# Cultural Heritage and Sciences

*M. Isabel Prudêncio*

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

Absolute dating, geochemistry and mineralogy are the main research domains, applied to archaeometry, environmental geology and paleoenvironmental reconstruction.

The principal research domains of CHS group include:

- Luminescence dating of archaeological contexts and artefacts.

- Optically stimulated luminescence applied to dating geological contexts.

- Thermoluminescence applied to archaeological / museum artefacts: authenticity of ceramic art objects.

- Chemical and mineralogical characterization and technological properties of Portuguese clays.

- Archaeometry of ceramic, metal and lithic materials.

- Environmental geochemistry: natural background and evaluation of anthropogenic inputs.

- 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) - comparative method, for chemical characterization, and luminescence dating (TL-OSL). Other methods, such as X-ray diffraction, SEM-EDS and Mössbauer are commonly used.

The application of ITN methodologies, unique in Portugal, is fundamental for solving problems of archaeological and geological nature. The research is developed through collaboration protocols, financed projects, 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 mineralogical phases assembly of archaeological specimens and raw materials (namely clays and silex) for the purpose of determining their provenance by using the above mentioned analytical techniques as tools. We are an archaeometrically oriented lab in Portugal with powerful analytical techniques in a single location. We also support geochemical research on the petrogenesis of various igneous, metamorphic, and sedimentary rocks, as well as environmental studies.

Research of CHS group also comprises luminescence techniques applied to various fields, archaeological and geological, including the study of natural radioactivity. New fields of application are in progress with the development of luminescence techniques, like a better knowledge of technological production of archaeological artefacts, allowing a better identification of post-burial phenomena. The Luminescence laboratory has incremented the variety of end users, supporting not only research projects, but also services to the community, like the authenticity of public and private ceramic art objects.

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, sample preparation, irradiation, and measurement and continuing through the statistical analysis and data interpretation, thus becoming well prepared to conduct research in academic and industrial environments.

## 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
P. FRANCISCO, graduation student
F. GEIRINHAS, graduation student
A. C. VERÍSSIMO, graduation student

### Technical Personnel

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

### Collaborators

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

# Luminescence dating of archaeological contexts and artefacts.

*M. I. Dias, M. I. Prudêncio, D. Franco, G. Cardoso, J. C. Waerenborgh*[1]
*A. Zink*[2], *A. C. Valera*[3], *C. Odriozola*[4], *V. Hurtado*[5] , *L. Osterbeek*[6]

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. The main goal of several running projects in this research area is to establish chronological references of human occupation levels. Beyond luminescence technique, the use of nuclear methods of analysis, namely INAA, using the RPI as neutron source is done, abling a more accurate paleodose in the luminescence absolute dating determination. These methods are complemented with XRD for mineralogical control.

The main steps evolved in this research domain include: (i) field work - sampling collection and in situ measurements of environmental radioactivity (gamma measurements); (ii) laboratory preparation of samples according to specific protocols; (iii) natural radioactive elements (K; U; Th) contents determination, by INAA, using the RPI as neutron source; (iv) OSL (Optically Stimulated Luminescence) dating of sediments and paleosoils; (v) TL (Thermoluminescence) dating of lithic materials (silex) and ceramics.

A relevant work developed in this research area was done in an urban context stratigraphy occurred along the Lisbon hill slope of Bairro Alto, at Lumiares Palace. A chronological stratigraphic differentiation by luminescence dating (TL-OSL) was performed, with special attention to the preserved paleosoil identified in the archaeological excavations. Several stratigraphic units were identified in the archaeological excavation, also differentiating geological formations. Chemical (INAA) and mineralogical composition (XRD), granulometry, calcimetry and organic matter content were obtained for the sediments of the identified units, as well as luminescence dating of sediments, soils and heated silex. In a geochemical point of view, the occupation levels developed above Miocene deposits present affinities with them, even with higher contents of organic matter and of chemical elements related with ferromagnesian minerals. The colluvium above paleosoil is detachable from other deposits by a more heterogeneous mineralogical composition and with higher amounts of heavy minerals and plagioclases. A better definition of the chrono-stratigraphic sequence was obtained by TL-OSL of both human occupation levels and geological deposits (Fig. 1), by establishing a chronological reference of the human occupation level in a late phase of early Neolithic (B-OSL of paleosoil samples and TL for related artifacts - silex were consistent: 6.000 years; error: ± 3%).

