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Technological Evaluation of Roof Tiles from the Monastery of São Bento, Rio de Janeiro

Caracterização Tecnológica das Telhas do Mosteiro de São Bento, Rio de Janeiro

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Abstract

The present work carried out the evaluation of the roof tiles belonging to the Monastery of São Bento, in the state of Rio de Janeiro, through the common analyzes of the technological characterization. The evaluation was performed by petrography, X-ray fluorescence (XRF), X-ray diffraction (XRD), physical and colorimetric properties and accelerated weathering tests using salt spray, súlfur dioxide, and ultraviolet rays. The results obtained through petrography, XRD, XRF, SEM, FTIR showed a mineralogical composition characterized in the internal part by hematite and in the external part presenting mullite, potassium feldspar, quartz and gypsum being the last one formed from physical-chemical changes of calcium. The analysis of the data obtained after the colorimetric analyzes and accelerated weathering (salt spray, SiO_2, UV), identified little variation in mass loss, however the colorimetric analyzes showed the darkening of the colors after the actions of SO_2 and their fading after the actions from salt spray and UV rays. The results obtained allowed a compositional characterization and the state of conservation of the roof tiles, allowing the evaluation of future interventions for the preservation of the se and/or possible substitute materials in accordance with the ceramic materials currently available.

Keywords: Historical building; Conservation; Ceramic materials

Resumo

O presente trabalho realizou a avaliação das telhas pertencentes ao Mosteiro de São Bento, no estado do Rio de Janeiro, através das análises comuns da caracterização tecnológica. A avaliação foi realizada por petrografia, fluorescência de raios X (FRX), difração de raios X (DRX), propriedades físicas e colorimétricas e testes de intemperismo acelerado usando névoa salina, dióxido de enxofre e raios ultravioleta. Os resultados obtidos por petrografia, DRX, FRX, MEV, FTIR mostraram uma composição mineralógica caracterizada na parte interna por hematita e na parte externa por mulita, feldspato potássico, quartzo e gipsita, sendo este último formado a partir de alterações físico-químicas do cálcio. A análise dos dados obtidos após as análises colorimétricas e intemperismo acelerado (névoa salina, SiO_2, UV), identificou pouca variação na perda de massa, porém as análises colorimétricas mostraram o escurecimento das cores após as ações do SO_2 e seu desbotamento após as ações da névoa salina e dos raios UV. Os resultados obtidos permitiram uma caracterização composicional e do estado de conservação das telhas, permitindo avaliar futuras intervenções para a preservação destas e/ou possíveis materiais substitutos de acordo com os materiais cerâmicos atualmente disponíveis.

Palavras-chave: Edificação Histórica; Conservação; Materiais cerâmicos

1 Introduction

The Monastery of São Bento do Rio de Janeiro, Figure 1, was founded in 1590, twenty-four years after the founding of the city, by two Portuguese monks. It was the second religious order established in Rio de Janeiro (the Benedictines were preceded only by the Jesuits), operating in a temporary building until the construction of the definitive building, designed by the military engineer Francisco Frias de Mesquita (Oliveira & Justiniano 2008; Mosteiro de São Bento do Rio de Janeiro 2024).

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The first phase of work on the São Bento Monastery and its church dedicated to Nossa Senhora de Montserrat began around 1620. In 1641, the chance land a *façade* with two bell towers and a triangular pediment were completed. By that time, two artist monks wanted to give the church its definitive form: the architect Frei Bernardo de São Bento and the sculptor Frei Domingos da Conceição. The first carried out a series of extensions to the original project, including the sacristy and side aisles, and these condidealized and partially executed the splendid internal ornamentation in the Portuguese national style that identifies the first phase of Luso-Brazilian Baroque. Mannerism, Baroque and Rococo are integrated in perfect symbiosis in the decoration of the Monastery of São Bento, a true example of the various phases of Luso-Brazilian carving (Oliveira & Justiniano 2008).

