

# Cultural Heritage and Sciences

*M. Isabel Prudêncio*

The Cultural Heritage and Sciences (CHS) group is especially voted to the **study of the Portuguese cultural assets and corresponding environment contexts**, through the **application of nuclear methods**, including chemical characterization and absolute dating.

Geochemistry, mineralogy and chronology (absolute dating), are the main research domains, applied to archaeometry, environmental geology and paleoenvironmental reconstruction.

The CHS research activities during 2007 were developed as follows:

- Portuguese clays: geochemical and mineralogical characterization with a view to technological applications
- Luminescence applied to dating geological and archaeological contexts and artefacts
- Luminescence applied to faiences and porcelains: Dating, authenticity, materials, pigments
- Archaeometry of ceramic, metal and lithic materials
- Geochemistry of the earth surface: natural background and evaluation of anthropogenic inputs
- Nuclear methods of analysis: development and application of luminescence techniques and neutron activation analysis

The analytical methods associated to the CHS group, include the instrumental neutron activation analysis (INAA) and luminescence dating (TL-OSL). Other methods, such as X-ray diffraction, SEM-EDS and Mössbauer are commonly used.

The application of these methodologies unique in Portugal, is crucial for solving archaeological and geological problems. The research is developed

through financed projects, protocols, collaboration with national and international laboratories and universities, and contracts/services with private and public institutions.

This group is specialized in trace-element analysis and geochemistry (i.e., geochemical fingerprinting) and mineralogy of geological and archaeological materials. We are an archaeometrically oriented lab in Portugal with powerful analytical techniques in a single location. We also support geochemical research mainly concerning superficial environments, related with weathering and sedimentary processes, as well as environmental studies.

Research of CHS group also comprises luminescence techniques applied to archaeology and geology, including the study of natural radioactivity. The luminescence laboratory has incremented the number of financed research projects in geology and archaeology: (i) coastal dunes and fluvial sediments; (ii) archaeological contexts; (iii) dating and authenticity of pottery from various chronologies (from pre-historical to contemporaneous materials).

Under the National Scientific Infrastructure Programme, an automated combined TL/IRSL/Blue-light OSL dating reader system, Risø TL/OSL-DA-15C/D, with a dual laser single grain OSL attachment, and a Integrated Gamma Spectrometer with automatic sample changer were installed and are fully running, increasing the analytical capabilities of the group.

One of the CHS group most important activities involves the education and training of students from national and international universities. Our students participate in the entire research work, including: field work and sampling, sample preparation for several types of analytical techniques, irradiations and measurements, and data management and interpretation. Thus, they become able to accomplish projects in fundamental and applied research.

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## Research Team

### Researchers

M. I. PRUDÊNCIO, Princ. Agregação, Group Leader  
M. I. DIAS, Invited Aux.

### Students

M. J. TRINDADE, PhD student, FCT grant  
A. JORGE, PhD student, U. Sheffield grant  
C. CAPITÃO-MOR, MSc student  
S. VILELA, MSc student  
L. PENG, MSc student  
A. CALADO, MSc student  
P. FRANCISCO, graduation student

### Technical Personnel

L. FERNANDES, laboratory technician  
R. MARQUES  
D. FRANCO  
G. CARDOSO

### Collaborators

M. A. GOUVEIA, Princ. Researcher  
(Retired)

## Portuguese clays – geochemical and mineralogical characterization with a view to technological applications

M.I. Dias, M.J. Trindade, R. Marques, M.I. Prudêncio, D. Franco, M.A. Gouveia, F. Rocha<sup>1</sup>

The characterization of clay raw materials from the Meso-Cenozoic southern and western borders of Portugal was performed using three approaches: granulometric, mineralogical and geochemical. The study of the behaviour of raw materials with firing (at temperatures from 300 to 1200° C) was also performed in laboratory experiments simulating the ceramics manufacture process, in order to enable future archaeometric studies. The main goals are: (i) better understanding the mineralogy and geochemistry of sediments, particularly of clayey levels, and (ii) a more detailed knowledge of the distribution of trace elements, namely rare earth elements (REE), in sediments and their correlation with mineralogy, particularly the clay minerals.



