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The adoption of pottery by north-east European hunter-gatherers: Evidence from lipid residue analysis

Ester Oras [a, e, *], Alexandre Lucquin [b], Lembi Lõugas [c], Mari Tõrv [d, e], Aivar Kriiska [e], Oliver E. Craig [b]

[a] *Institute of Chemistry, University of Tartu, Ravila 14A, 50411, Tartu, Estonia*
[b] *BioArCh, Department of Archaeology, University of York, Wentworth Way, Heslington, York, YO10 5NG, United Kingdom*
[c] *Archaeological Research Collection, Tallinn University, Rüütli 10, 10130, Tallinn, Estonia*
[d] *Centre for Baltic and Scandinavian Archaeology (ZBSA), Schleswig-Holstein State Museums Foundation, Schlossinsel 1, D-24837, Schleswig, Germany*
[e] *Institute of History and Archaeology, University of Tartu, Jakobi 2, 51014, Tartu, Estonia*

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ABSTRACT

Pottery was adopted by hunter-gatherers in the Eastern Baltic at the end of the 6th millennium cal BC. To examine the motivations for this cultural and technological shift, here we report the organic residue analysis of ceramic vessels from the earliest pottery horizon (Narva) in this region. A combined approach using GC-MS, GC-C-IRMS and bulk IRMS of residues absorbed into the ceramic and charred surface deposits was employed. The results show that despite variable preservation, Narva ceramic vessels were preferentially used for processing aquatic products. We argue that pottery was part of a new Late Mesolithic subsistence strategy which included more intensive exploitation of aquatic foods and may have had important implications, such as increased sedentism and population growth.

1. Introduction

The arrival of pottery marks a clear archaeological horizon in the Northern Eurasian prehistoric sequence, and is often synonymous with the period known as the Neolithic, despite predating agriculture by several thousand years. In the Eastern Baltic this transition occurred during the late 6th millennium cal BC, and may have been part of an east-west expansion of pottery technology from the Eurasian steppe (Dolukhanov et al., 2005; Piezonka, 2015) and even further east from the Palaeolithic ceramic cultures of Eastern Russia and Japan (Gibbs, 2015; Gibbs and Jordan, 2013; Silva et al., 2014). It has been proposed that pottery containers were part of a pre-farming package of technological innovations that led to more intensive food procurement, increased sedentism, higher population densities and the development of new exchange networks (Jordan and Zvelebil, 2009; Nordqvist and Kriiska, 2015). In this sense, pottery may have been as important as the agriculture in guiding Europe's subsequent cultural trajectory. The appearance of pottery certainly correlates with the flourishing of a broad range of late foragers across the Baltic in the 5th millennium cal BC, which also corresponds to the Holocene Thermal Maximum (e.g. Seppä et al., 2009). However, the motivations for the introduction of pottery at this juncture in Baltic prehistory remain unclear. Indeed, in the Eastern Baltic, there seems to be little evidence for dramatic changes in the subsistence economies or settlement patterns following the first appearance of ceramic vessels (Kriiska et al., forthcoming) and an alternative hypothesis is that pottery made a rather ‘silent’ impact despite their visibility in the archaeological record. Instead of transforming prehistoric societies and economies, pottery may have merely represented a small step in container evolution fulfilling niches already occupied by perishable containers.

To clarify their role and potential impact on the local economy, here we provide the first evidence of the use of pottery belonging to the earliest ceramic phases of the Eastern Baltic using lipid residue analysis. This approach involves the chemical and isotopic characterisation of lipids that become deposited within ceramic or

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[*] Corresponding author. Institute of Chemistry, University of Tartu, Ravila 14A, 50411, Tartu, Estonia.
E-mail address: ester.oras@ut.ee (E. Oras).

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charred surface deposits during their use (Evershed et al., 2001; Evershed, 2008; Craig et al., 2004, 2012). Lipid residue analysis has been applied to study pottery use by East Asian foragers (Craig et al., 2013; Lucquin et al., 2016) and by early farmers in Europe and the Near East (Copley et al., 2002; Cramp et al., 2014; Evershed et al., 2008b). However, far less attention has been given to the uptake and use of pottery by European foragers, although there has been some work on the western Baltic ceramic hunter-gatherers (Papmehl-Dufay, 2006; Isaksson, 2009; Craig et al., 2011).

Our study focuses on the Narva culture of Estonia dating from the late 6th to the 5th millennium cal BC (Piezonka, 2012, 2015; Kriiska et al., forthcoming), perhaps the best known example of pottery using foragers in the Eastern Baltic. Narva-type ceramics include several different forms, from pointed-base vessels and shallow oblong bowls and represent the earliest pottery complex of this region. They are characterised by striated or smoothed surfaces, but sometimes also decorated with pits, notches, grooves and comb impressions (Fig. 1). This pottery type is distributed across a range of hunter-gatherer societies throughout the Baltic countries, North-West Russia and Belorussia. Although, the technological aspects of Narva vessels have been well studied (e.g. Kriiska, 1996; Mikšaitė, 2005), direct chemical evidence of their use is currently missing.

2. Material and methods

2.1. Narva pottery and sites analysed

In Estonia, there are around 30 known Narva settlements, including the type site Narva Joaorg, and four Narva burial sites. Settlements cover coastal, island, lagoon and inland areas, and include both short-term seasonal camps and long-term habitation sites. Here we targeted a wide selection of sites from different environmental and habitational contexts. The multi-site comparative approach was chosen to determine whether the earliest pottery in this region had a common use or whether use varied by site type and environmental conditions. The dates from Narva stage sites in Estonia range from 5200 to 3900 cal BC with directly dated Narva sherds from 4850 to 4710 cal BC and 4590 to 4440 cal BC (Kriiska et al., forthcoming). The latter were also included in current analysis. In total 100 samples from 65 sherds and 12 different Narva sites all over Estonia (Fig. 2; Table 1) were analysed.

