Lipid residues in early hunter-gatherer ceramics from Finland

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Abstract

The adoption of pottery technology by hunter-gatherers in the Baltic region has mainly been placed within the concept of resource intensification, as a cultural choice that assisted an increased economic focus on aquatic resources. In Finland, a non-specialized pottery use has earlier been suggested on the basis of lipid residue analysis data for Early Neolithic[1] ceramics from a number of sites on the south-west coast that involve processing of both aquatic and terrestrial animal products. Our study is an attempt to further explore the relationship between the early pottery use, diet and environment in this particular region. For this purpose, we expanded the range of early pottery traditions and localities to be analyzed, and applied gas chromatographic and mass spectrometric (GC-MS) analysis on the absorbed lipid residues. The study material comprises ceramics of the Säräisniemi 1 (Sär 1), Sperrings 1 (Ka I:1) and Sperrings 2 (Ka I:2) pottery traditions, as well as of the Jäkärlä Ware group, from coastal and inland sites spanning a period from *ca*. 5100 to 4000 cal BC. Our results strengthen the above observation for Early Neolithic Finland and further suggest that both aquatic and terrestrial animal resources were processed in vessels belonging to most of the analyzed groups, irrespective of coastal/inland site division and pottery ware culture. This additionally indicates a variability of motives behind the introduction of pottery within the Baltic Sea region, considering pottery use evidence in the neighbouring Estonia, where the earliest local and contemporaneous Narva pottery culture had a more specialized use focused on the processing of aquatic resources.

Keywords: Early Neolithic, ceramics, hunter-gatherers, lipid residue analysis, biomarkers, GC-MS, Finland

Introduction

The early stages of pottery utilization by hunter-fisher-gatherer populations in various geographical regions and from different periods of time have been connected with subsistence strategies that involved new and more efficient ways to exploit the sustainable and nutrient-rich marine and freshwater resources (Jordan & Zvelebil 2009; Craig et al. 2013; Gibbs & Jordan 2013; Farrell et al. 2014; Taché & Craig 2015). In the West Baltic region, organic residue analysis of ceramic vessels indicated that pre-agriculture pottery-using populations living by the coast relied heavily on marine resources for their subsistence, and high aquatic food consumption was observed inland as well (Andersen 2008; Craig et al. 2011). Another more recent study of lipid residues conducted on the earliest ceramics from Estonia of the Narva Ware culture showed similar results that suggest a specialized use for the processing of aquatic products irrespective of site division, coastal or inland, despite the availability of land animal resources in the landscape (Oras et al. 2017). The authors argue that the findings invigorate the above hypothesis for the uptake of ceramics by foragers, and propose that pottery possibly supported the intensification of aquatic resource exploitation during seasons of higher productivity, which in turn led to increased sedentism and population densities (Oras et al. 2017). Moreover, an earlier study on Narva-type vessels from the coastal Sventoji site, Lithuania, although dating later, corroborates the above with evidence that also suggests a pottery use orientated to the processing of aquatic foodstuff (Heron et al. 2015).

In Finland, the application of organic residue analysis on archaeological ceramic vessels is still a novel line of research in the field of ceramic studies, although its potentials were fully realized quite early. There are so far only a few published studies with lipid identifications on organic residues from ceramics. The earliest concerned fatty acid analysis of charred surface deposits from Bronze Age sherds recovered from the Otterböte site, Åland, with the aim to investigate whether marine and specifically seal blubber lipids could be identified. One of the four analyzed samples showed positive results (Isaksson 1997). Typical and Late Comb Ware charred surface crusts from the Vantaa Maarinkunnas site were also analyzed for their lipid composition, with results suggesting mainly aquatic animal origins (Hopia et al. 2003; Leskinen 2003; Pesonen & Leskinen 2009). Another more comprehensive study was performed a few years ago, and included both molecular and stable carbon isotope analysis of individual fatty acids (Cramp et al. 2014). The analyzed material here covers a timespan from the fourth to the first millennium BC, comprising Typical Comb Ware, Corded Ware, Kiukainen Ware, Late Bronze Age and Early Iron Age Morby Ware ceramics. For the Typical Comb Ware pots, the results pointed to a predominant or exclusive use for the processing of marine resources. Dairy fats were mainly suggested for the Corded Ware, Bronze Age and Morby Ware pots, while Kiukainen Ware demonstrated mixing of ruminant and non-ruminant/marine fats (Cramp et al. 2014). The most recent study undertaken by Pääkkönen et al. (2016) was the first attempt on Early Neolithic material that allowed for interpretations about the function of ceramics at the initial stages of their introduction in this area. The analyzed residues derive from three coastal sites of the Early Comb Ware and Jäkärlä Ware cultures situated in south-west Finland and the results showed that both aquatic and terrestrial animals, basically ruminants, had been processed in the vessels. This

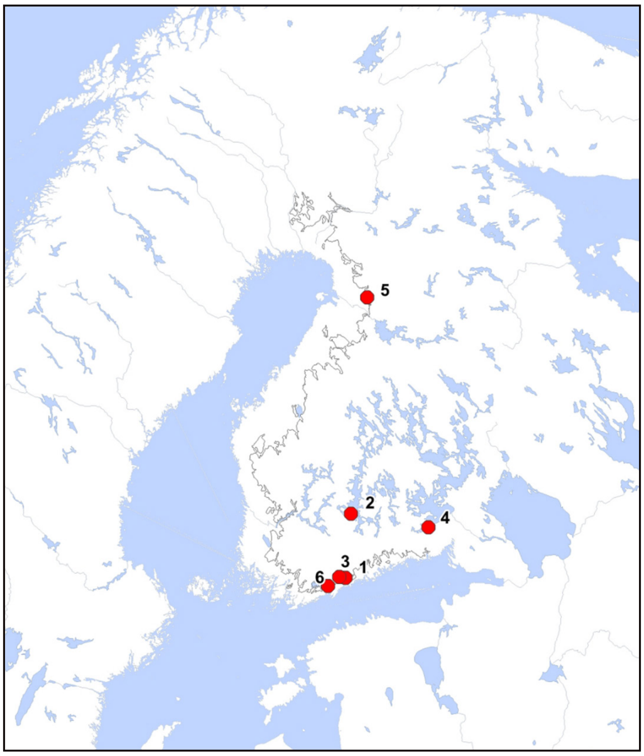

Figure 1. The sampled sites: 1– Vantaa Etelä- Vantaa 3/Mätäoja III (Palmu); 2– Padasjoki Leirintäalue; 3– Espoo Kläppkärr; 4– Lappeenranta (Etu-ja Taka) Muntero; 5– Oulu (formerly Ylikiiminki) Vepsänkangas; 6– Raasepori Telegrafberget. The grey line corresponds to the Littorina Sea phase of the Finnish coast ca. 6100 cal. BC (adapted from Tikkanen & Oksanen 2002).

gives another dimension for the early pottery use in the circum–Baltic region and challenges the hypothesized link of hunter–gatherer ceramics to subsistence/economic strategies connected with the exploitation of aquatic resources. Our study comes to contribute to this evidence with the aim to further explore the early pottery use and dietary habits in Finland through analysis of Early Neolithic ceramic sequences from different chronological phases and environments, coastal and inland, all located close to water systems (Figure 1; see also Appendix). We provide molecular (GC–MS) data of the absorbed residues.