Fig. 1. Bustos clay pit in Aveiro region (Upper Cretaceous level of the occidental border)

Mineralogy allowed distinguishing different types of materials, as well as a better understanding of the paleoenvironment and evolutionary history of the depositional environments. Elemental composition and ratios of immobile elements of both felsic and mafic sources contribute to establish the origin of the deposits, the degree of chemical weathering and the recycling of material coming from the upper continental crust. The major elements, certain trace elemental ratios (Th/Sc, Zr/Sc, La/Sc) and the rare earth elements patterns showed variations on sediments source areas and have evidenced the importance and responsibility of weathering, sedimentary calibration and diagenesis processes on the variation observed among and inside groups. After Sc normalization, it was possible to evaluate the geochemical nature of different units.

Temperature-induced mineralogical transformations of Algarve region clays were found to be dependent on the composition of the materials, particularly the type and content of carbonates (calcite and dolomite).

Detailed studies of fine fractions allowed identifying the trace elements incorporated in clay minerals by absorption / adsorption and/or substituting major elements.

The results obtained enabled to establish geochemical and mineralogical reference groups and fingerprinting clay raw materials, essential for contemporaneous and ancient ceramics applications.

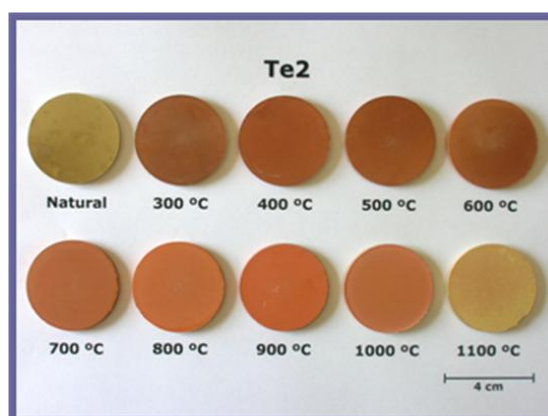


Fig. 2. Probes of powdered clay sample after firing at different temperatures.

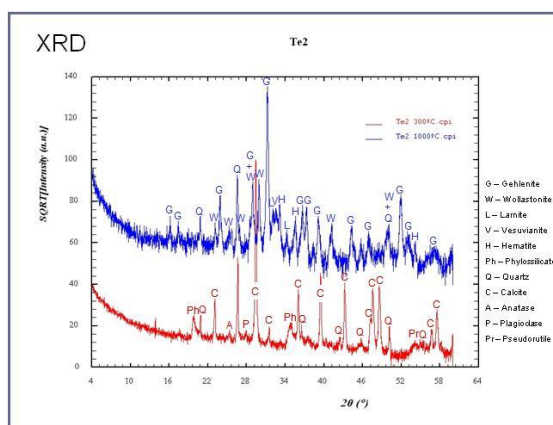


Fig. 3. XRD patterns of fired clays (300°C and 1000°C)

### Published work:

M. I. Dias, M. I. Prudêncio (2007), On the importance of using scandium to normalize geochemical data preceding multivariate analyses applied to archaeometric pottery studies, *Microchem. J.* (2007), 136-141, doi:10.1016/j.microc.2007.11.009

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**Optically stimulated luminescence applied to dating geological and archaeological contexts**

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An international intercalibration was done in order to validate analytical methods and results comparability, including uncertainties about luminescence mechanisms, measures reproducibility and precision, sample heterogeneity, humidity content and nuclide migration. In the case of a climbing dune, results on grain variations in burial dose rates were mainly due to spatial heterogeneity of radioisotopes contributing to the  $\beta$ -dose field and to non-uniform shielding of grains from relatively low activity. The effect of such heterogeneity is an increase in the scatter of equivalent dose values. The application of OSL to geology has contributed to a better knowledge of the Quaternary climate change scenario, based on multi-proxy investigations of paleosols, sediments and dunes. The TL-OSL absolute dating of archaeological contexts and related artefacts (ceramics and lithics) contributes to a better definition of the chrono-stratigraphic sequence of human occupation.



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**Luminescence applied to Portuguese Faience and Chinese Porcelain produced for the Portuguese market (XVI to XVIII centuries): Dating, authenticity, materials, pigments**

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This project focus on Portuguese faience and Chinese porcelain produced for the Portuguese market (XVI to XVIII centuries), aiming to contribute for a better knowledge of chronology precision, identification and, if possible, differentiation of production centers and technologies of ceramic production, as well as to characterize surface coatings (glazes and pigments). The main methodological approaches are INAA and XRF for chemical characterization, along with XRD for phase identification.



