Environmental and Analytical Chemistry

Maria de Fátima Araújo

The activities of the Environmental and Analytical Chemistry Group (EAC) combine fundamental and methodological research related to **Elemental and Isotopic Analysis** as well as their applications in the fields of **Environmental Geochemistry**, **Isotope Hydrology**, **Oceanography** and **Archaeometry**.

Research is developed by a multidisciplinary team constituted by Analytical and Inorganic Chemists, Geologists, Biologists and Conservation scientists. Different facilities have been implemented and maintained by the team: **Energy-Dispersive X-Ray Fluorescence**, **Light Isotope Mass Spectrometers**, **Radiocarbon Dating**, **Tritium Unit** and a recently installed **clean Laboratory** with a **High Performance Liquid Chromatograph/Inductively Coupled Plasma Mass Spectrometer**.

During the current year the project CISTER, financed by the "*Programa Nacional de Re-Equipamento Científico*", was executed by the installation and testing of the HPLC/ICPMS equipments, in the clean laboratory dedicated to isotopic and trace element research. Studies on the interferences of the polyatomic species and calibration procedures for trace analysis are being developed. Part of the stable isotopic ratio mass spectrometer to be associated with the laboratory dedicated to the sample preparation for AMS (Accelerator Mass Spectrometry) measurements was purchased.

Environmental Geochemistry and **Oceanographic** research was carried out under a multidisciplinary approach, including sedimentology, geochronology, absolute dating, meteorology and paleoecology. Research was focused in Sedimentary Geochemistry: to evaluate environmental changes occurred during the Holocene on the Portuguese coastal area, in particular at Minho and Sado estuaries and at the SW coastal area. Oceanographic studies concerning the marine reservoir effect, based on **Radiocarbon Dating** of pairs of samples (terrestrial and marine) were enlarged to new regions off the W margin of Iberian Peninsula – Aveiro / mouth of Minho river and Faro / Mouth of Guadiana river.

Isotope Hydrology research studies have been developed aiming at to contribute to a sustainable regional development and appropriate use of the water resources. Investigations were carried out in different environments: Urban Areas; High Mountain Areas; Arid and Semi Arid Zones and Gas Geochemistry in CO_2-rich Thermomineral Waters, including the geothermal potential evaluation and the seismo-volcanic hazard assessment at Azores archipelago. These investigations are essential to the exploitation and future development of regional water resources and to the delimitation of protection areas.

The **archaeometallurgical research** were focused on the materials characterization, involving different analytical techniques (EDXRF, micro-XRF, optical metallography, XRD and SEM-EDS) of artefacts and other remains from different archaeological sites of the Portuguese territory along the SW Iberian region and the North Central Portugal, in order to investigate the evolution of artefact production and metallurgical operations of Copper-based alloys during PreHistory.

The study of the Macao Scientific and Cultural Center Museum **Chinese copper cash** was focused on the microstructural characterization of imperial brass coins by optical metallography, micro X-ray fluorescence, scanning electron microprobe and micro X-ray diffraction.

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, P. Alvarez-Iglésias, M. G. Santos, D. Nunes, P. Valério, J. Martins

Objectives

Activities aim at the development and promotion of coordinate research on Earth and Environmental Sciences in conjunction with the implementation of the following analytical and absolute dating techniques: EDXRF multielemental analysis; Light isotope determination; Radiocarbon dating; Tritium dating and HPLC/ICPMS.

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; identify the recharge areas; calculate 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 estuarine and lagoon sediments provide information about the marine/ terrigeneous origin of the deposited materials and allow to identify changes occurred during the Holocene. During 2008, studies were mainly focused on: the Minho estuary; and on the salt marshes of the Sado, Mira and Guadiana estuaries. Study of Minho estuary sedimentary dated sequences reveal the influence of relative sea-level rise during the Holocene by the identification of marine inundation periods with alternating layers exhibiting a significant continental signature. Geochemistry of estuarine salt marsh sediments has permitted the establishment of natural baseline levels for many elements and the assessment of anthropogenic contamination. Baseline composition showed to be variable and highly influenced by the lithology of the catchment areas.

