Late Glacial Hunter-Gatherer Pottery in the Russian Far East: Presumed Diversity in Origin and Use

Authors: Shinya Shoda (1,2), Alexandre Lucquin (1), Oksana Yanshina (3), Yaroslav Kuzmin (4,5), Igor Shevkomud (6)†, Vitaly Medvedev (7), Evgeniya Derevianko (7), Zoya Lapshina (8), Oliver E. Craig (1), Peter Jordan (9).

Corresponding author: Shinya Shoda shinya.shoda@york.ac.uk

Affiliations:

1 BioArCh, University of York / Wentworth Way, Heslington, York, YO10 5NG, UK

2 Nara National Research Institute for Cultural Properties / Nijo 2-9-1, Nara, Nara, 630-8577, Japan

3 Peter the Great Museum of Anthropology and Ethnography, Russian Academy of Sciences, 199034, Saint-Petersburg, Universitetskaya Naberezhnaya 3, Russia

4 Sobolev Institute of Geology and Mineralogy, Siberian Branch of the Russian Academy of Sciences /Koptyug Avenue 3, Novosibirsk 630090, Russia

5 Laboratory of Mesozoic and Cenozoic Continental Ecosystems, Tomsk State University /Lenin Avenue 36, Tomsk 634050, Russia

6 Department of Archaeology, Khabarovsk State Museum, Turgenev Street 86, Khabarovsk 680000, Russia

7 Institute of Archaeology and Ethnography, Siberian Branch of the Russian Academy of Sciences / Lavrentiev Avenue 17, Novosibirsk 630090, Russia

8 Khabarovsk Theological Seminary / Turgenev Street 26, Khabarovsk 680000, Russia

9 Arctic Centre and the Groningen Institute of Archaeology (GIA), University of Groningen, Aweg 30, 9718CW, Groningen, the Netherlands.

† Deceased in September 2015

Abstract: During the Late Glacial, hunter-gatherers began using ceramic cooking containers in three separate geographic regions of East Asia: China, Japan and along the Amur River in the Russian Far East. While recent research has clarified the use of early pottery in Japan, very little is known about what led to the emergence of pottery in the other two areas, including the likely environmental, economic or cultural drivers. In this paper we focus on the Russian Far East, where pottery has been recovered from dated contexts that span circa 16,200 to 10,200 years ago (cal BP). Interpreting the use of early pottery along the Amur River has been difficult because the region's acidic soils make palaeo-economic reconstructions challenging. To address this gap in knowledge we undertook lipid residue analysis of 28 pot sherds from the sites of Khummi, Gasya, and Goncharka 1 on the Lower Amur River, and the Gromatukha site on the Middle Amur. Our results indicate that pottery was employed to process aquatic oils at sites on the Lower Amur, a pattern of use that aligns with similar results from Japan, and suggests that fishing – probably of

salmonids and freshwater fish – was becoming increasingly important during this period. In contrast, the results from the Middle Amur indicate a different pattern showing a significant contribution of ruminant animals. These regional differences in use are also mirrored in contrasting manufacturing techniques with pottery from the Middle and Lower Amur forming distinct ceramic traditions. These combined insights may point to greater local variability in the development and use of early pottery in East Asia than has previously been indicated.

Key words: Early pottery; Initial Neolithic; Lipid residue analysis; Compound-specific isotope analysis; Phytanic acid diastereomer; Osipovka Culture; Gromatukha Culture.

