



## Articolo

# Residue analysis of late Bronze Age ceramics from the archaeological site of Pilastrì di Bondeno (northern Italy)

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## Key words

- Terramare culture
- Middle-to-Late bronze age transition
- Pilastrì di Bondeno
- residue analysis
- wine

## Parole chiave

- cultura delle Terramare
- Transizione Età del Bronzo Medio e Recente
- Pilastrì di Bondeno
- analisi di residui organici nelle ceramiche
- vino

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## Summary

In this paper we present the results of the analysis of a first set of five fragments of pottery found at the late Bronze age site of Pilastrì di Bondeno, in the Po valley, northern Italian peninsula (15th-14th centuries BC). While the study of archaeological ceramics, in this area and for the involved period, has been traditionally addressed to building up chronological and cultural frameworks, GC-MS analysis revealed that the local pottery have absorbed and preserved organic residues that may reveal some aspects of the use of these vessels. Some vessels were used for the preparation and consumption of animal products, possibly broths. However, the most important aspect of the study is that it provides new insights on the production of fermented beverages and possibly wine in late Bronze age of northern Italy, and thus the most ancient date for the consumption of wine in the area in the transition between the Middle and the Late Bronze age. Moreover, the study shows the use of sulfur as possible coating agent, opening a discussion on its long-distance trade.

## Riassunto

In questo articolo si presentano i risultati preliminari di una prima serie di frammenti ceramici provenienti dallo scavo del sito di Pilastrì di Bondeno, nella valle del Po, Italia settentrionale (XV-XIV secolo aC). Mentre lo studio delle ceramiche archeologiche, in questa area culturale e nel periodo in questione, è stato, per tradizione, indirizzato soprattutto alla costruzione di sequenze crono-tipologiche e archeologiche, l'analisi mediante Gas-Cromatografia accoppiata a Spettrometria di Massa (GC-MS) indica che la ceramica dell'insediamento ha assorbito e conservato residui organici utili a rivelare alcuni aspetti delle funzioni alle quali i vasi erano destinati. Alcuni contenitori erano usati per preparare e consumare prodotti animali, probabilmente in forma di brodi. Tuttavia, l'aspetto più importante rivelato dallo studio è la produzione locale di bevande fermentate e probabilmente di vino nella transizione tra le età del Bronzo Medio e Recente dell'Italia settentrionale. Inoltre, lo studio indica l'uso dello zolfo come sostanza possibilmente usata per rivestire l'interno dei vasi, oppure antifermentativa, aprendo così la prospettiva di una antica rotta commerciale dalla Sicilia o dall'Italia centrale.

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## 1. Introduction

Almost forty years of investigations have shown that the study of organic residues trapped in the ceramic matrix can give information on different aspects of ancient life, and in particular the function of vessels, on the cooking and eating habits of the community who used them, on the body care, on ancient trade, and on technological aspects such as the lining and repair of ceramics (Evershed 1993, 2008; Garnier 2007; Pecci 2009; Regert 2011; Nigra et al. 2015).

Following such lines of investigation, this research tackles with the need of understanding the use and function of some ceramic containers from late Bronze Age sites of the so-called *Terramare* culture<sup>1</sup> of the Po floodplain, in the northern Italian peninsula (Bernabò Brea et al. 1997). So far, in fact, archaeologists have studied the pottery of this culture mainly in typological-chronological terms, with the purpose of refining the chronological and spatial-cultural implications of the sites. Many important aspects of the local technology remain undisclosed. In contrast, a fast developing emphasis on micro-economical adaptations, diets and food strategies, on the economic roles of household activities and female work as an historical agent made clear that certain shapes and sizes, or some technological features of prehistoric pots are particularly suitable to perform specific functions. However, in most cases, only with the study of the residues of the consumed substances it is possible to know the content of the ceramics and verify hypotheses regarding their use.

From residue analysis it is possible to understand, albeit with important limitations, what was eaten and what was stored, by determining the presence of products of vegetable or animal origin such as meat, milk, wine, oil.

