

ANALYSIS OF THE RESIDUALS IN GRAVE GOODS FROM THE VACCAEA ERA AT THE NECROPOLIS OF 'LAS RUEDAS' IN PINTIA.

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Introduction:

The archaeological site of Pintia (Padilla de Duero/Peñaflor, Valladolid), located at the oriental side of the vaccaea region, was occupied for more than thousand years, between IV century B.C and VII century A.C, by different cultures: Vaccaean, Roman and Visigothic. The Vaccaea era has yielded the more interesting archaeological records regarding the different functional areas that involve the oppidum: the fortified village of 'Las Quintanas' – surrounded by a wall and a moat-, the artisan neighbourhood of 'Caralaceña', the necropolis of Las Ruedas, etc.

Pottery, metal and bone/skeletal remains are the more common objects recovered. The pottery remains are very well preserved, so they are optimal for analysing and the subsequent study of the food contained, such as milk-based products, animal fats, beer, wine, etc.

The vessels from the funerary area of 'Las Ruedas' are specially interesting. They contained the food for the afterlife and therefore have a great symbolic value. The wealth of the grave, the absence or presence of objects or contents contributes significantly on the assessment of the deceased's profile (age, sex, social status, etc). Therefore, the analysis of the residuals found in the vessels becomes highly significant.

The use of three complementary instrumental techniques has been assessed as an analytical methodology. Light microscopy (visible and ultra-violet light), Environmental Scanning Electron Microscopy equipped with an Energy Dispersive X-ray Analysis system (ESEM-EDX) and micro-Raman spectroscopy (μ -RS) have been used in this work. Samples were set up on a common holder for all the techniques

Experiment: The specimens studied in this work were extracted from a crateriform vessel which belongs to the grave goods of the grave #154. The grave corresponds to a tall individual, probably female, buried in the II century BC.

Light microscopy analysis were carried out with a petrographic microscope Leica DMLP with both transmitted and epi-illumination. The environmental electron scanning microscopy was carried out with FEI-Quanta 200F equipped with a Genesis XM2i from EDAX. Micro-Raman spectra were acquired with a high resolution Raman spectrometer LABRAM HR 800UV

from Horiba-Jobin & Yvon Spex, equipped with an Olympus BX41 microscope and a XYZ-axis motorized stage. A x100 objective and backscattering configuration were used for Raman spectra acquisition. A 632.8 nm HeNe laser were used as excitation probe. The nominal laser power on the samples was 1mW and the irradiance was about 100 kWcm⁻². The spectral window was [200-3800] cm⁻¹, the integration time was [5-60] s and 2 accumulations were used as acquisition parameters. All this results in a spectral resolution less than 1 cm⁻¹.

Results and Discussion: The results show the presence of phytoliths, vegetal fibres and stems with parenchymal structures at different alteration degree. Low crystallization structures, with elemental composition of C, O and Ca were detected. They are probably related to oxalates that can come from milk-based derivatives or vegetal residuals.

Degradation and age of organic natural fibres can be assessed by the relative intensity relationship between the $\nu_{as}(COC)$, $\nu_s(COC)$ glycolic and $\nu_{as}(CH_2)$ bands at 1098, 1124 and 2917 cm⁻¹ respectively. We have obtained for I_{1124}/I_{1098} and I_{1098}/I_{2917} values of 0.9 and 0.6 respectively which are characteristic of old and degraded fibres.

The presence of organic materials with low crystallinity and order can be assessed by the study of the 1595 cm⁻¹ and 1386 cm⁻¹ bands, related to C-C bonds vibrations, and their relative intensities.

Raman bands assigned to compounds related to agricultural processes can be identified and therefore can be distinguished from the residuals of interest.

Conclusions: We have stated herein that the use of the Raman spectroscopy together with complementary high resolution analytical techniques becomes a powerful tool for the study about the contents and use of ceramic vessels from archaeological heritage.

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