

## Hot restoration technologies for external coating tiles: Nossa Senhora da Soledade Cemetery (Belém, Brazil)

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## 1. Introduction

Tiles are glazed ceramic pieces, with cultural value, frequently used in architecture. Depending on where they are fixed, tiles may need specific interventions of restoration, which could be performed through either hot or cold restoration. Each one presents positive and negative consequences, depending on the quality of the manufacturing process and environmental conditions to which the restored pieces will be exposed.

A conservation or restoration project must be based on the documentation (register and diagnose of conservation status) and scientific analysis of the material and its al-

## Abstract

In Belém, Brazil, during the 19th century, Portuguese tiles were used for the external facing of sepultures at the Soledade Cemetery. These pieces are fragile because of exposure to the tropical climate and of their location close to the ground. This research aims to obtain subsidies for the restoration of the glazed layer, through hot restoration, to serve as theoretical and technological references for future restoration projects. The samples consisted of fragments of Portuguese historical tiles from the Soledade Cemetery. Restoration was performed by cleaning (both mechanical and chemical ways) and re-firing (1000°C). Before and after the re-firing, samples were submitted to the analytical technique of X-ray diffraction. The results indicate that the re-firing eliminated defects and pathologies existent in the glaze and caused changes in the mineralogical composition of the ceramics. Used carefully, the hot technique is one of the alternatives to improve durability and protect the Soledade historical tiles.

**Keywords:** glazed ceramic tiles; restoration; firing.

terations (characterization). It should aim to reduce environmental effects, in order to: 1) rescue the aesthetic characteristics and functionality of the object (Fabbri, 2003); and 2) promote durability, because the repetition of interventions with short longevity may result in the loss of its original configuration or its essence (Oliveira, 2001).

In restorative interventions, materials to be used should have characteristics that are similar to the layers they will replace, so, in areas with glaze loss, the material used must be resistant to climatic actions, be impermeable, and adhere well to the ce-

ramic layer (Sanjad, 2009). Hot restoration was studied in various researches (Oliveira, 2001; Sanjad, 2002; Burgos, 2003; Schwarz *et al.*, 2003; Leite, 2011): 1) to remove dark stains (microorganisms) existent between the glaze and the ceramic; 2) to perform chromatic reintegration in the glazed layer, using glazes similar to the originals. In this kind of procedure, the tiles need to go through a new firing process with appropriate temperatures, respecting the instability gap of the minerals, in order to prevent any harmful alteration. Therefore, due to the recovery of the glazed layer, which provides

impermeability to tiles, the process ensures greater durability of the interventions carried out on outdoor pieces (exposed to actions of weathering) (Oliveira, 2001). It also guarantees the aesthetic compatibility of the intervention and the ancient material (Sanjad, 2009).

In Belém, tiles were frequently used to coat the façades of historic buildings, especially during the 19th century. In this period, the use of tiles in the mortuary architecture of Nossa Senhora da Soledade Cemetery stands out as the external coating of sepulchres. Despite the city's vast heritage of tiles, only a few tiled façades have been restored. Moreover, inappropriate procedures for tiles

used for external facing were applied in the interventions, due to low durability when exposed to weathering (Sanjad, 2007).

At this monumental romantic cemetery, built in 1850, it is possible to find Portuguese tiles decorated by the stamp technique. They are important components in the heritage of Brazilian tiles, due to their decorative patterns (artistic value) (Riegl, 2006) and they are inserted into a context in which the laying of tiles on cemetery structures was rare, even in Europe (Queiroz, 2012) (historical value) (Riegl, 2006). The tiles from the Soledade are fragile because of exposure to the tropical climate, human actions and their location close to ground.

They are favorable to the development of pathologies, such as glaze loss and proliferation of microorganisms between the glazed and ceramic layers, which accelerates deterioration.

Due to the historical and artistic importance of this material, a restorative intervention is required. The restoration must aim to extend the lifetime of the original material and to prevent them from being replaced by replicas.

The aim of this research is to obtain subsidies for the restoration of glazed layers, through hot restoration, to serve as a theoretical and technological reference for future restoration projects.