## 2. The site of Pilastrì di Bondeno

The archaeological area of lot “Verri” at Pilastrì di Bondeno (Fig. 1; hereafter “Pilastrì”: 8 m asl, 44° 56' 50" N, 11° 17' 07" E) was discovered by an amateur scholar in 1979, and was first excavated in 1989 (Desantis and Steffè 1995) (Fig. 1). The village flourished, according to preliminary pottery evidence, between the end of the Middle Bronze (hereafter MB3) and the beginning of the Recent Bronze ages (RB1), i.e. ca. between 1500-1300 BC or immediately after (terms and chronology after Cardarelli 2010, 450); when settlements of the *Terramare* culture were still widespread and densely clustered in the central-western Po floodplain of northern Italy. The site of Pilastrì is explored for getting further information on the stratigraphy and the spatial organization of the settlement; a particular focus is placed on a detailed study of the ancient diets and the subsistence strategies, in their interaction with the environment and the traditional ways of life of the local communities of the 2nd millennium BC<sup>2</sup>.

1 As widely reported, the word “*Terramare*” derives from the local Italian dialects of late XIX century. It was used by farmers for the dark archaeological soils, rich in ash, carbon, bones and broken artefacts that they systematically recovered from the interior of moated Bronze age sites: such “*terramara*” or “*terra marna*” was supposed to be optimal for fertilizing their fields. The term was later adopted by archaeologists to indicate the sites, and eventually the whole cultural phases of the 2nd millennium BC of the region.

2 The excavation is a socially shared project, part of Memory & Earthquake, a social response to a prolonged seismic emergency in the Po plains in 2012. We work after a three-years agreement set in 2014 between the Soprintendenza Archeologica dell'Emilia Romagna (Superintendents L. Malnati, M. E. Minoja and F. Gambari), the municipality of Bondeno, the associations Bondeno Cultura and the local Archaeological Group directed by D. Biancardi; and the Universities of Padua, Ferrara, Modena and of Calabria. The project is coordinated by V. Nizzo (General Direction for Museums, Rome), while the society PETRA, Padua, is in charge of the field direction.

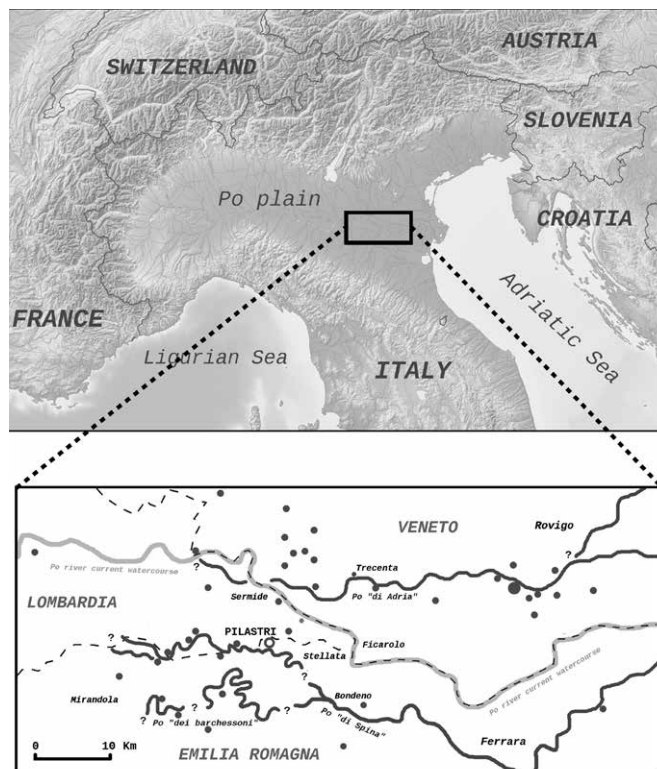


Fig. 1 - The site of Pilastrì di Bondeno and its location in the western Po plain (northern Italy). / Localizzazione del sito di Pilastrì di Bondeno nella Pianura Padana Occidentale (nord Italia).

