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Article / Articolo

Millet porridge and red fruits. Organic residue analysis of cooking wares from an upland site in the Dolomites (Busa delle Vette, 1850 m a.s.l., Belluno, Northern Italy)

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Key words

- Organic residue analysis
- Gas chromatography-mass spectrometry
- Biomarkers
- Cooking practices
- UPLanD project
- Busa delle Vette
- Dolomites

Abstract

Twenty-four (24) samples of cooking wares from the excavation of the hut at Busa delle Vette (Parco delle Dolomiti Bellunesi, Belluno Province, Veneto, Northern Italy) have been considered for organic residue analysis via GC-MS in order to better understand high mountain daily life in terms of food consumption and cooking practices during Early Middle Age (5th – 9th century AD). The results show abundant biomarkers and complex organic mixtures of animal and plant origin.

Parole chiave

- Analisi residui organici
- Gas cromatografia-spettrometria di massa
- Biomarcatori
- Pratiche culinarie
- Progetto upland
- Busa delle Vette
- Dolomiti

Riassunto

Ventiquattro (24) campioni di ceramica da cucina provenienti dallo scavo della capanna di Busa delle Vette (Feltre, Parco delle Dolomiti Bellunesi, Veneto) sono stati considerati per l'analisi dei residui organici tramite GC-MS al fine di comprendere meglio consumi e pratiche culinarie in alta quota durante l'Alto Medioevo (V – IX secolo d.C.). I risultati mostrano abbondanti biomarker e complessi composti organici di origine sia animale che vegetale.

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Introduction

Twenty-four (24) samples of cooking wares (Table 1) from the excavation of the hut at Busa delle Vette (1858 m a.s.l.; Parco delle Dolomiti Bellunesi, Belluno Province, Veneto, Northern Italy; fig. 1) have been considered for GC-MS analysis of the amorphous organic residue absorbed and kept in the ceramic pores (Roffet-Salque et al. 2017) with the aim of better understanding high mountain daily life in terms of food consumption and cooking practices in a still understudied historical period. All the vessels date to the Early Middle Age (5th – 9th century AD), corresponding to the phase of most intensive use of the structures, and belong to the typology of the *olla* showing blackened sooting traces on the surface (Ferronato 2019, fig. 2). For the molecular GC/C/IRMS and stable isotope analysis of further *olle* samples from the same context, see Mileto et al. 2023. On the archaeological context, see Cavulli et al. 2017.

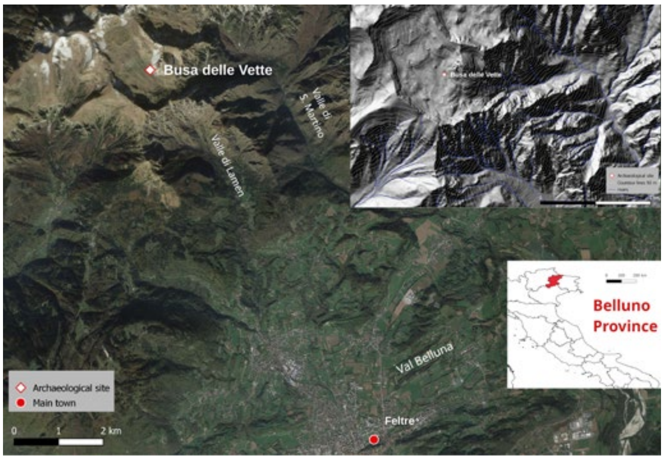

Fig. 1 – Location map of Busa delle Vette. / Fig. 1 – Localizzazione del sito Busa delle Vette.

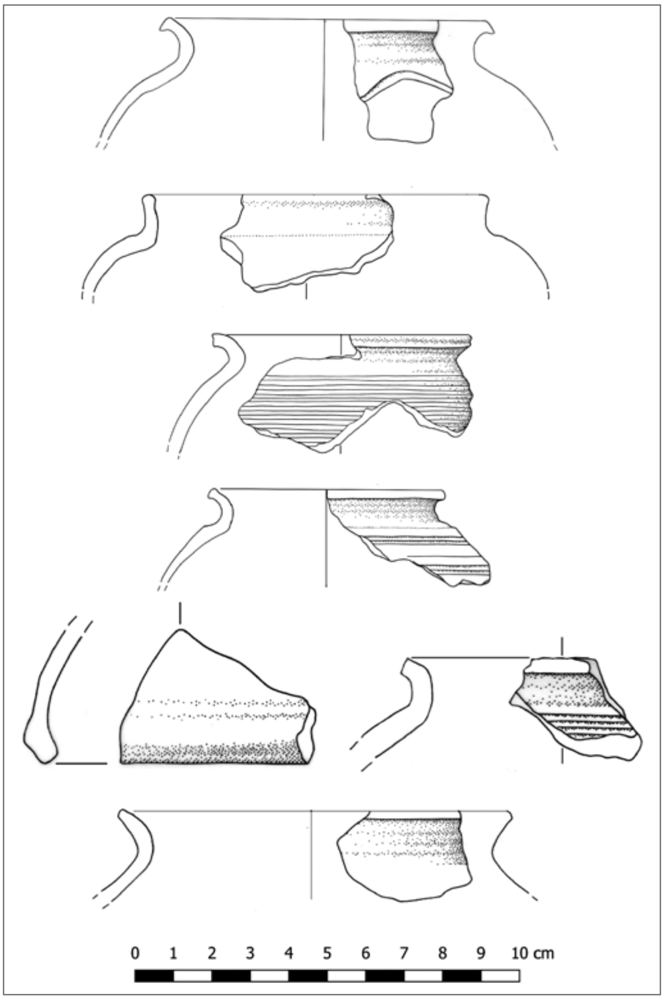

Fig. 2 – *Examples of analysed ceramic samples (drawings by Ferronato 2019). /* ***Fig. 2 –*** *Esempi di campioni analizzati (disegni di Ferronato 2019).*

