

## Cultural Heritage and Sciences

The “Cultural Heritage and Sciences” research group is mainly devoted to the valorisation and conservation of the Portuguese cultural patrimony, through scientific approaches. The application of nuclear and related methods of analyses consists on a basis of achievement the objectives. Several scientific domains are involved in this type of research, such as chemistry, physics, geochemistry, crystal chemistry, mineralogy, archaeology and geography. This research group, created in 1998, after a reorganisation of the scientific activities in ITN, has a multidisciplinary character, which is traduced naturally by the diversity of the know-how of the team – chemists, physics, geologists and geographers.

In order to increment the archaeology – archaeometry interaction with important long-term objectives, a protocol between ITN and the "Instituto Português de Arqueologia" (IPA) was signed in 1999.

The analytical methods implemented are unique in Portugal, namely the instrumental neutron activation analysis method and the radiocarbon dating. In addition, the installation of a luminescence dating unit started in 1999 (compromise assumed by ITN in the ITN-IPA protocol). The good performance of these laboratories is a major objective, with the maintenance of excellent quality in the results obtained to serve the national and international community.

Three main research domains, naturally related, cover the activities of the group: (1) archaeometry - characterisation of ancient materials and provenance, (2) conservation of the stone in the built patrimony, and (3) absolute dating.

The foremost objective of the research on archaeometry in ITN is the increase in Portugal of the study on ancient ceramics for several chronologies, in order to obtain a better knowledge of the production technologies through time. In addition the establishment of the principal productions *loci* in Portuguese territory is a major purpose. Several projects concerning archaeological ceramics are running and others are in preparation. Furthermore, these studies will provide a solid basis of comparison to similar studies of other archaeological sites in Portugal and in other European countries. ITN has already a large chemical data set (the larger in Portugal) and a main objective in short term is to organise a computer database to be available in the ITN web site.

The conservation of the stone in monuments must pass by a first step of identification of the causes and mechanisms of degradation of the stone. The identification of the quarries used to built and restore the buildings is essential, not only to evaluate the modifications on the alteration processes of the stones since their extraction from the outcrops and quarries, but also to have equivalent stone samples large enough to laboratory tests with conservation products. In this way, this research group is dedicated to the study of monuments built with igneous rocks (granites and volcanic rocks). The research will proceed increasing the study of the interaction between the stone and the building materials (mortars, cements, lime) and the percolating solutions.

The absolute dating through the radiocarbon method will be complemented in 2000 with the luminescence dating unit. ITN will become the only laboratory in Portugal able to date archaeological and geological materials in a large range of dates, serving the national and international archaeological and Quaternary geological communities. The study of the chronologies of materials derived from the “Parque Arqueológico do Vale do Côa” (PAVC) by employing the TL-OSL method, will be one of the first applications of this method after its installation and calibration.

The consolidation and development of new methods in order to contribute to the valorisation and conservation of the Portuguese cultural patrimony in particular, increasing the application of scientific methods to solve conservation and valorisation problems of our heritage, is a long-term goal. The contribution to the international community through the implementation of databases in the web site of ITN is a short-medium term strategy.

## Causes and mechanisms of degradation of igneous rocks in monuments from Minho, Alentejo and Azores\*

M.I. Prudêncio, M.A. Gouveia, J.C. Waerenborgh, M.J. Trindade<sup>1</sup>, M. Nasraoui<sup>2</sup>

### Objectives

The project aims to contribute for the detailed characterisation of the prevailing degradation state of the igneous rocks in monuments and of the responsible mechanisms, with the identification of the main decay agents. A better definition of preservation actions will be a major objective of the research results and interpretations.

### Results

Among the rocks studied a special attention was paid to trachytes behaviour in Azores monuments. This type of rock, commonly used in façades due to its colour and softness, has one of the worse behaviours in the buildings of these islands. The results obtained, through the chemical, physical properties and mineralogical studies including microscopic analyses (SEM and electron microprobe), showed that the intrinsic characteristics of the stones extracted from superficial levels (mainly the porosity) associated to the climate, lead to a undesirable rate of degradation in the buildings. Despite the possibility of the application of further conservation methods, one advice derived from these project results is the substitution in drastic situations of this type of stone for others such as the vacuolar andesite.

The major results of this project will be presented in five papers in the 9th International Congress on the Deterioration and Conservation of Stone, Venice, Italy in 2000, and were already been submitted for publication in the Proceedings of this Conference.