At present, we cleared an area of ca. 220 sqm at inside the western edge of the settlement (Fig. 2), focusing on its late phases of life (Nizzo 2013, 2014; Nizzo et al. 2015a, 2015b). What remains of the dwellings are patches of irregular floors of silt constantly inter-fingered with minor alluvial layers, and lenses produced by the gradual decay of mud walls, surrounded by ditches filled with domestic refuse and post-holes. The micro-stratigraphical excavation of a rectangular wooden construction with a floor in yellowish silt enclose the remnants of at least two kilns superimposed in time one on top of the other; one of which seems to have been used with some kind of ceramic tube or *tuyere*, and abandoned after a high-temperature ruinous collapse. The floor of one of the kilns is made of alternating layers of ash and heavily burnt chips of animal bones. Well preserved artisanal workshops are quite rare in the *Terramare* culture and this unusual evidence will help us to understand



Fig. 2 - A general view of the excavated trench. / Vista generale dell'area scavata.

**Tab. 1** - *Pilastrì di Bondeno, summary of information on the analyzed ceramic samples. / Pilastrì di Bondeno. Riassunto delle informazioni sulle ceramiche campionate.*

ID	Location	Description	Sampled portion
C1	Q 108 N, 92F Trench B, US 399	Fragment of carinated cup. External colour: pinkish white (5YR 8/2). Inner surface: pinkish grey (7.5YR 7/2). Strongly reduced in fresh fracture: dark grey (5YR 4/1). Compact body, no pores; lime-like (?), mica, and fine angular grog. Lower body moulded, mouth made with a clay strip. Mouth diam. 18.5 cm, th. 1.2 cm.	Lower wall
C2	Q 106N, 94E Trench B, [34] US 617	Fragment of a slightly carinated cup with "horned" upraised handle. Surfaces: pinkish grey (5YR 6/2). Strongly reduced in fresh fracture: grey (7.5YR 5/0). Inclusions are lime-like angular particles and tiny grey grog chips, abundant mica. Lower body moulded. Mouth diam. 18.5 cm, th. 0.9 cm	Wall, under the handle
C3	Q 102N, 96E Trench B, US 618	Fragment of a hemispherical bowl with everted rim and small conical lugs. Surfaces: light gray (7.5YR 8/1). Reduced in fresh fracture: dark reddish grey (10R 4/1-5/1). Homogeneous body with very fine grog. Mouth diam. 17.5 cm, th. 0.6 cm. Mouth diam. 15 cm, th. 1,1 cm.	Wall fragment
C4	Q 102N, 96E Trench B, US 613, cluster B	Fragment of the lower body of a coarse medium-sized pot. Surfaces: light gray (5YR 8/2). Coarse ware with grog and some chaff. Base diam. 13 cm, th. 1.7 cm.	Base and wall fragment
C5	Q 108 N, 86E Trench B, US 587	Wall fragment of a large cooking pot, with horizontal ridge in relief. Outer surface: pink (7.5YR 8/4). Inner surface: strongly reduced, black (2.5YR/0). In fresh fracture: brownish yellow (10YR 6/6). Coarse ware with large angular grog, quartzite flakes and rounded lithics. Th. 1.6 cm.	Wall fragment

the development of specialized crafts in the mature and late stages of the Bronze age settlements of the Po plains.