Materials and Methods

A total of 22 potsherds from Säräisniemi (Sär 1), Sperrings 1 (Ka I:1), Sperrings 2 (Ka I:2) and Jäkärlä Ware ceramic vessels (Table 1) were carefully selected for molecular analysis by gas chromatography – mass spectrometry (GC–MS) by excluding sherds with adhesives from previous conservation treatments and paying extra caution to avoid multiple sampling of singular vessels. They all seem to derive from large vessels judging from the flatness of the sherds (Figure 2). Coastal sites were sampled for the Sär 1 and Jäkärlä Ware pottery styles, and both coastal and inland ones for the Ka I:1 and Ka I:2. A sample amount of *ca.* 1.0–2.0 grams of ceramic powder was ground off from the interior surface by using a low–speed pottery grinder. Powder was collected on clean sheets of aluminium foil and transferred quantitatively in tubes after preliminary grinding and removal of the first ca. 1.0 mm of the surface to reduce possible interference of extraneous lipid sources, such as fingerprints or soil. All glassware used had previously been washed with dichloromethane (DCM).

Lipid extraction and derivatization

Before extraction, the pulverized samples were washed with a DCM/CH_3OH solution (2:1, v:v; 3*3.0 ml) to remove any remnants of exogenous lipids, following the protocol described by Lucquin et al. (2016). Lipid extraction and derivatization was accomplished by using a one–step acid extraction and methylation protocol as it was described in Papakosta et al. (2015). Briefly, an amount of 1.0 ml, or 2.0 ml, of CH_3OH acidified with concentrated H_2SO_4 (200 µl) as catalyst was used to release lipids, depending on the sample amount. The samples were heated for 4 h at 70°C. After heating, *n*–hexane was added (1.0 ml), and separated from CH_3OH after a three–time repeated centrifugation (3000 rpm, 5 min).

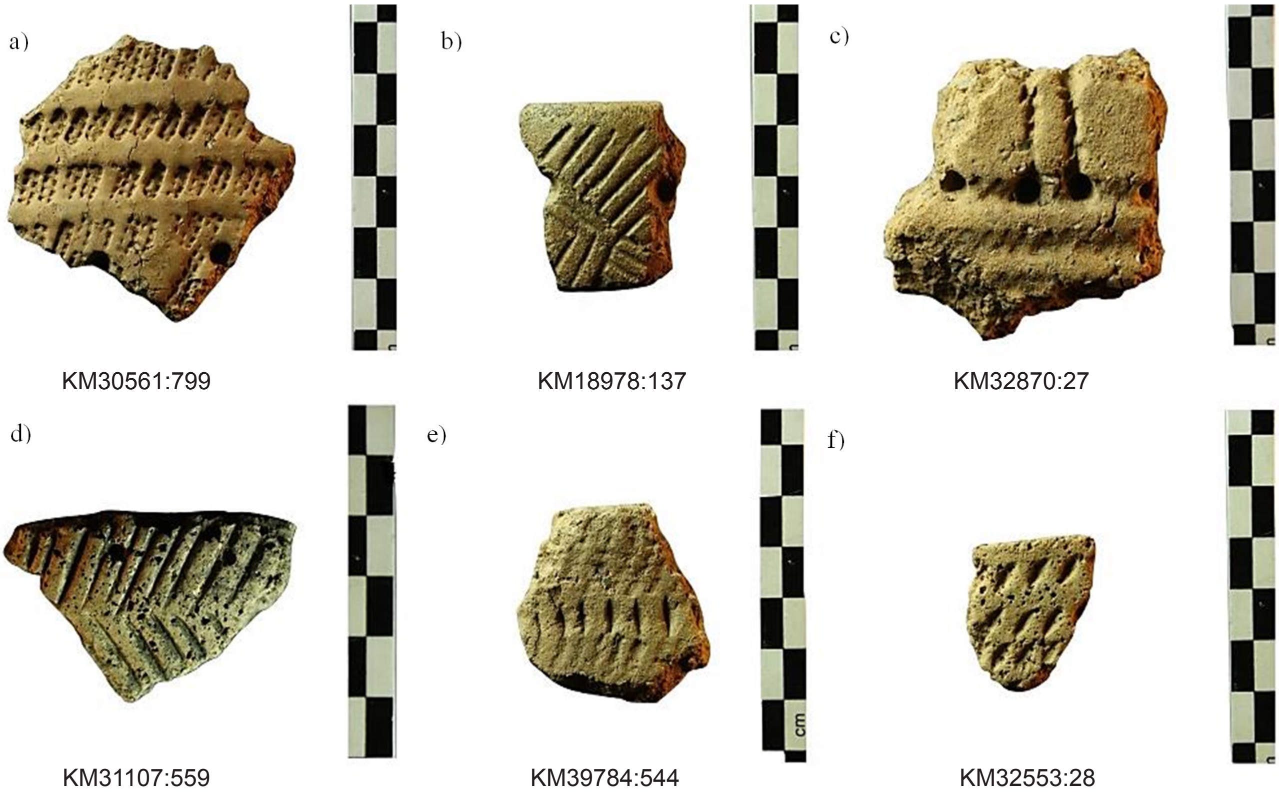

Figure 2. Examples of analyzed potsherds: a– Sär 1; b, c– KaI:1; d, e– KaI:2; f– Jäkärlä Ware (photos by P. Pesonen, modified by V. Papakosta).

The combined extracts were evaporated under a gentle stream of N_2 gas and treated with 100 µl of bis(trimethylsilyl)trifluoroacetamide (BSTFA) containing chlorotrimethylsilane (TMCS, 10%) to mask any underivatized hydroxyl groups. Then, they were heated at 70°C for 25 min, evaporated, and rediluted in *n*-hexane for GC-MS analysis.

