Lipids, pots and food processing at Hočevarica, Ljubljansko barje, Slovenia

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ABSTRACT – The paper presents the results of lipid analyses of pottery samples from Hočevarica (Ljubljansko barje, Slovenia). Total lipid extracts were subjected to high temperature gas chromatography (HT-GC), gas chromatography–mass spectrometry (GC-MS) and gas chromatography-combustion-isotope ratio mass spectrometry (GC-C-IRMS). The results show that some vessels were used for preparing ruminant meat and vegetable, but also the remains of aquatic food were identified. The processing of non-ruminant meat was detected in a few samples. A high number of pottery samples yielded the presence of beeswax lipids. The charred residual on pottery was AMS ¹⁴C dated.

IZVLEČEK – V članku predstavljamo rezultate analiz lipidov ohranjenih v keramičnem zbiru s Hočevarice. Lipide, ekstrahirane iz ostankov keramičnih posod, smo analizirali s pomočjo plinske kromatografije pri visokih temperaturah (HT-GC), plinske kromatografije sklopljene z masno spektrometrijo (GC-MS) in plinske kromatografije sklopljene z masnim pektrometrom za analizo stabilnih izotopov lahkih elementov preko sežigne enote (GC-C-IRMS). Rezultati kažejo, da so v posodah pripravljalih hrano iz mesa prežvekavalcev in zelenjave; redko iz mesa neprežvekavalcev. V drugih so pripravljalih hrano iz sladkovodnih rib. V številnih posodah je bil odkrito čebelj vosek. Karbonizirani ostanki na posodah so bili AMS ¹⁴C datirani.

KEY WORDS – lipid analysis; ¹⁴C dates; pottery; Eneolithic; Ljubljansko barje

Introduction

Hočevarica is located at the outfall of Hočevarica drainage channel into the Ljubljanica River between Blatna Brezovica and Verd on the western part of the Ljubljansko barje area (Fig. 1). A small trench (8m²) was excavated in 1998 (Velušček 2004a). The site was recognised as a pile-dwelling settlement embedded in the time span 3650–3520 calBC (for wood samples) (Čufar, Kromer 2004.283) and 3640–3530 calBC (for short-lived seed and carbonised grain samples) (Jeraj 2004.59).

The site stratigraphy consists of ten layers (Fig. 2). While some are of geological provenance, layers 4–8 relate to settling and can be associated with two settlement phases (Velušček 2004b.37–40; 2004c.213–217). Patches of burnt clay and daub (e.g., house remains) are deposited in well-defined stratigraphic superposition; they correlate with the distribution of vertical wooden piles, and depositions of pottery, stone and wooden tools within the stratified settlements’ layers (ibid. 40–47).

Palaeobotany and archaeozoology

More than 30000 remains of seeds and fruits of cultivated and gathered wild plants have been found in both settlement contexts. While cereal grains were carbonised, most of the remaining plant remains were unburned. The grains of cultivated Hordeum vulgare (six-rowed barley), Triticum monococcum...
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(einkorn wheat) and *Triticum dicoccum* (*T. turgidum* ssp. *Dicoccum*, emmer wheat) were identified; the most abundant cereal at Hočevarica is barley.

However, the remains of wild nuts, fruits and seeds predominated in the archaeobotanical assemblage, comprising *Quercus* sp., *Cupulae* (acorn), *Corylus avellana* (hazel nut), *Malus sylvestris* (crab apple), *Prunus avium* (wild cherry), *Cornus mas* (Cornelian cherry), *Cornus sanguinea* (common dogwood), *Rubus fruticosus* (blackberry), *Fragaria vesca* (wild strawberry), *Physalis alkekengi* (winter cherry), and *Trapa natans* (water chestnut). Along with *Papaver somniferum* (opium poppy) seeds, the only remains of an oily plant, *Chenopodium album* (goosefoot), which has seeds rich in oil and starch, were also gathered. Pulses such as *Lathyrus sativus* (grass pea) and *Vicia sp.* (*Vitis vinifera* ssp. *Sylvestris*, wild grapevine) were found in small numbers in the 1st settlement phase (*Jeraj* 2004.58–59; 2009.79–82).

It was suggested that while cereals were cultivated in fields situated on moist to damp soils close to the settlement, wild nuts, fruits and seeds were collected along the forest edges and in clearings around the settlement. The water plants were collected in small and shallow meso- to eutrophic lakes which warm up in summer. All the wild plants have been processed in settlement contexts (*Tolar* et al. 2011.216).