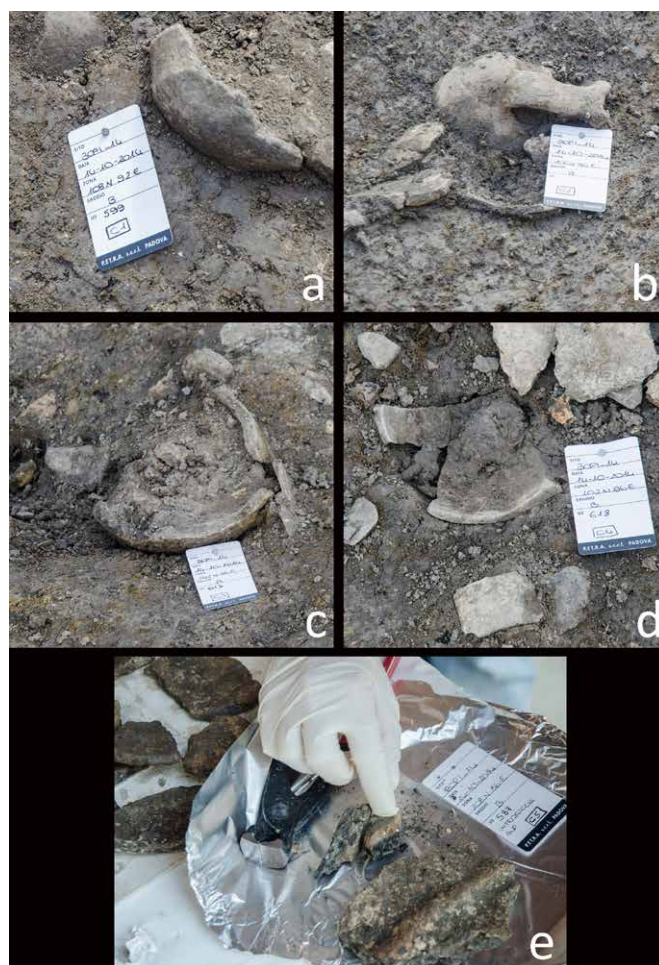
Within the ditches that surround the hut, and in the proximal open areas, we found large amounts of well preserved pottery and animal bones, discarded before the final abandonment of the site, together with lots of terracotta spindle whorls of various forms, bronze artefacts (knives, small daggers and a sickle), fragments and chunks of worked bone and stag horn, and broken and lost amber beads. The five pottery fragments here analyzed were selected because they were well preserved and typologically meaningful, as well as because they came from controlled stratigraphical units (outer dumping layers aside the wooden erection).

### 3. Materials and methods

A total of five samples was selected for testing how suitable was this type of analysis. The ceramics were sampled on the trench, taking fragments mostly from the wall, before they were washed (Figs. 3 and 4, Table 1); then they were powdered and analysed using different extraction methods aimed at identifying the different compounds preserved in the ceramic matrix: The so called total lipid extract was obtained on 2 g of ceramic powder following the procedure described by Mottram et al. (1999); its hydrolysis was performed following Pecci et al. 2013a; for the identification of wine markers, an extraction with KOH was performed on 500 mg of sample following Pecci et al., (2013b); finally, on some samples, an hydrolysis on the solid residue after the total lipid extraction was performed following Pecci et al. (2013a).

All the extracts were derivatised by adding 25 ml of N,O-bis(-trimethylsilyl)trifluoroacetamide (BSTFA, SigmaAldrich) at 70 °C for 1h.

The analysis was carried out using a gas chromatograph CP3800 (Varian, Walnut Creek, CA, USA) equipped with a DB5 30 m, 0.25 µm film thickness fused silica capillary column and a mass spectrometer Saturn 2000 (Varian, Walnut Creek, CA, USA) operated in the electron ionisation mode (70 eV). The mass range was scanned in the range of m/z 40-600. The GC oven temperature was held at 50 °C for 1 min, then increased at 5 °C/min up to 300 °C and held isothermally for 10 min.