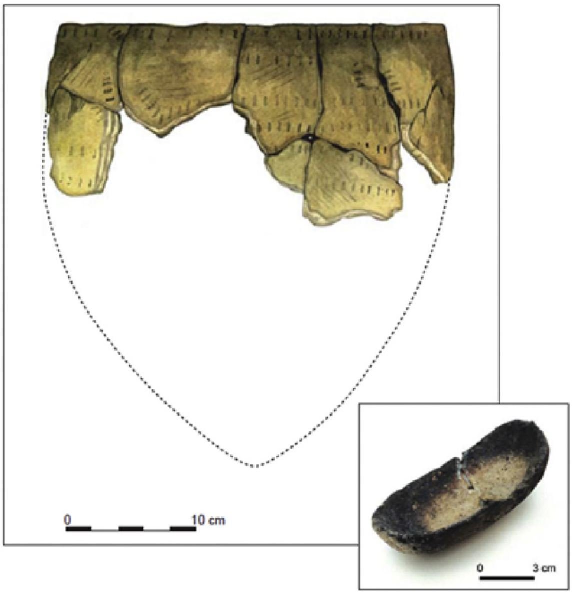

Fig. 1. Examples of Narva-type vessels from Estonia. Reconstruction of the Narva-type pot and shallow oblong bowl from Kääpa. Drawing by Diana Selli and photo by Kriiska et al., 1999.

2.2. Methods

Where available, both carbonised deposits or food crusts, visible on the pottery surface (ca. 20 mg of sample for GC-MS and 1 mg for bulk IRMS) and ceramic matrices (ca. 1 g) were mechanically removed. Lipids were extracted using a one-step acid/methanol extraction technique following previously established extraction protocols (Craig et al., 2013; Colonese et al., 2015; Heron et al., 2015; Papakosta et al., 2015). Briefly, acid catalysed lipid extraction and methylation with MeOH (70° C, 4 h) was conducted after which lipids were extracted with *n*-hexane (3 × 2 ml). The samples were dried up under the stream of nitrogen at 37.5° C, dissolved in 100 μl of *n*-hexane with the addition of 10 μg of internal standard of C36:0 (*n*-hexatriacontane) and analysed directly with GC-FID, GC-MS, and GC-C-IRMS.

General screening and quantification of the lipid extract was carried out by GC-FID (gas chromatography - flame ionization detector) using an Agilent 7890A gas chromatograph (Agilent Technologies, Cheadle, Cheshire, UK). The injector was splitless and maintained at 300 °C. One μl of sample dissolved in hexane was injected into the GC. The column used was a 100% Dimethylpolysiloxane DB-1 (15 m × 320 μm x 0.1 μm; J&W Scientific, Folsom, CA, USA). The carrier gas was hydrogen with a constant flow rate of 2 ml min^{-1}. The temperature program was set at 100 °C for 2 min, rose by 20 °C min^{-1} until 325 °C. This temperature was maintained for 3 min. Total run time was 16.25 min. The lower boundaries of interpretable archaeological lipid extract were 0.1 μg mg^{-1} of food crust corresponding roughly to 2 μg of extracted lipids (Craig et al., 2013) or 5 μg g^{-1} of ceramic powder corresponding roughly to 5 μg of extracted lipids (Evershed, 2008).

GC-MS (gas chromatography-mass spectrometry) analysis was performed with Agilent 7690A Series gas chromatography and Agilent 5975C Inert XL mass-selective detector with a quadrupole mass analyser with Triple-Axis Detector (Agilent Technologies, Cheadle, Cheshire, UK) were used. The splitless injector and interface were maintained at 300 °C and 280 °C respectively. One μl of sample dissolved in hexane was injected. Helium was the carrier gas at constant inlet pressure. The GC column was inserted directly into the ion source of the mass spectrometer. The ionization energy was 70 eV and spectra were obtained by scanning between m/z 50 and 800. All samples were analysed using a DB5-MS (5%-phenyl)-methylpolysiloxane column (30 m × 0.32 mm × 0.25 μm; J&W Scientific, Folsom, CA, USA) with the temperature program of 2 min at 50 °C, 10 °C min^{-1} to 325 °C and 15 min at 325 °C. The identification of compounds was conducted with the Agilent Chemstation software according to their mass spectrum, their retention time and with the help of NIST 2008 library of mass spectra.

GC-C-IRMS (gas chromatography-combustion-isotope ratio mass spectrometry) analysis of the samples with sufficient lipid preservation was conducted in order to estimate the ^{13}C/^{12}C ratio in two most abundant $C_{16:0}$ and $C_{18:0}$ fatty acids. This analysis provides further information for distinguishing different substances.

The samples were analysed using an Agilent 7890B GC (Agilent Technologies, Santa Clara, CA, USA) instrument coupled to an Agilent 5975C MSD and an Isoprime 100 IRMS (Isoprime, Cheadle, UK) with an Isoprime GC5 interface (Isoprime, Cheadle, UK). All samples were diluted with hexane and subsequently 1 μl of each sample was injected into a DB-5MS (30 m × 0.25 mm × 0.25 μm) fused-silica column. The temperature was set for 0.5 min at 50 °C, and raised