Reconstructing Phoenician pottery production in Sardinia: a multi-analytical characterization of samples from the site of Pani Loriga (8-7th century BC) and Monte Sirai (6-5th century BC)

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INTRODUCTION

Pottery represents one of the earliest materials created by humans as an expression of the cultural background (Rice, 2015). As one of the most complex and ubiquitous archaeomaterials, ceramics offer invaluable insights into past societies. Their analysis is essential for understanding the diverse cultures and functions they served, as well as the variety of materials and manufacturing technologies employed (Maritan, 2004; Hunt, 2017). However, the complexity of pottery's chemical and mineralogical composition requires a multi-analytical approach to fully understand its history. This approach combines physical and chemical analyses to determine raw material sources, explore technological aspects of pottery production, and define firing conditions, while also investigating post-burial processes (Cultrone et al., 2001; Barone et al., 2002; Belfiore et al., 2010; Hunt, 2017).

The present PhD thesis aimed to perform a multi-analytical archaeometric study on 53 pottery samples from two archaeological sites in Sardinia: Pani Loriga (7th-8th century BCE) and Monte Sirai (5th-6th century BCE), both from the Phoenician period. These sites are located in the South-West of the Sardinian region (Italy). Studying the history and the culture of these sites helps to reconstruct the extent of cultural interactions between the indigenous Nuragic people and the incoming Phoenician settlers, who played a crucial role in the exploitation of resources from the Sulcis inland area, establishing their control over the territory (Botto, 2016).

The main goal of this research was to explore the chemical, microstructural and mineralogical complexities of archaeological ceramics, providing specific guidelines for developing research strategies and formulating anthropological questions. The focus was to gather exhaustive information about the raw materials, provenance, and manufacturing techniques of the ceramics, with particular attention to firing temperatures and atmospheric conditions. This approach allowed a comprehensive characterization of the production processes involved.

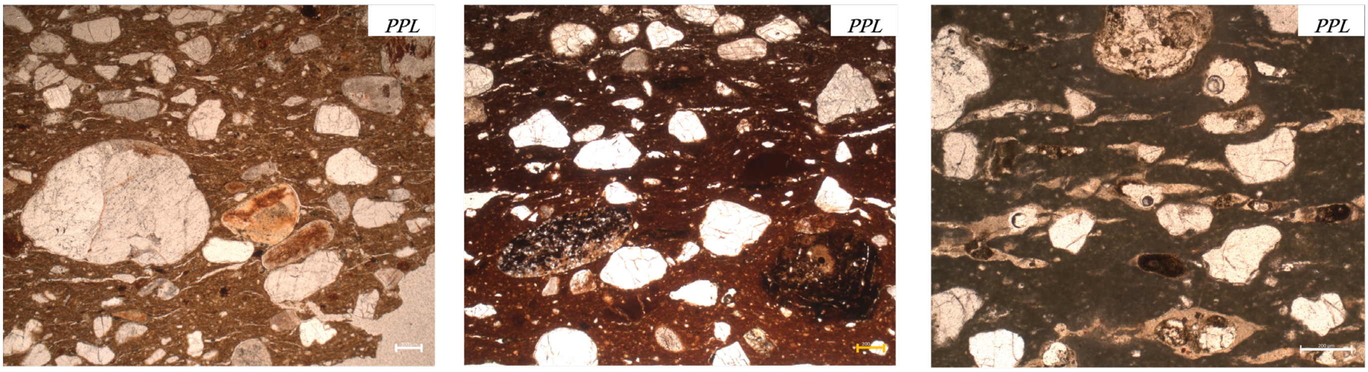
METHODS

The samples were examined with petrographical, mineralogical, chemical and archaeomagnetic approaches, and all the data gathered were crucial for understanding and reconstructing the cultural context of the Phoenician-Punic population. This study is particularly significant because the pottery from the Pani Loriga site had never been analysed using an archaeometric approach. The characterization protocol began analysing the mineralogical and microstructural composition of Monte Sirai samples, defining the best analytic procedure later applied to a wide set of samples from Pani Loriga.