The Monastery can be observed in the artistic productions of the famous French painter Jean-Baptiste Debret, as shown in Figure 2 (Baptista 2015). In this work, Debret portrayed the solemn landing of His Highness Leopoldina Carolina Josefa in Brazil at the beginning of the 19th century, with the Monastery of São Bento in the background.

Figure 1 São Bento Monastery of Rio de Janeiro.

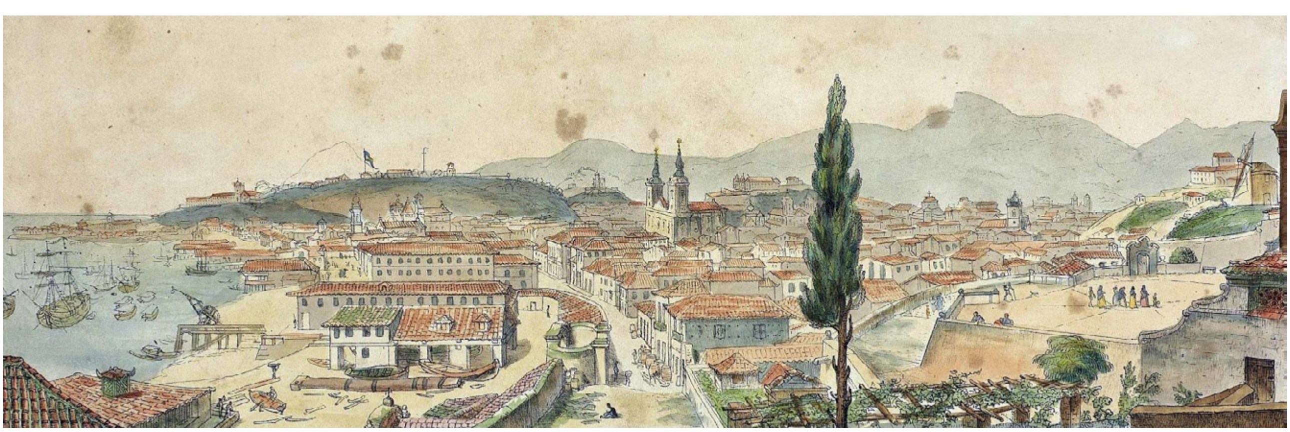

Figure 2 Painting by painter Jean-Baptiste Debret.

Colonial Brazil is marked by the construction of several religious buildings, related to the constructive and architectural pattern of Portugal, predominantly using the labor of enslaved Africans. The “French Artistic Mission” among them, Jean-Baptiste Debret, Figure 3, hired as a “history painter” whose various works portray the daily life of colonial Brazil, the enslavement of Africans, the fauna and flora in this period (Freitas 2009).

The São Bento Monastery in Rio de Janeiro is part of this context of colonial Brazil, being one of the most beautiful architectural complexes entirely preserved in Brazil. Built slowly, over a period of a century, which makes it difficult to accurately state the origin of the materials that make up the roof tiles, it is considered the best architectural project carried out in the city of Rio de Janeiro in the 17th century. The structure of the monastery features the Abbey of Monserrate, the traditional São Bento School and the São Bento University (Arruda 2007).

The Monastery of São Bento has great cultural and historical importance for Brazil, so it is essential to preserve this heritage.

Buildings located in open areas are vulnerable to physical, chemical, mechanical and biological weathering that contribute to the alteration of their structural properties. Thus, the stones, roof tiles and other materials that make up the Monastery building show some deterioration (Öztürk 1992).

The objective of the research focuses on the characterization of the roof tiles in order to understand their composition, as well as the possible causes of the changes observed, providing technological support for conservation and restoration actions.

Figure 3 Painting by painter Jean-Baptiste Debret.

2 Methodology and Data

The technological evaluation was performed on samples collected from an original roof tile, provided by IPHAN, with petrography, X-ray fluorescence (XRF), X-ray diffraction (XRD), determination of physical properties (porosity and absorption of water), colorimetry and accelerated weathering tests (salt spray, sulfur dioxid e and ultraviolet rays).

2.1 Petrography

The petrographic analysis was performed with the aid of a Carl Zeiss polarizing microscope with 2.5 to 60 objectives and X-ray diffraction (XRD).

