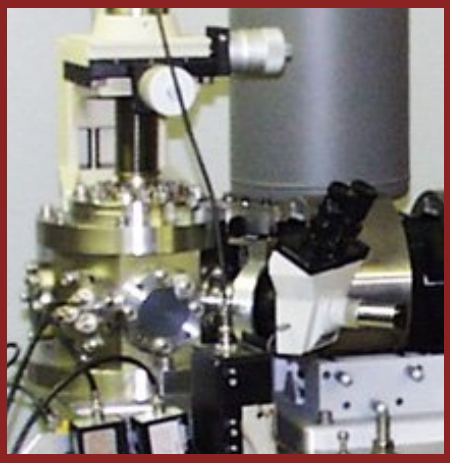
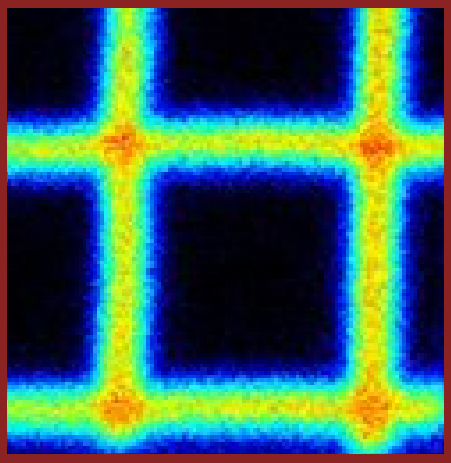
THE EXTERNAL ION BEAM FACILITY IN PORTUGAL FOR STUDYING CULTURAL HERITAGE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In 2008 an external ion microbeam analytical end-station became operational at the ion microprobe facility of the Laboratório de Feixe de Iões at Instituto Tecnológico e Nuclear, Portugal. Its availability adds a set of valuable analytical techniques for the community involved in the study and conservation of Cultural Heritage. With the external ion microbeam it is possible to analyze the elemental composition (in point, line or areal maps modes) and perform structural studies of different objects, large or small, using Ion Beam Analysis techniques in open air or helium atmosphere – i.e. without vacuum conditions – and without the need of sampling or any special preparation. In this article, the details concerning the external beam set-up and a selection of the results obtained from selected analyzed objects will be presented. These objects include glass fragments from a Roman villa and religious gilt objects from the XVI-XVIII centuries.

Introduction

Knowing the composition of an object is extremely important for the conservator-restorer's work. It can also indicate, for example, if the constituent elements are consistent with the ones used in the period to which the object is supposed to be. Furthermore, the trace element concentration can indicate in some cases the provenance or relate it with other objects of the same type. The possibility to analyze and identify corrosion products is also important, in order to better understand the mechanisms of degradation, which is essential for their preservation for the present and future generations.

There are several available analytical techniques that allow us to know the composition of materials, such as X-Ray Fluorescence (XRF) and X-Ray Diffraction (XRD) performed with portable equipments, UV-Visible and FTIR spectrometries and/or Scanning Electron Microscopy, etc. Some of them may provide information about the compounds that are present in the sample while others will determine their elements. Some of them are destructive, others are not. Generally speaking, we can say that for each object and depending on the information we are looking for, there is a set of analytical techniques that are more suitable than others. The choice is not always straightforward, but should be jointly determined by the different specialists involved.

One of the complicated choices refers to the sampling process. It is true that along the years the quantities of material needed for some techniques have been reduced substantially. In some cases, the quantities needed are in the range of micrograms and the resulting marks are not visible to the naked eye. But even under these conditions sometimes sampling is not possible. On the other hand, if we are interested in the material "core" of the object, there may be no other alternative and sampling is necessary.

There are also techniques where sampling is not needed although they may induce permanent changes to the objects' surface to be analyzed. For example, a surface preparation is needed or the technique may alter the surface composition such as those involving sputtering or laser ablation processes.

