

Environment and Analytical Chemistry

Maria de Fátima Araújo

Activities within the Environment and Analytical Chemistry Group mainly involve the study of the Biogeochemical Cycles of Chemical Elements and Light Isotopes in the Environment. Our expertise is in the fields of **Instrumental Analytical Chemistry, Environmental Geochemistry, Isotope Hydrology, Oceanography, ^{14}C Dating** and **Archaeometallurgy**.

Instrumental Analytical Chemistry is based on Energy Dispersive X-Ray Fluorescence Spectrometry, Mass-Spectrometry for Light Isotopes, Elemental Analysis coupled to Mass Spectrometry, ^{14}C and ^3H Dating techniques. These techniques are being used in studies involving quantitative elemental analysis, isotopic determinations and absolute dating of environmental and archaeological samples.

During the current year the clean laboratory for the ICP-MS (Inductively Coupled Plasma Mass Spectrometer) for isotopic and trace element research, financed by the “*Programa Nacional de Re-Equipamento Científico*” was installed. Also, a laboratory dedicated to the sample preparation for AMS (Accelerator Mass Spectrometry) measurements is underway.

Environmental Geochemistry and **Oceanographic** research was developed under a multidisciplinary approach, including sedimentology, geochronology, absolute dating, meteorology and paleoecology. Research was focused in Sedimentary Geochemistry: to evaluate environmental changes (natural and man-induced) occurred during the Holocene on the Portuguese coastal area. Moreover, studies concerning the marine reservoir effect off the W margin of Iberian Peninsula, based on **Radiocarbon Dating** of pairs of samples (terrestrial and marine) allowed the study of the variability of the coastal upwelling off Atlantic Iberia during the Holocene and its correlation with palaeoclimatic change.

Isotope Hydrology research studies were implemented taking into account the sustainable regional development and the appropriate use of the water resources and coastal management based on several European and national directives. Investigations in different environments namely: Urban Areas; High Mountain Areas; Arid and Semi Arid Zones and Gas Geochemistry in CO_2 -rich Thermomineral Waters, were reinforced. Besides, a new research area was initiated on the contribution of environmental isotopes hydrology to the geothermal potential evaluation and the seismo-volcanic hazard assessment at Azores archipelago. Their characterisation is being addressed to the exploitation and future development of regional water resources and the delimitation of protection areas.

Further works in **Archaeometallurgy** were implemented on the characterization of the technological and social conditions of Copper and Bronze Age metal production and circulation in Portugal. Cu and Au-based artefacts collections from the N and SW archaeological sites have been studied. Corrosion processes in archaeological Cu-based materials and surface enrichment processes were also investigated using optical metallography, micro-fluorescence and scanning electron microprobe. A study of the largest Portuguese collection of Cu-based Chinese coins was initiated in order to infer about its authenticity and the metallurgical development that they represent in its relation with the economic History of China. The EAC group is highly engaged in education and training of students from different Universities preparing their MSc and PhD thesis under our supervision.

Due to the specificity of the available equipment and expertise within the group, technical services are available to Universities and to Public and Private Institutions.

Research Team

Researchers

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Environmental Geochemistry – Elemental and Isotopic Research

*M.F. Araújo, P. Carreira, A.M. Monge Soares, P.G. Fernandes, C. Correadeira,
P. Alvarez-Iglésias, D. Nunes, P. Valério, R. Cardoso, J. Martins, A. Amaro, M. Correia*

Objectives

The development and promotion of coordinate research on Earth and Environmental Sciences by implementing analytical and absolute dating techniques:

1. Multielemental characterization ($Z > 10$) using X-Ray Fluorescence spectrometry;
2. Light isotope determination ($\delta^2\text{H}$, $\delta^{13}\text{C}$, $\delta^{15}\text{N}$ and $\delta^{18}\text{O}$);
3. Radiocarbon dating;
4. Tritium dating.