The mineralogical composition of the roof tile was evaluated by XRD analysis. It was performed in a Bruker-AXS D4 Endeavor diffractometer, with Co kα radiation (40 kV, 40 mA). Diffraction patterns were acquired from 4 to 80 (2θ) in 0.02 steps. Identification of all minerals was done with the Bruker-AXS DIFFRAC.EVA suite. X-ray fluorescence (XRF).

Chemical analysis by XRF was carried out on the roof tile sample using a Panalytical benchtop equipment, model Axiosm AX 4.0 kW.

2.2 Scanning Electron Microscopy-Energy Dispersive X-Rays (SEM-EDX)

The inner layer of the roof tile was evaluated using SEM-EDX analyses. For this, a Hitachi TM 3030 Plus scanning electron microscope was used. The instrument was equipped with a Bruker X-Flash energy dispersive X-ray spectrometer with MIN SVE detector and connected sweep generator.

2.3 Fourier Transform Infrared Spectroscopy (FTIR)

The outer and innerlayers of the roof tile sample were analyzed using a Perkin Elmer Spectrum 400 spectroscope, with a wave band of 4000-400 cm^{-1}. For this, the samples were prepared using a Fluxana VANEOX 40t automatic press. Background was measured with a sample of Potassium Bromide (KBr).

2.4 Water Absorption

The tests were carried out based on ABNT NBR 15310 (2005), to evaluate the water absorption of the roof tile material. Measurements were made at atmospheric pressure using a Marte AD2000 scale.

2.5 Colorimetry

The BYK (Spectro-Guide Sphere Gloss) portable spectro photometer was used to evaluate the color and gloss of the original roof tile sample. Measurements were performed on the outer na dinner layers of the roof tile sample.

The results were expressed in the three-dimensional reference of the CIELAB color space (Figure 4), where the L* axis represents the lightness of a color, from the darkest black (L* = 0) to the lightest white (L* = 100).; the a* axis denotes green/red of a color, with a* < 0 representing green, while a* > 0 denotes red; and the b* axis expresses the opposing colors blue/yellow, with negative b* values for blue and positive b* values for yellow. Consequently, when the values of a* and b* are equal to zero, true neutral Gray is expressed (NazdarInk Technologies 2016).

2.6 Hardness Determination

The non-destructive test was carried out in situ with the aid of a portable electronic hardness tester, model Equotip 3 from Proceq, with a type C probe. The hardness was evaluated in the Black part and in the red part of the roof tile sample.

2.7 Accelerate Weathering Tests

2.7.1. Salt Spray

Three roof tile samples were subjected to the salt spray test in a BASS USX-5000/2006 chamber. The test followed the ABNT NBR 8094 (1983b) standard.

2.7.2. Sulfur dioxide

Three roof tile samples were subjected to sulfur dioxide testing in a BASS UK-01 chamber. The test followed the ABNT NBR 8096 (1983a) standard.

2.7.3. Ultraviolet rays

Three samples of roof tiles were submitted to the ultraviolet rays test in a BASS UV/2006 chamber. The test followed the ASTM G53 (1996) standard.

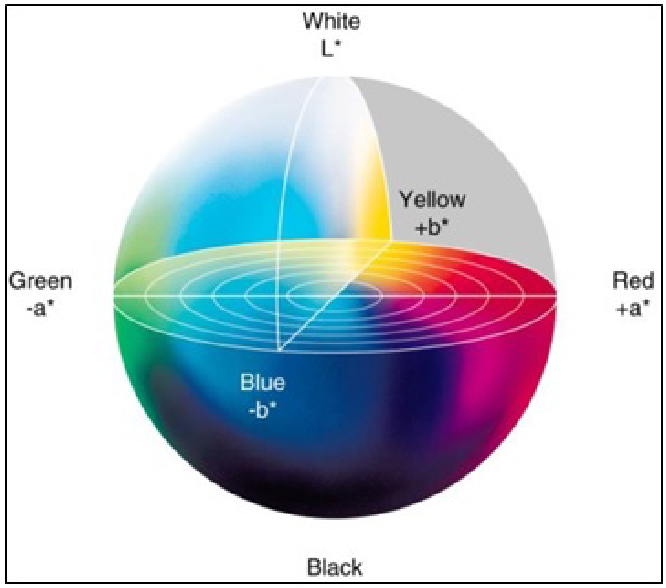

Figure 4 The CIELAB color space representing in three color coordinates L*, a* and b* (Eissa et al. 2013).

