# RADIART

FCT funded Project: Diagnosis, decontamination and conservation of cultural heritage: neutrons and ionizing radiation in artwork (PTDC/HIS-HEC/101756/2008)

# Non-destructive and micro-invasive techniques for cultural heritage diagnostics

## - a case study of glazed tiles from Portuguese historical buildings

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Among cultural assets, ceramics and particularly glazed tiles ("azulejos" in Portuguese and Spanish, from the Arab designation "al-zuléija" or "al-zulaiju") deserve particular attention in the Mediterranean region, where they have long been used to decorate buildings. "Azulejos" are present in many historical Portuguese buildings of the XVII to the XIX centuries. In fact, one may say that "azulejos" are everywhere in Portugal. They decorate everything from walls of churches and monasteries, to palaces and ordinary houses. Most of these "azulejos" present various degradation features, mainly due to exterior exposure in a range of different environments.

Scientific analysis / investigation of cultural heritage objects is ideally conducted in a manner that enables fail-safe transfer to late generations. A research methodology including the application of non-invasive (neutron tomography –NT) and micro-invasive techniques (XRF, XRD) for diagnostic of two types of "azulejos" is presented.

Materials studied: - Glazed tiles from the XVII century

NSA

MD

Madre de Deus Church, Lisboa (sample MD)

**BIOBURDEN ANALYSES OF TILES** 



#### Nossa Senhora dos Aflitos Church, Elvas (sample NSA)



## Chemical characterization by XRF

Sample		<b>Yellow</b> NSA	Blue NSA	Green MD
K Ka	136.8*	56 020	187 933	11 120
K Kb	118.2	11 642	19 971	1 375
fundo	134.0	548	1 109	374
Sb La	117.5	30 943	3 526	639
<b>Sn</b> Lb	114.4	3 469	2 352	1 234
Ca Ka	113.1	26 299	48 335	17 690
fundo	71.00	328	569	426
Cr Ka	69.36	558	678	609
Fe Ka	57.52	66 112	105 667	34 345
Co Ka	52.80	2 451	32 702	1 355
fundo	50.00	1 176	1 306	1 307
<b>Cu</b> Ka	45.03	2 904	5 375	43 780
<b>Zn</b> Ka	41.80	21 079	37 798	4 361
As / Pb	34.00	129 738	394 215	216 602
As Kb	30.45	2 327	7 034	2 446
Pb Lg	24.07	14 670	33 446	19 243
fundo	21.50	1 809	1 605	1 868

# Glaze

### Phases identification by XRD

## **Bindheimite** Yellow "Naples yellow" Ca<sub>2</sub> Sb<sub>2</sub> O<sub>7</sub>

Blue SnO<sub>2</sub> Green Amorfphous

#### **METHODS:**

## 1) DESTRUCTIBLE – MECHANICAL Destruction of samples item proportions Wash of the samples in physiologic serum with Tween 80 (0.1%) and homogenization by Stomacher® (mechanical procedure).

Spread aliquots of washing solution in Tryptic Soya Agar dishes and incubation at 30 °C for seven days. Colony forming units (c.f.u.) were counts along this period.

## 2) Swab – Swab scrub samples portions with a swab in 5 ml of physiolog serum with Tween 80 (0.1%).

Homogenize the swab in the wash solution by vortex (3000 rpm) during 1 minute.

Spread aliquots of washing solution in Tryptic Soya Agar dishes and incubation at 30 °C for seven days. Colony forming units (c.f.u.) counts along incubation

period. METHOD EFFICIENCY DESTRUCTIVE: 60 - 70%SWAB: 40 – 70%

**BIOBURDEN**: NSA:  $(9 \pm 4) \times 10^2 \text{ UFC/cm}^2$ MD :  $4.1 \pm 0.8 \text{ UFC/cm}^2$ 



Type X: yeasts

### BIOBURDEN CHARACTERIZATION (n = 169 isolates)

#### MOST FREQUENT MORPHOLOGICAL TYPES

Type IX: rods, gram -, oxidase -

■Type XI: fungi

# Ceramic body and mortar Phases identification by XRD NSA Counts Mortar 1600 Mic Gyp 400 Position [°2Theta Counts Ceramic body 900 10 20

NSA: Gram positive cocci, catalase positive (95%) MD: Gram negative rods, oxidase positive (64%)

## **Neutron Tomography (NT)**

Visualization of physical structures in the interior of an object without physically opening

Neutrons have the advantage to penetrate most metals (especially heavy ones) very efficiently and to detect hydrogeneous compounds very sensitively.

NT has application fields in moisture detection, test adhesive connections, structure analysis





Neutron radiography has a tradition since neutron sources and imaging devices became available. Although this method cannot compete directly with X-ray methods on a broad scale due to a smaller number of strong neutron sources (which are mainly stationary ones, such as research reactors or spallation sources), it has specific advantages:

neutrons have a high interaction probability with hydrogen (and other light elements like carbon) and a

lower attenuation in several heavy elements which are "black" for X-rays, such as Pb, Bi and U.



