

Preliminary Investigation into pre-Industrial Salt Production: an experimental approach

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Abstract – Salt, or halite, has been extracted from saline waters and sediments since the Neolithic period. Since then, the techniques used are natural evaporation (in salterns) and artificial evaporation (by applying heat from combustion). Within the study of archaeological salt-production areas, distinguishing between the two possible production processes allows for the inference of valuable insights into the socio-economic organization of the societies under investigation. Through the integration of mineralogical (XRD) and spectroscopic data (MIR and FAR-IR), this experimental work aims to identify geochemical markers of artificial evaporation processes in salt production from seawater.

- an anthropic surface extending for at least 70 m along the coastline, characterized by a substantial accumulation of processing waste (charcoal, pottery sherds, and lumps of burned soil).

I. INTRODUCTION

A. The archaeological question

This work has arisen from a specific case study: the proto-historic working area uncovered in 2008 along the shoreline of the Gulf of Baratti (Centro Velico, Piombino, Tuscany) [Baratti 2010, 2015, Baratti - Varenna 2022]. It was systematically excavated between 2008 and 2016 by the University of Milan, initially under the direction of C. Chiaramonte Trerè and subsequently of G. Baratti. In 2025, with funding from the Istituto Italiano di Preistoria e Protostoria, a multidisciplinary research team led by G. Baratti resumed investigations at the site, adopting a research strategy grounded primarily in archaeometric and geochemical methodologies.

The typology of the ceramic materials allows for the working area to be dated to the Final Bronze Age (ca. 10th century BCE), although radiocarbon dating on three charcoal samples is currently underway to confirm this chronology. This dating makes it one of the oldest known Italian sites of its kind. The site comprises:

- an anomalous fire installation, hypothesized to be a salt-working furnace based on parallels with quite similar installations identified in several *ateliers de briquetage*, in France.

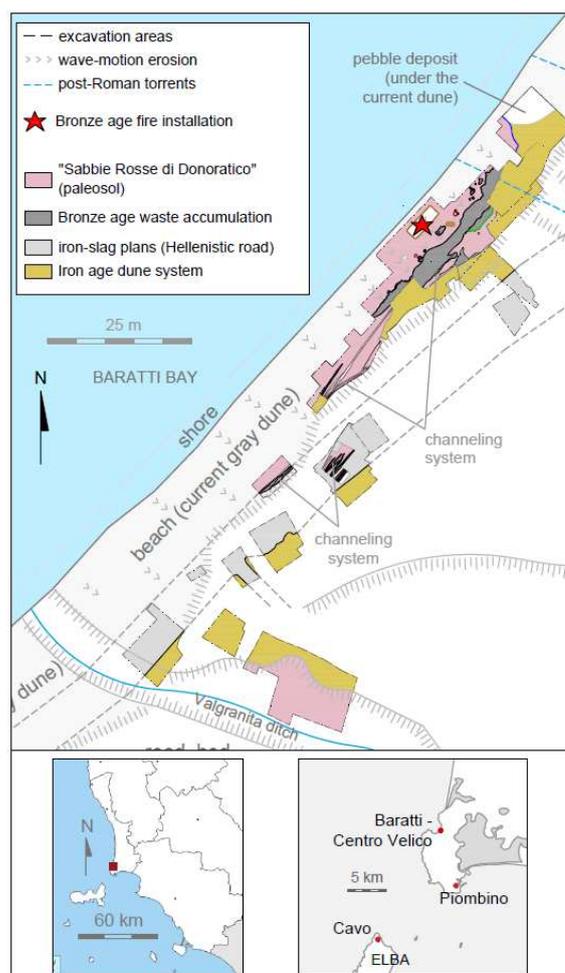


Fig. 1. Centro Velico site plan (Bronze age phase).

- a complex network of small channels running parallel to the shoreline - currently a unique feature - which may be interpreted as a saltern.
- remains of a series of other pit-based features of uncertain function.

The primary challenge presented by the study of this type of archaeological context lies in the possibility of reliably confirming the interpretation a facility for salt production or for salty food processing; more specifically, in demonstrating the use of artificially heat-induced evaporation techniques. Such interpretative issues are encountered at several other protohistoric site in Mediterranean area [Alessandri et al. 2019].

The aim of this work is to identify geo-chemical markers of these activities.

B. The sea salt production: natural and artificial evaporation

What is commonly referred to as “salt” can be chemically defined as sodium chloride (NaCl), potentially containing various impurities; from a mineralogical perspective, however, it is identified as “halite.”