Considering that another main goal consists in accurately dating the ceramics, also TL was done to pastes. TL has an important impact in the field of authenticity, contributing to well judge genuine and imitative objects. These approaches have contributed to ascertain chemical and mineralogical composition of pastes, to outline the technological features and to define the nature of coatings and pigments.



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**“Material Cultural Science”: Dating and provenance of archaeological ceramics from the Portuguese and Spanish Guadiana basin**

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This research project concerns a more accurate chronology establishment of Copper Age ceramics, as well as, a detailed study of production technology and consumption of artefacts from the Portuguese and Spanish Guadiana basin archaeological sites, namely Perdigões, Porto Torrão, Monte do Tosco and Mercador, and San Blas, La Pijotilla, Palacio III and Molino Perdido, respectively. Methodology involved includes physical and chemical characterization techniques (INAA, XRF and XRD), AMS and luminescence dating techniques. Compositional groups and categorization of productions as well as ritual and votive patterns were established. An important correlation between compositional grouping and the way these categories show a relationship with the spatial and chronological variations recorded in the use of archaeological materials was obtained.

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**Archaeometry of ceramic, metal and lithic materials**

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Nuclear methods of analyses were applied to several archaeometry studies, including ceramics, metal and lithic artifacts. The technological procedures during the manufacture process of making ancient pots, and its provenance were established for pre-historic ceramics from settlements and necropolis contexts of Beira Alta archaeological sites, contributing to a better understanding of continuities or innovations in ceramic production in a diachronic point of view.



Similar studies were done to other chronologies and regions, such as the Islamic ceramics from Silves, Algarve. Also ceramic and lithic raw materials resources exploitation strategies at local/regional scale were studied for several national archaeological sites, like silex artifacts characterization from Lisbon region.

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**Casa do Governador da Torre de Belém (Tagus estuary): Halieutical resources industry in Roman times**

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This project aims to understand one of the most important economic activities of the coastal areas of the former Roman Province of Lusitania, the salted fish and *salsamenta* production, exported in amphorae to other areas of the Roman Empire. By the study of the archaeological site “Casa do Governador da Torre de Belém” (Tagus estuary) and related contextual evidence, the aim is to provide a solid understanding of its organization and chronology. The archaeozoological study is done with a view to better understand the nature of the products and the kind of fishing activity involved. The archaeometric approach, using the ITN database of Lusitanian amphorae production centers, aims to identify the amphorae production center that supplied that fish industry. This running project also makes available data that can be used by researchers working in other areas of the Roman Empire, contributing to the establishment of the final destination of the Tagus estuary products.

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**Geochemistry of the Earth surface: natural background and evaluation of anthropogenic inputs**

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Geochemistry, crystal chemistry and mineralogy studies are applied to environmental projects. The expertise on geochemistry of the earth surface of CHS group researchers enables a detailed and complete overview in this kind of studies, with the characterization of the natural background and the evaluation of anthropogenic inputs. The CHS running projects, concerning the quality control and identification of pollutants of geological environments, refer mainly to lagoon environments. The geochemistry of the earth surface, particularly the geochemistry of trace elements in secondary natural systems, and the alterations due to pollution actions in sediments and soils, was applied to two case studies: the “Lagoa das Sete Cidades”, Açores, Portugal and the “El Melah” lagoon, Tunisia.



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**Nuclear methods of analysis: development and application of luminescence techniques and neutron activation analysis**

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The luminescence and neutron activation laboratories were reinforced with new equipment on behalf of the national Re-equipment Program by FCT (CONC-REEQ/590/2001).

Instrumental neutron activation analysis is a well-established method within this group since the seventies. Based on the use of the RPI (nuclear research reactor unique in the Iberian Peninsula), this analytical method together with luminescence techniques (complemented with other methods) supports most of the research activities of the CHS group. Public and private institutions often requested CHS group for services of both laboratories. The quality of the analytical procedures of INAA is periodically controlled through the analyses of international standards and inter-calibrations with other laboratories, in order to maintain and increment the good performance of the laboratories. The Luminescence Dating Laboratory participates in international comparisons, particularly with the Musée du Louvre (CNRS), France and IUX, Spain.