Environmental isotopes (δ^2H, δ^{18}O, $\delta^{34}S_{(SO4)}$, $\delta^{18}O_{(SO4)}$ and ^{3}H) have been be use in conjunction with conventional hydrogeological and geochemical approaches. The $\delta^{34}S_{(SO4)}$ and $\delta^{18}O_{(SO4)}$ values in soluble sulphates were used to conclude about its origin in groundwaters and the water contamination extent, in Caldas da Rainha and Oporto urban area. Nitrogen isotopes determination was used to identify pollutant sources in Beja groundwater systems: fertilizer and manure. The vulnerability of Aveiro aquifer to nitrate contamination was evaluated and data were used to identify nitrogen sources (agriculture, cattle-breeding, urban and industrial) contribution to nitrogen cycle.

Radiocarbon dating is an important tool to set up chronological frameworks for the past environmental changes. As usual, it is of much use in our research. A new step in the study of the marine reservoir effect was implemented during 2008 in order to get reliable ΔR values for two coastal zones in the Atlantic Iberia badly sampled previously – Aveiro / mouth of the river Minho and Faro / mouth of river Guadiana. Besides, ΔR values were determined, for the first time, for the coastal waters of Canaries Islands. Similarly to what was done for the Atlantic Iberia, the variability of the marine reservoir effect from the beginning of colonization of the Islands till nowadays will be investigated

Published work

D. Burdloff, M.F. Araújo, J.-M. Jouanneau, I. Mendes, A.M. Monge Soares, J.M.A. Dias. Organic carbon sources to the Northern and Southwestern Portuguese shelf sediments. *Applied Geochemistry*, 23 (2008) 2857–2870.

P.M. Carreira, J.M. Marques, R. Graça, L. Aires-Barros, Radiocarbon Application in Dating "Complex" Hot and Cold CO2 – Rich Mineral Water Systems: A Review of Case Studies Ascribed to the Northern Portugal. *Applied Geochemistry*, 23, 2817-2828.

C. Corredeira, M.F. Araújo, J.-M. Jouanneau, Copper, zinc and lead impact in SW Iberian shelf sediments: An assessment of recent historical changes in Guadiana river basin, *Geochemical Journal*, 42 (2008) 319 – 329.

Geochemical record of environmental changes in Minho Estuary (Portugal) since the Holocene

M.F. Araújo, P. Alvarez-Iglésias, T. Drago[1]

The overall objectives of this work are the recognition of the environmental changes which have occurred during the Holocene in the Minho estuary, by using a comprehensive study of the sedimentary record. Sedimentary record of transitional environments constitutes an archive of their temporal evolution, which can be assessed by multidisciplinary analytical approaches. The Minho River (about 300 km long) represents one of the main basins draining to the NW Iberian Peninsula. Its basin has around 17000 km2, and comprises from Paleozoic to Cenozoic rocks, being mainly constituted by granites, schists and greywackes.We have assessed the palaeoenvironmental evolution of the Minho Estuary for the last millennia by a detailed geochemical and sedimentological study of two long sediment cores (~30m) retrieved at the mouth of the Minho Estuary. Two percussion cores were collected at the Coura (Minho tributary) marsh. Samples were characterized by EDXRF and complemented with previously results obtained by INAA and sedimentological analyses. Data integration have pointed out that the evolution of the Minho Estuary for the last millennia showed a succession of four units: (1) fluvial: coarse grained, high Si, LREE enriched with a significant negative Eu anomaly; (2) marine: increased fine-grained particle content, high carbonate, S, Br, Cl, Ca, Sr and HREE and almost flat REE normalized profiles; (3) estuarine: alternating coarse and fine-grained layers, intermediate LREE/HREE ratios, significant Ca and Sr contents; and (4) sand barrier: constant sand content (high Si and Rb), intermediate LREE/HREE ratios.