RESULTS

Macroscopic observations of the shards were initially performed to assess their features, including form, colour, texture, surface treatment, and visible inclusions. These observations were essential for ensuring a comprehensive understanding of the samples' morphology and other macroscopic characteristics. Based on these analyses, five fabric groups were identified. Two of these groups, encompassing most of the samples, are characterized by coarse inclusions, suggesting minimal processing or purification of raw materials during the ceramic body preparation (Fig. 1). In contrast, the other three groups display purified clay, with minimal inclusions (Fig. 2). Optical petrography, combined with X-ray diffraction analysis, provided insights into the mineralogical associations, firing temperatures, and rock inclusions.

X-ray diffraction analyses further identified three distinct mineralogical groups based on their assemblages (Fig. 3). In the most populated groups, the presence of minerals such as quartz, plagioclases, feldspars, and hydro-silicates, compatible with igneous rocks from pyroclastic and epiclastic deposits (including dacites and rhyolites), suggests the local production of these samples. Conversely, a high calcite content, along with microfossils observed in thin sections, indicates that some samples were likely imported. The third group is charac-



Details of aggregates (qz, flds/pls, rock inclusions). Scale bar: 200 μm

Details of aggregates (qz, flds/pls, rock and iron inclusions). Scale bar: 200 μm

Details of isoriented and planar voids filled with secondary calcite. Scale bar: 200 μm

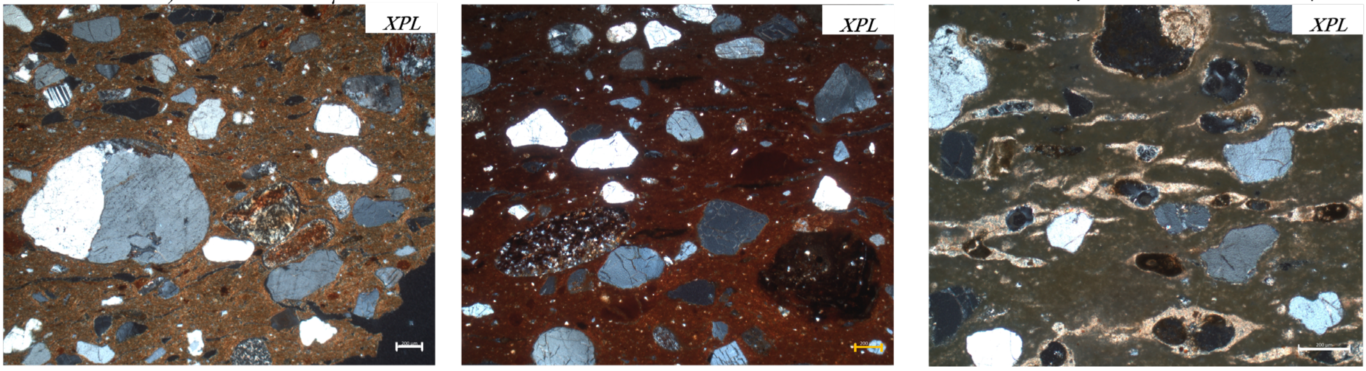
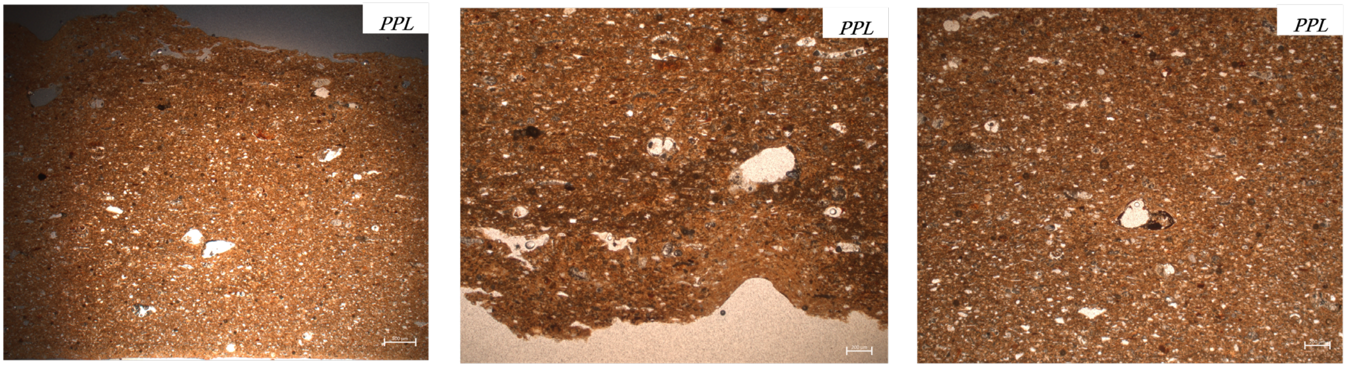