3 Results and Discussion

The macroscopic analysis, Figure 5, showed a sample thickness of around 0.4 to 1.5 cm, with a clear distinction of the ocher coloration in the external layer and the internal one showing a black color. The differentiation of such layers, in accordance with the results obtained in XRD, are represented in the external layer by quartz, potassic feldspar (microcline), mullite and gypsum and the internal layer represented by hematite.

The alterability of the roof tile outer layer was identified through the black gypsum crust formed by physical-chemical changes in the calcium present in the roof tiles in contact with the súlfur present in acid rain. However, such pollution was drastically reduced due to the removal of the Perimetral Avenue high way that passed close to the Monastery during the works to improve the city of Rio de Janeiro for the 2016 Olympics and the 2014 World Cup.

The microscopy performed on the sample, Figure 6, enabled the determination of the inner and outer layers. The inner layer (black color) is composed of a fine matrix, in which submillimeter and millimeter angular grains are dispersed, mainly quartz, feldspar and opaque minerals (associated with hematite as identified in the XRD). The outerlayer (ocher color) is composed of a fine matrix, with quartz, and eventually feldspar, submillimeter angular crystals.

(A) (B)

Figure 5 Macroscopic image obtained from a sample of roof tiles: A. Sample length; B. Sample thickness.

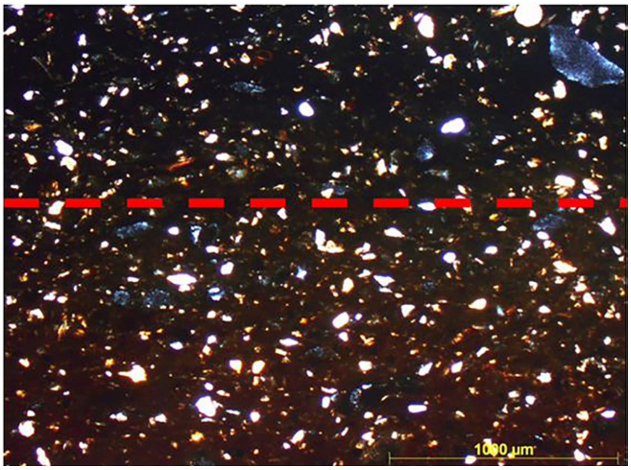

Figure 6 Microscopic image of roof tile sample.

The XRD analysis, Figure 7, showed representative spectra of the minerals quartz, potassium feldspar (microcline), hematite, mullite and gypsum. The mineralogical composition is in accordance with the materials used in civil construction as well as related weathering changes (Duggal 2017).

The XRF results are presented in Table 1, verifying contents around 60% related to sílica and around 30% related to alumina. Such oxides are present in the composition of potassium feldspar, mullite and quartz as observed in the XRD. There are also iron contents of around 6% associated with the hematite found inside the roof tile and around 1% of sulfur associated with atmospheric pollution, as previously described.