The animal bone assemblage consists of 4352 animal remains. About a third of them are fishes and birds, the remainder (63.4%) are mammals. The mammal bones (2757 total) are from at least 14 species (*Toškan, Dirjec* 2004.76–132). Roe deer (*Capreolus capreolus*) remains predominate, comprising a good third of the mammalian assemblage; the second most frequent was pig/wild boar (*Sus sp.*), accounting for one third. Other species were less frequent. Only red deer (*Cervus elaphus*), beaver (*Castor fiber*), dog (*Canis familiaris*), and the remains of sheep and goat (*Ovis s. Capra*) exceeded 5% of all finds. While *Sus scrofa domesticus* bones

### Tab. 1. Radiocarbon dates from Hočevarica. Dates marked with an asterisk (*) are inconsistently published (Hd-22139 as 4972±25 and Hd-20765 as 4746±26 in Čufar, Kromer 2004). Date for organic sediment (marked by **) by Jeraj (2004) is the same age as date for seed in phase 2. Since Jeraj does not cite lab codes for dates, it is possible that both are the same sample.

| Lab code | Material      | Phase | 14C Conventional age BP | Calibrated date calBC (2σ) | Reference   |
|----------|---------------|-------|-------------------------|----------------------------|-------------|
| Hd-18976 | wood          |       | 4822±39                 | 3695–3521                  | Čufar, Kromer 2004. Tab. 6.3.2 |
| Hd-22139 | wood          |       | 4867±26                 | 3702–3636                  | Čufar et al. 2010. Tab. 4* |
| Hd-20765 | wood          |       | 4748±26                 | 3636–3382                  | Čufar et al. 2010. Tab. 4* |
| Hd-22305 | wood          |       | 4825±25                 | 3656–3530                  | Čufar et al. 2010. Tab. 4* |
| ? organic sediment | |       | 4780±40                    | 3648–3383                  | Jeraj 2004.62** |
| ? seed | 2               | 4780±40       | 3648–3383                 | Jeraj 2004.59          |
| ? grain | 1               | 4810±40       | 3691–3518                 | Jeraj 2004.59          |
| Beta-391181 | food residue  | 2       | 4910±30                    | 3763–3642                  | Jeraj 2004.59 |
| Beta-391176 | food residue  | 1       | 4860±30                    | 3704–3539                  | Jeraj 2004.59 |
| Beta-391182 | food residue  | 2       | 4770±30                    | 3641–3519                  | Jeraj 2004.59 |
| Beta-391178 | bos taurus     | 1       | 4760±30                    | 3641–3384                  | Jeraj 2004.59 |
| Beta-391183 | ovis/capra    | 2       | 4740±30                    | 3639–3379                  | Jeraj 2004.59 |
| Beta-391185 | *Cornus* stone | 2       | 4720±30                    | 3635–3376                  | Jeraj 2004.59 |
| Beta-391180 | *Cornus* stone | 1       | 4680±30                    | 3623–3370                  | Jeraj 2004.59 |
| Beta-391177 | food residue  | 1       | 4780±30                    | 3635–3351                  | Jeraj 2004.59 |
predominate (38.2%) in the 1st settlement phase, *Ovis s. Capra* remains are the most frequent (19.7%) in the 2nd phase (*ibid.* 80).

The evidence of animal slaughter and further meat processing at the site are weak. The proportion of bones with cut and chop marks and/or traces of boiling or roasting was below 10%. However, the analysis of tooth wear showed that most of the pigs were slaughtered in the autumn at an assessed age of 17 to 22 months, and during winter or in early spring, at a probable age of 22 to 27 months (*Toškan, Dirjec* 2004.121). The fish remains consist of five species: common carp, rudd, pike, perch and roach. The carp and rudd remains predominate (*Govedič* 2004.133–151).

**Chronology**

The Hočevarica radiocarbon sequence is comprised of 13 AMS radiocarbon dates. In addition to the series of four dates on wooden piles used to anchor the dendrochronological sequence and two dates obtained on short-lived botanical samples, an additional two AMS radiocarbon dates on animal bones, two AMS dates on short-lived botanical samples and four dates of carbonised food residues on pottery were obtained recently (Tab. 1).

Complementary samples allow a better understanding of the chronology of activities at the site. The radiocarbon dates of bones and carbonised food/organic residues on pottery date events relating to the preparation and disposal of food, and thus complement the dates of the wooden structures relating to building and construction events. The floating oak chronology of 139 years from Hočevarica (HOC-QUSP1) is dated between 3685 and 3547 (±10) BC, which suggests an end to building activities after around 3550 BC (*Čufar et al.* 2010).

On the other hand, the majority of AMS dates on short-lived samples concentrate between 3630–3350 calBC (Fig. 3). The wide spread of values can be attributed to a wiggles in the calibration curve between 3620–3520 and 3480–3380 calBC. However, it seems that activities at the site reflected in the short-lived samples began well before the end of the building activities, before 3600 calBC, and continued for a few decades after building activities had ended. This long span of activities corresponds well with the two settlement phases.

Two dates on charred food residues on pottery are older than the oldest dates on the wooden piles. Lipid analysis on one sample (Beta-391176) from the first phase yielded a lipid concentration high enough (01HO; Tab. 2) to suggest that the pot was used to cook a ruminant/plant mixture. The concentration of lipids in the other sample (Beta-391181, 18HO; Tab. 2) was too low to allow a determination of foodstuffs. However, as this sample is associated with the second phase, it appears too old. At the moment, we have no dates on fish bones or food residues associated with aquatic foodstuffs that would demonstrate the presence of a reservoir effect. Therefore, both early dates could suggest earlier activities at the site or a reservoir effect.