The extraction of salt from seawater is complex, due to the generally higher presence of contaminants, in some cases with potentially harmful effects on health. On average, seawater consists of approximately 96.5% water, 2.5% various dissolved salts, and about 1% organic and inorganic particulate matter. The salts are primarily composed of ions of chlorine, sodium, sulphur, magnesium, calcium, and potassium, with concentrations that vary depending on geographic location and atmospheric conditions. All substances other than water and sodium and chloride ions can be considered contaminants. Some of these compounds (such as magnesium chloride, calcium chloride, magnesium sulphate, sylvite, bromine salts, and carnallite) can impart an unpleasant, bitter taste to the salt and so they are also known as “bittern salts” (or SWB = seawater bitterns) [Warren 2016, Abdel-Aal et al. 2017].

By employing slow evaporation processes, it is possible to remove a significant portion of contaminants. This is due to the phenomenon whereby different substances precipitate from solution at different concentrations [Vyas et al. 2022].

Salt has been extracted from saline waters and sediments since the Neolithic period [Harding 2021]. Since then, the techniques used are natural evaporation (e.g., solar and wind exposure in salterns) and artificial evaporation (by exposure to an artificial heat source, such as a fire); these methods may also be combined. In all cases, salt extraction involves the application of energy (from either natural or artificial sources) to a high-salinity aqueous solution (brine).

Along the Atlantic coasts, the rather harsh climate generally does not allow for salt production using the natural evaporation method, as the evaporation rates become negligible below approximately 10°C. In the Atlantic area, production facilities based on artificial evaporation techniques, such as the *briquetage* technique,

are in fact widespread [Harding 2021]. On the contrary, at least during the warm season, along the Mediterranean coasts it is possible to produce salt through entirely natural processes using the saltern method.

The *briquetage* involves at least four fundamental stages [Olivier 2024]:

1. Collection of brine (or of salted sediment, saltwater, or salt crusts),
2. Concentration (by natural or artificial evaporation) to obtain brine at the point of saturation (300 g/l),
3. Removal of the carbonate precipitate,
4. Shaping: further concentration using artificial heat within moulds, until compact salt cakes are obtained.

Although one might assume that the use of fire installations for artificial evaporation would be unnecessary in Mediterranean contexts, various evidence appears to challenge this assumption.

Based on indirect archaeological evidence, some authors [MONTAGNARI KOKELY *et alii* 2021] suggest that the exploitation of marine salt in Italy may have commenced in the Early Bronze Age, or even earlier, in connection with the diffusion of transhumant pastoralism and the development of techniques for processing food surpluses (notably cured meats and cheeses). Indeed, during these initial stages, extraction was likely conducted through exclusively natural methods, in simple forms organized at the household level, requiring minimal human intervention and leaving little to no evidence in the archaeological record.

Artificial salt production in Italy may date to the Middle–Late Bronze Age, as suggested by the *briquetage*-like evidence from the Gulf of Trieste, along the northern Adriatic coast, and from Isola di Coltano on the Tuscan coast. From the Final Bronze Age, sites interpreted employing pyrotechnological techniques for salt production increase markedly, especially along the coastal lagoons of central Tyrrhenian area. Here, the phenomenon expands rapidly during the Early Iron Age, peaking in the 8th century BC, before declining in the Archaic and Late Archaic periods, likely in favour of more complex saltern installations [ALESSANDRI *et alii* 2019; MONTAGNARI KOKELY *et alii* 2021; Alessandri – Attema 2021; BARATTI - VARENNA 2022; BULIAN *et alii* 2024].

From this scenario, a key question arises: why, between the Middle–Late Bronze Age and the early Iron Age, was salt along the Italian coasts processed through pyrotechnological methods?

Recent data suggest a strong correlation between this practice and the rise of proto-urban systems [Weller 2002; Cassola Guida - Montagnari Kokelj 2006; Cassola Guida 2016], wherein increasingly centralized and efficient control over key natural resources—such as salt—played a pivotal role in socio-economic development. In this framework, communities may have transitioned to more sophisticated salt production techniques, such as the *briquetage*, which yielded higher-

quality outputs compare to simple type salterns. Furthermore, the formation of hierarchically organized settlement networks, characteristic of proto-urban systems, would have facilitated trade across varying spatial scales. These networks provided structured exchange nodes governed by shared transactional norms, thereby enabling the circulation of high-value commodities—such as standardized salt cakes—that may have functioned as proto-currency within pre-monetary economies.