The archaeological context of Busa delle Vette

In the first few years (2013-2015) the archaeological research at Busa delle Vette focused on topographic and archaeological surveys and on the excavation of a dry-stone enclosure complex. The dating of charcoal and faunal remains found in test pits suggest that the enclosures were in use from the 10th to the 15th centuries. A second phase of the project (2016-2018) focused on a small mound a short distance from the enclosures. The stratigraphic excavation in this location (called Area B) revealed a partially sunken hut enclosed by a stone heap gently degrading and with abrupt limits. The internal area of the hut was filled with a thick black organic layer, a homogeneous soil difficult to differentiate on chromatic and granulometric basis in the field. This layer looks very similar to what is micromorphologically known as "dark earth" in medieval urban contexts (cfr. Brogiolo et al. 1988; Gelichi 2000), occasionally identified in mountain contexts as well (Nicosia & Devos 2020; Nicosia et al. 2017; Fondrillon 2009; Cammas et al. 1995; Carver 1987, pp. 40-46; Macphail 1981; 1990). The upper part of the hut was probably made of timber, and the roof was covered with limestone slabs. The artefacts uncovered and the radiocarbon dates reflect a long-lasting occupation starting from the 5th to the 9th century. Considering the elevation of 1850 m a.s.l., the site was seasonal occupied in summer and autumn.

The functional interpretation of the context is quite surprising because, despite the historical use of the area as grazing land, the amount of pottery and animal bones (about 95% sheep/goat) does not seem to be compatible with a shepherd occupation. The presence of pig bones, fragments of glass, bone combs, a bronze brooch, an iron chain, carved bone, numerous metal objects and a miniature eagle head (decorative element of a ceramic object) suggest a more complex and multifaceted function for this structure. Furthermore, the presence of Lombard military garments seems to indicate the presence of armed individuals or even elite members at the site during the 7th century (Martinelli et al. 2024).

Materials and Methods

The in 2017-18 unearthed samples come from the excavation area B (US 303, 304, 315 and 322) at Busa delle Vette. After surface-cleaning and crushing the sherds, two extractions protocols ([A] and [B]) have been applied (to all samples but VTres 2, 3, 4, 5, 6, 9, 10, 16, 19, 25) in order to extract lipids (Mottram et al. 1999; Pecci et al. 2013a: [A]), and other small organic acids (alkaline extraction according to Pecci et al. 2013b: [B]) from the powdered samples.

A. 2 g samples are extracted in 50 ml chloroform/methanol (2:1 v/v) for 2×15 min in an ultrasonic bath. The samples are then centrifuged for 10 min and the supernatant is evaporated to dryness under a constant stream of nitrogen. The samples are saponified with 2 ml sodium hydroxide solution (2M in MeOH) for 1 h at 70° C. After cooling, it is acidified with 15 drops of concentrated hydrochloric acid, the pH value is checked (pH 1). It is then extracted twice with 3 ml of chloroform. Then the solvent is removed under a constant stream of nitrogen and the sample is transferred to a sample vial with two times 50 µl of chloroform. The chloroform is removed under a constant stream of nitrogen and the sample derivatized

***Tab. 1 –** List of the analysed samples. / **Tab. 1 –** Lista dei campioni analizzati.*

Sample_ID	excavation area	Q	US	weight g	Description
VTres1	B	104/500	315	9,6	common ware shoulder
VTres2	B	106/502	315	10,6	common ware wall
VTres3	B	106/503	315	4,9	common ware wall
VTres4	B	101/503	315	12,5	common ware neck
VTres5	B	104/504	315	8,6	common ware shoulder/neck
VTres6	B	105/502	315	7	common ware comb-decorated wall
VTres7	B	103/500	315	16,1	common ware wall
VTres8	B	105/500	315	10,7	common ware wall
VTres9	B	101/504	315	9	common ware wall
VTres10	B	102/505	315	8,6	common ware wall
VTres11	B	103/504	315	7,8	common ware shoulder/neck
VTres12	B	103/503	315	33	common ware wall
VTres13	B	102/501	315	18	common ware wall
VTres14	B	102/505	304	4	common ware shoulder/neck
VTres15	B	101/504	303	5	common ware shoulder/neck
VTres16	B	103/503	303	3,3	common ware wall
VTres17	B	102/504	303	6,4	common ware comb-decorated
VTres18	B	103/505	303	8,7	common ware wall
VTres19	B	104/502	322	8	common ware comb-decorated wall
VTres20	B	102/504	304	18,1	common ware wall
VTres21	B	103/504	315	4,9	common ware bottom with external leafed surface
VTres23	B	102/501	315	5,8	common ware wall/bottom
VTres25	B	103/503	315	5,8	common ware bottom
VTres26	B	105/502	315	5,5	common ware wall/bottom

with 25 µl of BSTFA at 70° C for 1 hour. After the addition of 75 µl n-hexane and 5 µl internal standard (dotriacontane in hexane, for concentration see respective table of results under http://dx.doi.org/10.17169/refubium-40223), the samples are analysed by GC-MS.

B. 500 mg sample is extracted in 3 ml potassium hydroxide solution (1M in H2O) for 90 min at 70°C. The samples are then centrifuged for 10 min and the supernatant is acidified with 15 drops of concentrated hydrochloric acid, and the pH value (pH 1) is checked. Then the samples are shaken intensively with 3 ml ethyl acetate for 2 min and centrifuged for 10 min. This step is repeated 3 times in total. The supernatant (organic phase) is concentrated under a constant stream of nitrogen and transferred to a sample vial with two 50 µl portions of ethyl acetate. The solvent is then evaporated off and the sample is derivatized with 25 µl of BSTFA at 70° C. for 1 hour. After the addition of 75 µl n-hexane and 5 µl internal standard (dotriacontane in hexane, for concentration see respective table of results under http://dx.doi.org/10.17169/refubium-40223), the samples are analysed by GC-MS.