Figure 1 *Details of inclusions, matrix and voids of samples with a coarse matrix.*

terized by a pronounced presence of diopside and gehlenite. The identification of minerals such as hydro-silicates, (primary) calcite, gehlenite, and diopside reflects different firing conditions and allows the estimation of firing temperature ranges. Such temperature ranges are further supported by archaeomagnetic analyses, which provided additional insights into the thermal history of the pottery, offering additional data for their characterisation and confirming the firing process reconstructions based on XRD data (and the mineral stability fields).

After the first characterization of the ceramic samples, Scanning Electron Microscopy analysis was employed to perform micromorphological analyses in order to describe the textural and morphometric features of non-plastic inclusions, and SEM-EDS was used to perform microchemical analysis and maps, which allow describing the chemical variation within the ceramic body. To examine in depth the bulk chemical composition of the samples, elemental analyses were performed using a portable XRF instrument. All the ceramic bodies turned



Details of matrix, vesicles pores and silt siliceous inclusions (2.5x and 5x). Scale bar: 500 μm in the first left image and 200 μm in the others

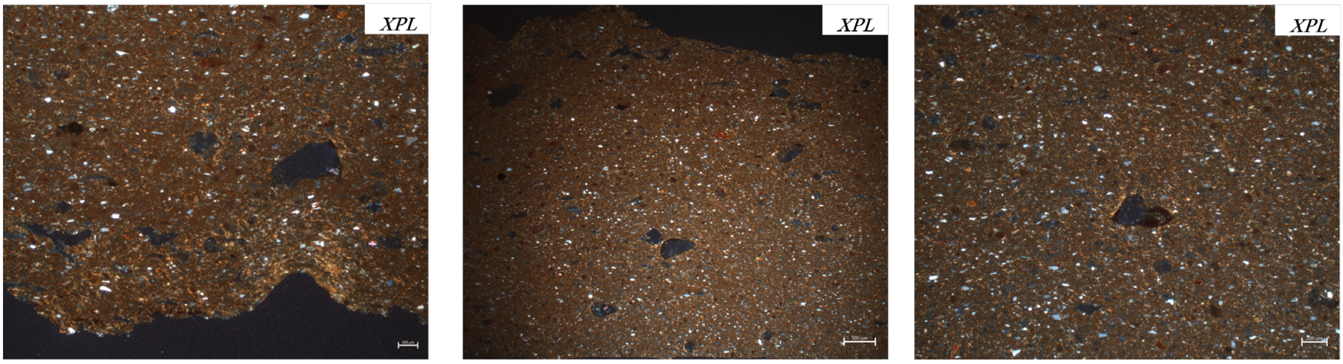


Figure 2 *Details of matrix and voids of the sample with a purified matrix.*

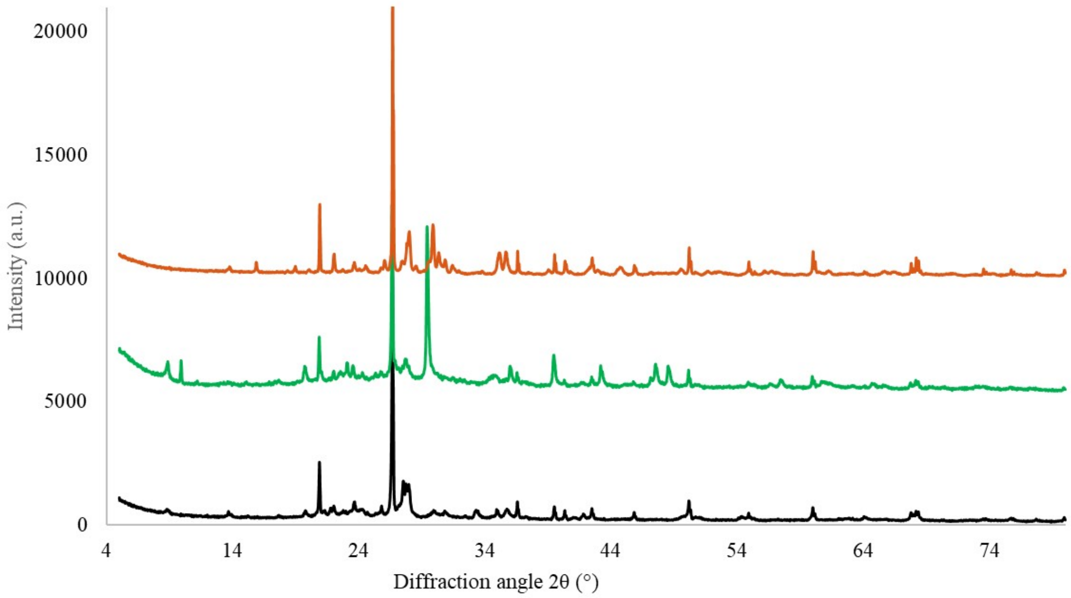


Figure 3 *XRD patterns of three different samples representative of each of the three different mineralogical associations.*