In this article, the characterization of different objects by means of Ion Beam Analytical (IBA) techniques is reported. These are a set of techniques used to study the composition and/or the quality of samples in a non-destructive way, using

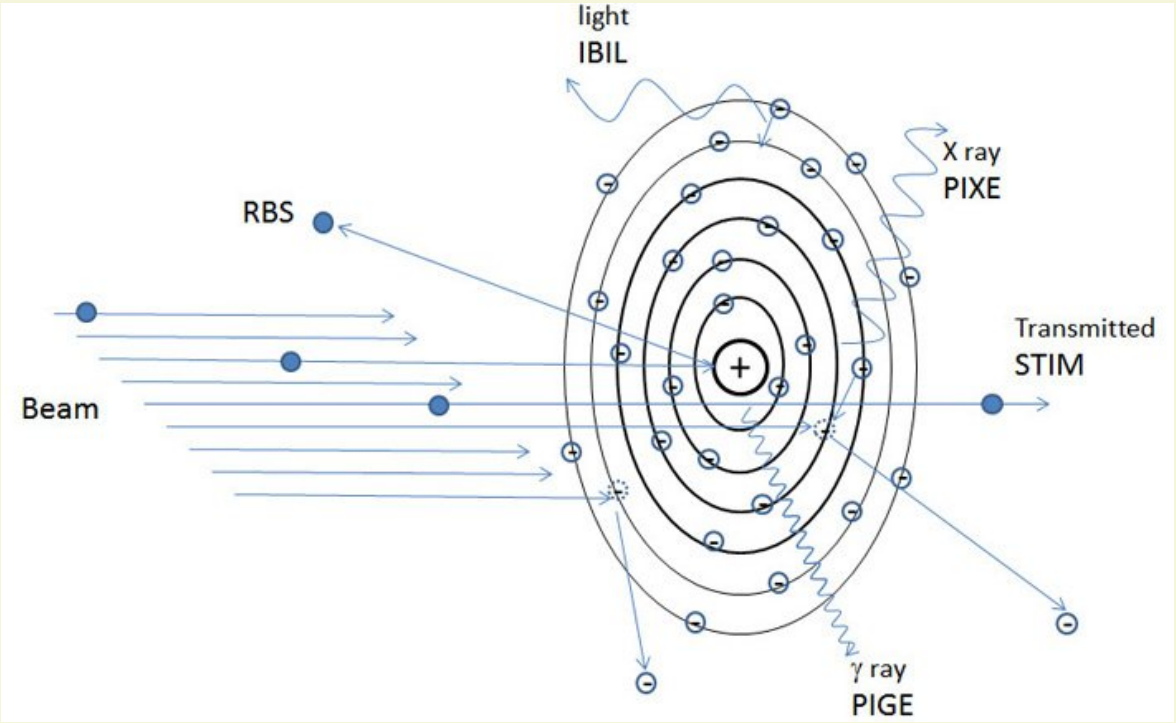


Figure 1. Scheme of interactions between ion beam particles and sample atoms with the corresponding IBA techniques.

a beam of high energy particles, typically of 1–3 MeV. Upon interaction in the sample, the beam induces the emission of secondary radiation and particles: depending on which one we choose to explore, there will be a specific IBA technique for each one (Figure 1).

When the X-rays generated by the sample are recorded and identified, the Particle Induced X-ray Emission (PIXE) technique is used. In the same way, Particle Induced Gamma Emission (PIGE) is used when gamma rays are involved. If the backscattered particles are recorded, Rutherford Backscattering Spectrometry (RBS) is performed. When visible light is emitted and recorded, the technique is called Ion Beam Induced Luminescence (IBIL). There are other IBA techniques that are not included in this introduction and we encourage the interested reader to read through the specialized literature [1, 2].

In our case, the most used IBA techniques are PIXE, PIGE and RBS. Each one can provide different information, but what is really remarkable is the information that can be extracted when they are combined.

A wide range of elements are automatically identified with PIXE, and the sensitivity is very high, typically in the range of some µg/g. However, the detection of elements with an atomic number less than 12 is poor, but the PIGE technique is an excellent alternative for their identification. With RBS the compositional depth profiles, i.e. the relative concentration of the constituents as a function of depth, can be determined. In this way, the combination of PIXE and RBS allows to obtain the concentration of the majority of elements present – from trace to major elements – and information on their depth distribution as well. And, if we add the PIGE technique then the concentration of almost every element of the periodic table may be obtained.

The experimental conditions involve low beam currents, in the order of 0.5-3 nA and short time,

around 10-20 minutes, is needed to acquire the spectra. Under these conditions structural damage or defect creation is minimized and IBA techniques are considered as non-destructive, but this consideration has to be contextualized since the measurements are usually done in a chamber under vacuum conditions:
- whatever the chamber dimensions are, there will always be a sample size limitation, implying that sampling may be necessary;
- working under vacuum conditions can induce mechanical damage: thermal, drying or charging effects can cause cracks or even detachment or sample fracture;
- some samples have complex geometry making them difficult to handle under these conditions.