**Fig. 2. Northern cross-section of the trench at Hočevarica (after Velušček 2004. Fig. 3.1.5).**
These new dates suggest a long and complex chronological sequence for the Hočevarica site. It appears that the site was settled for almost 200 years, had two distinct phases of occupation, and shows possible evidence of activities before the wooden structures were built.

The pottery

For the present study, we analysed 35 pottery samples from Hočevarica by hand lens to identify inclusions, their size and frequency, and the presence of voids. The samples were chosen on the basis of typology (see Velušček 2004d.169–212) and on the basis of the presence of charred food remains on the interior surface of the vessels. Most of the samples came from fragments of vessel rims and walls; only 9 samples were attributed to types according to their morphology: 3 pots, 4 dishes, and 2 bowls (Fig. 4; Tab. 2).

The vessel types are similar to the pottery assemblage from the contemporary site at Maharski pre-kop in the south-eastern part of Ljubljansko barje (Bregant 1974a; 1974b; 1975; Velušček 2004d.184–212). The majority of the vessels can be attributed to various types of pots (Velušček 2004d.186–194) and dishes (ibid. 196–203), but other forms are also present (cups, miniature vessels, hanging vessels and other special forms; ibid. 195, 203).

Similarly, the technological characteristics of the Hočevarica pottery assemblage are comparable to vessels from Maharski prekop (Žibrat Gašparič 2013. 153–155). The vessels are primarily dark grey, brown and black, and most were fired in a reducing atmosphere. Most of the pottery is poorly made and prone to mechanical decomposition; only the decorated vessels are of better quality and have polished surfaces or slips applied to the surface (Velušček 2004d.184–185).

We could divide the pottery samples into two technological groups according to their inclusions (descriptions after Horvat 1999): most of the samples have calcite/limestone inclusions (82.8%), while the
remainder are made of non-calcareous clay and have only quartz inclusions (17.2%). In the group with quartz, most of the inclusions comprise very fine (less than 0.25mm) or medium-size sand (0.25 to 0.50mm). Most of the samples with calcite/limestone have medium-size sand inclusions (0.25 to 0.50mm), but coarse sand is present (0.50 to 2.00mm) in a third of the samples.

The pottery samples from Hočevarica have voids, usually on both surfaces, in the size of medium to coarse sand fraction, and many have an angular shape similar to calcite crystals. This could be the result of calcite dissolved from the vessels. Such chemical changes in pottery are common post-depositional processes (Rice 1987:421). A similar situation could be observed at the contemporary site at Krašnja near Lukovica (Žibrat Gašparič et al. 2014).

All the pottery samples were handmade and their surfaces burnished; smoothing and polishing were also present. One of the vessels (10HO) was decorated with a grey-black slip on both the interior and exterior surfaces. They were fired in an incomplete oxidising (51.4%) and a reducing atmosphere (34.3%), while the other samples were fired in a reducing atmosphere with an oxidising atmosphere at the end of firing.

The pottery from the calcite/limestone group at Hočevarica has characteristics very similar to fabric MP-1 from Maharski prekop, which is a non-calcareous clay with frequent calcite grains added as temper and is the most common fabric found at that site (Žibrat Gašparič 2013:154). On the other hand, the group with quartz inclusions from Hočevarica differs from the fabrics described at Maharski prekop and could display a new technology in the later phase of the settlement, since the samples of the quartz group all come from the 2nd settlement phase at Hočevarica. This hypothesis would have to be tested with additional pottery samples, as well as with a petrographical analysis of thin sections.

Materials and methods

A total of 36 selected pottery samples were first cleaned to remove exogenous lipids, and then ground to a fine powder. For lipid extraction, about 2g of sample were transferred to a 50ml vial and 20μl of internal standard (n-tetratriacontane, 1mg/mL in n-hexane) were added. Lipids were ultrasonically ex-
extracted with a mixture of methanol and chloroform (1:2 v/v, 24mL, 2 x 30min). The solvent extract was removed into a glass flask and reduced to a small volume by rotary evaporation. The residue of solvent extract was transferred to a 2ml glass vial and evaporated to dryness under a gentle stream of nitrogen to obtain the total lipid extract (TLE). The aliquot (500μl) of the TLE was treated with BSTFA (N, O-bis(trimethylsilyl)-trifluoroacetamide, 40μl; 70°C, 60min), evaporated to dryness and re-dissolved in n-hexane. The resulting trimethylsilyl derivatives were analysed using high-temperature gas chromatography (HT-GC) and, where necessary, combined GC-MS analyses were performed to identify the structure of the components (Evershed et al. 1990). All HT-GC analyses were performed on Agilent Technology 6890N GC system equipment with DB-5HT capillary column (15m x 0.32m x 0.10μm). Temperature program: initial temperature 50°C (1min), increasing to 350°C (10 min) at a rate of 10°C/min. Helium was used as a carrier gas and a flame ionisation detector to monitor the column effluent.