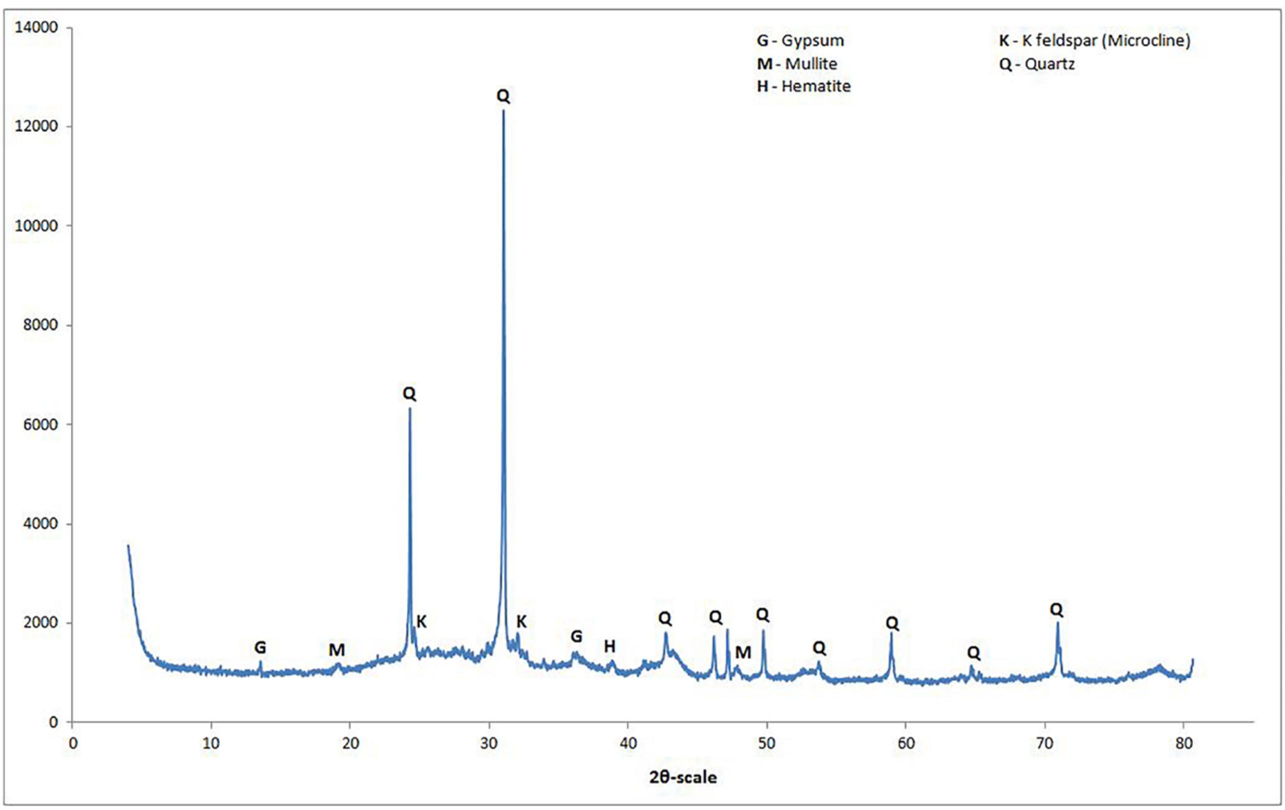

Figure 7 The x-ray diffractogram (Cokα) of roof tile sample.

Table 1 Chemical analysis (%) of the roof tile evaluated.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	Fe_2O_3
Roof tile sample	0.77	1.40	26.10	58.70	0.12	0.71	2.10	0.87	1.30	5.70

The results of the scanning electron microscopy with associated dispersive energy of the inner part of the roof tile are shown in Figure 8 and indicated a composition represented by oxygen, silicon, aluminum, iron, titanium, potassium, magnesium and sodium. This result is in agreement with that obtained by the XRF analysis.

The Figure 9 shows the infrared spectra of two specific points on the original roof tile, the red part and the black part, and it can be seen that they are very similar materials, in the case of clay minerals, given the presence of stretching of the Si-O-Si associated with peaks at 1080, 780 and 459 cm^{-1} associated with clay minerals according to observations by Medina-Dzul et al. (2015).

However, there is a more significant difference in the black part at 1390 cm^{-1}, related to the elongation of the C-O bond, that is, the black part is related to the clay burning process and corresponds to the CO not found in there placement roof tile.

Regarding the water absorption capacity, the maximum admissible limit for ceramic roof tiles, according to ABNT NBR 15310 (2005), is 20%. The test performed on the roof tile sample indicated 5.86% for this property. Therefore, the value is with in the water absorption limit defined by the standard. Low water absorption maybe associated with the presence of the inner hematite layer.