out to be composed mainly of Si, Al, K, Ca, Fe and Ti, a typical elemental composition expected in ceramic materials. This approach also provided experimental evidence for distinguishing locally produced ceramics from potential imports, as the chemical data plotted in the diagrams clustered into two distinct groups: one characterized by silica as the primary component, and the other diverging due to a higher calcium content (Fig. 4).

DISCUSSION

Petrographic analysis revealed significant variations among the different groups, offering insights into production techniques, raw material sources, and firing conditions. The first observations focused on the sherds' matrix composition, leading to a division into two main categories: silicate and carbonate matrices. In potteries with a silicate matrix, there is a high presence of quartz, feldspars/plagioclases, and notably dacites and rhyolites, demonstrating the local provenance, as proved by the geological setting (Assorgia et al., 1992; Barca et al., 2009). Conversely, samples with a carbonate matrix exhibit very low inclusions, primarily consisting of microfossils, suggesting a significant use of limestone or marl in the raw material. The different sources of raw material and the different manufacturing techniques of these sherds suggest they come from places where different production techniques were used.

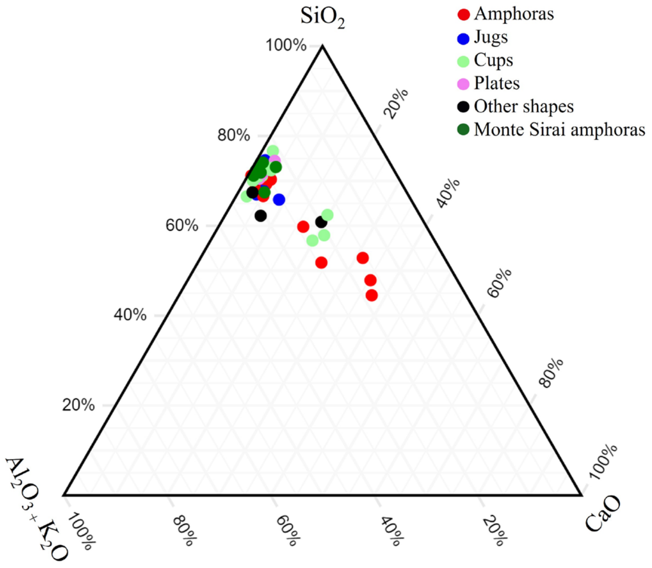


Figure 4 *XRF ternary diagram showing the relationship between SiO_2, Al_2O_3+K_2O, and CaO weight fraction (%).*