Another aliquot (500μL) of solvent extract was used to prepare free fatty acids methyl esters (FAMEs) by adding 100μL of BF3-methanol (14% w/v, Sigma Aldrich, 70°C, 60min). The methyl derivatives were extracted with n-hexane and analysed by GC-MS and GC-C-IRMS using standard protocols (Evershed et al. 1994; Mottram et al. 1999; Greg, Slater 2010; Ogrinc et al. 2012). For GC-C-IRMS (Isoprime GV system, Micromass, Manchester, UK) the accuracy of repeated measurements was ±0.3‰.

In addition, powder samples (~1mg) were analysed by elemental analysis isotope ratio mass spectrometry (IRMS) as previously reported (Ogrinc et al. 2012; Budja et al. 2013). Stable isotope results are expressed as δ13C or δ15N values in per mil (‰) relative to the VPDB and AIR international standard, respectively. The accuracy of measurements was ±0.2‰ for δ13C and ±0.3‰ for δ15N.

Results and discussion

The average and standard deviations from bulk potsherd samples are –28.3±1.6‰ and +4.5±2.0‰ for δ13C and δ15N, respectively (Fig. 5; Tab. 2). These data fall in the range expected for C3 plant and degraded animal tissues whose subsistence was based mainly on C3 plants. The δ15N values of terrestrial plant proteins are around +3‰, while proteins derived from terrestrial herbivores from temperate Europe should not exceed δ15N values of +7.0‰ (Richards et al. 2003), although protein derived from domestic animals (such as pigs) may be higher (Privat et al. 2002; Polet, Katzenberg 2003; Richards et al. 2003; Ogrinc, Budja 2005). At Hočevarica, only three samples (01HO, 03HO and 06HO) have δ15N values higher than +7.0‰. Thus the variations in the carbon and nitrogen isotope ratios in our sample show that a wide diversity of animal and plant food was processed in the vessels. No sample has an δ15N value greater than 9‰ consistent with processing aquatic products with a high trophic level (Fig. 5). However, data on fish species from modern and archaeological samples from lacustrine environments demonstrates a wide range of nitrogen values due to the diverse mixture of aquatic food sources. For example, the δ15N values of freshwater fish in Lake Baikal range from +7.3 to +13.7‰ (Katzenberg, Weber 1999). And Melanie J. Miller et al. (2010) reported that the modern fish δ15N values of Lake Titicaca range from +4.1 to +9.5‰, while the majority of the δ15N values in archaeological fish samples ranged from +5.1 to +7.7‰.

In order to obtain more reliable information on the processing of different commodities in pottery vessels from Hočevarica, more specific chemical and molecular analysis, including lipid analysis, were performed. Lipid preservation in our samples was very good, with more than 75% of potsherds containing appreciable quantities of lipid (Tab. 2).

Lipid biomarkers

Even-carbon number n-alkanoic acids that range from C12 to C22 were observed in analysed sherds (Fig. 6). In addition, monounsaturated fatty acids...
C18:1 were present in the lipid extracts of all samples (Tab. 1). The presence of odd number (C15:0 and C17:0) and/or a low amount of branched chain of C17:0 was determined in 50% of the pottery samples (02HO, 05HO, 14HO, 20HO, 21HO, 26HO, 27HO, 31HO, 33HO, 34HO, 36HO). The presence of these acids together with two double bonds positional isomers of C18:1 indicates ruminant animal fats that have been biosynthesised in the gut and rumen (Dudd et al. 1999; Regert 2011). The parallel biomarkers, i.e. triacylglycerols (TAGs) and their degradation products (diacylglycerols (DAGs) and monoacylglycerols (MAGs)) were detected in 9 sherds (02HO, 05HO, 14HO, 20HO, 21HO, 26HO, 27HO, 31HO, 33HO, 34HO, 36HO), confirming the presence of degraded animal fats (Tab. 2; Fig. 7). However, the TAG distribution could be identified in three sherds (20HO, 21HO and 26HO), while in the remaining samples only traces of TAGs were observed. The narrow distribution of TAGs in these three sherds, ranging from C12 to C52, indicates the presence of ruminant adipose or dairy fats.

The presence of saturated and monounsaturated fatty acids in a range from C20 to C24, together with a high proportion of C16:0 and minor amounts of C12:0 and C18:0 acids are indicative of aquatic oils and thus provide evidence that freshwater foods were processed in these vessels (Hansel et al. 2004; Craig et al. 2011; 2013; Cramp et al. 2014). Such a lipid profile was observed in 35% of the samples (04HO, 06HO, 09HO, 10HO, 11HO, 12HO, 13HO, 17HO and 18HO). In addition, in these samples 4,8,12-trimethyltridecanoic acid (4,8,12-TMDT) at low concentrations was also identified. This component is a characteristic lipid biomarker of aquatic resources (Hansel et al. 2004) (Fig. 6).