### References

1. Nasraoui, M, Trindade, M.J., Waerenborgh, J.C., Prudêncio, M.I., Figueiredo, M.O., Gouveia M.A. & Pereira da Silva, T. (1999). Geochemical variation of major and trace elements in trachyte from Portuguese monuments of Azores Islands. *Proceedings of the II Congresso Ibérico de Geoquímica / XI Semana de Geoquímica*, L. Aires de Barros, M.J. Matias e M.J. Bastos (eds.) , IST, p. 185-188.
2. Prudêncio, M.I. & Gouveia, M.A.. INAA applications on the study of degradation and conservation of stone in monuments. *Modern Trends in Activation Analysis, MTAA-10*, Washington, USA, 1999.

### Further work

This project ended in 1999. A detailed study of the interaction rock/percolation solutions should be subject of a future project.

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\* Funding: Contract PRAXIS 2/2.1/ CSH/254/95 (18 × 10<sup>6</sup> PTE), partners: LNEC, UM, IICT and CECRA.

<sup>1</sup> PRAXIS MSc student.

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## **Alteration processes and conservation methods of the built patrimony made of granite\***

M.I. Prudêncio, M.A. Gouveia, J.C. Waerenborgh, M. Nasraoui<sup>1</sup>, M. Gonçalves<sup>2</sup>

### **Objectives**

The main objectives are the following: (1) to study a type of monuments of great national importance – those built of granitic rocks; and (2) to contribute for the resolution of a large range of conservation problems in Alentejo region, being Évora cathedral our case study, on which very few investigation has been carried out.

The specific objectives of ITN team are: (1) to deepen the understanding of decay mechanisms of granitic rocks used in built monuments; (2) to manage the tools for the physic-chemical characterisation of materials with non-destructive methods and in special for in situ application and aiming at solving actual conservation problems; and (3) to contribute to a better understanding of the early alteration process that take place in quarry and to the identification of borrow areas.

### **Results**

Field and laboratory work was performed in order to identify the different types of stone of the “Sé” of Évora and the quarries used to built and restore the monument.

Taking into account the REE distribution pattern, we can broadly distinguish two kinds of cathedral stones: (1) Type 1 - include pegmatitic, aplitic granite, and the granite rich in biotite inclusions; the REE normalised patterns of these samples show a negative Eu anomaly. (2) Type 2 – do not present any significant Eu anomaly, show less fractionated REE pattern and are more rich in Ti, Mg, Fe and P.

Projected in this framework, the quarries samples have a consistent geochemical signature, well constrained by a number of conservative inter-element ratios as Ti, Mg and Fe which differ slightly from those of the church stones. Consequently, the quarries studied so far do not correspond to the church stones.

### **References**

1. Nasraoui, M., Clauer, N. & Prudêncio, M.I. (1999). Differential weathering of granitic quarry samples and monument stones. *Proceedings of the Ninth Annual Goldschmidt Conference*, Harvard Univ., Cambridge, Massachusetts, USA, p. 205-206

### **Further work**

A more extensive data set on quarries samples will be obtained as well as a more detailed chemical and mineralogical study of the monument stones.

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\* Funding: Contract PRAXIS 3/3.1/CEG/2516/95 (25 × 10<sup>6</sup> PTE), coordinator: LNEC.

<sup>1</sup> PRAXIS Post Doctoral.

<sup>2</sup> PRAXIS research grant.

## **Production and Technology of Roman Pottery: *Bracara Augusta* Case\***

M.I. Prudêncio, M.A. Gouveia, J.C. Waerenborgh

### **Objectives**

This proposal consists on a detailed study of roman pottery found in *Bracara Augusta* aiming to establish the town and trade patterns, the manufacturing techniques employed and production organisation. According to the different contexts of the roman town of *Bracara Augusta*, crossing a large chronological, a collection covering those variabilities was selected for compositional studies. In order to establish continuity or discontinuity in ceramics production, high medieval products found in Braga will also be studied.

This project has the following main goals:

1. To identify, to characterise and to classify the different groups of pottery present at *Bracara Augusta* (urban and neighbour area), including paste, pigments and surface treatment.
2. To contribute to the knowledge of the technology of ceramics manufacturing during the roman occupation.
3. To compare the roman ceramics with high medieval products found at Braga in order to establish continuity/discontinuity in ceramics production.
4. To characterise clay deposits (sedimentary and residual ones) attempting to find good mineralogical and geochemical (especially trace elements) fingerprints of the provenience.
5. To organise a collection of roman pottery reference samples, having in mind further comparative studies in the Braga region and in other areas.
6. To prepare a computer database of pottery materials in order to facilitate monographic studies.

