

Neutron techniques applied to better define conservation strategies of 16th – 18th centuries Portuguese glazed tiles

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The city of Lisbon has in situ sixteenth-eighteenth -century tiles that sometimes were decontextualized of its original architectural framework and landscape, owing to the deep urban changes operated in the following centuries.

There are several examples in the various city gardens and ancient buildings all over Lisbon and other cities such as: (i) 16th century tile panel near the floor in the Madre de Deus Church (belongs to the National Tile Museum) in so advanced state of degradation that have been taken off and replaced by new ones; (ii) 17th century set of tiles in the Torel hill, near the Campo de Santana, with advanced state of degradation and in the rear part of a new condominium; and (iii) 18th century glazed tile from the “N.S. Conceição dos Aflitos” Church (Elvas, Portugal).

These glazed tiles from different centuries have been placed in diverse environments along the last centuries. The 16th tiles were indoor close to the floor and given the proximity of the river and the low water level, they were seriously damaged by the effects of water ascendance by capillarity. The 17th tiles are outdoor exposed and without any kind of protection. The 18th tiles are indoor and are the less deteriorated.

Objective: to identify the degradation state and the main degradation processes of glazed tiles, so that they can be better overcome or at least reduced, giving tools to conservators so they can better choose the intervening strategy. In order to contribute to better design future actions of conservation/restoration of Portuguese glazed tiles, neutron techniques, namely neutron tomography and instrumental neutron activation analysis, were employed. X-ray diffraction was also used to identify the mineral phases of the ceramic body and mortar.

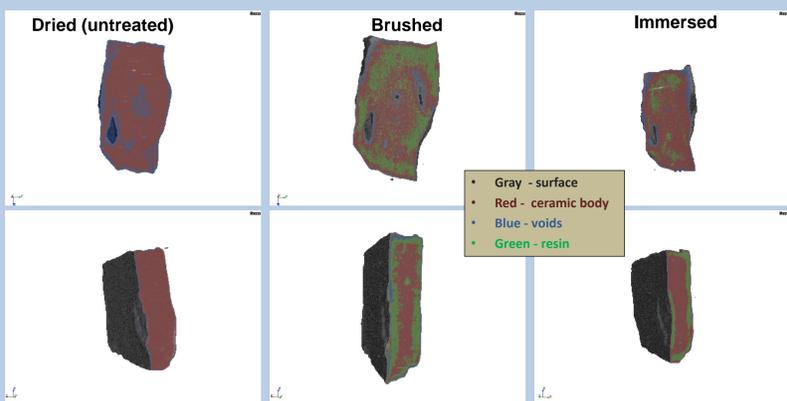
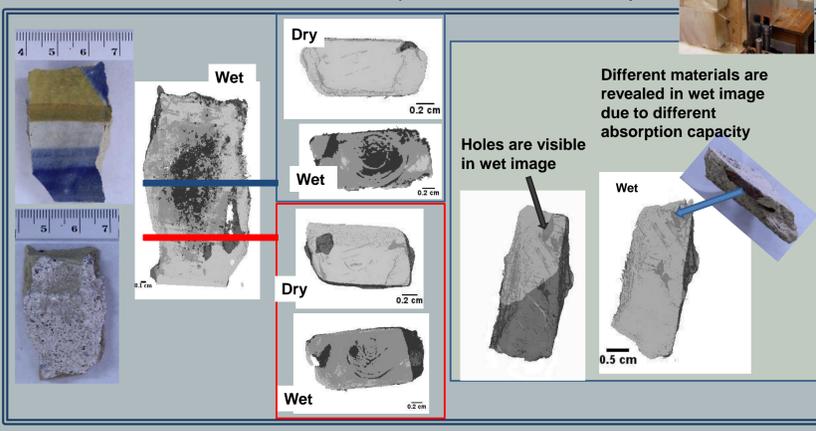
Neutron tomography (NT) –RPI (IST)

The tomography setup was installed at the horizontal access of the thermal column of the Reactor Português de Investigação (RPI).

Neutron flux at the irradiation position	2.2x10 ⁵ n cm ⁻² s ⁻¹
Beam diameter	5 cm
Mean energy	25 meV
Spatial resolution (µm)	323 (11)



Advantages of NT: neutrons have a high interaction probability with hydrogen which allows to evaluate the degradation state of the tile and visualize the water and consolidants penetration inside the object.