Alongside the identification of animal or aquatic fats, a high percentage of samples (81%) yielded the presence of beeswax lipids (Tab. 2). In five samples (20HO, 21HO, 25HO, 26HO, 36HO) the lipid distribution indicate the high content of degraded beeswax lipids, while in other samples only traces of wax lipids are present. Beeswax lipids may indicate the addition of honey to other foodstuffs or the application of beeswax to pottery vessels to improve impermeability (Regert et al. 2001; Kimpe et al. 2002; Copley et al. 2005). Although in most of the samples only trace levels of this particular commodity were detected, its presence indicates that beeswax was utilised at Hočevarica in pottery vessels associated with cooking/processing foodstuffs or applied as a coating.

Long-chain ketones (C31, C33 and C35) were observed in most samples with preserved lipids, except in 05HO. Long-chain ketones have been widely reported as components of the epicuticular waxes of higher plants (Walton 1990), but can also be formed from the condensation of fatty acids (C16:0 and C18:0) during the heating of vessels to temperatures in excess of 400°C (Evershed et al. 1999). The presence of long-chain ketones together with thermally pro-

Fig. 6. The representative GC-MS total ion chromatograms of the fatty acids methyl esters (FAMEs) with different C16:0 and C18:0 abundance extracted from the Hočevarica pottery samples 11HO and 14HO.
| Lab. sample no. | ID. No. | Context | 14C Lab. no. | 14C conv. age BP | Fabric group | Description | TLE (μg g⁻¹) | δ¹³C bulk (%) | δ¹⁵N (%) | δ¹³Ccalc (%) | δ¹⁸Ocalc (%) |
|----------------|---------|---------|--------------|-----------------|-------------|------------|-------------|--------------|----------|-------------|--------------|
| 01HO 126       | phase 1 | Beta-391176 | 4860±30       | calcite         | vessel wall |            | 36.5        | -26.8       | 7.2       | -29.0       | -29.1        |
| 02HO 165       | phase 1 | Beta-391177 | 4780±30       | calcite         | vessel wall |            | 25.3        | -29.1       | 4.9       | -28.0       | -29.2        |
| 03HO 073       | phase 1 | calcite    | vessel wall   | 48.3            | -27.0       | 7.5        | n/d         | n/d         |           |             |              |
| 04HO 075       | phase 1 | calcite    | vessel rim with wall | 32.9        | -27.7       | 6.6        | -31.0       | -25.7      |           |             |              |
| 05HO 080       | phase 1 | calcite    | dish          | 39.0            | -27.3       | 5.5        | -27.8       | -27.4      |           |             |              |
| 06HO 135       | phase 1 | calcite    | vessel wall   | 96.5            | -27.9       | 7.4        | -31.1       | -27.3      |           |             |              |
| 07HO 138       | phase 1 | calcite    | vessel wall   | 42.7            | -32.0       | -0.1       | -34.3       | -29.0      |           |             |              |
| 08HO 174       | phase 1 | calcite    | vessel rim with wall | 40.8        | -27.4       | 0.4        | -29.8       | -28.2      |           |             |              |
| 09HO 087       | phase 1 | calcite    | vessel rim with wall | 13.1        | -31.5       | 1.8        | -30.7       | -27.1      |           |             |              |
| 10HO 076       | phase 1 | calcite    | vessel rim with wall | 10.9        | -27.8       | 4.7        | -32.2       | -28.5      |           |             |              |
| 11HO PN0081    | phase 1 | calcite    | pot            | 71.3            | -29.1       | 3.5        | -29.8       | -27.7      |           |             |              |
| 12HO 068       | phase 1/2 | calcite | vessel rim with wall | 78.6        | -26.5       | 6.9        | -30.7       | -28.5      |           |             |              |
