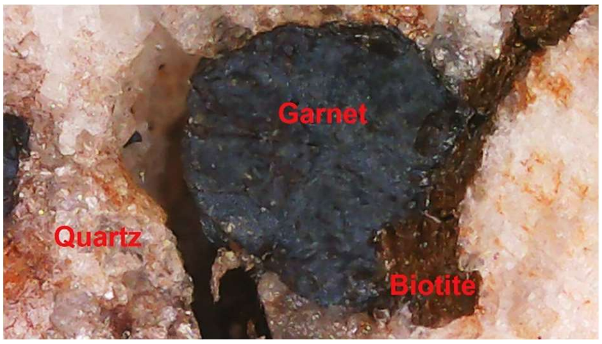

Figure 10: Detail of garnet-quartz contact with a fracture and garnet-biotite-quartz without fracture. Sample of LEP subjected to 1200°C for two hours and cooled naturally.

The time of permanence in the oven is another factor of cracking increase. The longer in the oven, the more penetrative the fractures.

Bulk density determination of the FAC scales resulted in 2,632 kg/m^3 (external) and 2,620 kg/m^3 (internal), slightly lower than the reference value (2,692 kg/m^3), which cannot be attributed just to the fire as the stone's condition before the fire is unknown.

For the LEP, the bulk density decreased with increasing temperature: from initial 2,623 ± 9 kg/m^3 to 2,529 ± 18 kg/m^3 (800 °C), 2,492 ± 13 kg/m^3 (1,000 °C) and 2,385 ± 23 kg/m^3 (1,000 °C). These results reflect the specimens' volume increase due to the cracks and fractures' development (Nemeth et al., 2021). In this work, it was not possible to analyse differences in bulk density decrease regarding the oven residence time of the specimens or the cooling method, as only one specimen was used for each testing condition (Figure 11).

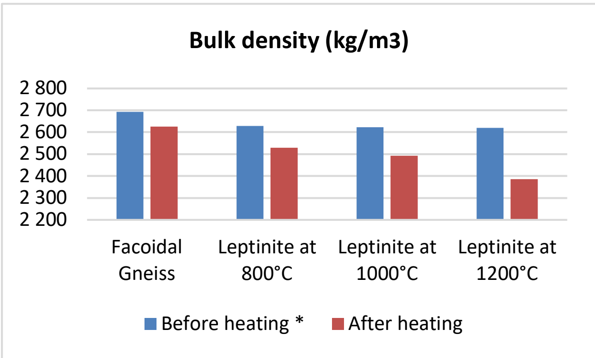

*Figure 11: Bulk density of the samples before and after heating. *FAC reference value from Castro et al., 2020.*

LEP apparent porosity after high temperatures exposure increased by around 500% (800 °C), 700% (1,000 °C) and 900% (1,200 °C). Water absorption at atmospheric pressure, directly related to porosity, followed the same trend (Figure 12). The higher the temperature, the more the microcracking, cracking and fracturing, as indicated in the stereoscopic magnifying glass description. The FAC specimens detached from the monument had higher porosity than the reference value for this lithotype, although, as explained before, the alteration degree of this stone before the fire is not known.

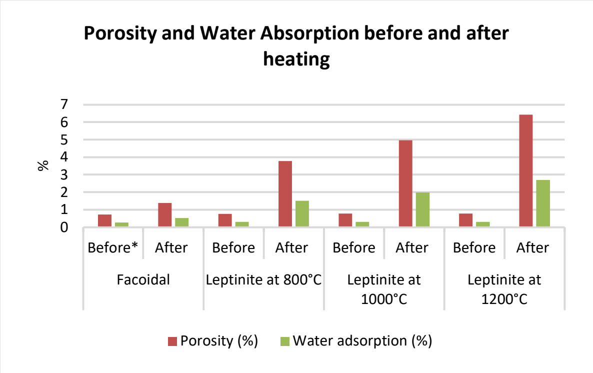

*Figure 12: Porosity and water adsorption of the samples before and after heating. *FAC reference values from Castro et al. (2020).*

Surface hardness

The Leeb surface hardness reference value for FAC is 690 ± 110 HLD. The fired monument sample measurements (20) resulted in 331 ± 153 (exterior scale) and 428 ± 90 (inner scale). For the LEP samples, the surface hardness value dropped from 802 ± 22 to 331 ± 76 HLD after heating, a more than 50% reduction of this parameter. Németh et al. (2021) also verified a 60% reduction in rebound hardness value after a 750 ºC heating treatment. The obtained individual hardness values did not allow the identification of any significant difference between groups by residence time or cooling method.

Ultrasonic wave velocity

The ultrasonic wave velocity (UPV, p-wave) for reference FAC and LEP is around 4,000 m/s. The effect of cracking for both the fired FAC and the heated LEP specimens was depicted by the low UPVs measured: 500 m/s parallel to the foliation and 600 m/s orthogonal to the foliation for FAC, and 800 m/s parallel to the foliation and 600 m/s orthogonal to the foliation for LEP.

5. Conclusions

The fire exposure of FAC in the monument to Pedro Alvares Cabral at Largo da Glória generated some damages. Macroscopically, they are reflected in visible cracking (fractures), exfoliation, loss of material (rounding off the edges), deposits (fumes and ashes), and staining (oil). In the samples studied in the laboratory, it was possible to identify intragranular and intergranular cracks in SEM-EDS images. The bulk density was slightly lower than that of sound rock, and porosity and water adsorption were a little higher but with similar values for the external and internal pieces. The XRD identification of sanidine points to a high temperature of the fire. This stone's condition before the fire is unknown, so that further investigations will be necessary.

The LEP exposure to high temperatures in the muffle furnace resulted in intense colour changes for all the specimens. All samples were red-stained by iron oxidation, at least to some extent, and whitened. Those colour changes were more pronounced as temperature increased. It was also observed that air-cooled samples were redder than water-cooled samples, which became whiter.

Cracks' density and intensity grew as the temperature rose: intragranular and intergranular cracks were observed in all the samples, and visible fractures developed at 1000 ºC and 1,200 ºC.

Biotite had a stewed appearance, and its colour turned golden. The garnets' colour became dark grey, and some crystals broke up at the maximum temperature. Quartz was intensely fractured and even appeared vitrified.

The mineral transformations and differences in thermal expansion caused a volumetric increase and initiated cracks in the stone, activating cleavage and foliation planes. The bulk density decreased by up to 10%, and porosity and water absorption increased by 900%. Surface hardness was reduced to less than half and the p-wave velocity to one-fifth, meaning that after a fire, those stones may endanger the stability of heritage buildings if they have a structural function.

Other analyses will be carried on so this work can serve as a basis to prepare better a future study of the National Museum gneisses decay state.

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