The abandoning of the *briquetage* system appears to coincide with the emergence of fully urban and state-level systems, which created the conditions for organizing production more efficiently through complex saltworks equipped with interconnected basin structures. These installations allowed for improved control over the fractional crystallization process and could yield abundant, high-quality salt. However, they required advanced knowledge of hydraulic engineering, which, as is well known, was developed in Etruria from the 7th–6th centuries BCE and subsequently adopted by the Romans. Moreover, although these advanced saltern installations also operated seasonally, they demanded year-round maintenance and control over extensive coastal areas. All of this may have contributed, at least from a certain chronological point onward, to the exploitation of enslaved labour and the development of forms of public control and monopoly, as is attested, for example, in the context of Archaic Rome [Capone 2015].

C. The archaeometric approach

Given this framework, it is clear it would be useful for archaeological interpretation to identify geochemical markers of artificial evaporation processes, to better understand the production cycles and the related socio-economic dynamics. These markers should be capable of long-term preservation and should be detectable on ceramic artefacts or within the sediments and soils from processing areas.

Unfortunately, not many works have been conducted on this topic. Until now, the study of halite crystallization as a marker of the process used in salt production has been conducted exclusively using SEM-EDS techniques on ceramic samples [Alessandri et al. 2019, 2024].

A more informative technique for characterizing halite crystals is far-IR spectroscopy, which allows for analysis even under conditions of micro-crystallinity and, at least in theory, would also enable the calculation of quantifiable indices of crystallinity - drawing on FT-IR methodologies already applied in the mid-IR range, for example, to calcite [Regev et al. 2010, Toffolo 2025].

Potentially, other mineral markers could also be investigated within the mid and far-IR range, such as the atomic order of aragonite [Toffolo et al. 2019, Toffolo 2021].

II. MATERIAL AND METHODS

The qualitative characterization of the mineralogical composition of salts is of fundamental importance from an archaeological perspective, as it contributes to a deeper understanding of the nature and function of the site under investigation. Techniques such as chromatography and atomic spectroscopy are commonly employed for the determination of salt content in aqueous media. In addition, X-ray powder diffraction (XRPD) is one of the most widely used methods for both the identification and quantification of mineral phases.

In this preliminary study, infrared spectroscopy was applied to characterize the different mineralogical phases present in salt samples prepared under various thermal conditions. Spectra were collected in both the mid-infrared (MIR) region (4000–400 cm⁻¹) and the far-infrared (FAR-IR) region (400–70 cm⁻¹) in order to obtain comprehensive information relevant to phase identification. Moreover, scanning electron microscopy images as well as the X-ray spectroscopic data coming from SEM-EDX investigation were used to confirm the presence of NaCl.

For the preparation of the experimental standards, seawater from the Bay of Baratti, located in front of the archaeological site of Baratti – Centro Velico, was used. Salt crystals were produced following a carefully designed analytical protocol. Five standards were prepared in the laboratory to simulate the temperature conditions believed to have been used in antiquity. Specifically, 200 mL of seawater were evaporated to dryness at temperatures of 25°C and 35°C, representing solar evaporation without artificial heat, and at 70°C, 80°C, and 90°C, simulating evaporation over a heat source.

To further simulate ancient salt production methods, additional evaporation tests were conducted under controlled temperature conditions using porous terracotta containers placed on embers, thereby closely replicating the *briquetage* technique.

The FTIR spectroscopy in the MIR region was performed using a Jasco FT/IR-4X spectrophotometer. Spectra were acquired using both KBr pellet and ATR (attenuated total reflectance) modes. The spectral resolution was set at 4 cm⁻¹, and each spectrum represented the average of 32 scans, ensuring a satisfactory signal-to-noise ratio. Component identification was achieved by comparing the acquired spectra with entries from both our internal spectral database and references from the scientific literature.

For the FAR-IR analysis, a Nicolet iS50 ABX FT-IR spectrometer was used.

The SEM-EDX analyses were conducted by a SEM Jeol JSM 5500 LV with a 500 IXRF Systems instrument.

The XRPD analyses were carried out on powdered samples using a Rigaku Miniflex 600 diffractometer, equipped with a Cu X-ray tube (operating at 40 kV and 15 mA). The scanning range was set between 5.00° and 90.00° 2 θ , with a step size of 0.02° and a scan speed of 3.00°/min.

III. DISCUSSION

A. Experimental observations

The experimental work has demonstrated that salt crystals formed in non-porous containers, such as glass, tend to rehydrate almost immediately after cooling, reverting to a state of hypersaline brine. In contrast, crystals produced in porous ceramic vessels appear to be more stable.