### **Results**

A large set of chemical and mineralogical data was already obtained for the different groups of pottery, as well as of the raw materials.

The database has been implemented.

Results obtained in 1999 will be presented in the 32<sup>th</sup> International Symposium of Archaeometry to be held in May of 2000 in Mexico City, Mexico.

### **References**

1. Gomes, A. & Prudêncio. The Roman painting ceramic from Bracara Augusta: an archaeometric study. *5th European Meeting on Ancient Ceramics*, EMAC'99, Athens, 1999.

### **Further work**

The chemical and mineralogical analyses will proceed and the computer database complemented.

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\* Funding: Contract PRAXIS / HAR / 13047 ( $6 \times 10^6$  PTE).  
other participants from, UA-UM, CCA-UM, and Câm. Mun. de V. Nova de Famalicão.

# Raw Materials Provenance and Ceramics Production Technologies of the Pre-historic Settlements of the Fornos de Algodres Region\*

M.I. Dias<sup>1</sup>, M.I. Prudêncio, M.A. Gouveia

## Objectives

The main purpose of this work is the global study of pottery production on a recent pre-historical settlement network during the 3rd millennium BC in Fornos de Algodres region, emphasising continuities or innovations in the production technologies and on the raw materials resources of local populations. It's important to enhance that for that period in Portugal only a very few works exists. This archaeometric study will also provide a solid basis of comparison to similar studies that can be done of the same chronologies in Portugal and the rest of the Iberian Peninsula.

## Results

The archaeological pottery classification revealed the presence of several typological groups. There are morphologies characteristic of the local Chalcolithic, like globular and hemispheric vessels, some with traditional decoration patterns such as combed incisions. Together with this traditional forms appear new types of recipients, namely pots with flat bases and beakers that mark the transition to the early Bronze Age in the region. An archaeometric study of the different ceramics groups has been undertaken through a chemical and mineralogical characterisation, by means of neutron activation analysis (NAA) and X-ray diffraction (XRD), as well as petrographic analysis. Clays from the area were also studied, in an attempt to identify the raw materials used for pottery production. The surrounding geological context of the settlements is granites with quartz and doleritic veins. The more argillaceous material found correspond to the weathered dolerite veins.

Chemical differences in pottery allowed the definition of several pottery groups according to their composition. It's also possible to establish certain correlation between some of the pottery analysed and local clay samples, but in some specific cases different origins for raw materials are suggested. According to the mineralogy (XRD) it's also possible to establish that pottery has not been submitted to very high temperatures during fire, possibly lower than 500°C. Another result of important relevance, is that a special typological group, like the beakers, presents also a special chemical and mineralogical behaviour, with a careful production technology.

Results obtained in 1999 will be presented in 2 papers in the 32<sup>th</sup> International Symposium of Archaeometry to be held in May of 2000 in Mexico City, Mexico.

## References

1. Dias, M.I.; Prudêncio, M.I.; Prates, S.; Gouveia, M.A.; Valera, A.C. (1999) - Tecnologias de Produção e Proveniência de Matéria-Prima das Cerâmicas Campaniformes da Fraga da Pena (Fornos de Algodres-Portugal). Actas do 3º Cong. de Arqueologia Peninsular, UTAD, Vila Real (in press).
2. Dias, M.I.; Prudêncio, M.I.; Sequeira Braga, M.A.; Gouveia, M.A.; Alves, C.A.; Valera, A.C.. Provenance and Technology of Pre-Historic Pottery from Fornos de Algodres (Portugal). Proceedings of the 5th European Meeting on Ancient Ceramics, EMAC'99, Athens, 1999 (in press).
3. Dias, M.I. (1999). Annual Report, Instituto Tecnológico e Nuclear, 40 pp.

## Further work

The study will proceed, and the computer database complemented. A project proposal enlarging the objectives and methodologies is in preparation to be submitted to SAPIENS'99 (FCT).

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\* Funding: PRAXIS XXI/BPD/18888/98.

Collaboration of GAFAL archaeologist (Câm. Mun. F. Algodres), Univ. of Minho and Univ. of Seville.

<sup>1</sup> PRAXIS Post Doctoral.

# Archaeometry: characterisation of archaeological ceramics<sup>\*</sup>

M.I. Prudêncio, M.A. Gouveia, M.I. Dias<sup>1</sup>

## Objectives

This research area includes projects submitted under the ITN-IPA protocol.

The foremost objective is the increase in Portugal of the archaeometric research on ancient ceramics for several chronologies, in order to obtain a better knowledge of the production technologies through time. In addition the establishment of the principal productions *loci* in Portuguese territory is a major purpose. Furthermore, this study will provide a solid basis of comparison to similar studies of other archaeological sites in Portugal and in other European countries.