Elemental, isotopic and dating techniques are being used in the understanding of natural environments, particularly aquifers, rivers, estuaries, lagoons, coastal waters and continental shelf. Our main goals are:

- 1) to establish the evolutionary patterns of coastal environments since the Holocene; identify sediment sources and evaluate the major driving factors controlling its distribution and composition; setting up regional backgrounds and the beginning and origin of anthropogenic inputs;
- 2) to determine the origin of different groundwater systems, definition of recharge areas and in the identification of mixture between different water bodies; assess the vulnerability of groundwater to surface pollution; understand and determine the origin of pollution in hydric systems;
- 3) to assess the variability of the W Iberian coastal upwelling; identify episodes of abrupt shifts in oceanic circulation, probably coupled with abrupt climatic changes.

Results

Geochemical and dating studies carried out in shelf, estuarine and lagoon sediments revealed to be essential tools in the study of transitional and marine environments, providing indication about the marine/terrestrial origin of the deposited materials and allowing to recognise and evaluate changes occurred during the Holocene. During this year, studies were mainly focused on: the SW Iberian shelf sediments (Gulf of Cadiz); the Minho estuary; the SW coastal lagoons and salt marshes of the Lima, Tagus and Mira estuaries. Geochemical and geochronological studies demonstrate that SW Iberian shelf sediments exhibit signatures of a continental origin. Main sources of pollution were identified as the mineral wastes resulting from the ore exploitation along the Iberian Pyrite Belt. A clear record of the beginning of "pollution" was detected in shelf sediments coincident with the mining exploitation reactivation during the XIX century. Palaeoenvironmental analysis on the Holocene evolution in Portuguese coastal zone

(Minho estuary and SW lagoons) reveal the existence of several sedimentary units identified as sequences of fluvial, marine and estuarine facies. These correspond mostly to the Holocene record influenced by climatic changes. Geochemistry of salt marsh sediments of different estuaries has permitted the establishment of regional background levels and to derive the enrichment caused by man induced factors.

Environmental isotope techniques applied to Caldas de Manteigas Spa thermal waters show that the upper valley of Zêzere river, should be faced as a potential recharge area of this system, where the main recharge seems to occur laterally. Besides, the $\delta^{13}\text{C}$ of the CO_2 gas measured in the thermomineral waters (NE of Portugal) indicate a mantellic origin, supported by the $^3\text{He}/^4\text{He}$ ratio measured in the gas phase. Also, isotope methods have allowed the identification of pollution sources: natural, industrial, agricultural, and domestic respectively inside Santa Margarida Military Training Campus, at Porto urban area and within Aveiro and Beja regions. Agricultural practices and high industrial areas with over exploitation of the systems were identified as sources of pollution to the environment: sediment and hydrological domains.



Radiocarbon dating has been used as a tool to set up chronological frameworks for the past environmental changes identified in our research. Besides this, the regional marine reservoir effect values, ΔR , for the 5th, 4th and 3rd Millennium BC were determined. Results enable a better clarification of the variability of the coastal upwelling off Atlantic Iberia, namely off western Portuguese coast. Also, a ΔR value was determined for an estuarine reservoir (of the river Tagus, near Muge, c. 7000BP), which allows a reliable and precise calibration of conventional radiocarbon dates from human skeletal material from the famous Mesolithic shell middens of Muge. Radiocarbon dating was also applied to aeolianite samples in order to set up a geochronologic framework to aeolian carbonate-rich sand deposits from the Lisbon region.

Geochemical record of environmental changes in the coastal zone (Portugal) since the Holocene*M.F. Araújo, C. Corredeira, P. Alvarez-Iglésias, S. Moreira¹, M.C. Freitas¹, C. Andrade¹, T. Drago²*