Molecular analysis by gas chromatography - mass spectrometry (GC-MS)

Separation and identification of the extracted lipids was performed on a HP 6890 Gas Chromatograph fitted with a SGE BPX5 fused-silica capillary column (15 m x 220 µm x 0.25 µm), coupled to a HP 5973 Mass Selective quadrupole detector. Samples were injected through a Merlin Microseal™ High Pressure Septum in pulsed splitless mode (pulse pressure 17.6 Psi, 325°C). The column oven was set to start with an initial isothermal at 50°C for 2 min, and then temperature increased to reach 360°C with a rate of 10°C per minute. A final isothermal hold was set for 15 min. Helium was selected as the carrier gas with a controlled constant flow of 2.0 ml/min. Ionization and fragmentation of the compounds were accomplished by electron impact (70 eV) with the aid of an ion source maintained at 230°C. The mass filter was set to scan between *m/z* 50 and 700 with a rate of 2.29 scans per second. Quantification was performed using the calibration line of the saturated C_{17} fatty acid methyl ester measured in different concentrations after treatment with the same protocol. Where present, compounds considered as contamination, mainly phthalates, were excluded from integration of the total peak areas of the total ion chromatograms.

Table 1. Sampled sites and weighed amount of ceramic powder from each of the selected samples.

Site/Site location	Sample Inv. no.	Sample Lab no.	Sample amount (in g)
Oulu (formerly Ylikiiminki) Vepsänkangas	KM30561:725A	Ou725A	1.9
/ Sär 1; Coastal	KM30561:725B	Ou725B	0.7
	KM30561:793	Ou793	1.8
	KM30561:799	Ou799	1.9
Vantaa Etelä-Vantaa 3/Mätäoja III (Palmu)	KM18978:139	V78:139	2.0
/ KaI:1; Coastal, close to river estuary	KM18978:146	V78:146	1.6
	KM18978:106	V78:106	1.7
	KM18978:137	V78:137	1.8
Padasjoki Leirintäalue	KM32870:27	P70:27	1.9
/ KaI:1; Inland, lakeside			
Espoo Kläppkärr	KM31107:559	E07:559	1.8
/ KaI:2; Coastal	KM31107:600	E07:600	1.9
	KM31107:468	E07:468	1.8
	KM31107:456	E07:456	1.8
Lappeenranta (Etu-ja Taka) Muntero	KM39784:11764	LPR11764	1.6
/ KaI:2; Inland, lakeside	KM39784:11935	LPR11935	1.9
	KM39784:544	LPR544	2.1
	KM39784:1629	LPR1629	1.9
	KM39784:322	LPR322	2.0
	KM39784:117	LPR117	1.9
Raasepori Telegrafberget	KM32553:66	R66	1.6
/ Jäkärlä Ware; Coastal	KM32553:28	R28	1.4
	KM32553:35	R35	1.9

Results and Discussion

The majority of the analyzed samples (55%) exhibit evidence of vessel use most probably associated with cooking, as the detected ω-(o-alkylphenyl)alkanoic acids (APAAs) are formed during heating of unsaturated fatty acids at 260–270 ºC (Hansel et al. 2004; Craig et al. 2007; Evershed 2008; Evershed et al. 2008; Heron et al. 2010). Besides APAAs, mid- to long-chain ketones were also detected in two of the samples (E07:559 and LPR11935) that might additionally indicate overheating of food lipids (≥300 ºC) (Evershed et al. 1995; Baeten et al. 2013). This observation seems to contradict a previous belief that this function may not have been central for these vessels (cf. Matiskainen 2008: 187).

The significant lipid preservation enabled identification of compounds diagnostic of their origin in most cases. Identification of aquatic resources (marine and/or freshwater) was supported by the presence of their corresponding biomarkers, although not in full set (at least one isoprenoid fatty acid and the C_{18} to C_{22} APAAs, or the C_{20} and C_{22} APAAs), the co-occurrence of broad series of diacids (DAs), *i.e.*, oxidation products of unsaturated fatty acids, the occasional persistence of the $C_{20:1}$, $C_{22:1}$ and $C_{24:1}$ fatty acids, and fatty acid distributions dominated by the saturated C_{16} homologue that give $C_{18:0}/C_{16:0}$<0.48 ratios (Tables 2-3; Figure 3) (Ackman & Hooper 1970; Regert et al. 1998; Hansel et al. 2004; Olsson & Isaksson 2008; Boudin et al. 2009; Cramp & Evershed 2014). In samples where these sets of compounds concurred with high $C_{18:0}/C_{16:0}$ ratios (>0.48), possible mixing with terrestrial animal fats is suggested (Romanus et al. 2007).

In the coastal Sperrings 1 (Ka I:1) Vantaa Etelä-Mätäoja III (Palmu) site, all samples demonstrate high $C_{18:0}/C_{16:0}$ ratios (>0.48) typical of terrestrial animal fats as the dominant components in the residues. Intriguingly, no aquatic biomarkers have been detected, such as isoprenoids and APAAs, that could point to a contribution of aquatic products as it would be expected considering the site location and the predominance of seal bones in the analyzed osteological material (Table 2–3; Appendix). Some possible vegetal input in the form of a plant-deriving oily substance could also be assumed by the short ranges of DAs (C_8-C_{10}) detected in three of the samples.

In the chronologically contemporaneous inland Ka I:1 Padasjoki Leirintäalue site, both terrestrial and aquatic animal products were processed in the single analyzed pot (P70:27), as it is inferred by the high $C_{18:0}/C_{16:0}$ ratio (>0.48) and the co-occurrence of phytanic acid with the full range of APAAs (C_{16}–C_{22}) in very low abundances and a broad series of short- to medium-chain DAs (C_7-C_{12}) (Table 2–3; Figure 3). It is worth noting that phytanic acid is not an aquatic biomarker in itself as it is also found in ruminant carcass fats and dairy products (Vetter & Schröder 2011; Cramp & Evershed 2014). The analyzed archaeofaunal material from the site is represented accordingly by both land and aquatic animals, with fishbone fragments being the most abundant (Appendix).