**Fig. 3** - *The analyzed ceramic samples, on the field (Samples C1-4, a-d) and in the archaeological lab (Sample C6, e). Samples are later cleaned and pulverized in the chemical laboratory. Photographs by Giulio Pola. / Campioni di ceramica sul sito (campioni C1-4, a-d) e nel laboratorio archeologico (campione C5). I campioni vengono successivamente puliti e polverizzati nel laboratorio chimico. Fotografie di Giulio Pola.*

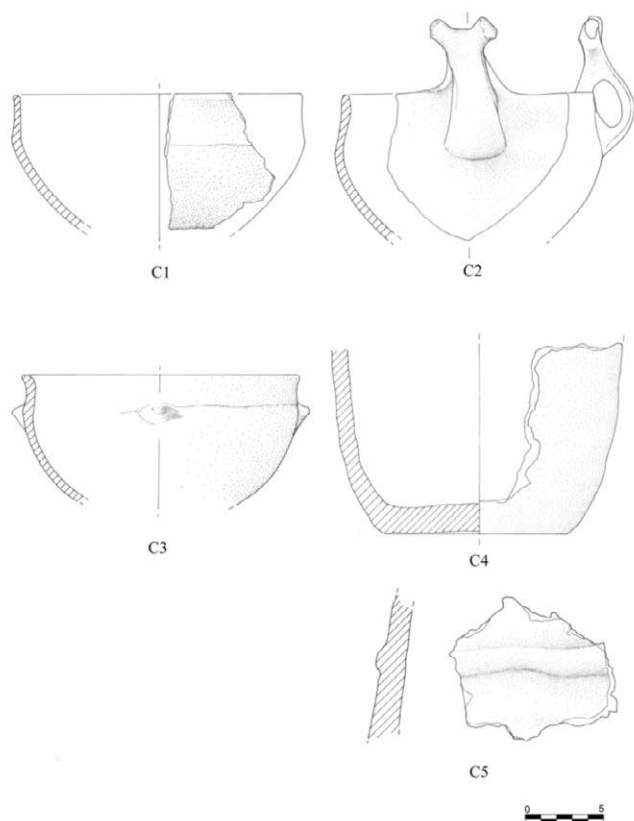


Fig. 4 - The five ceramic samples analyzed in the present study (drawing S. Tinazzo). / I cinque campioni analizzati (tavola di S. Tinazzo).

### 3. Results of the analyses

#### Sample C1

The results of the chemical analyses of Sample C1 (a carinated cup, traditionally considered a drinking vessel, Fig.5)<sup>3</sup> show the presence of tartaric acid, succinic, malic, maleic and hydrocinnamic acids (Fig. 5). Although other fruits, for example tamarind, may contain tartaric acid (Barnard *et al.* 2010), on the basis of the residues identified, and the presence of grape pips in the area (Mercuri *et al.* 2006) the vessel analysed may have contained wine. Sulfur is evident in the forms S<sub>6</sub>, S<sub>7</sub> and S<sub>8</sub> in the chromatogram of the total lipid extract. It is possible that sulfur was applied internally as a waterproofing agent of ceramics (as reported by Garnier *et al.* in 2011)<sup>4</sup>. To confirm this is the absence of resin or pitch, generally used to waterproof containers and found in Sample C3. Besides coating, sulfur might have been applied to wine as a preservative, anti-microbial agent and anti-oxidant, to reduce browning and delay fermentation, particularly in white wines (Jacobson 2006: 5, 1, 148; Henderson 2009; Rose 1993), or to disinfect the wine or the vessel. The absence of syringic acid, considered the marker of red wine (Guash Jané *et al.* 2004; Barnard *et al.* 2011) suggests the wine was white and could be related with the second hypothesis. The results of the analyses of this vessel are consistent with the fact that the vessel was a cup, probably used to drink.

3 This cup, like many others found at Pilastrì and the cup of Sample 2, might have had a upraised handle, possibly of the "horned" type, not preserved in the part of the vessel recovered in the dig.

4 Preliminary analyses carried out with portable XRF on the interior and exterior surface of the vessel allow to verify that sulfur is only present in the interior surface of the cup.