## 2. Materials and methods

### 2.1 Sampling

The samples consisted in fragments of Portuguese historical tiles, decorated by the stamp technique, collected from eight different graves of Soledade Cemetery (Figure 1). The collection was

authorized by the National Historic and Artistic Heritage Institute - IPHAN (*Instituto de Patrimônio Histórico e Artístico Nacional*). The samples were taken from areas where extraction was

easy, in which the pieces were already fractured and had weak adhesion to the support, in order not to compromise the artistic unity of the graves and avoid promoting new damages.

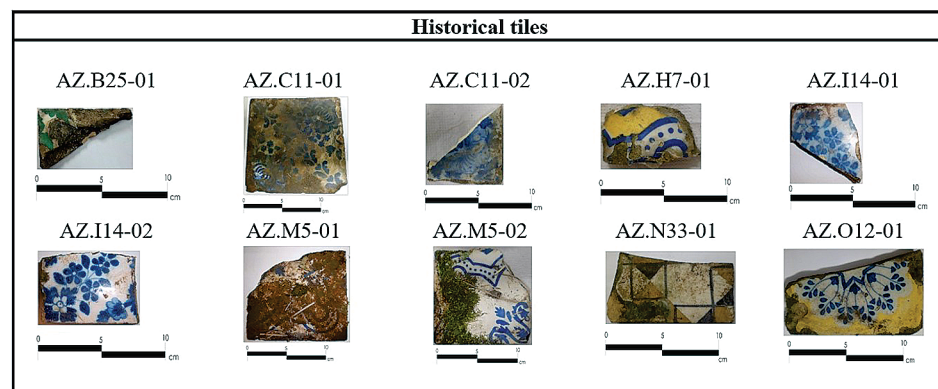


Figure 1  
Samples of deteriorated tiles fragments.

### 2.2 Restoratives procedures

The tiles were cleaned mechanically, using a surgical scalpel, and chemically, with a wash of distilled water and acetone application on decorative layers. The fragments were dried for 24 hours at 100°C in a Quimis, model Q317M-32 kiln, to remove moisture.

The re-firing was performed at 1000°C, during thirty minutes, in a Quimis, model G318M24 Quimis muffle oven. The use of elevated temperature, near the temperature of tile manufacturing, was chosen in order to: remove dark stains (bio-colonization)

that existed between the glaze and ceramic layers; restore adhesion of the glaze to the ceramic; and recast the vitrified layer in order to correct defects like vacuoles, microcracks and fissures. In samples with problems originating from this re-firing, a second re-firing was performed under the same conditions as the first.

Before and after the hot restoration, the mineralogical composition of the tiles was determined by Powder X-ray Diffraction Analysis, using a PANalytical X'Pert PRO MPD

(PW3040/60,  $\theta/\theta$ ) diffractometer with a ceramic X-ray tube (Cu anode,  $K\alpha_1 = 1.540598 \text{ \AA}$ ), Kb Ni filter, and an X'celerator PSD (Position-Sensitive Detector), belonging to the Laboratory of Mineral Characterization (LCM), at the Institute of Geosciences, UFPA. Instrumental conditions used are as follows: scan range from 5 to 100°,  $\gamma$ , tube power of 40 kV and 30 mA, step size of 0.02°, time/step of 60 s, divergence slit of 1/8°, anti-scattering slit of 1/4°, mask of 10 mm, and sample spinning with a rotation time of 1.0 s.

## 3. Results

### 3.1 Hot restoration of tiles

The cleaning of the tile samples removed the dirt deposits and superficial bio-colonization. The first re-

firing eliminated or softened the defects and pathologies that exist in the glaze (Figure 2). The pieces presented brighter

and more highlighted colors. The adherence of the glaze to the ceramic, previously weakened mainly due to dark stains

(microorganisms) in the interface zone between the layers (Oliveira, 2001), was

restored and this bio-colonization was also removed during the heat treatment.