| 13HO 067       | phase 1/2 | calcite | vessel wall | 57.8            | -27.0       | 3.7        | -32.4       | -27.6      |           |             |              |
| 14HO PN0135    | phase 1/2 | calcite | pot            | 51.5            | -27.5       | 1.8        | -25.5       | -27.9      |           |             |              |
| 16HO 049       | phase 1/2 | calcite | vessel wall | 108             | -27.3       | 1.5        | -36.0       | -29.9      |           |             |              |
| 17HO 082       | phase 2 | calcite    | vessel base with wall | 27.8        | -26.6       | 4.3        | -31.5       | -28.8      |           |             |              |
| 18HO 088       | phase 2 | Beta-391181 | 4910±30       | calcite         | vessel wall |            | 5.9         | -28.4       | 4.9       | n/d         | n/d          |
| 19HO 029       | phase 2 | calcite    | vessel wall   | 3.1             | -27.8       | 4.4        | n/d         | n/d        |           |             |              |
| 20HO 032       | phase 2 | quartz     | pot            | 211             | -30.7       | 4.8        | -27.3       | -33.9      |           |             |              |
| 21HO PN0049    | phase 2 | quartz     | dish           | 63.3            | -30.5       | 4.8        | -26.7       | -28.5      |           |             |              |
| 22HO 035       | phase 2 | Beta-391182 | 4770±30       | calcite         | vessel wall |            | 29.2        | -27.3       | 6.3        | -29.8       | -29.1        |
| 23HO 020       | phase 2 | calcite    | vessel wall   | 23.6            | -27.0       | 5.8        | -30.6       | -26.8      |           |             |              |
| 24HO 017       | phase 2 | quartz     | vessel rim with wall | 2.1          | -27.6       | 5.5        | n/d         | n/d        |           |             |              |
| 25HO 169       | phase 2 | quartz     | vessel wall   | 73.9            | -27.2       | 5.1        | -26.5       | -28.4      |           |             |              |
| 26HO 025       | phase 2 | quartz     | dish           | 53.3            | -27.3       | 5.2        | -28.4       | -29.2      |           |             |              |
| 27HO 019       | phase 2 | calcite    | vessel wall   | 15.9            | -28.2       | 4.6        | n/d         | n/d        |           |             |              |
| 28HO 120       | phase 1 | calcite    | vessel wall   | 1.6             | -28.2       | 4.6        | n/d         | n/d        |           |             |              |
| 29HO 121       | phase 1 | calcite    | vessel wall   | 6.0             | -29.3       | 5.6        | n/d         | n/d        |           |             |              |
| 30HO 089       | phase 1 | calcite    | bowl           | 4.7             | -26.5       | 6.7        | n/d         | n/d        |           |             |              |
| 31HO 085       | phase 1 | calcite    | pot            | 26.4            | -28.8       | 4.6        | -28.6       | -28.2      |           |             |              |
| 32HO 078       | phase 1 | calcite    | vessel wall   | 18.3            | -29.1       | 6.1        | n/d         | n/d        |           |             |              |
| 33HO 061       | planum 4/4 | calcite | dish           | 12.4            | -27.4       | 4.6        | -28.1       | -29.5      |           |             |              |
| 34HO 008       | SU 4/7 | quartz     | vessel wall   | 7.0             | -28.9       | 3.3        | -26.3       | -27.2      |           |             |              |
| 35HO 003       | SU 1/2 | calcite    | vessel wall   | 6.9             | -31.1       | 1.0        | n/d         | n/d        |           |             |              |
| 36HO PN0138    | E cross-section | calcite | bowl?        | 6.2             | -28.1       | 2.4        | -28.2       | -29.1      |           |             |              |