The roof tile hardness resulting in the layer without hematite is around 30 HLD and when evaluating the set containing hematite, the hardness increases substantially to 400 HLD, a value very similar to those obtained in granitic rocks, characterizing the great increase in mechanical resistance of this mineral roof tile.

Colorimetric tests were carried out to evaluate the color and brightness of the roof tile through five measurements in each layer. The outer layer of the roof tile sample tended to orange brown, while the inner layer tended to dark gray. In addition, both layers presented values close to zero for gloss, indicating that the roof tile materials are opaque.

The Figure 10 shows the differences between patterns L*, a* and b* for the internal and external area of the roof tile sample, as shown in Table 2.

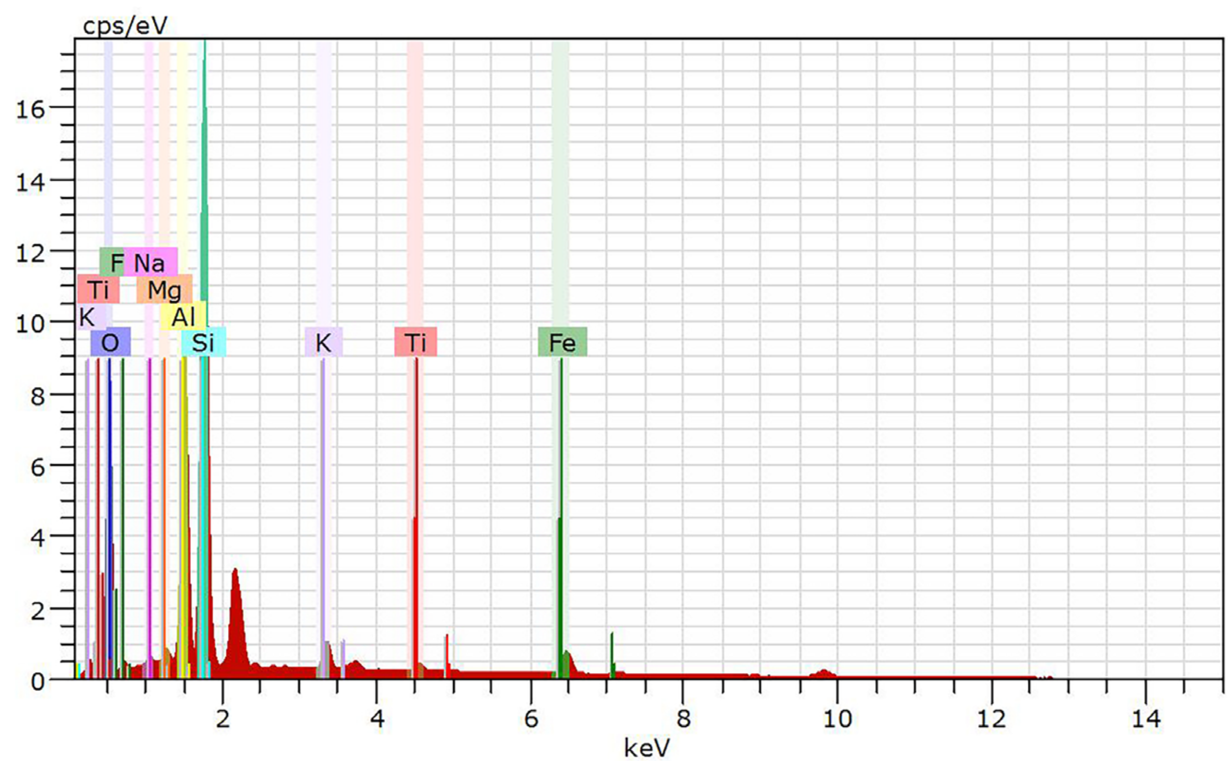

Figure 8 Scanning Electron Microscopy with Energy Dispersion (SEM-EDS) of the internal roof tile sample.