1. Introduction

Prehistoric hunter-gatherer societies in the Russian Far East (hereafter RFE) played an important yet poorly understood role in the emergence of the world's earliest pottery in the Late Glacial (ca. 16,000-10,000 years ago; hereafter – cal BP, e.g. Kuzmin 2015, 2017). Together with southern China and Japan, the RFE represents one of the three main clusters of early pottery production in East Asia, and there is now clear evidence that pottery was already in use at a range of sites on the lower and middle reaches of the Amur River from ca. 16,000 cal BP. This pottery was being used on a limited scale, with small numbers of sherds recovered from sites affiliated with two different archaeological cultures– the Osipovka and Gromatukha (Kuzmin 2002; 2017; Zhushchikhovskaya 2005; Derevyanko and Medvedev 2006; Shevkomud and Yanshina 2012; Yanshina 2017). The sites of Khummi, Gasya and Goncharka 1 are located in the extensive lowlands of Lower Amur River, and belong to the Osipovka culture. Around 700 km further westwards, the site of Gromatukha is located on the west bank of Zeya River, tributary river to the Amur, and provides the type site for the Gromatukha culture (Figure. 1). To date, no other Late Glacial pottery sites have been identified in the region under consiceration between areas occupied by these cultures.

While Russian archaeologists have long speculated about the likely economic factors that drove early pottery innovation in this area (e.g. Medvedev 1995; Zhushchikhovskaya 2005; Kuzmin 2013), there has been no direct evidence to indicate how the vessels were used. This is because of the region's acidic soil conditions which result in very limited preservation of organic materials and render detailed palaeo-economic reconstructions impossible. To address this gap in knowledge, our goal was to deploy lipid residue analysis to generate the first direct evidence for how the Late Glacial pottery was used in the RFE. The method has already been successfully employed at early pottery sites in Japan (Craig et al. 2013; Lucquin, Gibbs et al. 2016; Lucquin et al. 2018), Korea (Shoda et al. 2017) and Sakhalin Island (Gibbs et al. 2017), and emerging results from these areas indicates close links between the appearance of the first

ceramic cooking containers and the processing of aquatic resources. Our aim was to test whether the use of early pottery in RFE was also linked to processing of aquatic resources, or to other kinds of plant or animals.

2. Regional setting

Unlike the surrounding regions of East Asia, only one Late Paleolithic site - Golyi Mys 4 - is known from the Lower Amur River (Derevianko et al. 2006: 69–72). This site lacks pottery but yields microblade cores and scrapers. From 18,000 to 11,000 cal BP the Amur region experienced climatic amelioration, leading to the expansion of coniferous and mixed coniferous broad-leaved forests. Pottery starts to emerge at around the same time, but Russian archaeologists initially attributed these early ceramic layers to the Neolithic period and assumed that they dated to the Holocene. Radiocarbon dating has since demonstrated that the oldest pottery layers date to the Late Glacial (e.g. Kuzmin 2015, 2017). Sites with pre-Holocene pottery assemblages are widely scattered across the RFE, and are now defined as representing the onset of an 'Initial Neolithic' that is defined by the appearance of pottery and some other Neolithic novelties like polished axes, bifacially retouched or polished projectiles, new types of scrapers and art objects (e.g. Kuzmin 2002; Zhushchikhovskaya 2005; Derevianko and Medvedev 2006; Shevkomud and Yanshina 2012). Despite their neighboring disposition and synchroneity, these two cultures are distinguished quite sharply and separated by the vast empty area.

2.1. Sites and Pottery Assemblages of the Osipovka Culture

The Osipovka Culture includes around 70 archaeological sites, of which at least 15 have been excavated. All are located on the banks of the main Amur River and are associated with some of the earliest human occupation of the Lower Amur region so far discovered. The full time-range of the Osipovka Culture is 13,260 ± 100 to 9,890 ± 230 yr BP or 16,200-10,700 cal BP (Kuzmin 2006b; Kuzmin and Shevkomud 2003; Shevkomud and Kuzmin 2009; Shevkomud and Yanshina 2012). Two stages are tentatively outlined in the Osipovka culture (Table 1): the first stage is represented by the artifacts collected from disturbed layers while the second stage provides the majority of data including in situ contexts of stone tools and pottery. The most studied sites are Gasya, Khummi and Goncharka-1 (Figure. 1).