All samples were analysed using Agilent 7820A GC system and Agilent 5977 MSD, equipped with HP5-MS capillary column and EI as ion source. Initial oven temperature 50°C with a temperature ramp of 5 °C/min to 320 °C and hold time of 10 min. For this method a split ratio of 1:10 has been used.

Preliminary results

In general, the samples show a good preservation of organic residues, which has allowed the identification of several biomarkers. All the analysed *olle* show a very similar distribution of identified fatty acids. Nevertheless, the detected markers are mostly low-specific long-chain saturated fatty acids, present in both animal and vegetable products, e.g., palmitic (C16:0), stearic (C18:0) acids. Myristic acid (C14:0) may be related to dairy products but also to plant seed fats (oil) (Irto et al. 2022). Margaric acid (C17:0), an odd carbon number saturated fatty acid, may be related to bacterial degradation of ruminant fats (Evershed et al. 2002). The short/medium chain fatty acid caproic (C6:0), caprylic (C8:0) and capric (C10:0) acids, together with the predominance of palmitic and stearic acids, are characteristic for animal ruminant fats (Regert 2011) and could be found in all samples. Animal sterols, i.e., cholesterol and cholestane, have been detected in three samples (VTres1 VTres7, and VTres14). In the samples VTres14 and VTres15 deoxycholic acid, a byproduct of cholesterol, has also been detected. Even though cholesterol also may originate from (skin) contamination, cholesterol and cholestane may indicate an animal origin of the compounds (Whelton et al. 2021; Drieu et al. 2020b; Lundy et al. 2023) In general, cholesterol does not preserve well in archaeological ceramics due to degradation through oxidation and heating with fatty acids (Hammann et al. 2018). However, the processing of animal fats in these *olle* have been confirmed by the compound-specific isotopic analysis of the C16:0 and C18:0 fatty acids by Mileto et al. 2023.

The deoxycholic acid, a livestock-derived steroid, also is a faecal marker for ruminants, i.e., cattle and sheep (for example see Lerch et al. 2022 on the Mesolithic Stubai Alps): Thus, finding of deoxycholic acid might also indicate contamination from faeces deposition in the sediment of the burial environment.

The predominance of carboxylic acids, i.e., adipic, benzoic, glycolic, glutaric lactic, levulinic, oxalic, pyruvic, succinic acids, is attributable to fermentation/oxidation processes. Small carboxylic acids also result as breakdown products by heating carbohydrates (Matheson et al. 2022). However, fumaric, malonic and malic acids together with the latter discrete fermentation markers, may point to the processing of fruits in the vessels (Lundy et al. 2023). Furthermore, in 9 *olle* (VTres2, VTres3, VTres4, VTres6, VTres7, VTres12, VTres13, VTres15, VTres23, VTres25, VTres26) syringic acid, which derive from malvidin giving grapes and wine their red color and is therefore considered as a (controversial) indicator for red grapes/wine, has been detected (Guasch-Jané et al. 2004; Drieu et al. 2020a). It should be considered that free syringic acid is not only derived from malvidin but is also present in other plant products, i.e., cereals like barley and wheat, and may even occur in soil (Barnard et al. 2011). The adopted extraction protocol does not allow to distinguish syringic acid deriving from malvidin from free syringic acid (Pecci et al. 2020). Nevertheless, the not ubiquitous presence of syringic acid in the samples may exclude contamination from the soil. Other fruits than grapes characterised by a high amount of malvidin are blueberry (Barnard et al. 2011). The absence of tartaric acid, a polar molecule characteristic for wine, may be due to its high solubility in water. On the other hand, this may also point to other fruits than grapes, whereby malic acid is higher than tartaric acid, i.e., apple, plum, cherry or peach (Lundy et al. 2023 in Mediterranean context). Malic acid is not only related to fruits but also from the other plant products, such as Brassicas or leeks (Lundy et al. 2023). Among the carpological material from the site plum pits (*Prunus domestica insititia*), strawberry, juniper and also grapevine are attested (Castiglioni & Rottoli 2019). Blueberries are also common at these altitudes. However, from an historical and archaeological point of view the consumption of wine or the use of (red) wine or grapes juice in cooking can be corroborated by the military nature of the occupation of the high-mountain site as a strategic place and/or as traveller stopping place during the Early Medieval period, as Lombard soldier's garments and the huge amount of ceramic may confirm. Furthermore, aromatic compounds in the samples, e.g., cinnamic (VTres4, VTres5, VTres9, VTres10) and vanillic acid (VTres3, VTres4, VTres5, VTres6, VTres17, VTres18, VTres25, VTres26), are also contained in wine. Nevertheless, phenolic compounds like vanillic and syringic acid may also originated from decomposition of woody tissue, i.e., natural and anthropogenic alteration and degradation of lignin, and be indicative of lignin pyrolysis (Tamburini et al. 2016; Huber et al. 2022). Small organic aromatic compounds like phenol/benzenol, which is almost ubiquitous in the samples, may relate to coniferous resins. Nevertheless, caution is required in the interpretation of phenolic plant compounds (Whelton et al. 2021; Huber et al. 2022).

Short and medium chain and odd-carbon number fatty acids, i.e., valeric (C5:0), enanthic (C7:0), pelargonic (C9:0), lauric (C12:0) acids, could be detected and may be related to degraded plant fats (Irto et al. 2022). Salicylic acid may be also related to plants and plant-derived natural oils (Matheson et al. 2022). However, lauric acid together with even-numbered shorter chain fatty acids may indicate as well an animal origin of (dairy) fats. Compound-specific stable carbon isotope analysis of the major fatty acids, C16:0 and C18:0, as well as the presence of long-chain ketones in the same kind of *olle* (Mileto et al. 2023) seem to confirm the predominant animal origin of the detected compounds.