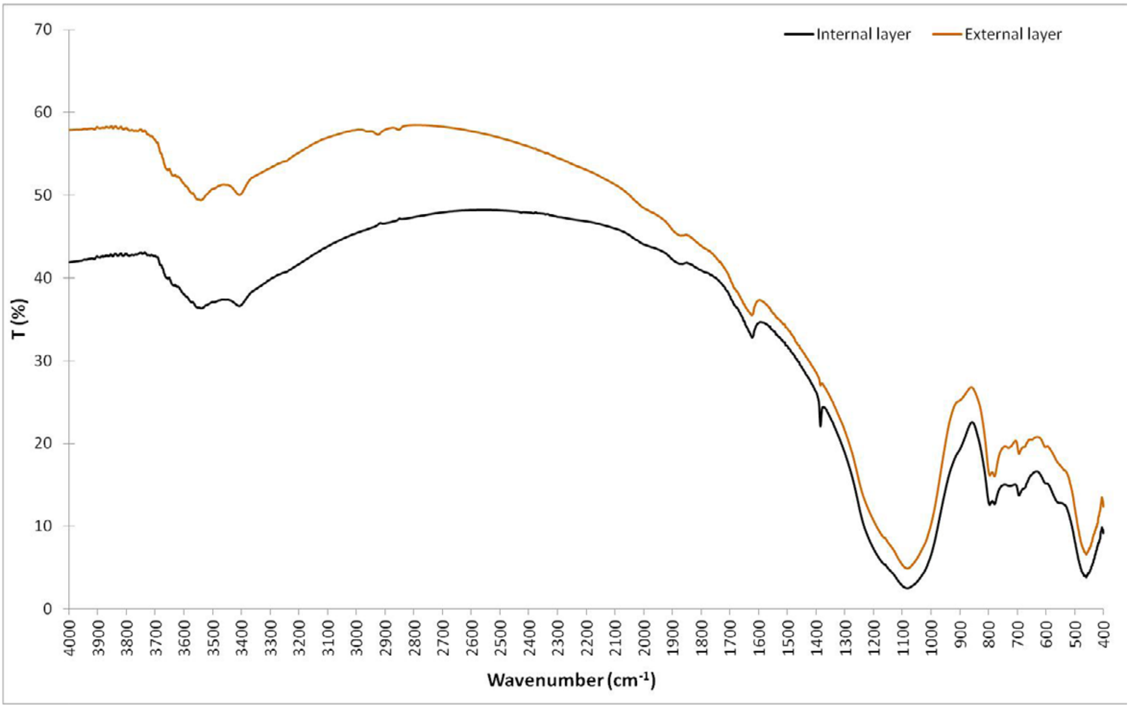

Figure 9 Fourier transform infrared spectroscopy (FTIR) of roof tile sample.