Another noteworthy observation is that crystals formed in ceramic containers tend to adhere strongly to the inner walls of the vessel, forming a hard, compact crust that is difficult to break. This finding appears consistent with data from previous experimental studies, suggesting that the extraction of salt cakes likely required the systematic breaking of the vessels [Campo 2012, Aranguren 2020].

A further observation confirms that the pyrotechnological process for producing salt cakes had to be initiated using hypersaline brines, rather than directly from seawater. This approach significantly reduces both processing time and fuel consumption, whereas the alternative proves unsustainable—for instance, achieving complete crystallization of just 200 ml of seawater in a ceramic container at 90°C requires approximately 4–5 hours.

These seemingly minor observations nonetheless allow us to infer significant insights into the organization of salt production in the Italian peninsula during the Protohistoric period, specifically in relation to the so called “opportunistic model”, according to which the *briquetage* activities may have been carried out by shepherding groups during the autumn months, when they descended from upland areas to temporarily settle along the coastal zones [Di Fraia 2006]. This model would nonetheless require a processing phase initiated at least by late summer, to obtain the brine necessary to trigger the pyrotechnological stage of the procedure. This observation is corroborated by our experimental data: without this preliminary step, the process would have demanded fuel quantities that were economically unsustainable, particularly in cases where a sustained level of production was sought that surpassed local subsistence requirements and could be mobilized within exchange networks. The requirement for this initial phase may account for the simultaneous presence, at the Centro Velico site, of installations indicative of *briquetage*-related operations and large-scale features—such as a system of channels—presumably employed for the concentration of brine.

Given this framework, it’s clear that a *briquetage* production area, also in case of an opportunistic model of use, have required a certain level of labour input and pretty regular activity during the summer months: it was necessary to establish the brine collection activities in proximity to stable settlements. For this reason, the opportunistic model has been challenged in relation to the Italian protohistoric context, based on the assumption that salt-working areas were located at a considerable distance from settlements, partly due to the risk of contracting malaria [Di Fraia 2006].

Notably, this is exemplified in the Gulf of Baratti, where the site of the Centro Velico is situated in close proximity

to a possible contemporaneous settlement located atop the nearby Poggio del Molino [Baratti – Varenna 2024]. At a comparable chronological horizon, a similar configuration appears to be identifiable in the Gulf of Trieste, where it’s suggested the possible existence of a coordinated salt production framework distributed among a series of functionally integrated sites [Cassola Guida 2016, pp. 39-40; Montagnari Kokelj *et alii* 2021].

In the proto-urban gestational phase (12/10th – 8th cen. BCE), it is plausible that artificial evaporation techniques were favoured by the interaction between their compatibility with seasonal pastoral mobility and the intensification of settlement centralization processes.

Future experimental work could expand the dataset by introducing further variations in salinity, the presence of contaminants, or other physical parameters, in order to better understand how these factors influence crystal formation, stability, and adherence to ceramic vessels. Such experiments would provide more nuanced insights into the range of conditions under which protohistoric salt production was feasible and help refine models of labour organization, seasonal scheduling, and resource management.

B. The archaeometric data

The qualitative analysis of the investigated samples was conducted through infrared spectroscopy—both in the mid-infrared (MIR) and far-infrared (FAR-IR) regions—as well as by X-ray powder diffraction (XRPD). The XRPD technique was specifically employed to support and confirm the interpretation of the FTIR spectra from a qualitative standpoint, providing complementary structural information.

The analysed samples predominantly consist of calcium carbonate (CaCO₃), which is present in two distinct polymorphic forms: calcite and aragonite. Each of these crystalline phases exhibits characteristic vibrational bands that were identified in the spectra. The diagnostic absorption bands for calcite appear at approximately 1798 cm⁻¹, 1420 cm⁻¹, 1082 cm⁻¹, 875 cm⁻¹, and 713 cm⁻¹. In contrast, the characteristic bands for aragonite are found at 1475 cm⁻¹, 856 cm⁻¹, 713 cm⁻¹, and 700 cm⁻¹.

Notably, a comparison of the spectra reveals some distinctive differences in the sample that was thermally treated at 90°C when compared to those treated at lower temperatures (as shown in Figure 2). Specifically, this sample exhibits the presence of absorption bands at 1442 cm⁻¹ and 874 cm⁻¹, which are indicative of aragonite and are absent in the other samples.