In order to improve these conditions and avoid the vacuum limitations, the particle beam must leave the chamber and meet the object in open air. That is, an external beam is required as well as the ability to perform measurements under atmospheric conditions. The following aspects should also be considered when working under these conditions:

- a thin window or barrier material, which can withstand the pressure difference between atmosphere and vacuum, and the buildup of radiation damage as the beam passes through it while interfering the least with its quality (energy and collimation/focusing), must be provided;
- air absorbs the low energy X-rays generated, and slows down the incident and backscattered particles from the sample;
- air contains Ar that is excited by the beam originating X-rays within the usual energy detectable range then interfering with the X-rays emitted from the sample;
- beam spatial resolution and detection limits will be degraded as compared with analysis performed under vacuum conditions.

In the next section the solutions adopted to resolve or minimize these effects will be presented and discussed.

Generally speaking, the use of ion beam analysis for the study of historical-artistic objects is very much linked to the development of the PIXE technique in the early 1970s. Another milestone was the implementation of the IBA techniques in air, allowing in situ analysis of objects of large sizes or too fragile to be in vacuum. The development of a focusing system and the use of ultra-thin exit windows enabled transforming it into a real extension of nuclear microprobes.

From the approximately 100 nuclear microprobe facilities in the world, only a few are entirely or partially dedicated to research in the fields of patrimony studies. This new facility in Portugal adds to a number of others in laboratories across Europe where these techniques were made available, namely the AGLAE (*Accélérateur Grand Louvre d´Analyse Elémentaire*) in Paris [3], LABEC (*Laboratorio di Tecniche Nucleari Applicate ai Beni Culturali*) in Italy [4], or the CNA (*Centro Nacional de Aceleradores*) [5] and the CMAM (*Centro de Microanálisis de Materiales*) [6], both in Spain.

The Portuguese External Beam Facility

Assembly of the external ion beam analytical end-station started in 2005 under the POCI/CTM/60685/2004 project funded by the Portuguese Foundation for Science and Technology (FCT). The first and main objective of the project was to install the external beam analytical end-station at the existing microprobe facility at Instituto Tecnológico e Nuclear (ITN), in operation since 1999 [7]. Figure 2 shows a photograph of the microprobe set-up. The proton beam is generated by a 2.5 MV singe ended Van de Graaff accelerator and directed