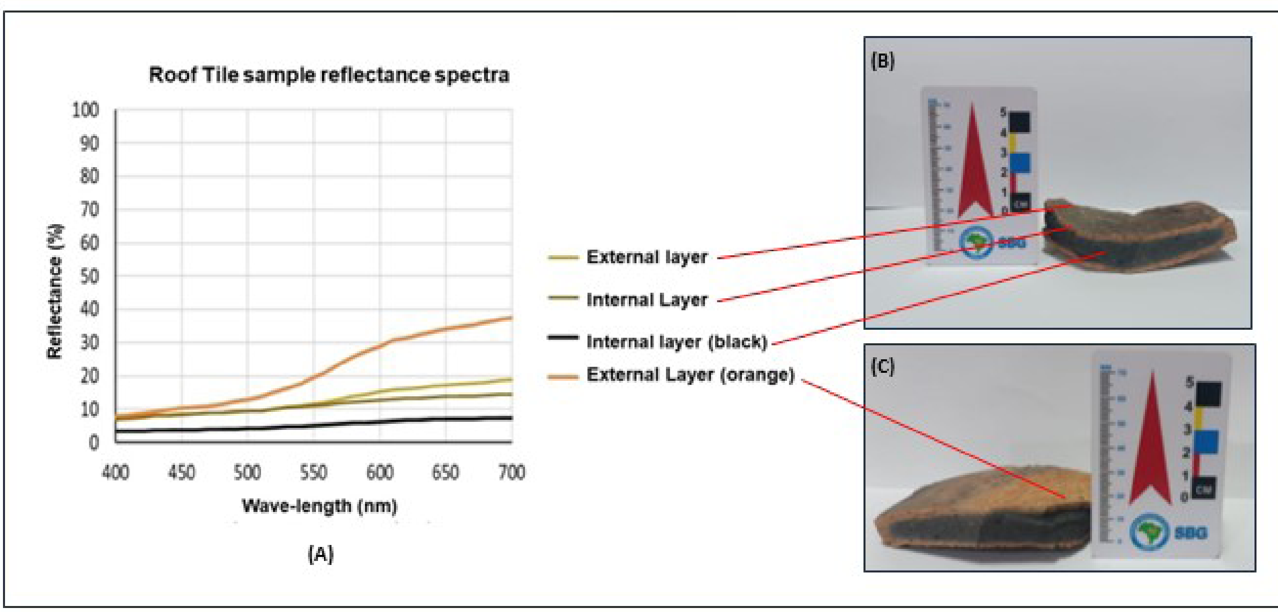

Figure 10 The differences between patterns L*, a* and b* for the roof tile sample: A. Reflectance Spectra Sample; B. Concave position of the roof tile sample; C. Convex position of roof tile sample.

Table 2 Colorimetric tests of roof tile sample.

Roof tile sample	L*	a*	b*	G (º)
External layer	41.55	6.90	11.01	0.30
Internal layer	39.76	3.40	8.57	0.00
Internal Layer (black)	27.39	4.21	7.81	0.10
External Layer (orange)	52.60	13.15	24.37	0.40

Regarding the accelerated weathering tests, them as of the sample was measured be fore and after the actions of SO_2 and saline mist, Tables 3 and 4, the colorimetric evaluations were carried out after the accelerated weathering tests, in order to compare the colors with the obtained before these tests. It was observed that after the actions of SO_2 the samples of roof tiles showed darker colors, after the actions of saline mistand UV rays the samples showed lighter colors. Results obtained from UV rays exhibited color fading compared to salt spray.

Table 3 Initial and final mass loss after salt spray analysis.

	Initial Mass (g)	Final Mass (g)	Loss Mass
Sample 1	289.00	289.00	0.00
Sample 2	303.08	303.00	0.08
Sample 3	289.44	289.41	0.03

Table 4 Chromatic roof tile standard before and after salt spray analysis.

	Initial			Final		
	L^*	a^*	b^*	L^*	a^*	b^*
Sample 1	46.41	7.73	12.69	49.12	14.88	11.90
Sample 2	39.99	9.33	6.92	40.11	16.89	9.89
Sample 3	44.44	8.90	11.99	45.89	12.90	9.99

The results obtained from mass losses were greater after the action of SO_2 compared to the salt spray. However, in both cases the mass losses were insignificant.

4 Conclusions

The analysis of the results obtained through petrography, XRD, XRF and FTIR showed that the roof tiles belonging to the Monastery of São Bento, Rio de Janeiro, have a mineralogical composition characterized by mullite, potassium feldspar and quartz, observed in the external layer and an internal part black in color, formed essentially of hematite, this layer was made at the time with the aim of increasing the tile's mechanical resistance, since the hardness of the material reaches 400 HLD, different from a roof tile without hematite which has only 30 HLD**.**

Mass losses after accelerated weathering, throughth eactions of SO_2 and salt spray, were insignificant. However, the colorimetric analyzes observed the darkening of the colors after the actions of SO_2 and their fading after the actions of salt spray and UV rays.

The results obtained provide knowledge about the material that makes up the monastery's roof tiles, allowing future measures to mitigate and/or reduce bad weather and/or conserve this historical heritage.

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Author contributions

All authors contributed to the preparation of the manuscript.

Conflict of interest

The authors declare no conflict of interest.

Data availability statement

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