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Geochemical and sedimentological characterisation of salt marsh sediments from Portuguese estuaries (Sado and Mira)

S. Moreira[1]*, M.F. Araújo, M.C. Freitas*[1]*, F. Fatela*[1]

Salt marshes are transitional zones located along the intertidal estuarine region, characterized by low hydrodynamic conditions that favour the accumulation of metals and other contaminants in sediments. Salt marsh sediments from two Portuguese estuaries located at the SW coastal area: Sado and Mira, have been studied. At Sado estuary, a total of 66 superficial samples collected seasonally (fall and spring) in the intertidal domain (tidal flat, low and high marsh) along four transects were characterized. At Mira estuary, studies were focused on short cores collected in the left margin in Vila Nova de Mil Fontes salt marsh. Research involved geochemical and sedimentological approaches, including elemental composition, organic matter, grain size, carbonates, pH determinations and sedimentation rates (for the core sediments). The superficial sediments of the Sado's salt marshes of Faralhão, Carrasqueira and Alcácer do Sal are in general muds with high levels of organic matter; despite in Tróia being essentially sandy sediments with diverse organic matter levels. Regional Cu, Zn and Pb background was assessed, which allowed to conclude that salt marsh areas are heavy metal enriched. A Pb increase is observed from the mud flat and low marsh to the high marsh in one transect. Cu and Zn enrichments are comparable, whereas the Zn enrichment for ALC-S transect (Alcácer do Sal) is higher. Cores from Mira were sub-sampled for sedimentological and geochemical analyses. Organic matter content was determined for all the subsamples, EDXRF analyses and the determination of sedimentation rates by ^{210}Pb and ^{137}Cs are in progress.

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The marine reservoir effect – its quantification and variability

A.M. Monge Soares, J.M. Matos Martins, J.M. Alveirinho Dias[1]

Following previous research (see 2007 ITN Report), a new step in the study of the marine reservoir effect was implemented during 2008 in order to get reliable ΔR values for two coastal zones badly sampled previously – Aveiro / mouth of the river Minho and Faro / mouth of river Guadiana. The first zone is in the transition between two regions of an intense upwelling (Aveiro / Cape of S. Vicente and Western Galician Coast), while the second one is in the transition between a region with a coastal upwelling of some strength (Cape of S. Vicente/ Cape of S. Maria) with a region with a non-existent upwelling (Eastern Gulf of Cadiz). In complement to the above mentioned research, ΔR values were determined, for the first time, for the coastal waters of Canaries Islands. Here the oceanographic conditions are different from those of the Atlantic Iberia and, similarly to what was done for this region, we will investigate the variability of the marine reservoir effect from the beginning of colonization of the Islands till nowadays. For this research we are having the collaboration of Spanish archaeologists, which have been providing pairs of samples from archaeological contexts located in different islands and with different chronologies.

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Hydrology in urban areas

P.M. Carreira, D. Nunes, P. Valério, M.F. Araújo M.J. Afonso[1,2]*, H. Chaminé*[1,2]*, J.M. Marques*[3]

Environmental isotopes, such as δ^2H, $\delta^{18}O$, $\delta^{34}S_{(SO4)}$, $\delta^{18}O_{(SO4)}$ and ^3H, are tools that can be used to complement the conventional hydrogeological and geochemical approach to evaluate the impact of urban recharge in groundwater.

Isotope techniques, mainly δ^2H and $\delta^{18}O$ measurements and environmental ^3H, have been employed in Oporto urban region in combination with major hydrogeochemical indicators. The isotopic depletion found in groundwater samples collected along gutters are ascribed to meteoric waters infiltrated along the granitic weathering zone. Besides, groundwaters resulting into a direct infiltration of meteoric waters along the fractured granitic rocks present higher ^3H concentrations. Besides the $\delta^{34}S_{(SO4)}$ and $\delta^{18}O_{(SO4)}$ values of dissolved sulphate will be used to conclude about the origin of sulphate in local groundwaters, in order to assess the extent of water contamination.

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Groundwater resources assessment by anthropogenic and natural contamination sources

P.M. Carreira, P.A. Fernandes, D. Nunes, L. Ribeiro[1]*, E. Peralta*[2]*, M.A. Marques da Silva*[3]*, M.T.C. Melo*[3]

Related to anthropogenic inputs that lead to the degradation of water resources, studies were focused on origin/sources of nitrate in water resources (surface and groundwater bodies) using nitrogen isotopes. Two areas, Aveiro and Beja have been investigated. The determination of nitrogen isotopes ($^{15}N/^{14}N$ ratios) offers a direct way to identify the pollutant sources in groundwater systems. In Beja two major sources of nitrate were identified – fertilizer and manure. The relative contribution of these two sources to groundwater or surface water was estimated by mass balance. At Aveiro region the isotope techniques were used to evaluate the aquifer vulnerability to nitrate contamination. Groundwater samples were collected from wells and springs for isotopic analysis. Nitrogen isotopes were used to identify nitrogen sources and assess agriculture, cattle-breeding, urban and industrial contribution to nitrogen cycle in Aveiro ecosystem.