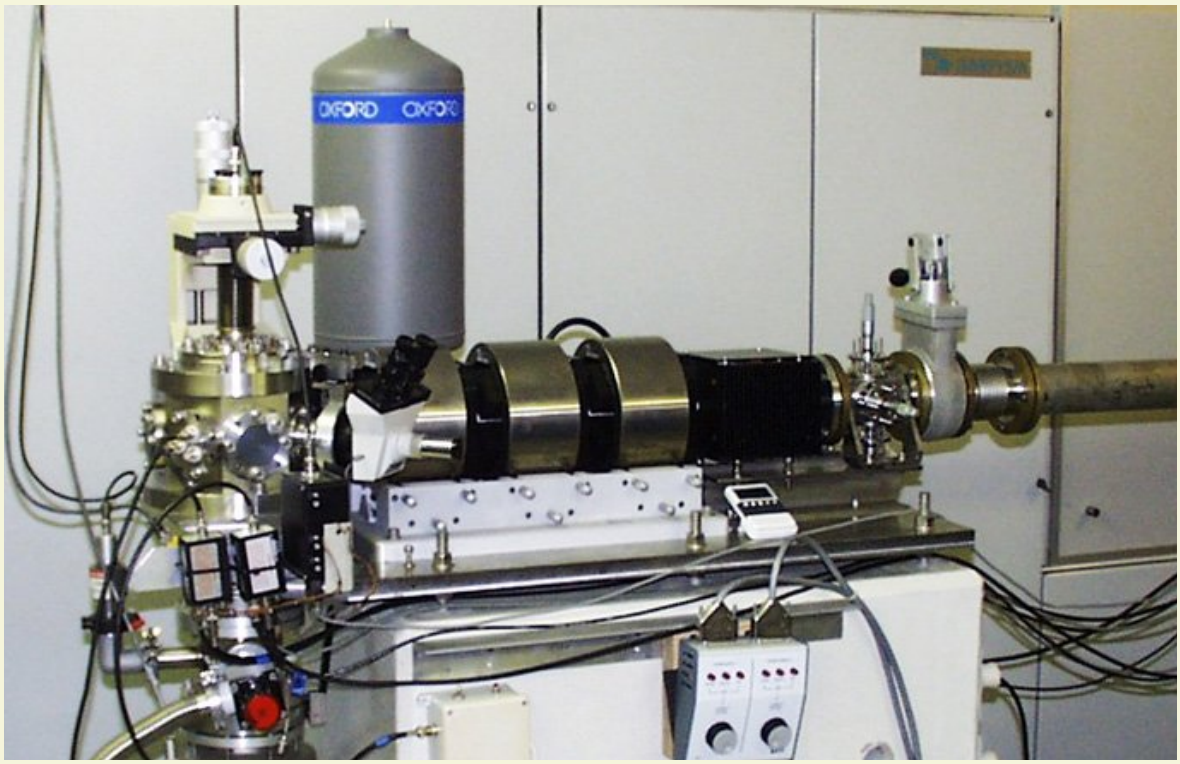


Figure 2. Microprobe installed at the Nuclear and Technological Institute.

to the microprobe beam line through a 90º bending magnet.

The microprobe collimator slits, scanning coils, lenses and chamber are mounted on a single concrete block sitting on a 1 cm thick plate of polystyrene foam to minimize vibrations. An Oxford Microbeams magnetic quadrupole triplet is used to focus the beam. The scanning coils located before the lenses allow to raster the beam over the sample surface, with a maximum area of 2.6x2.6 mm^2 when vacuum conditions and 2 MeV protons are used. Figure 2 also shows the vacuum chamber which can support up to eight different detectors and the cryostat needed for the X-ray detector.

The external beam set-up photograph is shown in figure 3. Each component will be described in the following paragraphs taking as reference the considerations made above in relation to the work under atmospheric conditions:

- The exit nozzle assembly is composed by two parts: one fixed to the chamber and one other, replaceable during the experiments if needed, having at its end a vacuum tight extraction window made of 100 nm thick Si_3N_4 membrane held in a 200 µm thick Si frame, allowing nearly 100% transmission with negligible energy loss. The size of this window (1x1 mm^2) sets the limit of the maximum beam scanning area.

- In order to reduce air interference during the measurements, a helium rich atmosphere is set by insufflating He gas towards the analyzed region by means of a nylon chamber, placed around the particles detector and connected to a He flow controller.

- To reduce the degradation of beam spatial resolution the distances should be kept as small as possible, while allowing the outgoing radiations to reach the detectors. The distance between the

