



Characterization of an ancient ‘chemical’ preparation: pigments and drugs in medieval Islamic Spain

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ABSTRACT

The analysis of the contents of an Islamic glazed ceramic pot found in Zaragoza (Spain) revealed an ancient ‘chemical’ preparation (11th century AD). The use of several analytical techniques (optical microscopy, scanning electron microscopy with X-ray energy-dispersive spectrometry and X-ray diffraction, and pyrolysis-gas chromatography/mass spectrometry) enabled us to identify mineral, organic and vegetal components: orpiment, a fig, and some grape seeds, together with a small amount of gypsum. The purpose of this mixture may have been for colouring because orpiment, the yellow sulphide of arsenic, has been used as pigment in painted decorations and manuscripts since ancient times. Nevertheless, since “yellow arsenic” was also one of the most common remedies in Arabic pharmacology and medicine, the components we identified may have been part of a medical treatment. Moreover, knowledge of the chemical composition of the contents of this pot provides a means of establishing the best way to conserve them.

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1. Introduction

The characterization of archaeological materials is very valuable in terms of what it reveals about the artistic and manufacturing techniques of the past. An in-depth analytical study of ancient products can reveal information on how advanced technologically ancient societies were, and also on their diet, trade, rituals, and everyday activities (Goffer, 2007). Knowing the composition of archaeological findings also enables us to assess the state of conservation and ongoing degradation processes, to set up exhibition and storage conditions, and to plan the right conservation treatments.

This paper reports the results of an investigation on the original contents recovered from a small glazed pot. The object was found during archaeological excavations from the ancient wall of Zaragoza (Fig. 1). This city wall was constructed in Roman times. For centuries some buildings were attached to the wall and part of these houses have remained buried in the sediments at the foot of the wall. The small glazed pot (Fig. 2) was found in these sediments, in the inner side of the wall, together with ceramics —*almagra* (red

slip) and manganese-decorated pottery— and a large number of glass fragments, that suggest they belonged to an Islamic context, dating to the 11th century AD or *Taifa* period (Carrasco et al., 2009).

The ceramic pot is 6.5 cm high, with a maximum diameter of 6.6 cm, completely glazed both inside and out, is yellow-honey in colour, and decorated with brown-black stripes. This type of decoration was common in Islamic ceramics of *Taifa* period, it was produced on an alkali-high lead glaze (around 3% K₂O + Na₂O and 45–48% PbO) and painted with manganese pigments (Pérez-Arantegui and Castillo, 2002); the pottery was made with calcareous clays, producing a cream body. Unlike in most other finds, part of the contents was blocking the mouth of the pot and it was not empty. This enabled us to study and analyze the complex contents.

The main objective of this study was to understand the composition, but also the purpose of the products contained in the ceramic pot, using samples as small as possible. The knowledge we acquired of this composition means that sustainable methods can be found in order to conserve the pot and its contents.

A multi-analytical approach, based on the use of complementary techniques, was carried out on the material collected from the pot in order to identify the materials in the object. Optical microscopy (OM) and scanning electron microscopy with an energy-dispersive X-ray spectrometer (SEM-EDX), together with X-ray diffraction (XRD), were all used to highlight the composition

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Fig. 1. The place where the object was found during the archaeological excavations in the city wall (Zaragoza, Spain).

and the nature of the inorganic components and the presence of vegetal components. Pyrolysis-gas chromatography coupled with mass spectrometry (Py-GC/MS) was complementary used to reveal the presence and the composition of other organic substances.

2. Materials and methods

Optical microscopy observation was performed using an Axio Imager (Zeiss), reflected light microscope. A JEOL JSM 6400 Scanning Electron Microscope (SEM) was used for the micromorphology study and for the identification of the chemical composition. The SEM was equipped with a system for Energy-Dispersive X-ray Analysis, EDXA (Oxford Instruments, INCAx-sight), with ZAF correction. For the SEM-EDX analysis of the samples, the acceleration voltage applied was 20 kV, with a 0.6 nA current probe. Bulk analyses were performed by focusing the electron beam on the sample and by acquiring data from different points. The powdered sample was prepared as carbon-coated and directly monitored to analyze and to observe the microstructure. The seeds and the fig microfragments were prepared as gold coated and directly monitored to observe the morphological characteristics, with an accelerating voltage of 5 kV and 0.1 nA probe current.

Crystalline phases from the yellow powder were identified by X-ray Diffraction (XRD), with a D-Max Rigaku diffractometer equipped



Fig. 2. Islamic glazed pot found during the archaeological excavations.

with a rotating Cu anode, working at 40 kV and 80 mA, and a graphite monochromator offering Cu K α 1,2 radiation. The acquisition was carried out between 5° and 70° (angle 2Theta), with 0.03° step⁻¹ and $t = 1$ s step⁻¹. The data were processed using the JCPDS-International Centre for Diffraction Data-2000 package.

Py-silylation-GC/MS analyses (few μ g of sample) were carried out at 550 °C (pyrolysis time: 20 s) (Pérez-Arantegui et al., 2009). The pyrolyser (CDS Pyroprobe 5000 series) was coupled online with a 6890N GC System Gas Chromatograph (Agilent Technologies) coupled with a 5973 Mass Selective Detector (Agilent Technologies) single quadrupole mass spectrometer. The pyrolyser interface was kept at 180 °C, the transfer line at 300 °C, and the valve oven at 290 °C. For the gas chromatographic separation, an HP-5MS fused silica capillary column (5% diphenyl-95% dimethylpolysiloxane, 30 m \times 0.25 mm i.d., J&W Scientific Agilent Technologies,) with a de-activated silica pre-column (2 m \times 0.32 mm i.d., J&W Scientific Agilent Technologies) was used. The split-splitless injector was used in split mode at 300 °C, with a split ratio 1:20. The chromatographic conditions were as follows: 30 °C isothermal for 8 min, 10 °C min⁻¹ up to 240 °C and isothermal for 3 min, 20 °C min⁻¹ up to 300 °C and isothermal for 30 min. The carrier gas (He, purity 99.9995%) was used in the constant flow mode at 1.0 ml min⁻¹.

3. Results and discussion

3.1. The contents of the pot

The small pot was emptied in the laboratory and its contents carefully observed under a microscope. The contents consisted of

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