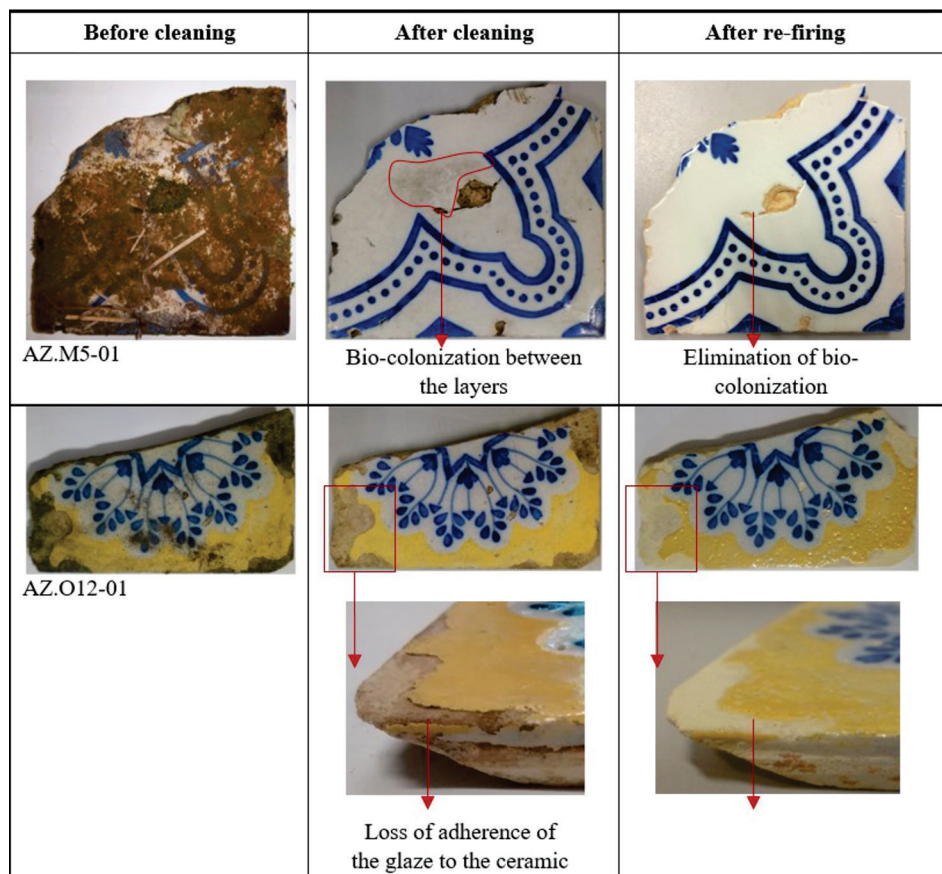


Figure 2  
Samples before and after the cleaning and firing treatment.

It was observed that some fragments developed new problems. In the vitrified layer, the formation of air bubbles was verified in the AZ.H7-01, AZ.I14-02 and

AZ.O12-01 samples. Regarding the ceramics, there was the appearance of deep fissures and a change in color and texture in the AZ.N33-01 fragment. In contrast, in

the second re-firing, performed in samples with defects in the vitrified layer, the glazes were made uniform with softening and/or disappearance of bubbles (Figure 3).