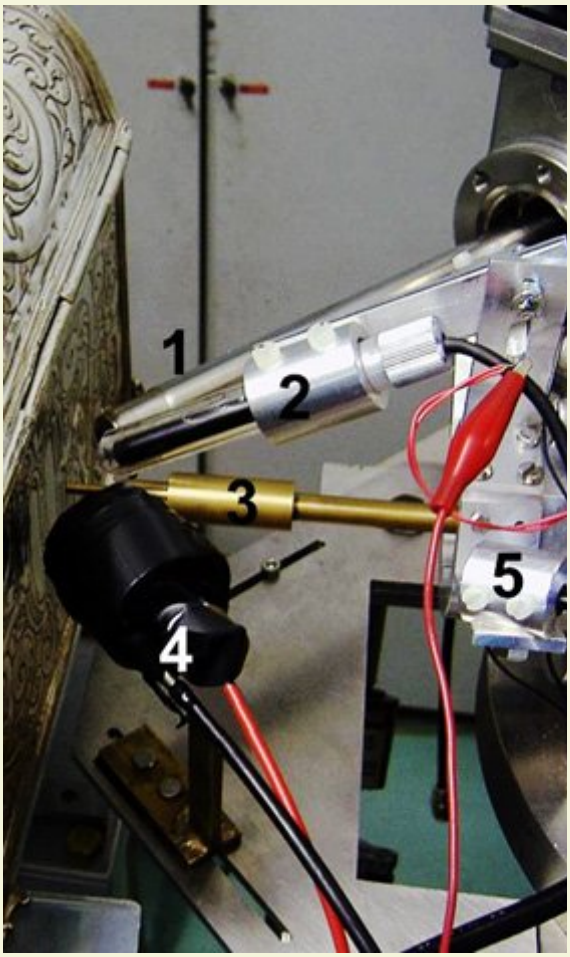


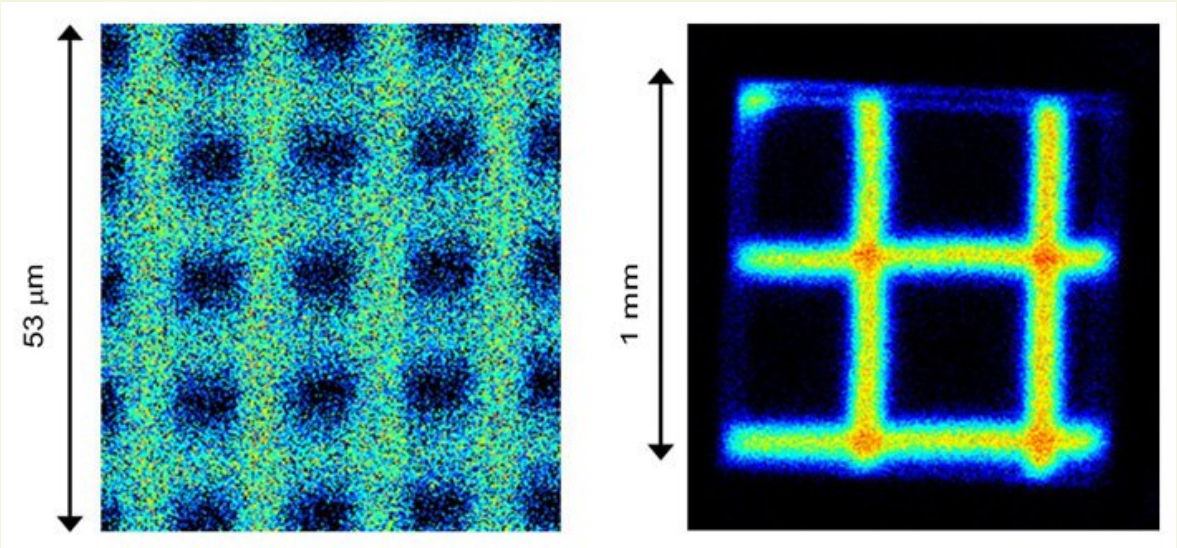
Figure 3. External beam set-up: 1. X ray detector; 2. mini-camera; 3. exit nozzle with a 100 nm thick Si_3N_4 window; 4. particle detector with He flux; 5. Positioning lasers.

beam exit window and the object is thus set to 3 mm, being controlled by reference to two intersecting laser beams. The object can be moved in the three directions and accurately positioned by means of a special x-y-z table. For assistance during the whole sample positioning procedure a mini-video camera is used.

The detectors are placed around the exit nozzle in different configurations according to the type of radiation to detect. The X-ray detector is a Bruker Si SDD detector with 8 μm Be window and 145 eV resolution at 5.9 keV. It is placed 2.8 cm from the sample at an angle of 45º to the beam direction. The backscattered protons are detected with a Si surface barrier detector placed at an angle of 47º to the beam direction, 2.2 cm away from the sample. When necessary, the gamma rays are detected with a large volume ORTEC HPGe detector with 45% efficiency and 1.9 keV energy resolution, placed at 45° to the beam direction.

Figure 4 shows images of a 2000 and a 50 mesh copper grids recorded under vacuum and external conditions, respectively, and under identical

Figure 4. Images for a 2000 mesh and 50 mesh copper grid recorded under vacuum and external ion beam set-up under identical experimental conditions.



experimental conditions (proton beams of 2 MeV energy and 1 nA current). For the former, the spatial resolution is 2x3 μm^2 while for the latter external conditions the best spatial resolution is 60x60 μm^2 when working under helium rich atmosphere.

In what concerns the analysis of the data generated by the techniques, there is specific software to analyze the different types of spectra and extract the required information. In the case of PIXE, the AXIL/QXAS [8] program is extensively used for X-ray lines deconvolution and peak areas extraction, and DATTPIXE [9] for quantification. GUPIX [10] software was also used for X-ray spectra deconvolution and quantification, and its results were compared with the ones obtained using AXIL+DATTPIXE showing a good correlation. As PIXE is not efficient for the detection and quantification of elements with low atomic numbers, namely for Na and Mg, these elements are detected and quantified by PIGE, in proton capture nuclear reactions, by considering the yields of the 440 keV and 585 keV gamma lines respectively in the gamma spectra of the daughter nuclei. Information on layered targets is gathered by means of the elemental depth distributions extracted from the recorded RBS spectra. The NDF code [11] is used for RBS spectra fitting and sample composition determined in a self-consisting way with PIXE data simulated by means of LibCPIXE code [12], an open-source library for multilayered samples that can work jointly with the NDF code.

Applications

The applications of IBA techniques to the study of Cultural Heritage objects are as varied as the objects themselves. Different objects were studied using the microprobe (under vacuum and also in external conditions) located at ITN. Some examples are Arraiolos tapestries [13], stained glasses [14], jewellery [15], and ceramics [16].