The **Gasya** site is located ca. 80 km downstream from the Khabarovsk on the cliff 13-16 m above the Amur river. The stratified site was excavated between 1975 and 1990 (Derevyanko, Medvedev 2006), leading to the recovery of cultural remains from different periods (e.g. Derevianko and Medvedev 2006). The ^{14}C dates for the charcoal samples from Osipovka Culture are in the range 12,960-10,875 yr BP or 15,870-12,660 cal BP. This age is further supported by thermoluminescence dating of the pottery itself (Kuzmin et

al. 2001). Several dozen potsherds of the Osipovka Culture have been recovered from the site, with major part of them derived from the lower cultural layers, while a few potsherds were dispersed throughout the upper layers, some in association with later artefacts. The oldest levels included fragments of crudely-made, plant fiber-tempered pottery with parallel grooves serving as rudimentary decoration (Figure 2). Among them, one vessel was reconstructed as a flat-bottomed container with a volume of ca. 5.5–6 litres. It was ca. 25–27 cm high, with thick walls of around 1.2–1.7 cm in width (e.g. Derevianko and Medvedev 2006; Kuzmin 2006a). There are traces of carbonised remains on both external and internal surfaces of the pottery, indicating that it had probably been used for cooking.

The **Khummi** site is the easternmost one of the Osipovka Culture, and was excavated between 1991-1997 (Lapshina 1999). The site is located ca. 20 km upstream from the city of Komsomolsk-on-Amur (Figure 1), on the ca. 30 m high bank of the Amur River. The cultural deposits are relatively homogenous, and contain materials primarily attributed to the Initial Neolithic. The ^{14}C dates for the Osipovka cultural stratum correspond with two stages of the Osipovka culture and span the broad range of ca. 13,260-10,375 yr BP or 16,240-11,820 cal BP (Kuzmin 1997). Only around 40 potsherds of the Osipovska Culture were recovered from this site, despite extensive excavation of the Late Glacial horizons (Figure 2), further suggesting that pottery was used on a very limited scale. Although it is difficult to separate these findings into earlier and later phases in terms of the layers, considering the design of potsherds, most of potsherds appear to correlate with the oldest stage of Osipovka Culture (Yanshina, Lapshina 2008; Shevkomud and Yanshina 2012: 195-207; 249).

The **Goncharka-1** is the best-studied site of the Osipovka Culture, and is located ca. 20 km upstream from the city of Khabarovsk, on the high terrace situated ca. 20 m above the river (Shevkomud and Yanshina 2012). The lower horizon (layers 4-5) of the site represents the earliest stage of the Osipovka Culture and have ^{14}C dates on charcoal that fall between 12,500-12,055 yr BP or 15,070-13,750 cal BP. The upper horizon (layer 3B, Russian labelling is "3Б") belong to the late stage of the Osipovka Culture, and has been dated to 11,340-9890 yr BP or 13,300-10,650 cal BP (on charcoal samples) and 11,650-10,060 yr BP or 13,590-11,330 cal BP (on foodcrusts) (Kuzmin 2006b; Kuzmin and Shevkomud 2003; Shevkomud and Kuzmin 2009; Shevkomud and Yanshina 2012). Most artefacts, household structures and evidence of ritual activities are found in layer 3B. More than 2000 potsherds of the Osipovka Culture have been recovered from the site, but only 130 of these are derived from the oldest layers (4-5) (Shevkomud and Yanshina 2012).

In general, there is substantial variability within the pottery of the Osipovka Culture, though all pottery appear to have had flat bottoms and thick walls. Their shapes are either conical or slightly restricted in the upper part. The clay paste was tempered with diverse materials, including rocks, dried clay or grog, with

plant tempers used infrequently at the early stage. Scraping with hard comb-like tools inside and sometimes outside vessels is the most specific way of pots processing and meets at both stages of the Osipovka culture. Besides, the earliest potsherds have featureless cord marks on the outer surfaces instead of decoration, while the latest vessels are decorated in different patterns typically by comb-like tools (Figure. 2-3) (Yanshina 2017).

2.2. Sites and Pottery Assemblages of Gromatukha Culture

The Gromatukha Culture has been less well researched. To date, only eight sites have been discovered, and only three have been excavated. The sites are situated in different kinds of landscapes compared to those of Osipovka Culture, with some directly on the banks of Amur River, but others also situated on the banks of its smaller northern tributaries. The chronology of the Gromatukha Culture overlaps to some extent with the Selemdga Palaeolithic Culture, suggesting that pottery was in use at some sites but not at others.