Tab. 2. A summary of the organic residues detected in pottery samples from Hočevarica, Ljubljansko barje region. Key: MAG – moniacylglycerols; DAG – diacylglycerols; TAG – triacylglycerols; A – n-alkanes; OH – n-alcohols; K – ketones; WE – wax esters; (tr) – trace; n/d – not detected.
| Δ13C/‰ | C_{16:0}/C_{18:0} | Fatty Acids (FA) | Other lipids | Predominant commodity type | Reference |
|---------|--------------------|------------------|--------------|-----------------------------|-----------|
| 0.0     | 1.48               | C_{16:0}, C_{18:0}, C_{17:0}, C_{19:0}, C_{20:0}, C_{22:0} | K, WE         | mixture ruminant, plant     | Not published |
| -1.2    | 1.45               | C_{16:0}, C_{18:0}, C_{17:0}, C_{19:0}, C_{20:0}, C_{22:0} | K, WE, DAG(tr), TAG(tr) | ruminant | Not published |
| n/d     | n/d                | K                | n/d          | Not published              |           |
| 5.3     | 1.50               | C_{18:0}, C_{16:0}, C_{17:0}, C_{19:0}, C_{20:0}, C_{22:0} | K              | freshwater | Not published |
| 0.4     | 0.74               | C_{16:0}, C_{18:0}, C_{17:0}, C_{19:0}, C_{20:0}, C_{22:0} | WE, DAG, TAG   | mixture ruminant, non-ruminant | Velušek 2004.Pl. 4.1.57 |
| 3.8     | 1.44               | C_{16:0}, C_{18:0}, C_{17:0}, C_{19:0}, C_{20:0}, C_{22:0} | K, WE(tr)      | freshwater | Not published |
| 5.3     | 2.80               | C_{16:0}, C_{18:0}, C_{17:0}, C_{19:0}, C_{20:0}, C_{22:0} | K, WE(tr)      | non-ruminant | Not published |
| 3.6     | 3.79               | C_{16:0}, C_{18:0}, C_{17:0}, C_{19:0}, C_{20:0}, C_{22:0} | K, WE(tr)      | freshwater | Not published |
| 3.7     | 1.57               | C_{16:0}, C_{18:0}, C_{17:0}, C_{19:0}, C_{20:0}, C_{22:0} | K, WE(tr)      | freshwater | Not published |
| 2.1     | 0.94               | C_{16:0}, C_{18:0}, C_{17:0}, C_{19:0}, C_{20:0}, C_{22:0} | K, WE(tr)      | freshwater | Velušek 2004.Pl. 4.1.3.2 |
| 2.2     | 1.15               | C_{16:0}, C_{18:0}, C_{17:0}, C_{19:0}, C_{20:0}, C_{22:0} | K, WE(tr)      | freshwater | Not published |
| 0.4     | 0.74               | C_{16:0}, C_{18:0}, C_{17:0}, C_{19:0}, C_{20:0}, C_{22:0} | K, WE(tr)      | freshwater | Not published |
| 3.8     | 2.22               | C_{16:0}, C_{18:0}, C_{17:0}, C_{19:0}, C_{20:0}, C_{22:0} | K, WE(tr)      | freshwater | Not published |
| 2.7     | 1.35               | C_{16:0}, C_{18:0}, C_{17:0}, C_{19:0}, C_{20:0}, C_{22:0} | K, WE(tr)      | freshwater | Not published |
| n/d     | n/d                | n/d              | n/d          | Not published              |           |
| n/d     | n/d                | n/d              | n/d          | Not published              |           |
| -6.6    | 1.19               | C_{16:0}, C_{18:0}, C_{17:0}, C_{19:0}, C_{20:0}, C_{22:0} | A, OH, K, WE, MAG, DAG, TAG | mixture ruminant dairy fats and degraded beeswax | Velušek 2004.Pl. 4.1.9.10 |
| -1.8    | 1.26               | C_{16:0}, C_{18:0}, C_{17:0}, C_{19:0}, C_{20:0}, C_{22:0} | A, OH, K, WE, MAG, DAG, TAG | mixture ruminant fats and degraded beeswax | Velušek 2004.Pl. 4.1.8.1 |
| 0.7     | 2.22               | C_{16:0}, C_{18:0}, C_{17:0}, C_{19:0}, C_{20:0}, C_{22:0} | K              | mixture ruminant, plant    | Not published |
| 3.8     | 2.02               | C_{16:0}, C_{18:0}, C_{17:0}, C_{19:0}, C_{20:0}, C_{22:0} | K              | non-ruminant               | Not published |
| n/d     | n/d                | n/d              | n/d          | Not published              |           |
| -1.9    | 1.93               | C_{16:0}, C_{18:0}, C_{17:0}, C_{19:0}, C_{20:0}, C_{22:0} | A, OH, K, WE   | mixture ruminant fats and degraded beeswax | Not published |
| -0.8    | 2.28               | C_{16:0}, C_{18:0}, C_{17:0}, C_{19:0}, C_{20:0}, C_{22:0} | A, OH, K, WE, DAG, TAG | mixture ruminant fats and degraded beeswax | Velušek 2004.Pl. 4.1.10.2 |
| -1.1    | 0.85               | C_{16:0}, C_{18:0}, C_{17:0}, C_{19:0}, C_{20:0}, C_{22:0} | K, DAG(tr), TAG(tr) | ruminant | Not published |
| n/d     | n/d                | n/d              | n/d          | Not published              |           |
| 0.4     | 1.11               | C_{16:0}, C_{18:0}, C_{17:0}, C_{19:0}, C_{20:0}, C_{22:0} | K, WE(tr)      | mixture ruminant, plant    | Velušek 2004.Pl. 4.1.3.3 |
| n/d     | n/d                | n/d              | n/d          | Not published              |           |
| -1.4    | 1.67               | C_{16:0}, C_{18:0}, C_{17:0}, C_{19:0}, C_{20:0}, C_{22:0} | K              | ruminant                   | Velušek 2004.Pl. 4.1.7.1 |
| -1.0    | 0.24               | C_{16:0}, C_{18:0}, C_{17:0}, C_{19:0}, C_{20:0}, C_{22:0} | K, TAG, WE(tr) | ruminant | Not published |
| n/d     | n/d                | n/d              | n/d          | Not published              |           |
| -0.9    | 0.81               | C_{16:0}, C_{18:0}, C_{17:0}, C_{19:0}, C_{20:0}, C_{22:0} | A, OH, K, WE, DAG(tr), TAG(tr) | mixture ruminant fats and degraded beeswax | Velušek 2004.Pl. 4.1.7.3 |
duced \( \omega \)-([\( \alpha \)-alkylphenyl])alkanoic acids implies that their formation is mainly related to heating to high temperatures.