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Arid /semi arid zones hydrology

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Overexploitation of coastal aquifers and pollution are among the main problems related to groundwater resources assessment and management in Santiago Island (Cabo Verde). Brackish groundwater for agriculture and human supply is being provided to numerous parts of the Island, as the only type of available water. Results indicate water-rock interaction mechanisms as the major process responsible for the groundwater quality (mainly calcium-bicarbonate type), reflecting the lithological composition. Anthropogenic contamination was identified, in several points of the island. Isotopic techniques combined with geochemistry provided comprehensive information on groundwater recharge, and identification of salinization mechanisms (e.g. seawater intrusion, salt dissolution, and marine aerosols) of the groundwater systems, at Santiago Island. At Essaouira Basin (Morocco) a multidisciplinary approach has been applied to evaluate the hydrogeological potential of arid zones and estimate groundwater supply of the region.

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High mountain areas in catchment water resources

P.M. Carreira, D. Nunes, J.M. Marques[1], J. Espinha Marques[2], H. Chaminé[3]

Serra da Estrela Mountain "normal" groundwaters belong to the Na-Cl and HCO_3-Na *facies*, with low TDS, considered as good signatures of local recharge and hydrolysis of Na-plagioclases, respectively. The high Na-Cl concentrations found in some of the waters of this group could be ascribed to the local use of NaCl to promote snowmelt in the roads during the winter season. Mineral waters from Caldas de Manteigas Spa are characterised by: high pH values (≈ 9), EC values around 300 μS/cm, the presence of reduced species of sulphur ($HS^- \approx 1.7$ mg/L), silica contents of about 50 mg/L and high F^- concentrations (up to 7 mg/L), indicate that the reservoir rock should be mainly the granite. The most important isotopic signature is ascribed to the fact that no influences of a lighter isotope component in the groundwater samples from winter campaigns was found and explain the mixing process between different groundwater bodies, with the tendency to the homogenisation of the isotopic composition.

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Gas geochemistry in CO_2-rich thermomineral waters a contribution to the geothermal potential evaluation and the seismic-volcanic hazard assessment

P.M. Carreira, D. Nunes, J.M. Marques[1], M.R. Carvalho[2], G. Capasso[3], F. Grassa[3], J.C. Nunes[4], P. Madureira[5]

The chemical and isotopic data of HCO_3-Na-CO_2-rich thermomineral waters (NE Portugal) revealed that the CO_2-rich thermomineral waters are part of an open system to the influx of CO_2 gas from a deep-seated reservoir. The chemical composition of both hot and cold mineral waters suggests that water-rock interaction reactions is mainly controlled by the amount of dissolved CO_2(g) rather than by the water temperature. The $^3He/^4He$ and $^4He/^{20}Ne$ ratios measured in the gas phase are consistent with a two-component mixing of prevailing radiogenic and a mantle origin. This hypothesis is supported by the stable carbon isotope data that corroborates an external source of CO_2 of inorganic origin. $\delta^{13}C_{CO2}$ values ranging between -7.2°/₀₀ and -5.1°/₀₀ are consistent with a two-component mixture between crustal and mantle-derived CO_2. In parallel two sampling campaigns were performed in Azores archipelago in order to: i) characterize the chemical and isotopic composition of groundwaters, fumaroles and gas emissions related with hydrothermal/geothermal systems and volcanologic/seismic settings.

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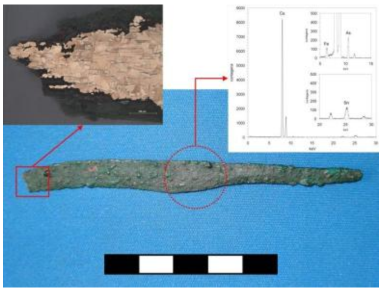
Radiocarbon dating applied to archaeological contexts – building up the South-Western Bronze Age Chronology

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

As we said in the 2007 Report, 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 and Early Iron 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, with prolongations to Early Iron Age contexts in some of them. 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. Since the beginning of this research a few tens of radiocarbon dates were already obtained, strongly changing the previous situation concerning the absolute chronology for the Proto-History of SW Iberia.