Three out of four samples from the contemporaneous coastal Sär 1 Oulu (Ylikiiminki) Vepsänkangas site demonstrate high $C_{18:0}/C_{16:0}$ ratios (>0.48) indicative of terrestrial animal fats (Table 2). However, the lipid profile of one of them (Ou725B) does not seem to represent food residues, but most probably coniferous resin or tar, according to the presence of dehydroabietic acid that was the most abundant compound (Pollard & Heron 1996: 240-5). These substances

Table 2. Summary of the GC-MS results for each of the analyzed samples. SFAs= saturated fatty acids, LCALs= long-chain alkanols, ALs= alkanes, BRFAs= branched fatty acids, MUFAs= monounsaturated fatty acids, DAs= diacids (dicarboxylic acids), LCKs= long-chain ketones, APAAs= ω-(o-alkylphenyl)alkanoic acids, DT= dehydroabietic acid, tr.= traces. Cx:y (x denotes the carbon-chain length, y denotes the degree of unsaturation). A C18:0/C16:0 >0.48 ratio indicates a fatty acid distribution indicative of terrestrial animal fats. T= terrestrial animals, A= aquatic animals, P= plants.

Sample	Lipid concentration (µg g^{-1})	$C_{18:0}$/ $C_{16:0}$ (>0.48)	Compounds detected	Interpretation
Ou725A	150	1.3	SFAs ($C_{10:0}$-$C_{26:0}$), LCALs ($C_{22:0}$, $C_{24:0}$), ALs ($C_{14:0}$-$C_{25:0}$), BRFAs (C_{15}, C_{17}, C_{18}), MUFAs ($C_{16:1}$, $C_{18:1}$), DAs (C_6-C_{12}), phytanic acid, APAAs (C_{16}-C_{22}), cholesterol, 3, 5-cholestadienone, (2-TMS-oxy) $C_{16:0}$, DT tr.	T A
Ou725B	70	1.0	SFAs ($C_{12:0}$-$C_{26:0}$), LCALs ($C_{22:0}$, $C_{26:0}$), ALs ($C_{16:0}$-$C_{24:0}$?), BRFAs (C_{15}, C_{17}), MUFAs ($C_{18:1}$), DAs (C_8, C_9), b-sitosterol, DT	Coniferous resin/ tar
Ou793	30	0.9	SFAs ($C_{9:0}$-$C_{28:0}$), ALs ($C_{15:0}$-$C_{20:0}$), BRFAs (C_{15}, 10-methyl-, 14-methyl-C_{17}), MUFAs ($C_{16:1}$, $C_{18:1}$), DAs (C_8, C_9), DT tr.	T
Ou799	140	0.3	SFAs ($C_{9:0}$-$C_{24:0}$), ALs ($C_{16:0}$-$C_{18:0}$, $C_{22:0}$-$C_{24:0}$), BRFAs (C_{15}, C_{17}, C_{18}), MUFAs ($C_{16:1}$, $C_{18:1}$), DAs (C_7-C_{12}), phytanic acid, APAAs (C_{16}-C_{22})	A
V78:139	40	0.9	SFAs ($C_{12:0}$-$C_{26:0}$), ALs ($C_{16:0}$-$C_{26:0}$), LCALs ($C_{22:0}$, $C_{26:0}$), BRFAs (C_{15},C_{16}), MUFAs ($C_{18:1}$), DAs (C_8-C_{10})	T P
V78:146	40	1.0	SFAs ($C_{12:0}$-$C_{26:0}$), ALs ($C_{16:0}$-$C_{27:0}$), BRFAs (C_{15}-C_{17}), MUFAs ($C_{18:1}$ tr.), DT tr.	T P
V78:106	80	0.9	SFAs ($C_{9:0}$-$C_{28:0}$), ALs ($C_{14:0}$-$C_{25:0}$), LCALs ($C_{22:0}$, $C_{26:0}$), BRFAs (C_{15}-C_{18}), MUFAs ($C_{16:1}$, $C_{18:1}$), DAs (C_8-C_{10}), DT tr.	T P
V78:137	50	0.9	SFAs ($C_{10:0}$-$C_{26:0}$), ALs ($C_{15:0}$-$C_{26:0}$), BRFAs (C_{15}-C_{17}), MUFAs ($C_{16:1}$,$C_{18:1}$), DAs (C_8, C_9), DT tr.	T P
P70:27	40	0.8	SFAs ($C_{12:0}$-$C_{24:0}$), ALs ($C_{16:0}$-$C_{24:0}$), BRFAs (C_{15}, C_{17}, C_{18}), MUFAs ($C_{18:1}$), DAs (C_7-C_{12}), phytanic acid, APAAs (C_{16}-C_{22}), DT tr.	T A
E07:559	1010	0.36	SFAs ($C_{9:0}$-$C_{24:0}$), ALs ($C_{13:0}$-$C_{17:0}$), BRFAs (C_{13}-C_{19}), MUFAs ($C_{18:1}$), DAs (C_7-C_{12}, C_{14}, C_{16}, C_{18}), phytanic acid, APAAs (C_{16}-C_{22}), LCK (C_{31}, C_{32}), 5-oxo-($C_{16, 18, 20, 22}$), 3, 5-cholestadienone	A
E07:600	300	0.2	SFAs ($C_{9:0}$-$C_{28:0}$), ALs ($C_{15:0}$-$C_{20:0}$), BRFAs (C_{15}-C_{18}), MUFAs ($C_{16:1}$, $C_{18:1}$, $C_{20:1}$, $C_{22:1}$, $C_{24:1}$), DAs (C_7-C_{14}), phytanic acid, APAAs (C_{18}-C_{22})	A
E07:468	110	0.22	SFAs ($C_{10:0}$-$C_{24:}$0), ALs ($C_{14:0}$-$C_{23:0}$), BRFAs (C_{15}-C_{18}), MUFAs ($C_{16:1}$ tr., $C_{18:1}$ tr.), DAs (C_9-C_{14}), APAAs (C_{16}?, C_{18})	A
E07:456	20	1.0	SFAs ($C_{10:0}$-$C_{28:0}$), ALs ($C_{15:0}$-$C_{24:0}$), LCALs ($C_{22:0}$-$C_{26:0}$), BRFAs (C_{15}, C_{17}, C_{18}), MUFAs ($C_{18:1}$), DAs (C_9)	T
LPR11764	180	0.23	SFAs ($C_{10:0}$-$C_{24:0}$), ALs ($C_{15:0}$-$C_{17:0}$), BRFAs (C_{15}-C_{18}), MUFAs ($C_{16:1}$?, $C_{18:1,}$ $C_{22:1}$), DAs (C_7-C_{12}), phytanic acid, APAAs (C_{16}-C_{22})	A
LPR11935	1150	1.8	SFAs ($C_{9:0}$-$C_{26:0}$), ALs ($C_{15:0}$-$C_{18:0}$), LCALs ($C_{22:0}$-$C_{26:0}$), BRFAs (C_{15}-C_{18}), MUFAs ($C_{18:1}$), DAs (C_8-C_{18}), LCK (C_{31}-C_{33}), phytanic acid, APAAs (C_{16}-C_{18}), (2-TMS-oxy) $C_{18:0}$	T P
LPR544	200	5.5	SFAs ($C_{12:0}$-$C_{28:0}$), ALs ($C_{15:0}$-$C_{20:0}$), LCALs ($C_{22:0}$-$C_{26:0}$), BRFAs (C_{15}-C_{18}), MUFAs ($C_{16:1}$, $C_{18:1}$), DAs (C_9), phytanic acid, APAAs (C_{16}, C_{18}), cholest-7-ene?, (2-TMS-oxy) $C_{18:0}$, DT tr.	T
LPR1629	30	1.0	SFAs ($C_{12:0}$-$C_{26:0}$), ALs ($C_{16:0}$-$C_{19:0}$), BRFAs (C_{15}-C_{19}; 10-methyl-, 14-methyl-C_{17}; 10-methyl-, 14-methyl-C_{18}; 10-methyl-, 14-methyl-C_{19}), MUFAs ($C_{16:1}$ tr., $C_{18:1}$), DAs (C_7-C_{12}), DT tr.	T P
LPR322	10	0.9	SFAs ($C_{12:0}$-$C_{26:0}$), ALs ($C_{16:0}$-$C_{19:0}$), BRFAs (C_{15}-C_{19}; 10-methyl-, 14-methyl-C_{17}), MUFAs ($C_{16:1}$, $C_{18:1}$), DAs (C_9), DT tr.	T
LPR117	200	0.48	SFAs ($C_{9:0}$-$C_{26:0}$), ALs ($C_{16:0}$-$C_{19:0}$), BRFAs (C_{15}-C_{18}), MUFAs ($C_{18:1}$), DAs (C_7-C_{12}), APAAs (C_{16}-C_{22})	A
R66	400	0.5	SFAs ($C_{9:0}$-$C_{24:0}$), ALs ($C_{16:0}$-$C_{20:0}$), BRFAs (C_{15}-C_{18}), MUFAs ($C_{16:1}$, $C_{18:1}$), DAs (C_7-C_{12}), pristanic acid, phytanic acid?, APAAs (C_{18}-C_{22}), (2-TMS-oxy) $C_{18:0}$	A
R28	30	0.8	SFAs ($C_{12:0}$-$C_{26:0}$), ALs ($C_{15:0}$-$C_{25:0}$), BRFAs (C_{17}), MUFAs ($C_{18:1}$ tr.)	T
R35	500	0.7	SFAs ($C_{9:0}$-$C_{26:0}$), ALs ($C_{14:0}$-$C_{25:0}$), BRFAs (C_{13}, C_{15}-C_{18}), MUFAs ($C_{16:1}$, $C_{18:1}$), DAs (C_7-C_{12}), pristanic acid, phytanic acid, APAAs (C_{16}-C_{22})	T A