In this section two case studies selected among the works performed are presented. They show the versatility of the set-up since different detectors, software and experimental conditions were used to study each specific case.

The case studies refer to Roman glasses from Museu Municipal de Arqueologia da Amadora (MMAR), and religious gilt objects dated from the XVI to the XVIII centuries belonging to Casa-Museu Dr. Anastácio Gonçalves (CMAG). Both museums are located in Portugal.

Roman Glasses

The Roman glasses from MMAR are referred to different occupation times of a Roman *villa* during the III and IV centuries A.D. at Quinta da Bolacha, Portugal. This Roman *villa* was discovered in 1979 during the prospection of a Roman aqueduct in Amadora.

The archaeological works made possible identifying sealed contexts that are attributed to the III and IV centuries A.D., together with revolved contexts of uncertain dating. The study intended to materially characterize the occupation periods, resorting to analyzes of glass fragments, as well as to associate the fragments from revolved contexts with those from other contexts, trying to determine its possible chronological attribution. The poor state of preservation of these glasses strongly advised against analysis in vacuum, leaving the external beam as the only suitable alternative option.

Results were obtained with the use of a proton beam of 2 MeV of energy and 1 nA of current.

Results were constantly compared for each sample with those obtained using Corning standard reference glasses with well known composition.

Figure 5 shows two fragments that were attributed to the first occupation period (III and IV centuries A.D.) of the Roman *villa*, and considered by archaeologists as belonging to the same object. The analysis performed using the external microprobe set-up showed different compositions for both fragments. The larger fragment is abnormally rich in K, as compared to other samples, while being low in Na, contrarily to the smaller one [17]. Therefore these two fragments should not be considered as belonging to the same original object (as opposed to what was initially thought).

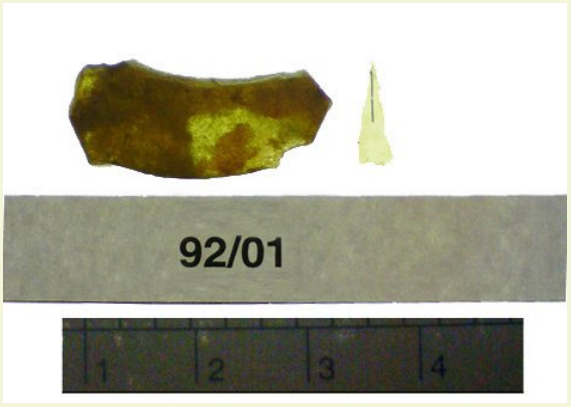


Figure 5. Fragments recovered from excavation site in Amadora, Portugal, attributed to the first occupation period of the Roman villa (III-IV centuries A.D.).

A very interesting capability of the external microprobe applied to the study of these objects is the possibility to perform scan analysis. Elemental scans are presented in figure 6 that show the distribution of Si, Ca and Mn in a region of a glass fragment partially covered by an evident corrosion over layer. The corrosion region correlates to a higher content of Mn due to leaching and surface redeposition, and also Fe, probably from the soil contamination. There is also anti-correlation to the contents of Si and Ca, probably due to the leaching of these elements from the glass matrix.

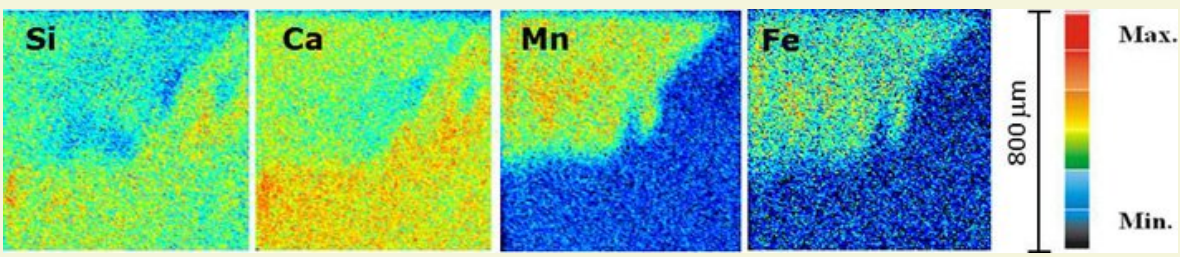
Contrary to the glass fragment referred to above, analysis of the remaining selected glasses from the different contexts showed moderate to high contents of Na, together with reduced contents of K and Mg, which are typical of soda-lime-silica glasses produced by resorting to natron as a source of alkali. Specific contents of Sr and Mg, along with absence of Zr, indicate the use of coastal Mediterranean sands as raw material.