**Stable carbon isotope composition of fatty acids**

Further information regarding the source of the organic residues was obtained by measuring the stable carbon isotope ratio of saturated fatty acids C_{16:0} and C_{18:0} preserved in sufficient quantities in the pottery samples. The results were compared with modern reference animal data obtained from the literature presented in Figure 8 (Evershed et al. 2002; Copley et al. 2005; Craig et al. 2007; 2012).

Twelve samples (04HO, 06HO, 07HO, 08HO, 09HO, 10HO, 11HO, 12HO, 13HO, 16HO, 17HO and 23HO) yielded \( \delta^{13}C \) values closer to those of lipid extracts from modern pottery vessels used to prepare freshwater and non-ruminant animals (Copley et al. 2005) (Fig. 8). Although nine of them (04HO, 06HO, 09HO, 10HO, 11HO, 12HO, 13HO, 16HO, 17HO) have aquatic biomarkers present, their use cannot be resolved more specifically. Non-ruminant, terrestrial animal contribution/origin could not be excluded, since the animal bone assemblage contains a high percentage of boar/pig (>30%) (Toškan, Dirjec 2004).

35% of samples (02HO, 14HO, 21HO, 25HO, 26HO, 27HO, 33HO, 34HO, 36HO) plot in the range for ruminant adipose fats (Fig. 8). The C_{16:0}/C_{18:0} ratios of fatty acids for these samples range between 0.74 and 2.28 values (Tab. 2) typical of ruminant adipose fat (Copley et al. 2005). The distribution of the data (Fig. 8) and \( \delta^{15}N \) values of samples (average value 4.4±1.2‰) suggested that the population at Hočevarica used diverse domesticated (goat, cattle) or wild (deer) animal products in their diet. The sample 20HO plots in the region typical of ruminant dairy fats. The processing of dairy products in this pottery vessel is further supported by the distribution of lipids (Fig. 7).

A further 15% of the samples (01HO, 05HO, 22HO, 31HO) fall close to the limit value between non-ruminant and ruminant fat (\( \Delta^{13}C = \delta^{13}C_{18:0} - \delta^{13}C_{16:0} = 0\% \)). However, not all samples could be assigned to meat mixtures exclusively. In vegetable oils, for example, the C_{18:1} fatty acid is enriched in \( ^{13}C \) compared to C_{18:0} (Spangenberg, Ogrinc 2001). A \( ^{13}C \)-enrichment of C_{18:1} (up to 2.3‰) compared to C_{18:0} acid was also observed in three pottery vessels (01HO, 22HO and 31HO) suggesting an admixture of plant-animal fats.

**Conclusions**

The results of stable isotope data and the more specific product identification based on available lipids indicate varied vessel use: pots were used to cook both aquatic and terrestrial products.

The ruminant animal fats of either domestic (cattle, goat) or wild (deer) origin were the most frequently processed products preserved in the Hočevarica pottery samples (Tab. 2; 02HO, 05HO, 14HO, 21HO, 22HO, 25HO, 26HO, 27HO, 31HO, 33HO, 34HO, 36HO). These samples come from all the analysed settlement phases at Hočevarica and display a variety of different types and technologies (both the calcite/limestone group and the quartz group). This confirms that ruminant animal fat was processed in a variety of vessels, such as pots (14HO, 31HO), dishes (21HO, 26HO, 33HO) and bowls (05HO, 36HO) (Fig. 4).

The processing of non-ruminant animal fats was detected in only three samples from Hočevarica that come from both main settlement phases, all made from the most common technological group with added calcite/limestone inclusions (Tab. 2; 07HO, 08HO, 23HO).

**Fig. 7. Partial high-temperature gas chromatogram showing total lipid extracts from pottery sample 20HO from Hočevarica that is characteristic of a mixture of ruminant dairy fat and degraded beeswax.**
Only one decorated pot with an appliqué (20HO) indicates the processing of dairy fat. This pot dates to the 2nd settlement phase at Hočevarica and was made with the less common fine-grained fabric with quartz inclusions (Fig. 4; Tab. 2).

The appearance of aquatic biomarkers is associated with nine samples (04HO, 06HO, 09HO, 10HO, 11HO, 12HO, 13HO, 16HO and 17HO), indicating that these vessels were used in the preparation of aquatic resources such as fish and molluscs (Tab. 1). One of the samples with aquatic biomarkers is a pot with an appliqué (11HO; Fig. 4). Most of the samples come from the oldest settlement phase at Hočevarica and have similar technological characteristics in terms of their inclusions (calcite/limestone group), surface and firing treatment. This group of vessels also includes the only samples with a grey-black slip on the surface (10HO).

Moreover, we found that three of the pottery samples (01HO, 22HO and 31HO) were used to process both plant and animal fats. These samples also come from all the settlement phases and are made with calcite/limestone inclusions. Sample 31HO is also a pot with an appliqué and comes from the same context as pot 11HO, which showed the presence of aquatic biomarkers (Fig. 4; Tab. 2).

The presence of beeswax in the vessels suggests either the storage of honey or the use of beeswax as a waterproofing agent. Beeswax was detected in five samples (Tab. 2; 20HO, 21HO, 25HO, 26HO, 36HO), of which four come from the 2nd settlement occupation phase and fall into the group with quartz inclusions. As to their morphology, the samples with preserved beeswax include two dishes (21HO, 26HO), one pot that was also used to process dairy fat (20HO), and one bowl (36HO) (Fig. 4). These results suggest that the use of beeswax as a waterproofing agent or the use of honey in the preparation of food was more common in the younger settlement phase at Hočevarica and/or connected to special types of vessels made with a different ceramic fabric.

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