The **Gromatukha** site was used to define the Gromatukha Culture and provides almost all the available information for it (Okladnikov and Derevyanko 1977). The site is situated on the high bank of the Zeya River, a tributary joining the middle course of the Amur River (Figure 1). The main excavations took place in the 1960s (Okladnikov and Derevianko 1977), and smaller-scale work has been done in the 2000's to 2010's (Derevyanko et al. 2004; Derevianko et al. 2017). The ^{14}C dates for the lowest cultural component of the Gromatukha site were run on charcoal and fall in the range of 12,380– 9895 yr BP or 14,820-11,200 cal BP. Direct 14C dating of pottery using oxygen and oxidation temperature of 400° C resulted in ages of ca. 13,240–13,310 yr BP or 15,900–16,000 cal BP (O'Malley et al. 1999), confirming the Late Glacial age of the Gromatukha pottery. Sixteen ^{14}C dates on foodcrusts fall within 12,400–9150 yr BP or 15,010-10,190 cal BP (Derevyanko et al. 2017). Based on pottery analysis (Shevkomud and Yanshina 2012: 213-230) and parallel ^{14}C dating, two stages of the site occupation could be tentatively recognized. The earliest stage is in the range of 12,530–12,170 yr BP or 15,120-13,900 cal BP, while the latest one has ages of 10,060–9,150 yr BP or 11,970-10,190 cal BP (Derevyanko et al 2017).

Several hundred pottery fragments have been recovered (Okladnikov and Derevianko 1977; Shevkomud and Yanshina 2012: 213-230). The vessels have a slightly conical shape, both flat and round bottoms, thick walls of ca. 0.7-1.3 cm tempered with layers of grass additives with some stabbing patterns on the surfaces. The pottery vessels also have cord marks on their surfaces, with grooves on both internal and external surfaces (Figure 2). Dense zigzag lines arranged in horizontal bands adorn the vessels from top to bottom. However, pots in the later horizon are characterized by significant reductions in the amount of plant fibers

additives, cord marks and zigzag patterns (detailed reviews of this subject can be found in: Shevkomud and Yanshina 2012: 207-228; Yanshina 2017).

3. Materials

To investigate the use of the earliest pottery in the RFE, absorbed lipid residues were extracted from one pottery sherds from Khummi (n=1), Gasya (n=3), Goncharka-1 (n=19) and Gromatukha (n=5) sites (Table 2). All the samples were small sherds (ca. 3-5cm square) without carbonized deposits on the surface. While these samples are partly correlated with the clear-defined stages of each of the two cultures, some of them are lacking their contextual information.

Pottery sherds from Khummi and Gasya have no contextual information while ^{14}C dates of these sites show very wide time ranges: 13,260-10,375 yr BP or 16,240-11,820 cal BP in the former, 12,960-10,875 yr BP or 15,870-12,660 cal BP in the latter. Therefore, the analyzed samples can be dated only within these broad limits, although both sites have the oldest dates for the Osipovka culture and the smallest collections of potsherds.

The contexts of the samples from Goncharka-1 are summarized in table 4. The estimated ages of each sample are based on the ^{14}C dates of charcoal, stratigraphy, and the contexts. Most of the samples belong to the horizon 3B and correspond to the late stage of the Osipovka culture while some of them belong to lower layers.

All the Gromatukha sherds analysed are from the excavation in 1965-1966 without the information of grids and layers. Given their appearance (intensively corded surfaces, greater amount of grass additives and the design pattern), all five samples are associated with the early stage of the Gromatukha culture and therefore with the Bølling-Allerød period. Direct ^{14}C dates of such kind of pottery vary from 12,530-11,440 yr BP or 15,117-13,136 cal BP although later dates cannot be excluded (Derevianko et al. 2017).