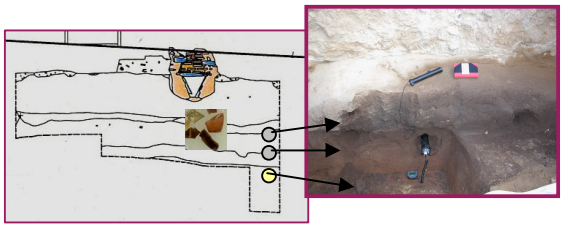

Fig. 1 – Luminescence dating of stratigraphic units identified in P. Lumiares Lisbon urban context.

A new application of luminescence techniques was undertaken in order to better estimate firing temperatures of archaeological ceramics. The identification of clay minerals in some ceramics (XRD), as well as the significant presence of iron oxides (essentially hematite) with nanometre granulometry (Mössbauer), suggests that pottery was not fired at temperatures above 500ºC, or had had post-burial alterations. The already obtained TL results have been more enlightening: (i) reflecting maximum firing temperatures lower than 550ºC (Fig. 2); (ii) pointing to the same firing temperature (550ºC), but with modifications in the crystalline net that could reflect post-burial alterations.

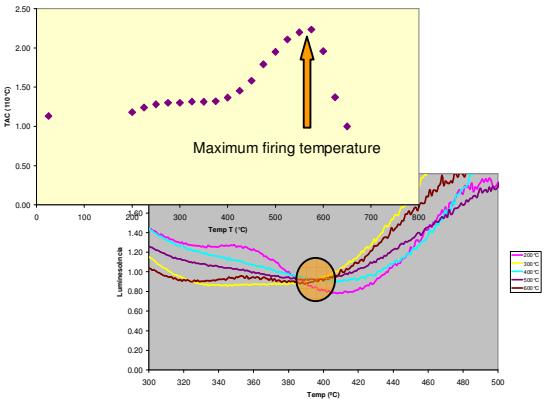


Fig. 2 – Glow-curves (TAC and annealing) for archaeological ceramic.

## Published or in press work:

G.O. Cardoso, M.I. Prudêncio, A. Zink, M.I. Dias, J.C. Waerenborgh, Determinação da temperatura de cozedura de cerâmicas arqueológicas: Luminescência – potencialidades e limitações. *Proc. Congreso Ibérico de Arqueometria.* Girona, Espanha (in press).

[1] Solid State Group of ITN, [2] CNRS, Musee do Louvre, Paris, France, [3] Era Arqueologia S.A. Portugal
[4] ISCME, CSIC, Seville, Spain, [5] Univ. Seville, Spain
[6] Instituto Politécnico de Tomar, Portugal.

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

*M.I. Dias, M.I. Prudêncio, D. Franco, G. Cardoso, R. Marques, D. Mosquera[1], J. Sanjurjo[1], A. Martins[2], L. Rebelo[3]*

Research undertaken in this field by the CHS group involves the analysis of geological contexts, namely former coastal environments. Research is mainly devoted to palaeosol and sediment studies by absolute dating (OSL), chemical (INAA) and mineralogical (XRD) characterization. Recent and current projects have included working with other national and international laboratories and universities. Also services to private and public institutions were done. The application of OSL method to geological investigation has a huge contribution to the palaeoenvironmental reconstruction, investigating and explaining environmental change on different spatial and temporal scales. Our contribution assists landscape evolution, particularly the Portuguese coast line, in order to determine the geological processes that control the evolution of the beach and the effects that this evolution has in the shoreline displacement. Thus, these research fields aim to contribute to a better knowledge of the Portuguese Quaternary climate change scenario, based on multi-proxy investigations of paleosols, sediments and dunes. These investigations constitute the research component into long-term environmental change of climate and shoreline.

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**Thermoluminescence applied to archaeological / museum artefacts: authenticity of ceramic art objects**

*M.I. Dias, M.I. Prudêncio, D. Franco, G. Cardoso, A. Zink[1], M O. Figueiredo[2], T. Silva[2]*

Authenticity testing by thermoluminescence have been implemented and applied to art objects in ITN laboratory. This technique provides a very powerful independent judgment of what is genuine and what imitative. Research work has been performed in order to optimize the laboratory protocols for ceramics and porcelains. Services to private collectors were done to art objects of various chronologies and provenances. Recent collaboration with other laboratories and museums was established, specially aiming a better chronology precision, identification and differentiation of national production centres and technologies of ceramic production, and characterization of surface coatings (glazes and pigments).