#### Sample C2

In Sample C2, belonging to slightly carinated cup with short "horned" handle (Figs. 3 and 4); one of the most common forms at Pilastrì and a distinctive chronological marker of the early RB age of the region) there are very few residues. Animal origin products are indicated by traces of cholesterol and stearic acid. There are also long-chain hydrocarbons and alcohols (C<sub>24</sub>-C<sub>30</sub>) (indicated by \* in the Figures) compatible with waxes or contamination (Fig. 6). Tartaric acid is absent, but malic, maleic, hydrocinnamic and succinic acids are there, being especially abundant maleic acid. This may indicate the presence in the cup of fermented substances, but different from wine, possibly derived from a different fruit. Therefore, the hypothesized function of drinking is not confirmed, but cannot be excluded.

#### Sample C3

In Sample C3 (Fig. 3), signals are very low, as if the vessel had contained almost nothing identifiable with the analysis carried out. There are only some markers of possible fermentation (maleic, succinic and hydrocinnamic acids) in extraction c. However, due to the absence of tartaric acid, it is not possible to state whether the vessel had contained wine.

#### Sample C4

As for Sample C4 (Figs. 3 and 4d), from the lower body of a coarse medium-sized pot, the presence of cholesterol in the hydrolysis of the total lipid extract and of relatively high amounts of stearic acid, indicate the presence of animal origin products in the vessel (Fig. 7). There are also other acids, such as succinic, oxalic and

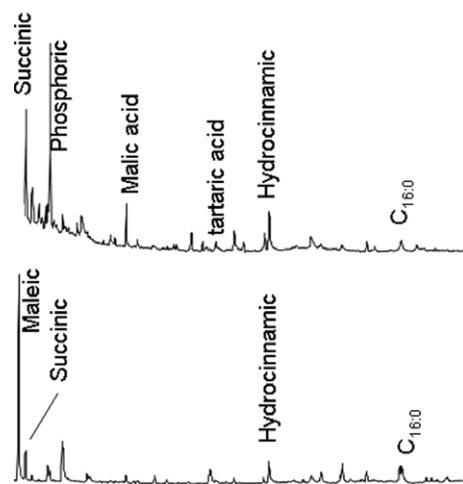


Fig. 5 - Particular of the chromatograms obtained with the analysis of the extract (c) to identify wine residues and extract d. of Sample C1. / Particolare del cromatogramma dell'analisi dell'estratto (c) per identificare i marcatori del vino e dell'estratto d. del campione C1.

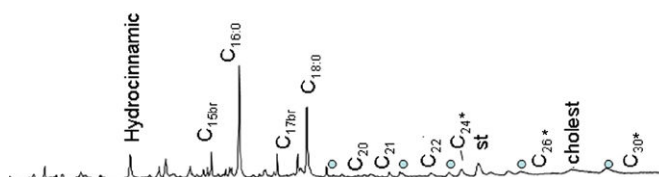


Fig. 6 - Chromatogram of the hydrolysis of the total lipid extract of Sample C2. / Cromatogramma dell'idrolisi dell'estratto lipidico totale del campione C2.

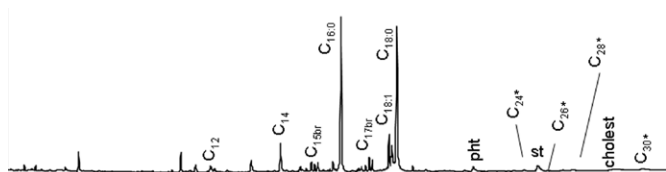


Fig. 7 - Chromatogram of the hydrolysis of the total lipid extract of Sample C4. / Cromatogramma dell'idrolisi dell'estratto lipidico totale del campione C4.

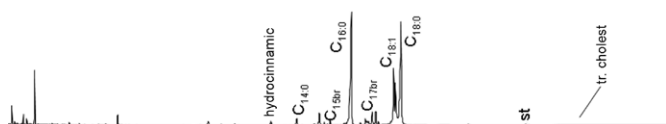


Fig. 8 - Chromatogram of the hydrolysis of the total lipid extract of Sample C5. / Cromatogramma dell'idrolisi dell'estratto lipidico totale del campione C5.

hydrocinnamic acids which can be linked to fermented products. However, we do not consider them as markers of specific food/beverage. Long-chain alcohols ( $C_{24}$ - $C_{30}$ ) compatible with waxes, and possibly deriving from post depositional contamination, are present in the hydrolysis of the total lipid extract.