4. Methods

4.1. Lipid residue extraction from ceramic powder

Lipid residue analysis was conducted following established acidified methanol protocols (Craig et al. 2013, Papakosta et al. 2015). Briefly, methanol was added to the drilled ceramic powder sample (4 ml of methanol to 1 g of sample) which was then sonicated for 15 minutes. Concentrated sulfuric acid (800 μl) was added to acidify the samples which were then sealed and heated at 70 °C for 4 hours. After cooling to room temperature, lipids were extracted with *n*-hexane (3 x 2 ml) and directly analyzed by Gas Chromatography Flame-Ionization Detection (GC–FID) for the quantification, Gas Chromatography – Mass Spectrometry

(GC–MS) for the biomarker identification as well as Gas Chromatography–combustion–Isotope Ratio Mass Spectrometry (GC–c–IRMS) for the measurement of compound-specific carbon stable isotopic ratios.

Additionally, where additional sample was available, solvent extraction was conducted following established protocols (Evershed et al. 1990). Here, lipids were extracted using DCM/MeoH (2:1 v/v, 3 x 2ml). The solvent was removed, dried under a gentle stream of N_2 and silylated with N, O-bis(trimethylsilyl)trifluoroacetamide with 1% trimethylchlorosilane (70 °C, 1 hour). The resulting total lipid extract (TLE) was dried under N_2. The extracts were dissolved in *n*-hexane before analysis by GC–MS.

4.2. Gas Chromatography (GC–FID)

General screening and quantification of the lipid extract was realised by GC-FID (gas chromatography - flame ionization detector). Analyses were carried out using an Agilent 7890A gas chromatograph (Agilent Technologies, Cheadle, Cheshire, UK). The injector was splitless and maintained at 300 °C and injected 1 μl of sample into the GC. The column used was a 100% Dimethylpolysiloxane DB-1 (15 m x 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 minutes, rose by 20 °C min^{-1} until 325 °C. This temperature was maintained for 3 minutes. Total run time was 16.25 minutes. The lower boundaries of interpretable archaeological lipid extract were 5μg g^{-1} of sherd sample powder (Evershed et al. 2008).

4.3. Gas Chromatography – Mass Spectrometry (GC–MS)

GC–MS analysis was carried out using an Agilent 7890A series chromatograph attached to an Agilent 5975C Inert XL mass-selective detector with a quadrupole mass analyser (Agilent technologies, Cheadle, Chershire, UK). A splitless injector was used and kept at 300 °C. Helium was used as the carrier gas and inlet/column head-pressure was constant. A DB-5ms column coated with 5% phenyl-methylpolysiloxane column (30 m × 0.250 mm × 0.25 μm; J&W Scientific, Folsom, CA, USA) was used for general scanning. The oven temperature was set at 50 °C for 2 min, then raised by 10 °C min^{-1} until it reached 325 °C where it was held for 15 min until the end of the run. The GC column was inserted directly into the ion source of the mass spectrometer. The ionisation energy of the mass spectrometer was 70 eV and spectra were obtained between m/z 50 and 800.

To obtain the ratio of phytanic acid diastereomer (SRR/RRR) (Lucquin, Colonese, et al. 2016) and detect aquatic biomarkers (Evershed et al. 2008) a DB-23(50%-Cyanopropyl)-methylpolysiloxane column (60 m

× 0.250 mm × 0.25 μm; J & Scientific, Folsom, CA, USA) was used with the mass spectrometer in selected ion monitoring (SIM) mode. The oven temperature was set at 50 °C for 2 min, then raised by 10 °C min^{-1} until it reached 100 °C, then raised by 4 °C min^{-1} to 140 °C, then by 0.5 °C min^{-1} to 160 °C, then by 20 °C min^{-1} to 250 °C where it was maintained for 10 min. The first group of ions (*m/z* 74, 87, 213, 270) monitored 4,8,12-trimethyltridecanoic acid (TMTD) fragmentation, the second group of ions (*m/z* 74, 88, 101, 312) correspond to pristanic acid, the third group of ions (*m/z* 74, 101, 171, 326) corresponding to phytanic acid and the fourth group of ions (*m/z* 74, 105, 262, 290, 318, 346) corresponding to u-(o-alkylphenyl) alkanoic acids with carbon length C_{16} to C_{20}. Helium was used as the carrier gas with a flow rate of 2.4 mL min^{-1}. The relative abundance of two diastereomers of phytanic acids is quantified by the integration of the *m/z* 101 ion. This is reported as %SRR = SRR/total phytanic acid x 100.