The overall objectives of this work are the recognition of the environmental changes (natural and man-induced) which have occurred during the Holocene and the prediction of those that are likely to occur in the future in selected areas of the Portuguese coast, by using a comprehensive study of the sedimentary record. Studies were focused in NW (Minho estuary), in the SW (wet dune slacks) coastal region and in the SW Iberian Continental Shelf. Sediment records of Minho estuary (~20 m long) were studied to establish stratigraphic succession of Holocene sediments. Geochemistry, in particular the elemental distribution in terrigenous (Si, Al, Fe and Ca) and palaeosalinity indicators (Cl and Br) has contributed to recognize the evolution of the northern area of Portugal since 18,000 years ago by identifying fluvial, marine and estuarine sedimentary facies. Sedimentological, geochemical and dating proxies of SW wet dune slacks (Poços do Barbaroxa and Lagoa da Sancha) allowed to establish major steps of coastal evolution, environmental changes and relations with sea-level throughout the Holocene. Results of textural analysis, organic matter and geochemistry (lithogenic and palaeosalinity indicators) have been used in the definition of 4 lithostratigraphic units and 3 sedimentary environments – marine, lagoonal and fluvial, in the last 10,000 years. At the SW Iberian continental shelf downcore profiles on elemental concentrations and sedimentation rates determined by the excess of ²¹⁰Pb pointed out to a register of the beginning of heavy metal pollution in shelf sediments (~200-250 years ago) coincident with the mining exploitation reactivation occurred in the region during the XIX century.

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Geochemical and sedimentological characterisation of salt marsh sediments from Portuguese estuaries (Lima, Tagus and Mira)*R. Cardoso, M.F. Araújo, M.C. Freitas¹, F. Fatela¹*

Salt marshes are transitional zones located along the intertidal estuarine region, characterized by low hydrodynamic conditions and an alluvial substrate which is colonised by halophyte vegetation acting as natural barriers which diminish the impact of floods and prevents coastal erosion. In these environments, the prevailing conditions favour the accumulation of metals and other contaminants in sediments. Salt marsh sediments collected along salt in 7 different settings areas of three Portuguese estuaries (Lima, Tejo and Mira) were characterized by using geochemical and sedimentological approaches, including elemental composition, organic matter, grain size, carbonates and pH determinations. Results demonstrated that samples from high marsh sectors are usually metal enriched, due to the bioaccumulation effect of vegetation. Heavy metals show an enrichment trend in fine fraction, and might also be related to organic matter. Seasonally there are differences in metal concentrations in sediments, as a result of the interaction between climate conditions, salt marsh productivity and metal accumulation by plants. Although some of the enrichment factors calculated have a natural origin such as the presence of specific geologic formations, others are the result of human impact on these environments. There are variations in sediment chemical compositions according to their sampling location in each estuary and significant differences are also found between the three studied estuaries.

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The marine reservoir effect off the western margin of the Iberian Peninsula*A.M. Monge Soares, J.M. Matos Martins, J.A. Alveirinho Dias¹*

Following previous research (see 2005 and 2006 ITN Reports), the sampling program increased during 2007 in order to get reliable ΔR values for the time interval 5000 BP – 3000 BP. It must be noted that this time interval corresponds to Late Neolithic, Chalcolithic and Bronze Age, cultural prehistoric periods that are object of an intense archaeological research by Portuguese archaeologists and, consequently, reliable and precise chronologies are needed. Also, during this time interval, i.e. after the Holocene Optimum, some climatic change is known that certainly will be reflected in the behaviour of the west Iberian coastal upwelling or in the marine reservoir effect values. Besides this research or in complement of it, a ΔR value was determined, for the first time, for the Mesolithic estuarine environment related to the famous and important shell middens of Muge. This ΔR value is different from the correspondent value determined for the open sea environment off the Portuguese western coast and allows the precise calibration of conventional radiocarbon dates from human skeletons discovered in these shell middens, since the $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ of bone collagen of the skeletons show a mixture of marine and terrestrial diets of the Mesolithic individuals.

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Using radiocarbon dating to test contemporaneity of aeolianite formation