### Sample C5

Judging upon the dark carbon-like enrichment of the inner surface (Fig. 3), and the absence of soot on the exterior, this coarse pot might have been used for cooking by the means of red-hot pebbles and sherds inserted from the mouth. The abundance of fats in this sample (especially of animal origin) may support this hypothesis. In fact, in Sample C5 – a fragment of a large coarse ware restricted cooking pot – we found traces of cholesterol in the hydrolysis of the total lipid extract, together with high stearic and palmitic acids, indicating the presence of abundant animal products in the sample (Fig. 8). In the sample there are traces of waxes indicated by the long-chain alcohols and hydrocarbons. In the extract for the identification of wine markers, traces of dehydroabietic acid are present. They are markers of Pinaceae products probably used for coating the interior of the vessel.

The application of a resinous coating is possibly related to the need of waterproofing the vessel. This practice, although well known for Roman amphorae, has been testified by residue analyses also on cooking, storing and serving vessels of different periods (Colombini et al. 2005; Pecci 2006, 2009; Zifferero et al. 2011). The data support the hypothesis that the pot was used to cook broths based on animal products, possibly conditioned with fermented beverages or sauces.

## 4. Discussion

Although only a very limited number of ceramic fragments was analyzed, the study allows to propose some hypotheses on the use of the original late MB-early RB age ceramic containers. For example, the abundant animal products in Sample 5 witness the use of the vessel for boiling broths and/or soups. The carbonized layer on the inner wall suggests the possibility that the pot was not set on a fireplace, but some stones and/or ceramic sherds, previously red-heated outside, were inserted in its boiling or broiling fluid contents. This hypothesis needs to be verified through further dedicated studies.

Animal products are also present in Sample C4, a coarse pot, that might also have been used for the preparation of broths or soups, such as those prepared in Sample C5, perhaps conditioned with fermented beverages/sauces. More animal products and resi-

dues of possible fermented beverages were recorded in Sample C2, thus suggesting uses similar to Sample C4. The absence of residues in the bottom of the bowl C3 may indicate that this container was used to drink fermented beverages different from wine. At present, however, the most relevant evidence are the probable markers of wine detected within Sample 1.

## 5. Main results

The study of the first sample (carinated cup C1), in fact, hints to the presence of wine or derivatives. This might be the earliest material evidence of wine consumption in the northern Italian peninsula. While the agriculture of the *Terramare* sites inherited the traditional local neolithic patterns (being based on different forms of wheat, barley, millet and flax, with a limited contribution leguminous plants), they also exploited a wide range of wild resources, among which chestnuts (*Castanea*), cornelian cherry (*Cornus mas*), elderberry (*Sambucus ebulus/nigra*), blackberry (*Rubus* sp.), wild cherries of *Prunus* sp., wild apples (*Malus sylvestris*), hazelnuts (*Corylus avellana*) and wild grapevine (*Vitis vinifera sylvestris*) (Nisbet and Rottoli 1997; Mercuri et al. 2006; Marchesini et al. 2010).

Some of these fruits, including cornelian cherry, could be transformed in fermented fluids and beverages, but their chemical indicators are not detailed in the published records.

It is generally assumed that grapevine was one of the arbustive species of economic interest that during the Sub-boreal period (ca. 3800-700 cal. BC) gradually spread under the intensified exploitation by local communities. In spite of a general difficulty of distinguishing wild from domesticated pip forms, we know that in northern Italy *Vitis vinifera* was gradually domesticated during the Bronze age or IInd millennium BC (Forni 1996; Zohary and Hopf 2000; Marchesini et al. 2010: 236).