Table 3. Summary of the GC-MS results by pottery tradition and site location.

Pottery tradition	Site location	No. vessels analyzed	No. vessels yielding aquatic biomarkers[a]	No. vessels with pure[b] aquatic animal signals	No. vessels with indications of pure[c] terrestrial animal signals	No. vessels with indications of mixing (aquatic + terrestrial animal fats)	Aquatic to terrestrial animal ratio (a/t)
Sär 1	Coastal	4	2	1	1	1	1.0
Ka I:1	Coastal	4	-	-	4	-	0.0
	Inland/Lake	1	1	-	-	1	1.0
Ka I:2	Coastal	4	2	3	1	-	3.0
	Inland/Lake	6	2	2	4	-	0.3
Jäkärlä Ware	Coastal	3	2	1	1	1	1.0

[a] At least one isoprenoid fatty acid acid and ω-(o-alkylphenyl)alkanoic acids (C_{18}-C_{22}), or ω-(o-alkylphenyl)alkanoic acids (C_{20}-C_{22}).
[b] No terrestrial animal signals detected. Plant residues may be present.
[c] No aquatic signals detected. Plant residues may be present.

could have been applied to seal the walls of the vessel for waterproofing. No signs of vessel restoration using resin as glue are visible on the potsherd. Indications of the processing of aquatic products occur in two of the samples, either as pure components (Ou799), or in mixture with terrestrial animal fats (Ou725A) (Table 3). In the sample Ou793, the proposed terrestrial animal signals would possibly originate from ruminants based on the presence of positional isomers of the C_{17} branched fatty acid besides the C_{15} homologue. European elk (*Alces alces*) and reindeer (*Rangifer tarandus*) are likely components according to the recorded archaeofaunal material from the site. Generally, a variety of possible components could be attributed to the three of the analyzed vessels that yielded food signals inferring from the wide diversity of species reported in the archaeofaunal dataset from the site, which is composed of terrestrial and aquatic mammals, fish and waterfowl (Appendix).

Aquatic resources seem to dominate in the analyzed pots from the coastal Ka I:2 Espoo Kläppkärr site, while one sample (E07:456) shows indications of terrestrial animal fats. In the pots from the contemporaneous inland Ka I:2 Lappeenranta Muntero site, only two of the six analyzed samples show clear indications of aquatic products as sole components. The presence of phytanic acid and the C_{16} and C_{18} APAAs in two of the samples (LPR11935 and LPR544) having high $C_{18:0}/C_{16:0}$ ratios (>0.48) cannot be used as safe indications of mixing with aquatic products, since these compounds could also originate from ruminant animals (Ackman & Hooper 1970; Craig et al. 2007; Heron et al. 2015) (Figure 3). An aquatic contribution could be possible for the samples LPR11935 and LPR1629 based on the broad sets of DAs detected, however, as these could also derive from plant oils, in the absence of other biomarkers it is not safe to form such an inference. Ruminant origins are proposed for terrestrial animal residues where wide ranges of branched fatty acids and positional isomers were detected. It is also unknown whether our findings agree with the zooarchaeological material from the site since it has not been analyzed.