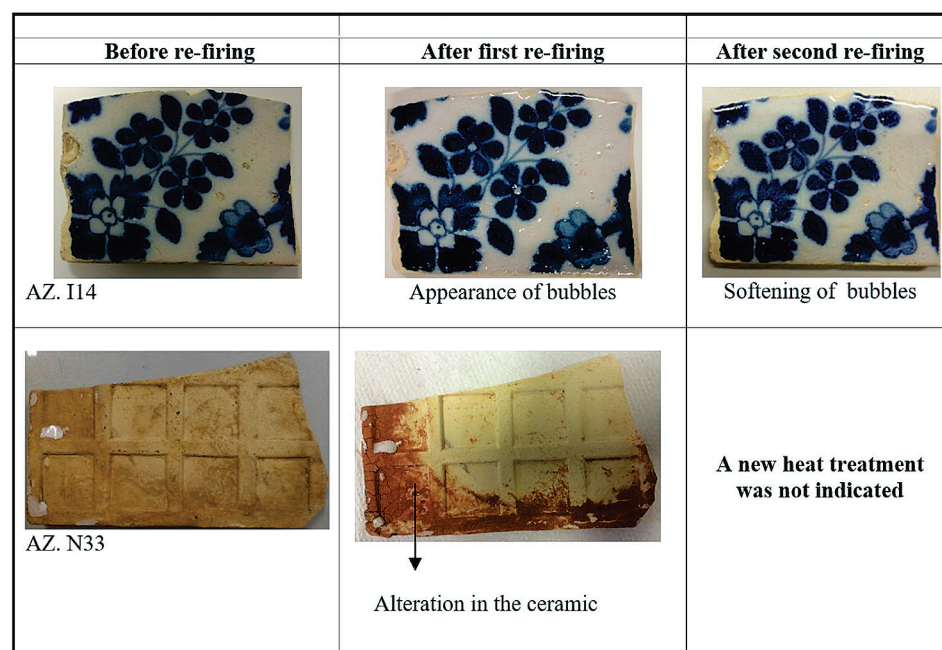


Figure 3  
Samples AZ.I14 and AZ.N33 before and after the heat treatments.

The X-ray diffraction analyses show that before the firing, the samples basically contained: quartz, gehlenite, calcite, and Ca,Mg-minerals as diopside (AZ.I14, AZ.M18, AZ.P3). After the firing, the

(AZ.C11, AZ.H7, AZ.M5, and AZ.N33 AZ.O12) and anorthite (AZ.I14) and Ca,Mg-minerals as diopside (AZ.I14, AZ.M18, AZ.P3). After the firing, the

calcite, originally present in most of the samples, disappeared. There was formation of diopside in AZ.B25, AZ.C11 and AZ.M5; and anorthite in AZ.C11 (Figure 4).

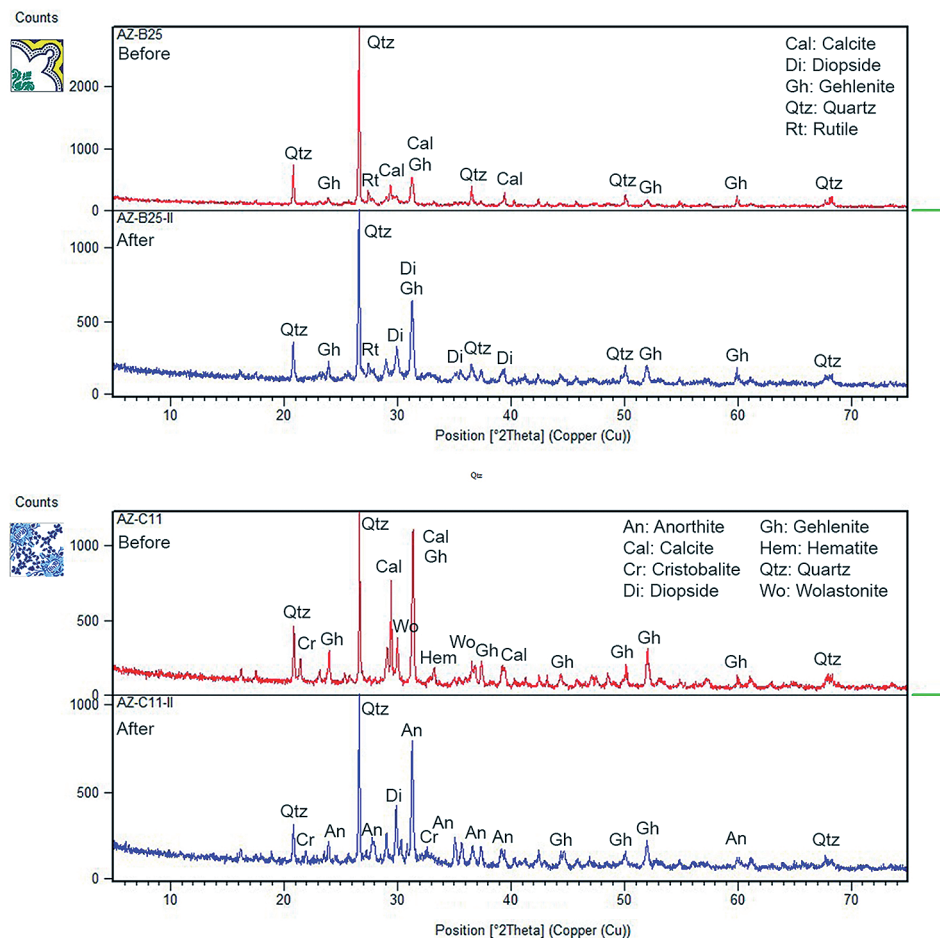


Figure 4  
XRD patterns of the samples before and after the re-firing.