4.4. Gas Chromatography – combustion – Isotope Ratio Mass Spectrometry (GC–c–IRMS)

In order to compare with modern and archaeological authentic animal/plant samples, stable carbon isotope ($\delta^{13}C$) values of two major saturated fatty acids ($C_{16:0}$ and $C_{18:0}$) were analyzed by GC-c-IRMS, following a published procedure (Craig et al. 2012). An Isoprime 100 (Isoprime, Cheadle, UK) linked to a Hewlett Packard 7890B series GC (Agilent Technologies, Santa Clara, CA, USA) with an Isoprime GC5 interface (Isoprime Cheadle, UK) was used, with a DB-5MS ultra-inert fused-silica column (60 m x 0.25 mm id x 0.25 μm film thickness). One μL of the acid/methanol extracts, diluted in hexane, was injected using the splitless mode where it was vaporized at 300 °C. The temperature was set for 0.5 min at 50°C, then increased by 25°C min^{-1} to 175 °C, 8 °C min^{-1} to 325 °C and held for 20 min. As the carrier gas, ultra-high purity grade helium was used with a flow rate of 3 ml min^{-1}. The gas flows eluting from the column were split into two streams. One was directed respectively into an Agilent 5975C inert mass spectrometer detector (MSD), for the sake of sample identification and quantification, while the other was directed through the reactor tube to oxidize all the carbon species to CO_2. A clear resolution and a baseline separation of the analyzed peaks were achieved.

Eluted products were ionized in the mass spectrometer by electron impact and ion intensities of *m/z* 44, 45 and 46 were recorded for automatic computing of the $^{13}C/^{12}C$ ratio of each peak in the extracts. Computation was made with IonVantage and IonOS software (Isoprime, Cheadle, UK) and based on comparisons with standard reference gas (CO_2) of known isotopic composition that was repeatedly measured. The $\delta^{13}C$ values obtained are expressed in per mill (‰) relative to the Vienna Pee Dee Belemnite (V-PDB) international standard. The accuracy (< 0.3‰) and precision (< 0.5‰) of each instrument was determined on *n*-alkanoic acid ester standards of known isotopic composition (Indiana standard F8-3). Each sample was measured in replicate (S.D. 0.1‰ for each fatty acid). Values were also corrected subsequent to analysis to account for

the methylation of the carboxyl group that occurs during acid extraction. Corrections were based on comparisons with a standard mixture of $C_{16:0}$ and $C_{18:0}$ fatty acids of known isotopic composition processed in each batch under identical conditions. For a comparison with archaeological data, values were adjusted for the effects of the variation of the atmospheric $\delta^{13}C$ between the Pleistocene and Holocene (Schmitt et al. 2012).

5. Results

The results show interesting contrasts in early pottery use between the middle and lower Amur sites. All samples yielded interpretable amounts of lipids (i.e. > 5μg g^{-1} sherd) with a mean value of 346μg g^{-1} and maximum value of 5009μg g^{-1}(Table 3). The acidified methanol extracts mainly consist of short to long-chain fatty acids, dominated by mid-chain saturated fatty acids such as palmitic acid ($C_{16:0}$) and stearic acid ($C_{18:0}$), unsaturated fatty acids such as oleic acid ($C_{18:1}$), as well as mid to long chain *n*-alkane. A typical partial chromatogram of these samples is shown in Figure 4.