Generally, fish bones are often reported in high proportions on inland lakeside sites (Ukkonen 1996). Seal bones are also found today in the entire region that was previously covered by the waterbody of the Lake Saimaa, on the shore of which the site was located (Ukkonen 2002). In the coastal Raasepori Telegrafberget site of the Jäkärlä Ware group, both terrestrial animal and aquatic resources seem to be equally represented in the three analyzed pots, either as main components or in mixture (Table 2-3).

The graphic representation (Figure 4) provides a visual overview of our data and shows that aquatic and terrestrial animal resources were both processed in almost all analyzed vessel groups, irrespective of pottery ware culture, time period and environmental setting. More specifically, in the Sär 1 and Jäkärlä Ware ceramics from the coastal sites, and the Ka I:1 ones from the inland, aquatic and terrestrial animal resources have been identified in balanced proportions. In the Ka I:2 ceramics from the coastal site aquatic animal resources are found in higher proportion, and in lower in those from the inland site of the same culture. Our results are generally in agreement with the available zooarchaeological assemblages of the sites, though the relative proportions of aquatic and terrestrial animal resources in the bone material and the analyzed residues differ. A significant divergence was observed in the Ka I:1 ceramics from the coastal site, where despite the clear prevalence of seal bone fragments in the osteological material, no aquatic signals were detected in any of the analyzed pots. Despite the water-centric character of the site and the observed importance of sealing in the local economy, the inhabitants seem not to have used their pots to process seals and generally aquatic resources. This does not necessarily mean that aquatic resources were not of significant dietary importance for the site occupants, since these could have been processed and consumed by means that do not involve pots (Olsson & Isaksson 2008). The importance of fish for their subsistence, however, cannot be estimated since

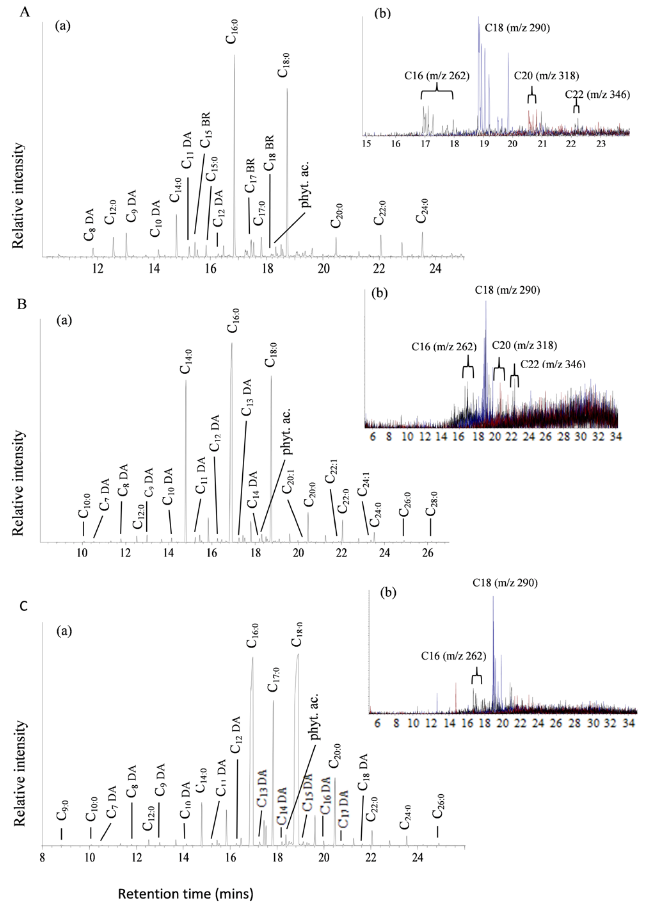

Figure 3. Partial GC-MS chromatograms showing the distribution of fatty acids (m/z 74) and derivatives interpreted as representing, A(a): mixture of aquatic and terrestrial animal products (P70:27), B(a): aquatic products (E07:600), and C(a): terrestrial animal products (LPR11935). A(b), B(b), and C(b) show the range of APAAs detected in each sample.

fish remains do not occur in the archaeofaunal assemblage, perhaps due to the lack of sieving at the early times of the excavation. The degree to which a preferential pottery use towards terrestrial animal resources at this site is valid might be tested through analysis of more samples as we may have missed to sample pots with aquatic residues.

Conclusions

Molecular (GC-MS) analysis of absorbed lipid residues from Early Neolithic hunter-fisher-gatherer pottery sequences from Finland enabled interpretations on pottery use thanks to the