It was also possible to determine from the X-ray spectrum (Figure 7a) significant levels of Sb and Pb in one fragment, a deep blue tessera shown in Figure 7b, indicating the use of opacifying agents which were in use until the IV century A.D., confirming the time interval of the villa's occupation.

Gilt Objects

Another interesting example is the study of religious gilt objects belonging to the CMAG Collection:

Figure 6. Elemental distribution of Si, Ca and Mn. On the corroded areas there is a higher concentration of Mn and Fe.



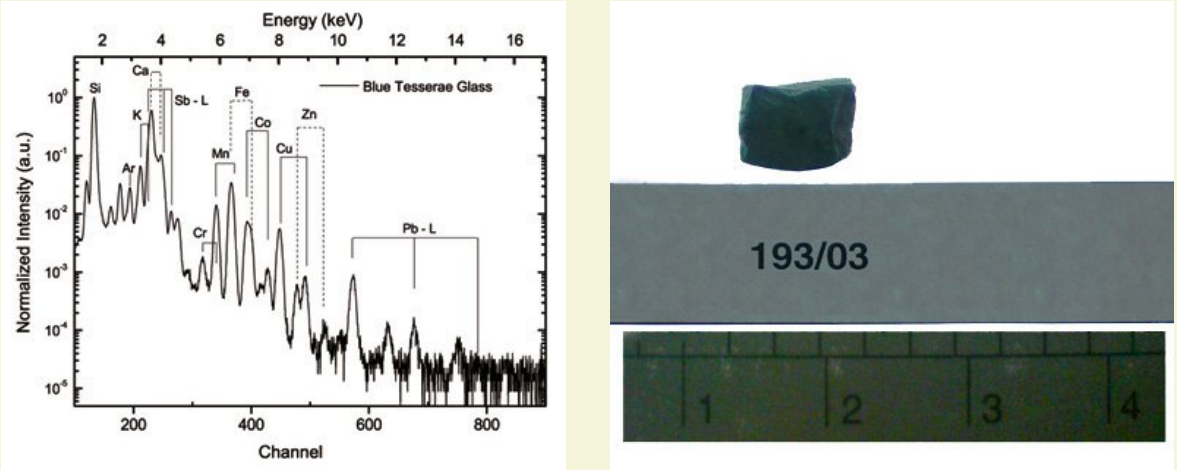


Figure 7. a) Normalized PIXE spectrum recorded under atmospheric conditions; b) Blue tessera glass.

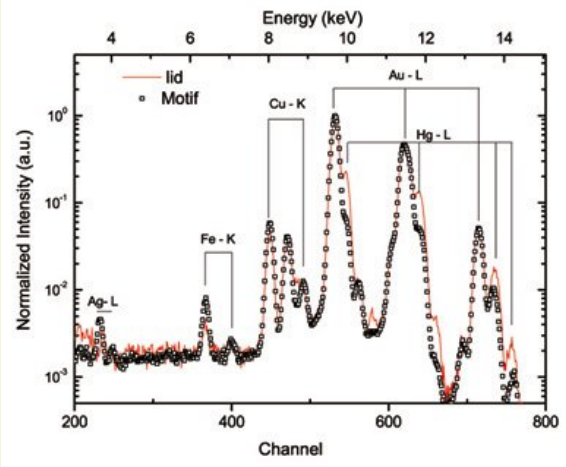


Figure 8. Normalized PIXE spectra from inside the cup (red) and an external gilt motif (black) of the ciborium (CMAG 1180 Collection).

a reliquary (CMAG 1194) from the XVI century, which has two visible hallmarks (AR SII) on the base and on the lid, and it is believed to be of Spanish origin; an *ostensorium* (CMAG 1164) from the mid-XVIII century with a visible hallmark indicating the goldsmith (J.P./C.) and Portuguese origin (a crown L, from Lisbon); a *ciborium* (CMAG 1180) with an oval base and partially gilt with several religious motifs.

Regarding the experimental conditions, the X-ray spectra were acquired with a 350 μm thick Mylar foil in order to filter the Au and Hg M-lines and a He flow was used to improve the resolution. Two reference samples with known composition, brass NBS 1105 and Ag–Cu (80–20) alloy were analyzed throughout the measurements.

The gilt method has been used since ancient times to make an object look like cast gold and at the same time to improve the surface of the object for corrosion resistance. The technique has been developed and improved along the centuries. The method used for these objects is the mercury gilt, also known as fire gilding. It is based on the application of an amalgam composed of gold and mercury onto a metal surface [18], then heating it to 250-300 ºC for a short time (few minutes), and cooling down, followed by polishing until the object shows a smooth and brilliant surface.