In addition, all samples contain various dicarboxylic acids, i.e., azelaic, pimelic, sebacic, suberic acids, indicating fermentation/oxidation processes of unsaturated fatty acids (Copley et al. 2005), and, in a few cases, also unsaturated fatty acids, i.e., oleic acid (C18:1), likely suggesting a plant (oil?) origin of the compounds. The use of *Brassicaceae* (seed oil) could be suggested by the presence of undecanedioic acid, a frequent oxidation byproduct of aged erucic acid (C22:1), an unsaturated fatty acid which is a chemical fingerprint of *Brassicaceae* seed oil together with gondoic acid (C20:1) (Colombini et al. 2005; Irto et al. 2022). Beta-Stigmasterol, a phytosterol, considered a plant biomarker (Irto et al. 2022) has been detected in VTres1, VTres8, VTres15. Arachidic acid (C20:0), a long chain saturated fatty acid, can be related to a plant origin of the compounds, e.g., vegetable oils (see arachidic/eicosanoic acid from *Brassicaceae* seed oil in Colombini et al. 2005).

Lignoceric (C24:0) (VTres1, VTres14, VTres15, VTres17, VTres18, VTres20, VTres23) and behenic acid (C22:0) (VTres1, VTres6, VTres7, VTres8, VTres13, VTres15, VTres17, VTres18, VTres23, VTres26), plant derived high molecular weight fatty acids, are commonly found in beeswax, which mostly comprises higher fatty acid and long chain alcohols (Chasan et al. 2021). Miliacin, a triterpenoid marker of broomcorn millet (*Panicum miliaceum*; Heron et al. 2016; Ganzarolli et al. 2018; Standall et al. 2022), has been detected in three samples (VTres13, VTres15, VTres17). A preparation of millet as porridge may have led to the absorption of miliacin in the cooking pot (Rageot et al. 2019).

Millet seeds are present in the macrobotanical remains on the site (Castiglioni, Rottoli 2019). The direct consumption of millet in this kind of *olle* from the same context has been also confirmed on the basis of miliacin detected by Mileto et al. 2023. In particular, the abundant markers and complex organic mixtures in samples VTres4, VTres6, VTres7, VTres12, VTres13, VTres23, VTres25, VTres26 suggest the preparation and consumption of cereals cooked together with fruits/wine (?), plant food (fava beans are for instance abundantly attested at the site) and probably dairy products and/or meat. Only a few samples (VTres8, VTres19 and VTres20) may indicate, based on degraded animal fats, the processing/cooking of probably only animal (adipose/dairy) products.

***Tab. 2** – List of the samples and interpretative hypothesis according to the detected compounds. See the complete results under http://dx.doi.org/10.17169/refubium-40223. / **Tab. 2** – Lista dei campioni e ipotesi interpretative sulla base delle sostanze identificate. Per i risultati completi vedi http://dx.doi.org/10.17169/refubium-40223.*

Sample ID	Probable source of compounds
VTres1	animal and plant fats
VTres2	(red) fruits; animal fats?
VTres3	(red) fruits; animal fats?
VTres4	(red) fruits; plant oils?/fats?
VTres5	animal and plant fats?
VTres6	fruits?; plant (seed oil)?; animal fats?
VTres7	(red) fruits; plant (seed oil)?; animal fats
VTres8	insufficient/ plant fats?
VTres9	Insufficient
VTres10	Insufficient/ degradated animal or plant fats?
VTres11	Insufficient/ degradated animal or plant fats?
VTres12	(red) fruits?; plant (seed oil)?; animal fats?
VTres13	(red) fruits; plant fats (oil?); animal fats; millet
VTres14	animal and plant fats (oil?)
VTres15	(red) fruits; plant fats; animal fats; millet
VTres16	Insufficient
VTres17	animal and plant fats; millet
VTres18	animal and plant fats (oil?)
VTres19	Insufficient
VTres20	degradated animal fats; plant fats?
VTres21	Insufficient/ plant (oil)? / animal fats?
VTres23	(red) fruits?; plant (seed oil)?; animal fats?
VTres25	(red) fruits?; plant (seed oil)?; animal fats?
VTres26	(red) fruits?; plant (seed oil)?; animal fats?