A.M. Monge Soares, J.M. Matos Martins, A. Ramos Pereira¹

Several consolidated sand-dunes (aeolianites) of Late Pleistocene age are known along the south-western Portuguese coast, namely in the coastal region of Lisbon Peninsula. Praia Azul, S. Julião, Samarra, Magoito, Aguda, Praia das Maças and Oitavos are some of these aeolian sand deposits. For two of them, Magoito and Oitavos, reliable dates for their formation were determined. Magoito was formed during the Pre-Boreal, whilst the Oitavos aeolianite was generated during the last interstadial of OIS 3 (ca. 32000 BP). If it is postulated that the carbonate-rich sand that is deflated from the dune field to form the new aeolianite had an apparent radiocarbon age that is characteristic of that dune field at the time when the deflation occurred, then the radiocarbon dates obtained from samples collected at the aeolianite will indicate its relative chronology in relation with the aeolianites from the same region which ages are known. Samples from Magoito and Oitavos aeolianites, and also from S. Julião and Aguda aeolianites were collected in order to prove the postulate referred to above is correct and to determine their relative chronologies. Aguda and Magoito have the same apparent age (ca. 20000 BP), suggesting that Aguda is also of Holocene age. All the samples collected at different places at S. Julião have the same apparent age (ca. 40000 BP). Consequently, this aeolianite is probably of the same age of that of Oitavos.

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Hydrology in urban areas

P.M. Carreira, D. Nunes, H. Chaminé^{1,2}, M.J. Afonso^{1,2}, J.M. Marques³, M.M. Abreu⁴, P. Figueiredo⁵

In order to assess surface water / groundwater interaction in Porto urban area, several sampling campaigns were performed along the underground galleries excavated to conduct spring waters. Isotopic signatures from Paranhos spring collection chambers indicate that: i) samples collected along gutters present a homogeneous composition, ii) an isotopic depletion of about 1‰ in ¹⁸O and 7.5‰ in ²H was found, ascribed to groundwater samples collected from the granitic fractures. Coupled isotopic and geochemical signatures indicates that i) groundwaters collected along the gutters are meteoric waters infiltrated along the residual granitic soil; ii) groundwater samples from the granitic fractures could be ascribed to random precipitation events, resulting into a directly infiltration along the fissured granitic rocks. Besides this, geochemical and isotope techniques have greatly contributed to the assessment of the possible damage caused by the military training activities at Santa Margarida Military Camp, where environmental problems have been identified: in the Ervideira area, surface waters (from the stream) and shallow groundwaters (from the spring) present evidences of anthropic contamination. The results so far obtained indicate that the signatures of anthropic contamination found in the Military Camp are mainly the result of diffuse pollution ascribed to: some particular urban waste disposal, pasture ground, a small duck's lagoon, poultry farming and cattle breeding.



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Arid /semi arid zones hydrology

P.M. Carreira, P.A. Galego Fernandes, D. Nunes, P. Valério, F. Monteiro Santos¹, A. Pina², A. Mota Gomes², J.M. Marques³, M. Bahir⁴

An evaluation of the groundwater systems at Santiago island (Cabo Verde) was carried out through the application of environmental isotopes and geochemical data in order identify: origin and mechanisms of groundwater recharge; relation between the hydrochemical evolution of the groundwater systems with the geological matrix (minerals dissolution) or mixture with seawater and aerosol marine influence; identification of seawater intrusion mechanisms and, determination of the apparent groundwater "age". The isotopic composition of the groundwater samples collect at Santiago Island range from -4.72 to -2.61‰ in $\delta^{18}\text{O}$ and from -44.6 to -15.8‰ in $\delta^2\text{H}$, with a mean isotope content of $3.80 \pm 0.53\text{‰}$ in $\delta^{18}\text{O}$ and $-26.6 \pm 5.8\text{‰}$ in $\delta^2\text{H}$ vs. V-SMOW. Two groups of groundwater samples are identified based on the $\delta^{18}\text{O}$ and $\delta^2\text{H}$. Besides, at Esssaouira Basin (Morocco) a multidisciplinary approach has been applied to evaluate the hydrogeological potential of arid zones and environmental / climatic change. Considering the importance of the Esssaouira aquifer in the groundwater supply of the region, the study was conducted in order to comprehend this aquifer groundwater evolution.