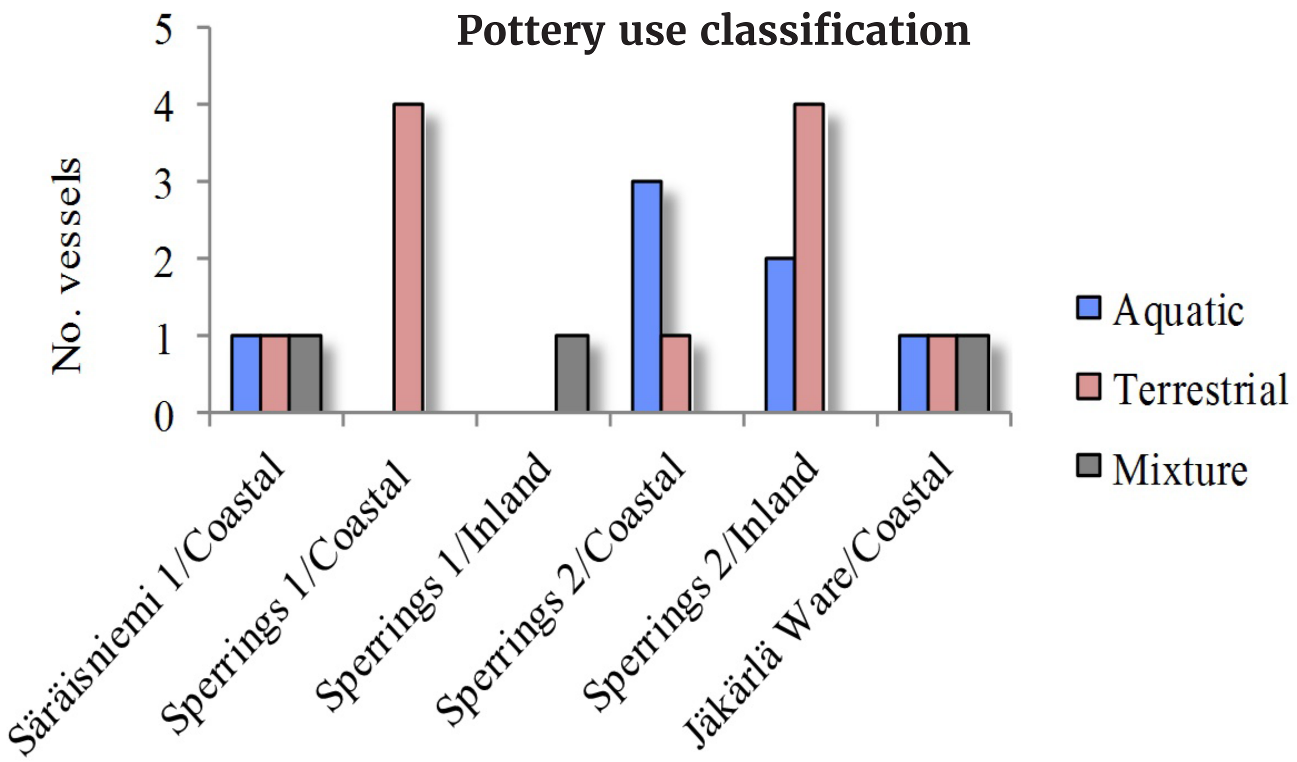

Figure 4. Relative representation of aquatic and terrestrial animal signals in the analyzed vessels by pottery tradition and site location.

significant lipid concentrations and persistence of compounds specific to their origin. The majority of the analyzed residues (55%) showed evidence of a vessel use most probably associated with cooking as it was inferred by the presence of compounds that form through protracted heating at high temperatures.

Moreover, we suggest that the early pottery use in Finland was not specialized for the processing of aquatic resources. Instead, aquatic and terrestrial animal resources were both identified as components in most of the analyzed vessel groups, irrespective of coastal/inland site division and pottery ware culture. Considering also the possible plant-deriving signals, it seems that site occupants were exploiting their local natural environments at the optimum of what it could give them for nourishment with pottery serving as useful implement in their nutritional habits. Our molecular evidence are in accord with the available archaeofaunal assemblages, although the relative proportions of aquatic *versus* terrestrial animal resources in the bone material and the analyzed residues differ. It is only the results from the Ka I:1 ceramics from the coastal Vantaa Etelä-Vantaa 3/Mätäoja III (Palmu) site that show a divergent feature with aquatic signals being absent from all analyzed residues, despite the site location and the predominance of seal bones in the archaeofaunal material.

Our observations are in line with those made by Pääkkönen et al. (2016) in a previous study on Finnish Early Comb Ware and Jäkärlä Ware pots from coastal sites concentrated in south-west Finland, and together challenge the idea of a generalized concept that associates the adoption of ceramics by hunter-fisher-gatherers in the Baltic region and elsewhere with practices related to intensified aquatic resource exploitation (e.g., Andersen 2008; Jordan & Zvelebil 2009; Craig et al. 2011; Craig et al. 2013; Taché & Craig 2015; Oras et al. 2017). Although this idea would apply in particular cultures and geographical regions, we argue that the uptake and use of ceramics is a more complex phenomenon that requires to be studied on a localized scale for interpretations of higher resolution. For instance, considering that the earliest, and contemporaneous to our material, ceramics in neighbouring Estonia, named Narva, were found to have had a more specialized use for the processing of aquatic resources, the results from Finland indicate most probably the existence of different motives behind the uptake of this technology in these two regions

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Endnote

1) The paper follows the Eastern European terminology where the Neolithic marks the introduction and use of ceramic vessels and is dissociated from farming.

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Appendix: Description of the sampled sites

1. Vantaa Etelä-Vantaa 3/Mätäoja III (Palmu)
Coordinates (EUREF-FIN): N 60.261247442, E 24.854878139, Z 31 m asl
Research history: Surveys 1962, 2000 (V. Lehtosalo, K. Lesell), excavations 1971–1973 (P. Purhonen, L. Väkeväinen)

The site was originally located at the ancient coast of the Littorina Sea in southern Finland, near Helsinki, and close to the Vantaanjoki River estuaries of that time. Excavations took place in the early 1970s, and besides Sperrings 1 (Early Comb Ware, Ka I:1), Morby Ware pottery from the Early Metal Period has also been found. The osteological material is composed mainly of seals, but regarding the early times of the excavation and the lack of sieving, the fish bones may possibly be underrepresented. The site has also yielded quartz, stone tools (axes, adzes, slate pendants) and a few pieces of decorated clay figurines of the so-called Paimio type. Today, there is a modern settlement at the area where the site was situated (Leskinen & Pesonen 2008).

Radiocarbon dates: Ua-32194: 5925±45 BP (-24,80‰ °13C), charred crust (Sperrings 1). One of the earliest dates of Sperrings 1 in Finland.

Osteological material (Vantaa Etelä-Vantaa 3/Mätäoja III (Palmu)):

Animal species	**No. of bone fragments**
Seals (*Phocidae*)	211
Eurasian beaver (*Castor fiber*)	1
Mountain hare (*Lepus timidus*)	3
Ducks (*Anatidae*)	2
Unidentified birds (*Aves.*)	7

2. Padasjoki Leirintäalue (campsite)
Coordinates (EUREF-FIN): N 61.36479359, E 25.29168219, Z 85 m asl
Research history: Survey 1971 (M. Huurre), excavations 1999, 2001–2002, 2006–2007, 2013 (S. Vanhatalo, N. Strandberg, K. Luoto, T. Karjalainen, P. Kouki)

The site is located by the Lake Päijänne, one of the biggest lakes in central Finland, on a woody terrain between the lake-shore and a steep rocky hill in the west. The surroundings of the site are highly aquatic but would also permit fair access to terrestrial resources. Besides Sperrings 1 (Ka I:1), the site has also yielded Sperrings 2 (Ka I:2) and Typical Comb Ware sherds. The find material consists mainly of quartz, ceramics and burnt bones. Pieces of a crucible indicate a later (Metal Period) use of the site.

Radiocarbon dates: The site has not so far been C14-dated.