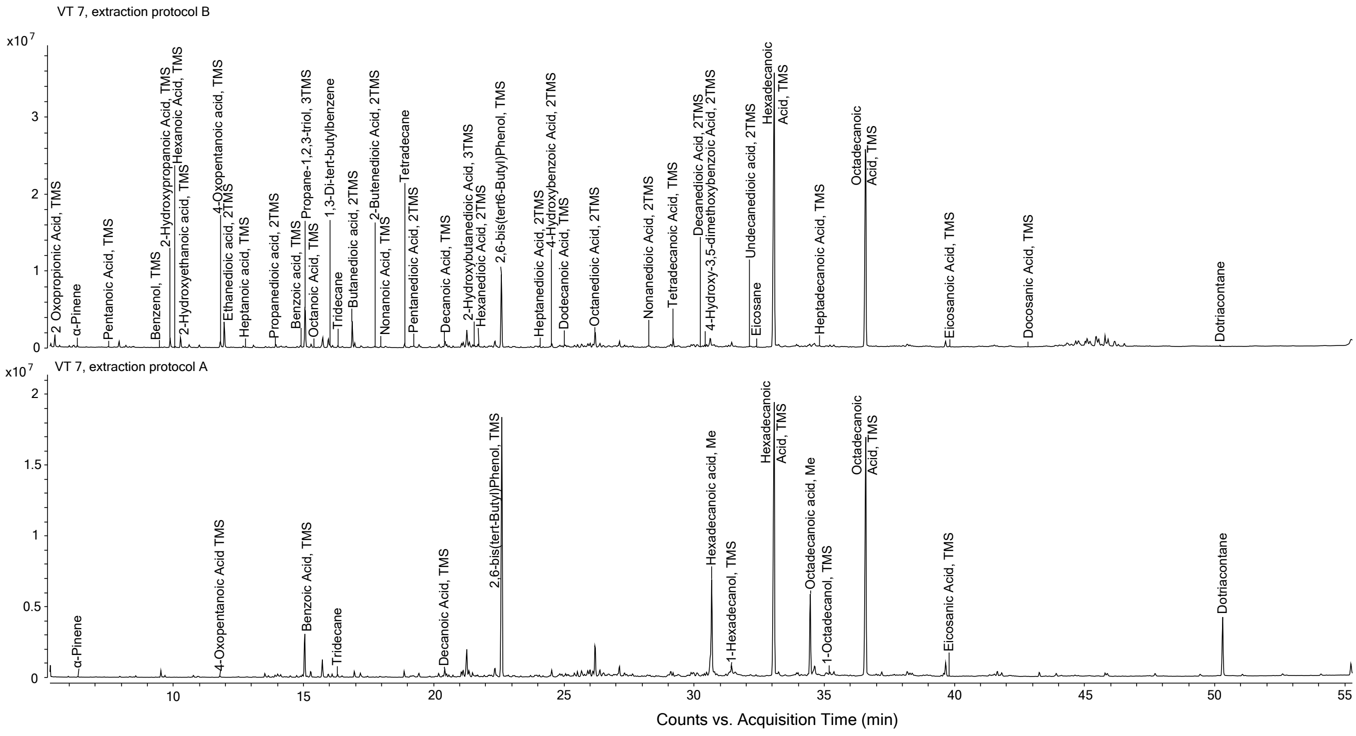

Fig. 3 – *Exemplary chromatogram of sample VTres7 (VT7 for brevity) according to the applied protocol. /* ***Fig. 3 –*** *Cromatogramma esemplificativo relativo al campione VTres7 (per brevitá VT7) secondo il protocollo di analisi applicato.*

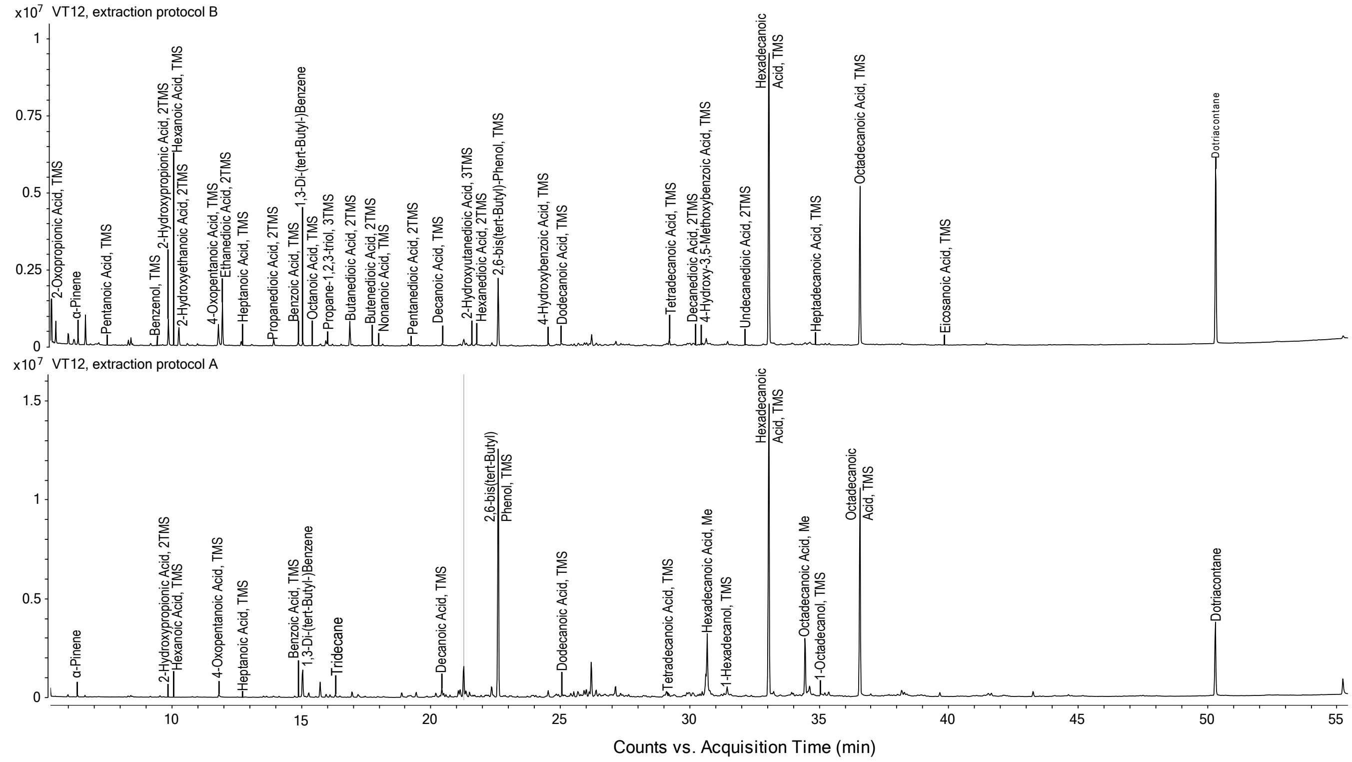

Fig. 4 – *Exemplary chromatogram of sample VTres12 (VT12 for brevity) according to the applied protocol. /* ***Fig. 4 –*** *Cromatogramma esemplificativo relativo al campione VTres12 (per brevità VT12) secondo il protocollo di analisi applicato.*