Both the find of archaeobotanical macro-remains and variations in pollinic spectra reveals that the exploitation of *Vitis vinifera*, in important sites, intensified just in the transition MB3-RB1 (Cardarelli 2014: 106, with further references) in landscapes already heavily impacted by farmers and deforested through slash and burn practices (Cremašchi 2010). In one of the best documented sites, observing that by 1400 BC seeds of *Vitis* became more common than stones of *Cornus*, Mercuri et al. (2006: 55) proposed that "...as both fruits are used to prepare fermented drinks, the inversion could suggest a cultural shift in alcoholic drinks from cornelian cherry wine to grape wine". They also hypothesized that the new technology of vinification might have been inspired by contacts with Mycenaean traders.

For contemporary settlements, Cardarelli (2014: 843) specifically suggests an established production of wine and even a ritual, formalized use of the drink in funerals. Thus, the discovery of traces of wine in Sample 1 fits very well, and consolidate the emerging picture of the earliest wine-making practices in the northern part of the peninsula.

The presence of sulfur in the same cup (Sample C1) deserves further comments. Recently, the identification of sulfur in several vessels from the Iberian oppidum of Puente Tablas in Jaén, Spain has been interpreted as related to rituals carried out at the sanctuary of the site, where it would have been used as a purifying and healing agent (Parras et al. 2015). However, these vessels did not show other residues. At Pilastrì, preliminary analyses suggest that sulfur is present only in the inner part of the vessel. However, further studies are in progress to verify this datum. In this case, being sulfur present together with wine or its derivatives, it may have been used to waterproof the container or to relent the spoiling of wine, although this issue, too, needs further investigation. Also Sample 5 was waterproofed, but in this case with *Pinaceae* resins, a much more common practice than the use of sulfur and abundantly testified in the peninsula by many finds, since Etruscan times to the Middle Ages. The use of *Pinaceae* resin/pitch in cooking vessels such as sample 4 has been observed ethnographically by Shiffer et al. (1994) among

the Kalinga in the Philippines, and, as suggested by the authors, could be related to the possibility of reaching high cooking temperatures as well as reducing the amount of fuel consumed.

As for the sulfur, several protohistoric communities of the Italian peninsula were quite familiar with hot springs and fumaroles, sometimes flowing in caves, whose waters were rich in sulfur. Such sites were often frequented for cultural reasons (among others, Grifoni Cremonesi 2007). However, as far as we presently know, deposits of high purity, marketable sulfur were exploited and transformed mainly in Sicily since the Early-Middle Bronze age: between the XVIIth-XVth century BC, Sicilian sites like Thapsos, Cannatello and the sanctuary-cum-sulphur extraction workshop of Palma di Montechiaro, Monte Grande were probably involved in tramping-like exchange routes. Sicilian products like alum, sulphur, rock salt were exchanged for prestigious objects and valuable materials imported from Aegean and Cypriot sources (Russel 2011). In Sicily, sulphur seems to have been used as fuel, possibly for cultural practices, since the EB-MB age culture of Castelluccio (see Ianni 2009: 249). Sulphur, melted in workshop areas such as that of Monte Grande (Castellana 1998, 1999; Caminnecki 2014) and refined for export in standardized preforms, could be used in metallurgy and possibly in vinification processes (Castellana 2000: 167).

Being the ceramic vessel typically local, as we cannot verify the origin of the sulfur at this stage, it is possible to hypothesize that the sulfur identified in Sample 1 came from this area. The trade of refined sulfur to northern destinations could have been supported by the terminals of the amber trade along the Po river entrepôts and workshops. Eventually, in such light, the residues detected in the pots of Pilastrì suggest that in the Po floodplains, in the XIVth century BC, pottery making, vinification, the trade and use of vegetal resins and of sulphur might have been technically intertwined and linked, in turn, to wider production and exchange networks. This – if and when this emerging picture will be substantiated by new data – might help us to better focus a crucial aspect of the economy of the local complex societies. Ultimately, the residues analysis shows that even the study of a small part of the abundant materials available in the Terramare sites might open new pathways to be investigated, leading to quite different, historical scenarios.

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