Instrumental Neutron Activation Analysis (INAA)

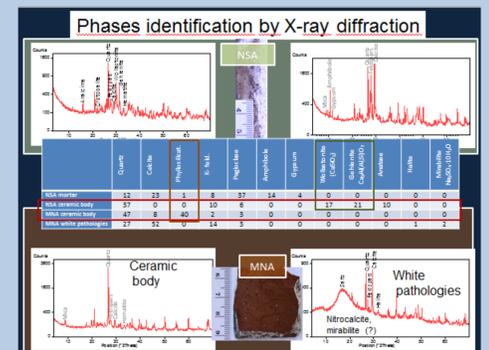


Advantages of INAA:

- Simultaneous determination of several elements with different geochemical behaviours (good fingerprints)
- Extremely sensitive, able to detect many elements in minor and trace amounts.
- Good precision and accuracy (in general within 5%)
- Small amount of sample required for analysis (micro-invasive).

Consolidation was conducted using Paraloid B-72® (copolymer of methyl acrylate and ethyl meta acrylate), a acrylic adhesive known for its stability and reversibility.

Paraloid B-72 is dissolved in acetone (1:9) to produce a low viscosity solution that may be applied by brush or immersion. To facilitate impregnation the piece is first soaked or immersed in the solvent, to aid the diffusion of the resin in the interior of the ceramic body.

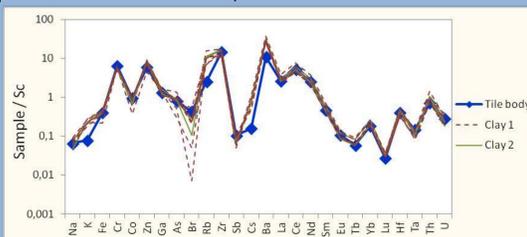


INAA showed that geochemical patterns (together with mineralogy), are a very useful approach for the knowledge of nature of these works of art, as they characterized the original materials and the alteration products. These results allowed identifying the chemical and physical conditions which favored endogenous and/or exogenous processes of decay.

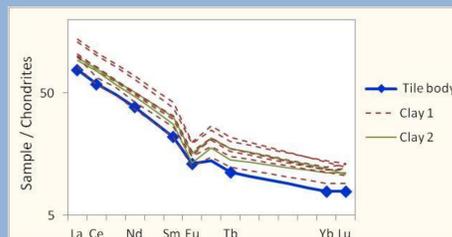
NT, enabling an inner visualization and the light elements content, particularly hydrogen, is particularly useful to evaluate the degradation state of each tile.

NT showed that brushing technique to apply consolidants appears to be more efficient than immersion technique, used in the National Tile Museum.

- Quinta do Torel tile and clays from Lisbon region (two Miocene units: **Clay 1**: M²_{Iva} – “Argilas do Forno do Tijolo”; **Clay 2**: M¹_{II} – “Areolas da Estefânia”)

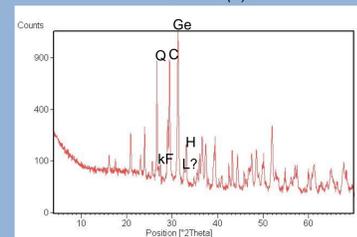


▪ Ceramic body has similar geochemical pattern to Lisbon clays, but with **lower alkalis contents and generally higher Br content** - Br enrichment due to organic matter contamination.



▪ Ceramic tile body has similar chondrite-normalized REE pattern to Miocene Lisbon clays, but with **slightly lower Eu anomaly and, in general, lower concentrations of all REE**

Ceramic tile body: Gehlenite (Ge) > Calcite (C) > Quartz (Q) > Hematite (H) > Potassium Feldspar (kF) > Larnite (L)?



▪ Raw materials: carbonated clays (calcite-rich)
▪ New minerals with firing: gehlenite, larnite and probably hematite
▪ Firing Temperatures: 800 – 900 °C

➔ Neutron techniques allowed establishing a methodological approach to help conservators to select conservation practices of tiles in different environmental conditions. **Brushing technique** appears to be a suitable technique for consolidation, which favors the conservation procedures of the tiles in situ, like in the Quinta do Torel panel.