The data obtained will be organised in a computer database.

## Results

Chemical characterisation were performed by neutron activation analysis of selected sherds included in the following projects:

1 – Study of the ceramic pastes of the Ajuda roman kiln (Peniche). Leaders: G. Cardoso, J. Gonçalves and J. Rodrigues.

2 – Characterisation of archaeological ceramics – Lusitanas amphorae – of the Roman *Villa* of Quinta das Longas (Elvas). Leaders: A. Carvalho and M.J. Almeida.

3 – Roman ceramics workshop of the Tejo estuary: production centers – Porto dos Cacos, Quinta do Rouxinol and archaeological site of the Rua dos Correeiros. Leader: J. Raposo

4 – Characterisation of the archaeological ceramics of Sellium (Tomar). Leader: S. da Ponte

5 – Zambujalinho – Ceramics production center of the Sado Valley. Leader: I. Fernandes

6 – The Santarém "Alcáçova" from Iron Age to roman times. Leader: A. Arruda and C. Viegas.

7 – Ceramics study of the pre-historic sites (Neolithic – Late Bronze Age) in the Côa Valley Archaeological Park (CVAP). Leader: A. F. Carvalho.

Corresponding to the following archaeological sites:

PENICHE: Roman kiln of Ajuda

ELVAS: Quinta das Longas

VALE DO TEJO: Porto dos Cacos;

Quinta do Rouxinol;

Correeiros st (Lisbon);

TOMAR: Forum of Sellium

VALE DO SADO: Herdade do Pinheiro;

Zambujalinho

SANTARÉM: Alcáçova de Santarém

CVAP: Quebradas; Quinta da Torrinha; Tourão da Torrinha; Tourão da Ramila; Castelo de Algodres;

Barrocal Tenreiro; Fumo; and Castelão

## Further work

Under the ITN-IPA protocol, three new projects of chemical characterisation of archaeological ceramics ("Escavações no Centro Histórico de Tavira", "Evolução das Paisagens Culturais na Plataforma do Mondego na Pré-História Recente" e "PALCAS: A Alcáçova de Santarém durante a Idade do Ferro") will run during 2000.

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<sup>\*</sup> Funding: IPA (1.88 × 10<sup>6</sup> PTE).

<sup>1</sup> PRAXIS Post Doctoral.

## **Roman marbles in Portugal: artistic, petrographic, chemical and isotopic characterisation<sup>\*</sup>**

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### **Objectives**

This project aims the chemical and isotopic characterisation of marbles used in the production of Roman works of art that are conserved in Portuguese museums, in order to determine their provenance.

### **Results**

Firstly, marbles from different quarries located in the Alto Alentejo region have been studied by neutron activation analysis and mass spectrometry. The results obtained so far showed that it is possible to distinguish those marbles from other marbles used in Classic Antiquity namely the Carrara marble.

Secondly, marbles used in the production of Roman works of art whose provenance was well established have been characterised by the same methods. The results obtained confirmed what was expected.

### **Further work**

Isotopic analysis of marbles used to make two dozens of Roman works of art from Portuguese museums will be carried out in the very near future. Based on the results obtained, their provenance will be determined.

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<sup>\*</sup> Funding: PRAXIS XXI 2/2.1/CSH/819/95 (10<sup>6</sup> PTE).

other participants: Univ Évora, Univ. Nova de Lisboa, Univ. de Coimbra.

<sup>1</sup> PRAXIS graduate student.

<sup>2</sup> Univ. Nova de Lisboa.

<sup>3</sup> Univ. de Coimbra.

<sup>4</sup> Univ. de Évora.

## **Paleobiology and Paleodiets from the Mesolithic to the Final Neolithic: the Portuguese case. Trace elements and stable isotopes contribution \***

E. Cunha<sup>1</sup>, C. Umbelino<sup>1</sup>, J.M.P. Cabral, M.C. Freitas

### **Objectives**

The main purpose of this project is to characterise the dietary patterns and the human biology of Portuguese populations from the Mesolithic to the Final Neolithic. Portugal owes some of the largest skeletal collections from the Mesolithic period as well as from the Neolithic, providing an excellent opportunity to evaluate the effects of the introduction of agriculture on the human biology through a comparative analysis of these Portuguese skeletal series.