Furthermore, it is possible to characterize the specific type and degree of aragonite crystallinity through the calculation of the crystallinity index (CI). This parameter provides insight not only into the crystalline nature of the aragonite phase but also into the crystallization temperature, which in turn enables researchers to infer the approximate preparation temperatures of ancient salt-based materials in archaeological contexts.

Figure 2 presents a comparative overview of salt samples prepared at five different temperatures: 25°C, 35°C,

70°C, 80°C, and 90°C, highlighting the spectral variations associated with thermal treatment.

The nucleation process of aragonite and calcite in aqueous solutions has not yet been fully understood and is influenced by several factors, such as temperature, concentration, and the chemical composition of the solution. For instance, high magnesium concentrations – as in the case of seawater – can promote aragonite formation. It was therefore observed that temperatures approaching the boiling point favor the formation of aragonite [Jones 2017, Toffolo 2021]. This observation supports the hypothesis that thermal conditions play a crucial role in the polymorphic transition of calcium carbonate during salt crystallization processes.

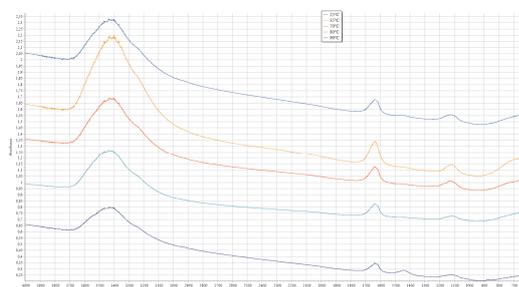


Fig. 2. IR spectra of salts treated at different temperatures

Additionally, the XRPD data enabled the confirmation of analcime phases, which had previously been identified through FTIR spectroscopy, thereby reinforcing the reliability of the spectroscopic interpretation.

In the far-infrared (FAR-IR) spectral region, characteristic peaks of aragonite are clearly distinguishable, particularly in the sample of salt that was allowed to evaporate at 90°C. These spectral features provide further evidence of the temperature-dependent formation of this specific crystalline phase.

In future studies, the application of this analytical approach is planned to be extended directly to archaeological ceramic samples, with the aim of better understanding the technological processes related to salt production and processing. This could provide valuable insights into ancient production techniques and contribute to the reconstruction of historical manufacturing practices.

To enhance the interpretation of the spectroscopic dataset, future research could employ statistical or machine learning techniques to classify and discriminate the spectra according to thermal treatment, crystallinity, or other mineralogical features. Such methods have been positively applied to other similar studies, specifically on several samples from Riparo Mochi and Gaban neolithic archaeological sites [Guglielmi et al 2024; Santiglia et al 2025]. This approach would allow for more objective and reproducible identification of temperature-dependent phase transitions, such as the formation of aragonite, and could be extended to archaeological ceramic samples to reconstruct ancient production techniques with higher confidence. Additionally, combining this approach with

expanded experimental datasets—including variations in salinity and other parameters—would create a robust framework for modelling the technological choices behind salt production.

IV. CONCLUSION

Through the combined application of analytical techniques – including X-ray powder diffraction (XRPD) and infrared spectroscopy in two distinct spectral ranges (mid-infrared (MIR) and far-infrared (FAR-IR)) – it was possible to identify the presence of aragonite and observe variations in the mineralogical composition among the different prepared standards. The study further demonstrated that higher temperatures promote the formation of aragonite, which can thus serve as a spectral marker for identifying salt subjected to boiling conditions.

This novel analytical approach, presented in the current work, has been specifically developed for the characterization of archaeological materials and soil samples from the Bronze age site of Baratti – Centro Velico, located in the province of Livorno, Italy. It represents a valuable contribution to the field of archaeometry, offering new insights into ancient salt production practices through the integration of mineralogical and spectroscopic data.

From a pure archaeological point of view, our experimental work can offer a contribution to a better understanding of the dynamics involved in protohistoric salt exploitation. Further experimental research is also planned, involving field-based trials to produce salt cakes using larger quantities of hypersaline brine.

Building on the findings presented here, future work aims to integrate experimental expansions and advanced analytical approaches, including machine learning classification of spectroscopic data. In particular, by comparing the data obtained from the analysis of the ceramics and sediments recovered at this archaeological site with those we have already examined (Riparo Mochi and Riparo Gaban), it is possible to further integrate the database we have developed on archaeological materials subjected to pyrotransformation. Applying these methods to larger-scale experimental trials and directly to archaeological ceramics could provide unprecedented insights into the technological strategies, environmental adaptations, and socio-economic organization of ancient salt exploitation.

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