The void arrangement also differs between these identified fabrics. Planar and iso-oriented voids are indicative of pottery manufactured using a pottery wheel. On the other hand, the absence of iso-oriented voids suggests that this sample was likely produced without a pottery wheel, which prevented the formation of planar and iso-oriented voids. The matrix optical properties, mainly governed by the hydrosilicate behaviour, can reveal the temperature range at which the pottery was fired. If the matrix is optically active and shows birefringence, it indicates that the pottery was fired at temperatures below 800-850°C, a range at which the clay matrix generally loses its birefringence. In contrast, when samples display an optically inactive matrix, it suggests that the firing temperature was above 800-850°C (Gosselain, 1992; Ferrara et al., 2016; Amicone et al., 2021; Quinn, 2022). For carbonate samples, the firing temperature was determined by examining the structure of microfossils, which are not completely destroyed by the firing process, and well-formed traces of the shells are clearly visible. The microfossil degradation *status* can thus support the hypothesis of a firing temperature not above 900°C. Another group contains samples which exhibit a fully vitrified matrix, where optical activity has disappeared. This particular matrix appearance suggests that these samples were likely fired at a temperature around 1000-1200°C (Gosselain, 1992; Ferrara et al., 2016; Amicone et al., 2021; Quinn, 2022). The XRD mineralogical analyses also suggest different firing conditions between the samples, which range from low-temperature (maximum temperature $T_{max} < 850$-900°C), as shown by the co-presence of hydro-silicates (group one) and carbonates (group two), to high-temperature firing conditions ($T_{max} >$ 1000°C), with newly-formed phases such as Ca-(Mg,Fe)-pyroxenes and gehlenite (group three). Archaeomagnetic analysis allowed refining the firing temperature estimation, adding precision to the XRD interpretation. The hysteresis cycle of the three samples investigated, each representing a different mineralogical group, was measured at increasing firing steps, up to 700°C, and changes in magnetic behaviour were analysed to determine thermal alteration thresholds. The sample belonging to the silicate low-firing group begins to change its magnetic behaviour after heating to 500°C, suggesting an original firing temperature lower than the maximum one estimated by X-ray diffraction. In contrast, the behaviour of the carbonate-rich sample displayed a slight alteration of its hysteresis curves above 300°C, but this change was not pronounced enough to conclusively determine the original firing temperature (Rasmussen et al., 2012; Matau et al., 2013; Vaknin et al., 2023). A different pattern was observed for the over-fired sample, where the hysteresis curves remained unchanged throughout the heating process, indicating that the material had already been subjected to higher temperatures.

Pottery samples with slips were then analysed by SEM-EDS, which provided interesting results. Micro-chemical analyses revealed that the slip is more iron-rich if compared to the body of the samples analysed. Despite this slight variability, no other significant differences were found in the chemical compositions between the body and the slip. Additionally, from a morphological point of view, no visible differences between the clay grains of the slip and the body are detectable. This variation in iron content could be related to the addition of hematite in the slip.

Chemical analyses performed using portable XRF provided further confirmation of the sample composition observed with other analytical techniques. Most of the samples are characterised by the predominance of SiO_2, Al_2O_3 and K_2O, which is a common feature in ceramic as these elements are correlated to the abundance of detrital quartz grains and other silicate minerals or rock fragments. In addition, SiO_2 is also present in the clay minerals composing the finer groundmass. However, a relevant compositional differentiation is observable in the distinct group, which is characterised by a high calcium content, which suggests, again, the use of different clay sources. The consistency in the composition, even from a chemical perspective, suggests a standardized production technique and common raw material sources among most of the pottery samples. Conversely, the outliers may represent different sources, indicating the existence of trade and pottery exchanges.