#### 4. Discussions

Based on the results, it was possible to improve the visual aspects and fix manufacturing defects and alterations due to re-vitrification of the decorative layer, since the temperature reached was sufficient for a new fusion of the materials. The dark stains of bio-colonization in the interface area between the layers (Oliveira *et al.*, 2001) were also removed during heat treatment due to the carbonization of the organic material (Sanjad, 2002).

The air bubbles in the glaze of some samples were possibly caused by the release of carbon dioxide after the breaking down of the calcium carbonate structure and/or of organic matter in the ceramic (Costa *et al.*, 2013). In the second new firing, these defects in the vitrified layers were minimized, probably due to: 1) re-accommodation of the glaze, which with the high temperature passed to liquid state, causing a re-vitrification; 2) the low release of CO<sub>2</sub>, since most of the organic particles were eliminated from the ceramic during the first new firing and due to inexistence (or low quantity) of carbon dioxide.

As for the change in the ceramic of the AZ.33-01 sample, this result shows

certain fragility due to the alterations suffered by the tile, which are probably related to the area of the grave in which the tile was inserted. Since this tile was part of a panel fixed on the lateral of the N33 grave, in direct contact with the ground, the piece may have absorbed too much humidity, as well as organic and inorganic components.

Despite giving durability to the elements exposed to weathering and the use of materials and techniques similar to the original, it is observed that the firing treatments can also present risks to the material according to its chemical and mineralogical composition or level of alteration. Tests should be conducted on small fragments prior to a mass intervention.

From the point of view of the material safeguarding, the hot restoration results are contributive. However, this procedure has some conceptual limitations, based on classic restoration theories. Brandi (2008), defines that “restoration must aim to reestablish the potential unity of a work of art, as long as is possible without producing an artistic or historical forgery, and without erasing every trace of the passage of time on the artwork”, and “restoration

must be reversible”. Hot restoration goes against some of these principles because it is irreversible; can compromise the marks of time in the original piece, due to the new fusion of the glaze; and may change the composition of the original material. Despite this, contemporaneous restoration theories are more flexible about these concepts, and seen as subjective, since they depend on a point of view.

According to Viñas (2004), restoration is not a neutral activity, since it involves a series of technical and ideological elections. In some cases, the conditions of the artifact may require the sacrifice of part of the material’s consistency in order to safeguard it. Due to the fact that the work of art, in tiles, is present in the painting, the preservation of the glazed surface is a priority (Oliveira, 2001), which was secured through the hot restoration.

Changes in the mineralogical composition of the ceramics occurred due to the mineral transformations that may happen because of re-firing temperature, re-firing time, and rate of temperature increase (Riccardi *et al.*, 1999; Cultrone *et al.*, 2004; Vaz *et al.*, 2008; Cultrone and Madkur, 2013). It is very likely that the

calcite, a mineral with fusion temperature of about 900 °C, present in most samples before re-firing, was derived from the mortar (Sanjad *et al.*, 2004; Costa *et al.*, 2013), since the tiles had minerals in their composition such as gehlenite, formed

at about 1000°C (Sanjad *et al.*, 2004). Thus, the calcite disappeared after the re-firing at 1000°C. According to the re-firing temperatures, it is possible that silica reacts with: 1) calcium compounds that originate wollastonite; 2) calcium and

magnesium compounds which originate diopside; and 3) gehlenite and aluminum phases, resulting in anorthite (Riccardi *et al.*, 1999; López-Acre *et al.*, 2003; Tratoré and Kabré, 2003; Sanjad *et al.*, 2004; Koch, 2008).

## 5. Conclusions

Due to differences between the various types of tiles, such as their country of origin, fabrication techniques, where they were placed, and deterioration level, it is important that the tiles be scientifically analyzed before performing hot restorative intervention.

Otherwise, the restoration may compromise the integrity of the piece, causing irreversible damages.

Used carefully, the hot technique is one of the alternatives to improve durability and protect the heritage of the Soledade tiles and other external coating

tiles in the same deterioration situation, mainly concerning the re-vitrification of the decorative layer. However, it is necessary to evaluate the kind of alteration before submitting the historic tiles to this restorative intervention, because of its risks.

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