Diet inferences are made through trace elements and stable isotopes analysis in human bones and complemented with some macroscopic analysis of the human skeletal remains, such as oral pathologies. The basic assumption for the use of trace elements analysis in the inference of ancient diets, relies on the fact some of the elements found in human bones reflect the types of food ingested during life, since the different classes of food present distinct elemental levels. Some trace elements, namely strontium and zinc, can be interpreted and used for the determination of the relative amount of meat versus vegetable foods. Other elements such as Ba, Ca, Cu, Fe, Mn and Mg are also considered to be informative for dietary inferences.

### **Results**

The elemental content of human bone is being determined by using the INAA technique. A total of 180 human bone samples from six Neolithic sites and from the Sado and Muge shell middens (Mesolithic) have been irradiated in the RPI reactor and most of the irradiated samples measured in a  $\gamma$ -spectrometer.

Stable isotopes analysis is being carried out in a Canadian laboratory.

The last results for trace elements and stable isotopes are awaited for the beginning of March 2000. So, a presentation and discussion of the results obtained so far would be premature.

### **Further work**

The discussion of the total data will be done as soon as the experimental work is completed.

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\* Funding: PRAXIS PCNA/C/BIA/114/96.

<sup>1</sup> Instituto do Ambiente e Vida, Univ. de Coimbra.

## **Analysis of ancient coins and medallions**

J.M.P. Cabral and L.C. Alves

### **Objectives**

This project aims the non-destructive determination of the chemical composition of the alloys used for the production of ancient coins and medallions, in order to study their metrology.

### **Results**

Twenty nine Visigothic coins, from Sisebut (612-621), Reccared II (621), Suinthila (621-631) and Sisenand (631-636), have been analysed by PIXE.

Eleven medallions from the Aboukir hoard, pertaining to the Gulbenkian Collection, have also been analysed by the same technique.

### **Further work**

The results are being discussed and they will be published in the near future.

## **Analytical methods development and application:**

### **1. Instrumental neutron activation analysis**

### **2. Radiocarbon dating**

### **3. Luminescence dating**

M.A. Gouveia, M.I. Prudêncio, A. Monge Soares<sup>1</sup>, A. Zink<sup>2</sup>

#### **Objectives**

The analytical capabilities of this research group are rather unique: (1) the instrumental neutron activation analysis (developed by members of this group in Portugal since the seventies) is based on the use of the nuclear research reactor (unique in the Iberian Peninsula) and (2) the environmental isotopes laboratory (radiocarbon dating) is also unique in Portugal. In addition, in 1999 a decision was made to install a TL-OSL dating unit in ITN. This decision was made under a protocol established between ITN and the Instituto Português de Arqueologia (IPA). This method will serve not only the archaeological community but also to the Quaternary geological community.

The good performance of these laboratories is a major objective with the maintenance of excellent quality in the results obtained.

#### **Results**

The quality of the analytical procedures is periodically controlled through the analyses of international standards and inter-calibrations with other laboratories.

These analytical methods (complemented with other methods) support all the research activities of the HCS group and are also used by other ITN researchers. Besides the absolute dating commonly serves the national and international communities.

Services have been done to industry, namely in the chemical characterisation of environmental samples in the vicinity of an urban solid residues power plant (CTRSU, S. João da Talha) for VALORSUL and fuel samples for PETROGAL.

#### **References**

1. Kin, F. D., Prudêncio, M.I., Gouveia, M.A., Magnusson, E. Determination of rare earth elements in geological samples: a comparative study of instrumental neutron activation analysis and inductively coupled plasma mass spectrometry. *Geostandards Newsletters*. **23** (1) (1999) 47-58.
2. Gouveia, M.A., Prudêncio, M.I., New data on sixteen reference materials obtained by INAA, *Journal of Radioanalytical and Nuclear Chemistry, Articles* (in press).
3. Correia, J.D.G., Fernandes, C., Marques, F., Martinho, E., Domingos, A., Gouveia, A., Patrício, L., Santos, I. Novel <sup>186</sup>Re complexes prepared using the irradiation conditions available at the RPI: synthesis, characterisation and evaluation of their stability. *International Symposium on Research Reactor Utilisation, Safety and Management*, IAEA-SM-360, Sesimbra, Portugal, 1999.

#### **Further work**

The installation of the TL-OSL laboratory is a main objective for 2000. The adequate installation of this unit will be achieved with the collaboration of two experts with complementary know-how, namely Dr. Antoine Zink (PRAXIS/BPD/22024/99) and Dr. Daniel Richter.

The analytical work involved in the contract "Monitoring program of trace elements in the vicinity of CTRSU, S. João da Talha" (VALORSUL), as well as the chemical analysis of fuel samples for PETROGAL.

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