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Groundwater resources assessment by anthropogenic and natural contamination sources

P.M. Carreira, P.A. Fernandes, D. Nunes, L. Ribeiro¹, E. Peralta², M.A. Marques da Silva³, M.T.C. Melo³

According to field practice the major cause of pollution in the rural area of Beja comes from fertilizers. The application of stable nitrogen isotopes ($^{15}\text{N}/^{14}\text{N}$ ratios) offers a direct way to identify the pollutant sources in groundwater systems. In the studied area two major sources of nitrate were identified, fertilizer and manure which present different isotopic $\delta^{15}\text{N}$ signatures. The relative contribution of these two sources to groundwater or surface water was estimated by mass balance. The analysis of nitrate $\delta^{18}\text{O}$ together with $\delta^{15}\text{N}$ improves the ability to trace nitrate sources and cycling. The isotope results are not conclusive about the possibility that major source of nitrate-N in groundwater comes from agriculture as expected. Also some studies performed at Aveiro region were carried out in order to use some isotope techniques to evaluate the aquifer vulnerability to nitrate contamination. Groundwater samples were collected from wells and springs for isotopic analysis ($\delta^{18}\text{O}$ and $\delta^{15}\text{N}$ from NO_3 and $\delta^2\text{H}$ and $\delta^{18}\text{O}$ from H_2O). Nitrogen isotopes were used to identify nitrogen sources and assess agriculture, cattle-breeding, urban and industrial contribution to nitrogen cycle in Aveiro ecosystem, based on the fact that the main sources of nitrate in the area have isotopic distinct $\delta^{15}\text{N}$ and $\delta^{18}\text{O}_{\text{NO}_3}$ signatures.

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High mountain areas in catchment water resources

P.M. Carreira, D. Nunes, J.M. Marques¹, J. Espinha Marques², H. Chaminé³

Sampling campaigns were performed at Serra da Estrela Natural Park to understand the i) recharge and discharge processes and ii) role of snowmelt as a source of groundwater resources. Preliminary geophysical studies (a 600m dipole-dipole profile with a dipole distance of 10m) were carried out in order to investigate one of the main potential recharge areas. The seasonal variation of the precipitation stable isotopic composition is reflected in the comparatively low δ -values in the snowfall, which will be reflected in the $\delta^{18}\text{O}$ and $\delta^2\text{H}$ contents of meltwater. This shift provides a very useful tracer signal for hydrogeological studies in mountainous areas. The recharge of the thermal waters was studied based on the spatial and temporal variability of the isotopic composition of water ($\delta^2\text{H}$, $\delta^{18}\text{O}$ and ^3H). According to the isotopic gradient in the study area ($-0.10\text{‰}/100\text{m}$ of altitude) and considering the mean isotopic composition of the thermal waters ($\delta^{18}\text{O}_{\text{mean}} = -7.8\text{‰}$ vs. V-SMOW), the upper valley of Zêzere river, should be faced as a potential catchment area of this thermal water system, where the main recharge seems to occur laterally. The Bragança-Vilariça-Manteigas fault zone should play an important role in conducting the laterally infiltrated meteoric waters towards the discharge area at the Caldas de Manteigas Spa.

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Gas geochemistry in CO₂-rich thermomineral waters a contribution to the geothermal potential evaluation and the seismo-volcanic hazard assessment

P.M. Carreira, D. Nunes, J.M. Marques¹, M.R. Carvalho², G. Capasso³, F. Grassa³, J.C. Nunes⁴, P. Madureira⁵



In Portuguese mainland the most important $\text{HCO}_3\text{-Na-CO}_2$ -rich thermomineral waters issue in the Northern part of the country, within the Galicia-Trás-os-Montes region, linked to one of major NNE-trending faults, the so-called Penacova-Régua-Verin megalineament. From the interpretation of chemical and isotopic data, it is evident that the CO_2 -rich mineral waters are part of an open system to the influx of CO_2 gas from a source other than redox reactions. Isotopic data suggests that mantle-derived CO_2 is actively reacting with the groundwater. Stable carbon isotopic data indicate that the external CO_2 is of inorganic origin. The values obtained for the $\delta^{13}\text{C}$ of the CO_2 gas indicate a mantellic origin, supported by the $^3\text{He}/^4\text{He}$ ratio measured in the gas

phase. Also, research at Azores archipelago studies are starting in order to: i) characterize the chemical and isotopic composition of ground waters, fumaroles and gas emissions related with hydrothermal/geothermal systems and volcanologic/seismic settings.