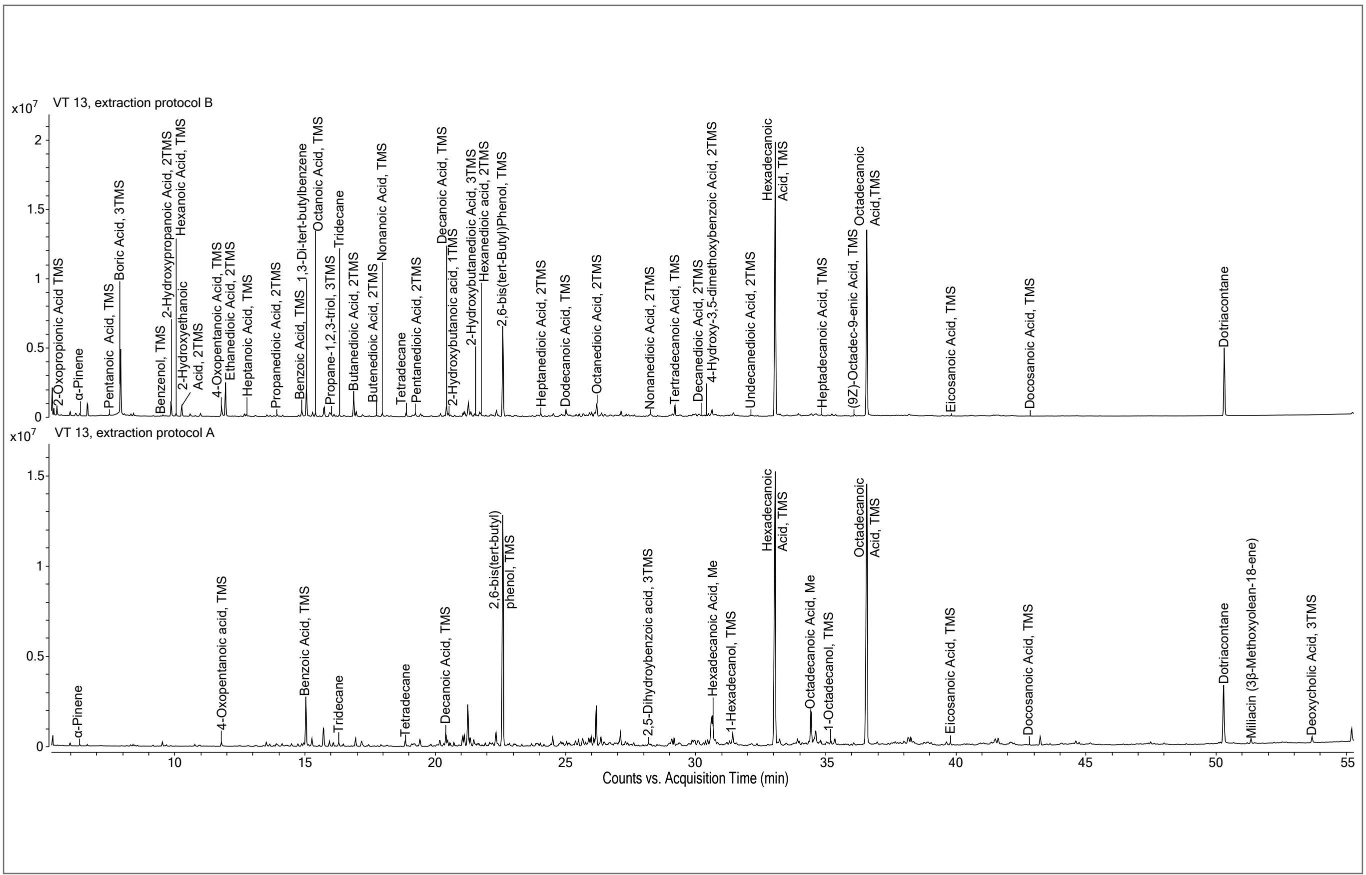

***Fig. 5 –** Exemplary chromatogram of sample VTres13 (VT13 for brevity) according to the applied protocol. / **Fig. 5 –** Cromatogramma esemplificativo relativo al campione VTres13 (per brevitá VT13) secondo il protocollo di analisi applicato.*