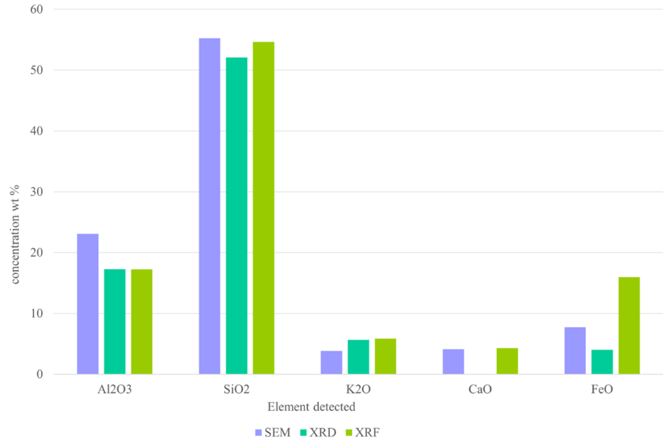


Figure 5 Graphic representation of the chemical content of one silicatic sample, as obtained by SEM-EDS, XRD and XRF. Ca-bearing phases were not here detected by XRD.

XRF data also acquire additional strength when directly compared to the results obtained by SEM and XRD analyses, as they offer compelling insights into the reliability and consistency of the methods employed and underscore their complementary nature. The convergence across different methodologies is significant, as it suggests that despite the inherent differences in how each technique operates—SEM and XRF providing direct chemical data, while XRD Rietveld analysis the mineralogical phases—they can offer a reliable characterisation of the material composition (Fig. 5). Moreover, the possibility of comparing chemical and mineralogical analyses opens new possibilities for estimating also the raw material composition.

CONCLUSIONS

In this research project, we addressed several challenges in archaeometry with the aim of expanding our understanding of the Phoenician-Punic archaeological world of Sardinia. A multi-analytical approach was employed to disclose hidden details about these ceramic shards. As a first step, the combination of XRD and optical microscopic analysis was essential for understanding the mineralogical content and structural features of these samples, thereby defining their main characteristics. By combining microtextural features and mineralogical assemblages, the samples were divided into three groups based on their phase content, which was influenced both by the raw materials and the firing environment. Some phases, such as hydro-silicates and carbonates, indicated lower heating (below 800°C), while the presence of pyroxenes and gehlenite suggested higher firing temperatures (over 950°C). Additionally, techniques such as SEM and XRF provide further information about point and bulk chemical composition of the samples. While macroscopic observations did not allow a robust distinction of the samples, the instrumental analysis indicated the presence of two distinct production groups. The first group, characterized by the occurrence of dacites and rhyolites, suggested the use of local raw materials from the Sulci region. The second group, with a carbonate-rich composition, pointed to different sources. The manufacturing techniques further distinguished the two groups: one group had an abundance of coarse temper, indicating a rougher production process, while the other showed a higher degree of purification, suggesting a more refined and controlled manufacturing technique. The presence of planar and oriented voids in the coarse-matrix group suggested the use of a pottery wheel, whereas the more purified samples, which lacked such orientation, likely indicated handcrafting methods. Chemical analysis, particularly p-XRF, corroborated the data obtained by other methodologies, reinforcing the division of the samples into two main compositional groups.

The analyses on the shards suggest that the Phoenician-Punic population of Sardinia predominantly produced their utilitarian ceramics locally, using basic production processes with minimal purification and under less controlled firing conditions. However, some samples, particularly those rich in carbonates and microfossils, indicate trade and commerce with other Mediterranean regions. These higher-quality, highly purified ceramics likely represent luxury goods exchanged with other colonies, as evidenced by the different raw materials.

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