[1] CNRS, Musee du Louvre, Paris, France, [2] IGM, INETI, Portugal

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**Chemical and mineralogical characterization and technological properties of Portuguese clays**

*M.I. Dias, M.I. Prudêncio, M.J. Trindade[1], R. Marques, D. Franco, M.A. Gouveia, F. Rocha[2], J. Coroado[3], M.A. Sequeira Braga[4],*

Detailed studies of Portuguese clays of different geological contexts are in course, including geochemical, mineralogical and technological characterization for two main fields of application, namely ceramic production (ancient and present-day) and waste-fills liners. In Portugal ceramic handicraft has a long tradition. Detailed studies of raw materials will benefit this economic sector. An inventory of availability of potential raw materials for ceramic production is running in the southern and occidental Portuguese borders, followed by an evaluation of chemical (INAA) and mineralogical (XRD) transformations of clay materials of different nature, during ceramics production processes, and its implications on provenance and technological procedures studies of ancient and present-day ceramics. In Portugal, due to the replacement of old traditional waste-fills by new ones for domestic and other common wastes, according to EU norms, we need to find in each region of the country appropriate clay deposits for exploitation in order to use them as liners. The mineral and chemical composition and other relevant properties required for clay liners, such as: heat diffusion, cooling rate, fluid-rock interaction, hydromechanical behaviour and permeability are studied. Smectite is considered as the more adequate clay mineral for this kind of application. In Portugal, heavy clays utilized for structural ceramics are quite common. These clays containing smectite-illite-kaolinite assemblages and being considered, from the hydrogeologic point of view, as presenting extremely low permeability, will be our main target.

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

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Archaeometry is one of the main research fields of CHS group, mainly applied to archaeological ceramics. The application of nuclear methods of analyses to archaeometry was extended to other types of materials, like lithic and metal artifacts. The main goals include: (i) establishment of the technological procedures during the manufacture process of making ancient pots, and its provenance, in order to contribute to the establishment of continuities or innovations in ceramic production; (ii) ceramic raw materials resources exploitation strategies at local/regional scale; (iii) characterization and provenance of lithics; (iv) metalworking process including ore samples, crucible fragments and slag production; (v) ore sources of archaeological artifacts; (vi) to reconstruct the smelting process based upon qualitative and quantitative analysis of the materials; (vii) geological reappraisal of regional metal sources, contributing for the knowledge of geographical sources of raw materials and metal alloys used.

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**Environmental geochemistry: natural background and evaluation of anthropogenic inputs**

*M.I. Prudêncio, M.I. Dias, R. Marques, D. Franco, J.C. Waerenborgh[1], F. Ruiz[2], E. Galan[3]*

Geochemistry, crystal chemistry and mineralogy studies are applied to environmental projects, with a view of the preservation of classified natural environments and valorization of natural resources. The expertise on geochemistry of the earth surface of CHS group researchers enables a detailed and complete overview in this kind of studies. The characterization of the natural background is a main goal which is essential for the evaluation of the anthropogenic input in the geological environment. The CHS running projects, concerning the quality control and identification of pollutants of geological environments, refer mainly to lagoon environments focused in the study of the geochemistry of the earth surface, particularly the behavior of trace and major elements in secondary natural systems, and the alterations due to pollution actions.

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**Development and application of luminescence techniques and neutron activation analysis**

*M.I. Prudêncio, M.I. Dias, R. Marques, M.A. Gouveia, D. Franco, G. Cardoso*

The main objective of the Luminescence Dating Laboratory is the development and application of luminescence dating techniques (TL, G-OSL, IRSL, B-OSL) in collaboration with archaeologists and geologists, and assisting curators and private collectors in establishing chronologies. In addition, fundamental studies on the luminescence properties of minerals, which could be exploited for dating application, has been an important part of the work. In order to assure the quality of dating results and to optimize lab and measurements protocols, the ITN luminescence dating laboratory participates in international comparisons, particularly with the Musée du Louvre (CNRS), France and IUX, Spain. Instrumental neutron activation analysis is a well-established method within this group since the seventies. Based on the use of the nuclear research reactor (unique in the Iberian Peninsula), this analytical method (complemented with other methods) supports most of the research activities of the CHS group and is also used by other ITN researchers. Public and private institutions often requested CHS group for services, considering the high performance of the chemical analysis. Most of the activities concern archaeometry and environmental monitoring studies. The INAA is fundamental for the research activities of the group, allowing determine trace elements, especially Rare Earth Elements, which are excellent geochemical fingerprints. 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.

Both laboratories (TL-OSL and INAA) were reinforced with new equipment on behalf of the national Re-equipment Program by FCT (CONC-REEQ/590/2001).