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Archaeometallurgy – Provenance, technology and use of metallic artefacts

M.F. Araújo, P. Valério, E. Figueiredo, A.M.M. Soares, R. Silva¹, J.C. Senna-Martinez², L.C. Alves³

This research combines analytical and archaeological investigations regarding metallurgical activities during the Copper, Bronze and Iron Ages. Metallic artefacts and metallurgical remains (e.g. ores, crucibles and slags) are being characterized by EDXRF, micro-XRF, micro-PIXE, optical metallography and SEM-EDS. The obtained results are integrated with the archaeological evidence in order to resolve specific archaeological issues. In the current year, the research regarding the Southwest Region included the study of gold buttons from Castro dos Ratinhos (Early Iron Age), which present a homogeneous alloy composition and exhibit silver and copper contents within the characteristic EIA values ($10\% < Ag < 20\%$ and $Cu > 1\%$). The welding process used is still under study – no solder was identified, which might indicate solid state diffusion welding, a process still not identified in such an early period of the Iberian Peninsula. Copper ores with tin impurities and a mould for flat axes with Cu, Sn and Pb enriched in the carving, all from Casarão da Mesquita 3, attest the practice of the bronze metallurgy during Late Bronze Age at this site. Regarding the Central Portuguese Region, metallurgical productions from Fraga dos Corvos and Canedotes (Late Bronze Age) were found to be made of bronze alloys ($8\% < Sn < 14\%$) with low lead contents ($< 2\%$). The microstructures of the studied artefacts exhibit sulphide inclusions and establish the use of thermo-mechanical operations that finished either with annealing or mechanical deformation processes.



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Characterization of an ancient Cu-based Chinese coin collection

M. João Furtado, M.F. Araújo, R. Silva¹

Almost four hundred Chinese copper cash (round coins with a square centred hole) part of the collection of the Macao Scientific and Cultural Centre’s Museum (Lisbon, Portugal) are being studied in order to characterize their alloys and main superficial corrosion products. Although the opening question was whether the coins were official or counterfeit, because of their unknown source, soon other issues arose. So far, the coins were analysed by Energy Dispersive X-Ray Fluorescence, a non-invasive analytical technique, which permitted to obtain some interesting results: the presence of very heterogeneous alloys - bronzes up to the 14th century and mostly brasses after that – almost all of them with high lead percentages, possibly because lead was available and cheaper than copper or tin/zinc. Iron and nickel, are present in considerable percentages, and this could be an indication that the alloys may be ancient. Also, it was found that in coins with substantial superficial corrosion products there was commonly an elemental variation between the obverse and reverse. The correlation between obtained analytical information and similar existing or on-going studies, both analytical and historical ones, will increase the knowledge of the Chinese monetary system. Furthermore, the study will also focus on the metallurgy development in China and as such, contribute to the understanding of Chinese technologic and economic History.

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Radiocarbon dating applied to archaeological contexts – building up the South-western Bronze Age Chronology

A.M. Monge Soares, J.M. Matos Martins

Since the beginning of the radiocarbon laboratory, twenty years ago, one of its important tasks has been to build up reliable and precise prehistoric chronologies in closed cooperation with archaeologists. Nevertheless, some cultural periods still fall short of absolute dates that allow a reliable chronology for their phases and sub phases. It is the case for the Bronze Age of the South-western Iberian Peninsula. Recent mitigation works, as a result of Environmental Impact Assessments related to the Alqueva Dam Project, have been carried out by archaeological companies that led to the discover of several Bronze Age settlements and burials. These archaeological contexts, at first sight, seem to be largely diachronic belonging to different cultural phases from Early to Late Bronze Age. Organic samples, i.e. charcoal and bones, have been recovered after archaeological excavations that will allow a good precision and reliability for the chronologies that are actually in process of building up. It must be stressed that this is possible because the excellent cooperation that exists between the radiocarbon laboratory and the archaeological companies and archaeologists that dug or are digging those prehistoric and protohistoric settlements.