Archaeometallurgy – Provenance, technology and use of metallic artefacts

M.F. Araújo, P. Valério, E. Figueiredo, A.M.M. Soares, R.J.C. Silva[1], F.M. Braz Fernandes[1], J.C. Senna-Martinez[2]



The archaeometallurgical research combines material characterization, involving different analytical techniques (EDXRF, micro-EDXRF, optical metallography, XRD and SEM-EDS), with archaeological evidences, in order to investigate the evolution of artefact production and metallurgical operations of Copper-based alloys during PreHistory on the SW Iberian region and on North Central Portugal. Metallic artefacts from Late Bronze Age and Early Iron Age archaeological sites of the South-western Iberian Region, namely Castro dos Ratinhos, Santa Margarida, Salsa 3 and Casarão da Mesquita 3, were found to be made of binary copper-tin alloys (Sn~ 8-12%) with low lead content (Pb<1%). The presence of copper and tin residues aggregated to the inner surfaces of several Late Bronze Age crucibles from Entre Águas 5, Martes and Casarão da Mesquita 4, establish the practice of bronze metallurgy at those sites. Regarding the Central and Northern Portuguese regions a large set of artefacts, fragments, scraps, and other metallurgical remains (crucibles, slags and moulds) from Bronze to Iron Age recovered from different archaeological sites (Castro de Nossa Senhora da Guia de Baiões, Castro de Santa Luzia, Castro de São Romão, Cabeço do Couço and Dólmen de Fiais da Telha in Beira Alta region, and Fraga dos Corvos in Trás-os-Montes) have been analysed. Analyses show during the Late Bronze Age artefacts from the Beira Alta region had very low Pb contents (Pb<1%) wherever the bronze corresponding to an early Iron Age period from the Trás-os-Montes region had frequently ~2%Pb. The tin content for both periods and regions is regularly around 8-13%. Artefacts microstructures were variable, exhibiting characteristic evidences (e.g. annealing twins and deformation bands) of different thermo-mechanical finishing operations, involving for instance mechanical deformation and/or annealing.

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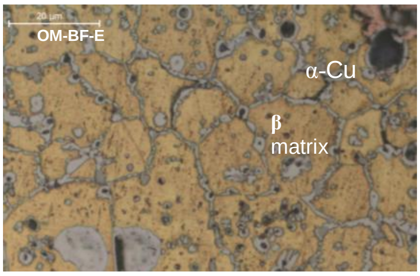
Composition and microstructures of imperial brass Chinese coins

M.J. Furtado, M. Fátima Araújo, Rui Silva[1]

On the sequence of the EDXRF analyses on the c. 400 coins last year, copper cash (round coins with a square centred hole) from the Macao Scientific and Cultural Centre’s Museum (Lisbon) the study of the collection has continued by the characterization of their (bulk) composition and microstructures through micro-EDXRF, SEM-EDS and micro-XRD. About 25 selected coins (brasses) were prepared for analyses, with minimal damage to the coins surface: the coins were mounted on resin and c. 1mm^2 of the rim was abraded to access the metallic (patina/corrosion free) surface. The EDXRF results of the cleaned area did not show significant variation from the previously obtained, except a slight increase in the zinc content. This is in agreement with the observed microstructures that have clearly indicated a superficial dezincification. Microstructures observed are typical of as-cast structures although very fine due to their size and thickness. Lead was found to form globules in the structure and EDS analysis reveals the presence of S, that wasn’t detected with EDXRF. The main phases are *alpha-beta* of Cu-Zn system (in brasses) and *alpha-delta* in Cu-Sn systems (bronzes). Other elements as Pb, Fe, Sb or As tend to group together in grain boundaries. An important find was that the presence of small amounts of Sn in brass alloys (up to 3%) inducing the appearance of a third phase, Sn or Sb rich. This phase is congruent with Cu-Zn-Sn ternary system (*gamma$_1$*) has been identified through micro-XRD.



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