As it is expected, differences in composition in the object were found according to the different provenances and manufacturing dates. The gilt results were very dependent on the goldsmith experience, since the temperature and times were "visually controlled" and at the same time they are crucial on this process. For example, the time was controlled as "when the amalgam changes colour from grey to dull yellow" [19]. Because of that it was not surprising finding different Hg and Au/Ag concentration ratios for each piece, as is shown in table I [20].

Table I. Results of X-ray diffraction analysis of greywacke rocks from Wadi Hammamat.

	Ag (%)	Au (%)	Hg (%)	Impurities
Reliquary	10	75	10	Cu, Ca, Fe, Pb, Zn
Ostentorium	10	77	12	
Ciborium	12	78	8	

These differences in concentration were found not only between objects, but also in different parts of an object. One clear example of this is the *ciborium*. Figure 8 represents the X-ray spectra recorded in two different parts of the *ciborium* which corresponds to the inside of the cup and to one of the gilt external motifs, respectively. Both areas are gilt with homogeneous distribution but with different composition: the inner part shows an average composition of 25% Ag, 60% Au and 15% Hg while the external motif has an average composition of 9% Ag, 84% Au and only 5% Hg. This difference in concentration can be attributed to the different temperatures achieved during the gilt process according with the Au-Hg phase diagram [21], or to the Au layer thickness, being lower inside the *ciborium*. Another possibility is the handling of the piece once the external parts are more predisposed to handling that the inner parts, or to the cleaning process with different products.

In figure 9a, the elemental distribution of Hg, Au, Ag and Cu is presented, covering a 800x800 μm^2 area corresponding to the fastener of the reliquary (figure 9b). In fact, it was found that not only the fastener, but also the hinge show a quite similar elemental distribution as the one shown in figure 9a.

From these elemental distribution maps the relationship between the Hg/Au and the Ag/Cu atoms can be extracted. The Au and Hg elements are associated and they follow the same pattern in the studied region. On the other hand, the copper follows the silver distribution. The addition of Cu to Ag was used to improve the hardness of the silver, a method that is still used nowadays.

Conclusions

The external ion beam analytical end-station at ITN, Portugal, is a valuable facility for studying a

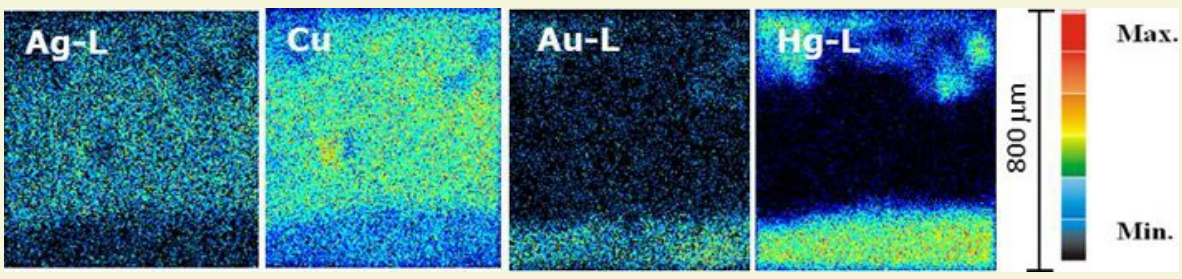


Figure 9. a) Elemental distribution of Ag, Cu, Hg and Au; b) View of the exit nozzle and the object to be analyzed (the fastener of the reliquary (GMAG 1194).

wide variety of objects related to our common Cultural Heritage and History. The technique itself is non-invasive and it is expected to become a standard tool available to the conservator and conservation-scientists. Different examples of applications have been shown in order to illustrate the versatility of the Portuguese set-up. It is expected that in a near future the techniques it provides can be increasingly used as standard tool accessible for the cultural heritage professionals community.

Acknowledgments

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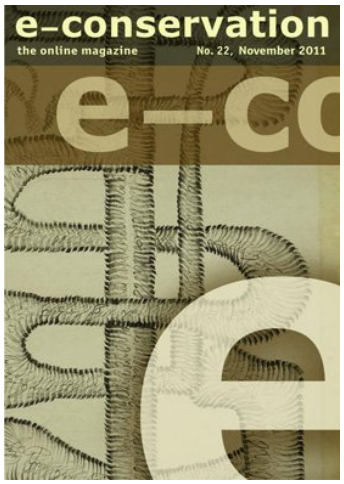

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