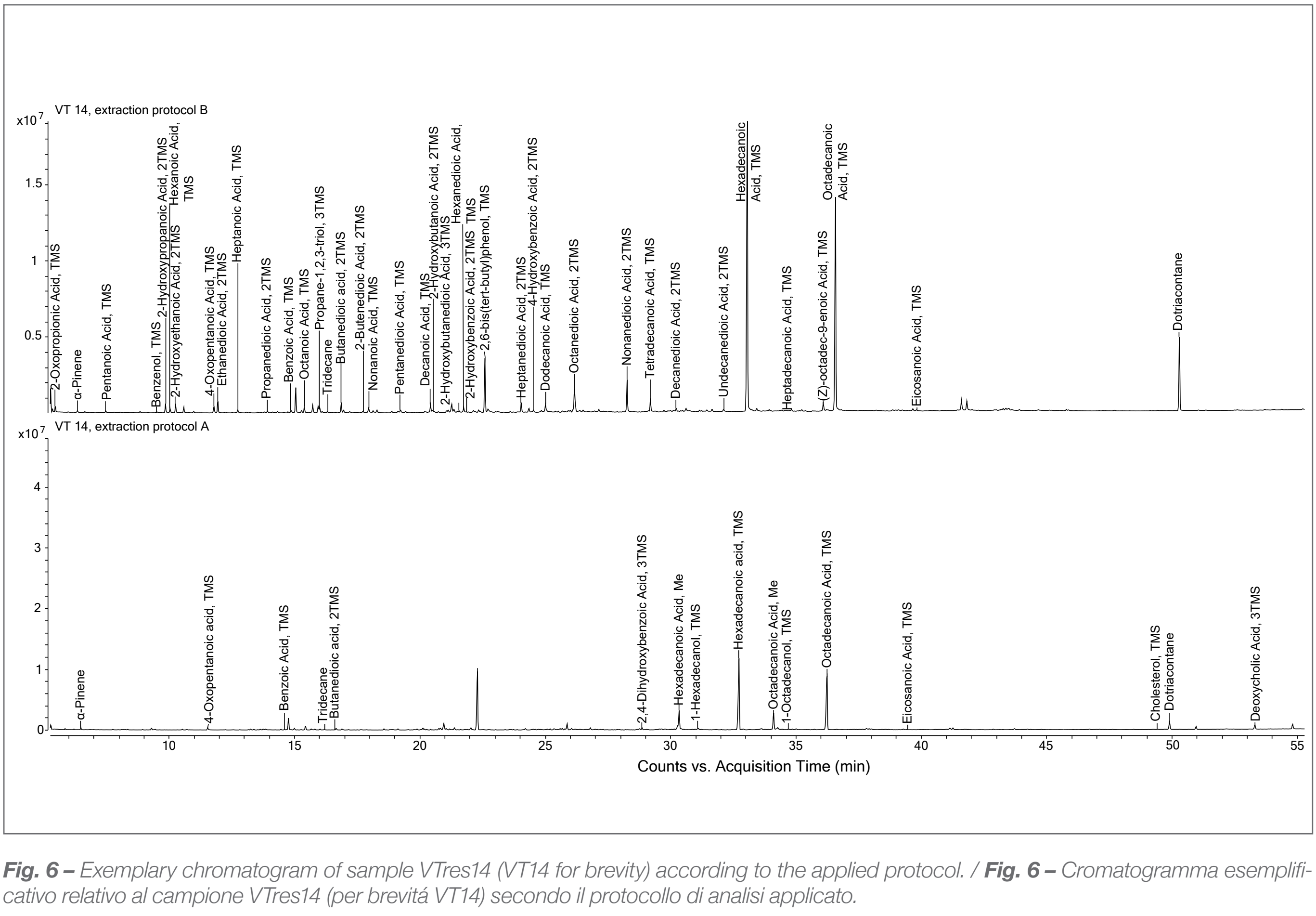

***Fig. 6 –** Exemplary chromatogram of sample VTres14 (VT14 for brevity) according to the applied protocol. / **Fig. 6 –** Cromatogramma esemplificativo relativo al campione VTres14 (per brevitá VT14) secondo il protocollo di analisi applicato.*

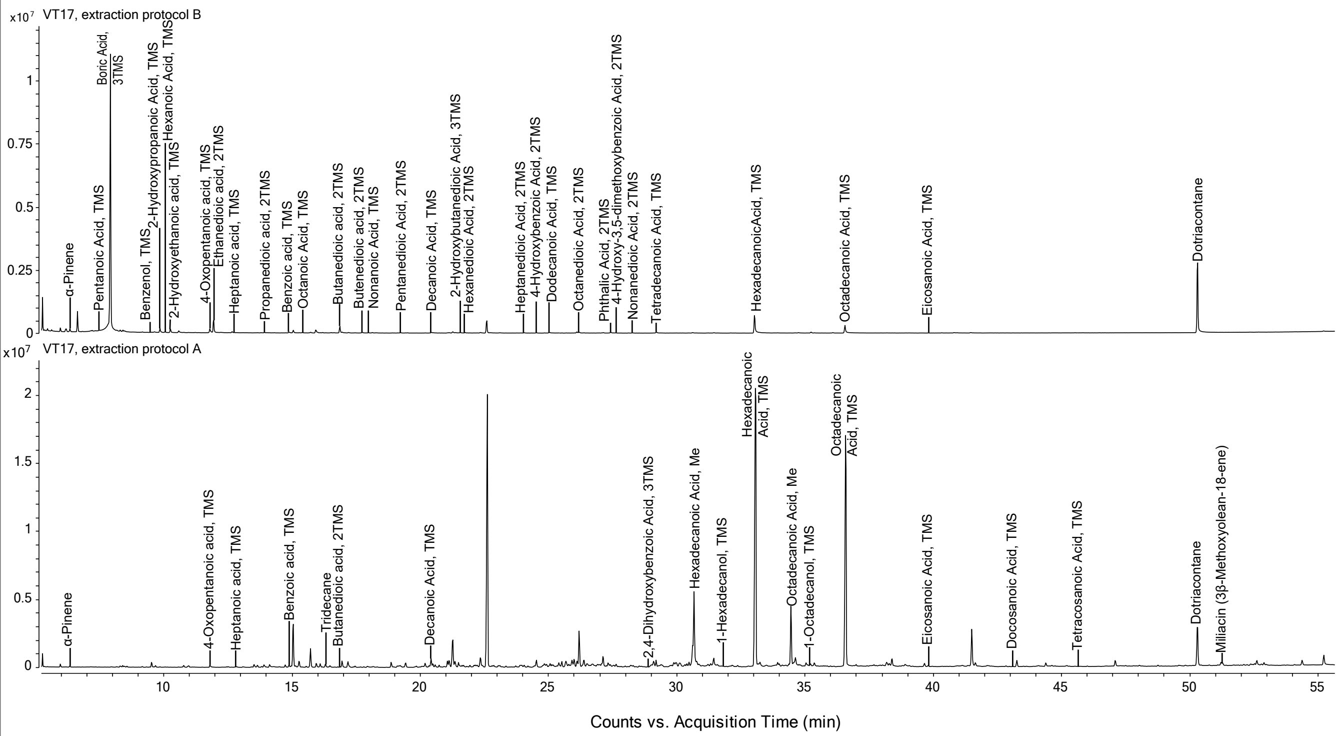

***Fig. 7 –** Exemplary chromatogram of sample VTres17 (VT17 for brevity) according to the applied protocol. / **Fig. 7 –** Cromatogramma esemplificativo relativo al campione VTres17 (per brevitá VT17) secondo il protocollo di analisi applicato.*