Osteological material (Padasjoki Leirintäalue):

Animal species	No. of bone fragments
Moose (*Alces alces*)	5
Red fox (*Vulpes vulpes*)	1
Eurasian beaver (*Castor fiber*)	23
Pike (*Esox lucius*)	49
Perch (*Perca fluviatilis*)	15
Percid fish (*Percidae* sp.)	2
Cyprinids (*Cyprinidae*.)	14
Burbot (*Lota lota*)	2
Unidentified fish (*Teleostei*)	171

3. Espoo Kläppkärr
Coordinates (EUREF-FIN): N 60.20824798, E 24.58405355, Z 25–35 m asl
Research history: Surveys 1934, 1962, 2004 (E. Kivikoski, M. Huurre, H. Jansson), excavations 1998–1999 (J. Fast)

The site is located in southern Finland, west of Helsinki. Kläppkärr lies near the type site of Sperrings, on the shore of a former Littorina Sea bay. Today, it is partly located at an open land area and woodland. There have been small-scale excavations at the site in the 2000s. The find material includes Sperrings 2 (Ka I:2) and Corded Ware ceramics. The stone tool inventory is composed of a series of axes, adzes, a slate pendant and whetstones (Europaeus-Äyräpää 1930).

Radiocarbon dates: Hela-3173: 5439±43 BP (–26,00‰ δ13C), charred crust (Sperrings 2).
Osteological material: The osteological material has not so far been analyzed.

4. Lappeenranta (Etu- ja Taka-) Muntero
Coordinates (EUREF-FIN): N 61.04727687, E 28.05227743, Z 80–83 m asl
Research history: Surveys 1974, 1976, 1988, 1993, 1994, 1997, 1998, 2006, 2007, 2010, 2012 (T. Miettinen, M. Huurre, P. Pesonen, J. Luoto, T. Jussila, E. Mikkola, P. Kankkunen, J. Lagerstedt), excavations 1998, 2004, 2006–2009, 2012–2013 (J. Luoto, P. Kankkunen, E. Mikkola, P. Pesonen, T. Rostedt)

The site is located on the shore of a small pond that was formerly part of the Ancient Lake Saimaa. Lake Saimaa was the largest inland water body in Holocene Finland. The site has yielded material from several periods of the Stone Age, e.g., Sperrings 1 Ware (Ka 1:1), Sperrings 2 Ware (Ka I:2), Early Asbestos Ware and Typical Comb Ware. There have been small-scale excavations between 1998 and

2013, of which the latest was the largest so far. This excavation concentrated on the Sperrings 2/Early Asbestos Ware phase of the site, and yielded a mass of ceramics and burnt bones with some quartz material. The sampled sherds are from this phase of the excavations.

Radiocarbon dates: Hela-2231: 164±30 BP (-25,50‰ δ13C), charcoal, irrelevant date; Hela-2232: 1077+/-30 BP (-26,70‰ δ13C), charcoal, irrelevant date; Hela-2295: 5818+/-41 BP (-26,20‰ δ13C), burnt bone (meso mammal), connected with the Sperrings 1 (Ka I:1) phase of the site; Hela-2296: 5783+/-39 BP (-27,50‰ δ13C), burnt bone (mammal), connected with the Sperrings 1 (Ka I:1) phase of the site.

Osteological material: The osteological material has not been analyzed so far.

5. Oulu (formerly Ylikiiminki) Vepsänkangas
Coordinates (EUREF-FIN): N 64.99060160, E 26.22206459, Z 79 m asl
Research history: Survey 1989 (M. Mäkivuoti), excavations 1992, 1996-1998 (M. Mäkivuoti, S. Koivisto)

The vast settlement site was originally located at the north coast of the Littorina Sea, but also in close proximity to terrestrial resources. Today, it is surrounded by marshlands. Sand extraction has destroyed a big part of the site. There have been extensive excavations in the 1990s. The site is a pure Säräisniemi 1 site, at least no ceramic material from other periods has been recovered as of yet. Besides ceramics, the site has yielded stone tools, quartz, flint arrowheads (Slettnes-type), burnt bones, and pieces of "chewing resin", i.e. birch bark pitch pieces with teeth marks (Koivisto 1998; Torvinen 2000).

Radiocarbon dates: Hel-4126: 2810±90 BP (-26,70‰ δ13C), charcoal; Hel-4127: 6170±90 (-25,90‰ δ13C), charcoal; Hela-128: 5995±65 (-22,20‰ δ13C), charred crust; Hela-129: 6020±80 (-27,20‰ δ13C), "chewing resin"; Hela-235: 6065±75 (-27,50‰ δ13C), "chewing resin"; Hela-236: 6120±75 (-26,30‰ δ13C), charred crust (Säräisniemi 1 ceramics); Hela-312: 5990±60 (-27,30‰ δ13C), "chewing resin"; Hela-313: 3130±70 (-26,50‰ δ13C), charcoal. It seems that most of the charcoal datings are not related to the Stone Age settlement, while charred crust and birch bark pitch datings are well in line with each other and the Säräisniemi 1 context.

Osteological material (Oulu Vepsänkangas):

Animal species	No. of bone fragments
Seals (*Phocidae.*)	193
Moose (*Alces alces*)	11
Reindeer (*Rangifer tarandus*)	1
Eurasian Beaver (*Castor fiber*)	22
Mountain Hare (*Lepus timidus*)	1
Other mammals (*Mammalia* sp.)	24
Common Teal (*Anas crecca*)	13
Mallard (*Anas platyrhynchos*)	14
Eurasian Wigeon (*Anas penelope*)	8
Long-tailed duck (*Clangula hyemalis*)	4
Velvet scoter (*Melanitta fusca*)	2
Smew (*Mergus albellus*)	6
Diving ducks (*Aythya* sp.)	7
Common eider (*Somateria mollissima*)	1
Ducks (*Anatidae*)	2
Divers (*Gavia* sp.)	8
Grebes (*Podiceps* sp.)	1
Other birds (*Aves*)	47
Pike (*Esox lucius*)	68
Cyprinids (*Cyprinidae.*)	10
Unidentified fish (*Teleostei*)	75

6. Raasepori Telegrafberget
Coordinates (EUREF-FIN): N 60.01547696, E 23.68625724, Z 29 m asl
Research history: Survey 1999 (J. Fast), excavation 2001 (J. Fast)

The site is located at the southern coast of Finland in a Littorina Sea bay. There has been only a small test excavation at the site, and it may well be that most of it has been destroyed due to sand extracting. The ceramics found are exclusively of the Jäkärlä Ware type accompanied with quartz lithics.

Radiocarbon dates: Hela-3168: 5211±40 BP (-26,80‰ δ13C), charred crust (Jäkärlä Ware), one of the few crust dates produced from Jäkärlä Ware contexts.

Osteological material: The osteological material has not so far been analyzed.