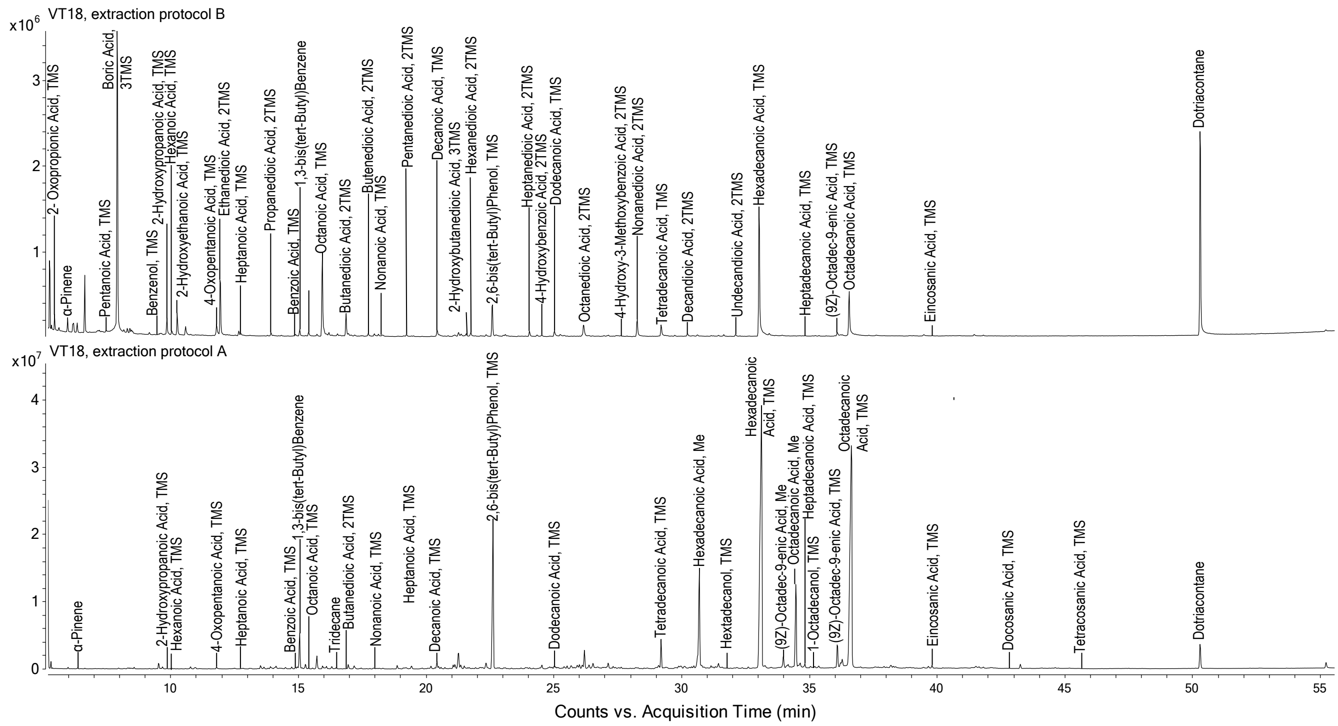

***Fig. 8 –** Exemplary chromatogram of sample VTres18 (VT18 for brevity) according to the applied protocol. / **Fig. 8 –** Cromatogramma esemplificativo relativo al campione VTres18 (per brevitá VT18) secondo il protocollo di analisi applicato.*

The detection of α-pinene can be interpreted as residue of firewood from coniferous trees, since the *olle* had a blackened surface due to direct contact with fire. Other characteristic markers of coniferous resins, i.e., dehydroabietic acid and other diterpenic markers, that could be related to the coating of the ceramic surface, are lacking (on the same conclusion also Mileto et al. 2023). The history of the site seems to be marked by various fire events indicated by numerous fragments of charred wood.

The complete data obtained by GC-MS according to the applied extraction protocol, are available under http://dx.doi.org/10.17169/refubium-40223 in tabular form, where sample ID for brevity corresponds to VT01, etc. The results and interpretative hypotheses according to the detected compounds are resumed in table 2 and exemplary shown in the chromatograms fig. 3- 8.

Concluding remarks

In conclusion, the analyses of the organic residue were able to identify abundant biomarkers and complex organic mixtures suggesting a multifunctional use of the *olle* for cooking animal (meat/dairy), plant products (cereals like millet) and (*Brassicaceae*) plant seed oils (?) together in the form of boiled soups and stews or porridge. The consumption of millet was common in Early Medieval Northern Italy (Adamson 2004; Castiglioni & Rottoli 2013; Ganzarolli et al. 2018; Montanari 2019; Ragno 2023 on statistical analysis of first millennium CE cereal farming practices in the Italian Peninsula). Furthermore, some fruits markers have been detected indicating the cooking use of (red) fruits/grapes (Lundy et al. 2021). Even though specific markers of wine are lacking, also wine cannot be excluded from an archaeological point of view considering the military strategic nature and intensive occupation of the site during the Lombard period despite the conspicuous elevation of the site and the difficult access from the valley.

The diversified use of local plant and animal resources in Early Medieval Northern Italy, e.g., cereals polyculture, several types of cultivated and spontaneous fruits, vegetables and legumes, the breeding of ruminants and pigs, has been reassessed and discussed in the context of political and economic change by Rottoli (2014). Nutrition and foodways in Early Medieval Italy is a topic that has been treated in the last two decades from various points of view (Brecciaroli Taborelli 2005; CISAM 2016). Together with human isotopic data providing dietary evidence (see recently Maxwell 2019 on diet and migration in Late Antiquity to the Early Medieval Veneto), the analysis of organic residues from ceramic vessels has the potential to complement historical, archaeological, botanical and faunal studies on the transition of patterns in the use of resources by focusing on cooking practices and direct consumption.

Credits

Fabio Cavulli and Francesco Carrer, coordinators of the UPLanD project, sampled the potsherds for the analysis and provided a description of the site in this paper. Silvia Polla, Andreas Springer, Niklas Limberg and Heiko Stukenbrok carried out the laboratory analysis and wrote the Introduction, Material and Methods and Preliminary Results. All the authors contributed to the Conclusions.

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