5.1. Identification of aquatic derived lipids

Previous analysis of organic residues of Late Glacial pottery in Japan have demonstrated that a high proportion of sherds were used for processing aquatic resources (Craig et al. 2013; Lucquin, Gibbs et al. 2016, Lucquin et al. 2018). Here, in the Amur region, the full range of aquatic biomarkers, i.e. ω-(o-alkylphenyl) alkanoic acids containing 18 and 20 carbon atoms with at least one isoprenoid fatty acids (Evershed et al. 2008), were identified in only two samples, one from Khummi (KHM1) and one from Goncharka 1 (Amur3). A further four samples (GSH3; GCK09; and Amur 4, 5 and 10) contained fatty acids relatively enriched in ^{13}C and consistent with measurements made on modern marine fish and salmonids that migrate into the Lower Amur River. These samples also have a higher relative amount of the SRR diastereomer of phytanic acid (i.e. >80 %) which is also typical of aquatic organisms (Lucquin, Gibbs et al. 2016). To summarize, the Lower Amur samples all bear evidence for the processing of aquatic resources.

5.2. Identification of non-aquatic derived lipids

The isotope characterization of individual lipid molecules can also be used distinguish whether pottery was used to processes ruminants or non-ruminant fats. The difference in $\delta^{13}C$ values between the two major fatty acids ($C_{16:0}$ and $C_{18:0}$) was calculated ($\Delta^{13}C$) for each sample. Samples with $\Delta^{13}C$ values of less than -1 ‰ are considered to have been derived from ruminant fats (e.g. Dudd and Evershed 1998, Copley et al. 2003, Craig et al. 2012, Robson et al. 2019), as the $C_{18:0}$ fatty acid is relatively depleted in ruminant tissues due to bacterial processing in the rumen (Copley et al. 2003). Although $\Delta^{13}C$ values are a relative measure

considered to be independent of local stable carbon isotopic variation, values obtained from East Asian authentic reference ruminant fats confirm the validity of this criteria (Lucquin, Gibbs et al. 2016; Craig et al. 2013). This approach enabled us to identify ruminant adipose fats in a number of the RFE samples (i.e. GSH01, 02; GCK01, 02, and 08; Amur4 and 5; and GMT01, 02, 04, and 06). Samples with lower $\Delta^{13}C$ also have lower relative amounts of the SRR isomer of phytanic acid, more typical of measurements made on ruminant tissues, the other major source of this compound (Lucquin, Colonese et al. 2016). Interestingly, there is a clear difference in the %SRR between pottery from the different reaches of the Amur river (Figure 5) - samples from the middle Amur (Gromatukha) have a lower %SRR (mean 68.0%, median 66.6%), while pottery from sites in the lower Amur (Khummi, Gasya and Goncharka 1) (mean 88.3%, median 87.3%), supporting the interpretation that ruminant products were processed in the Middle Amur pottery whilst there were absent or at much lower frequency in the Lower Amur pottery.

5.3. Investigating the mixing of resources

The results from our lipid residue analysis of Late Glacial pottery from sites on the Amur River indicate spatial variability in use. It appears that aquatic resources like salmonids predominated in the resources processed in pottery at the Lower Amur sites, while ruminants formed the main resource processed at the Middle Amur site. Additionally, other kinds of plants, freshwater fish or wild non-ruminants may also have been processed in the vessels. In order to investigate the extent of resource mixing at the different early pottery sites we applied a concentration dependent mixing model (Fernandes et al. 2014) that used the $\delta^{13}C_{16:0}$ and $\delta^{13}C_{18:0}$ values, and %SRR as proxies (Lucquin, Colonese et al. 2016).   This model was used to examine the likely proportions of lipids derived from plants (acorns and chestnuts), freshwater fish, wild boar, wild ruminants and salmonids to each pot. The model assumes that the pottery vessels were used multiple times, and thus averages a number of individual measurements made on authentic reference fats. It provides a more accurate overview of how a pottery vessel has been used, as it accounts for uncertainties in specific measurements while taking into account of the fact that fatty acid content can vary between different foodstuffs.   The model outputs percentage values in terms of lipid contribution by weight of salmonids versus ruminants (Table S1). The results indicate that ruminant fats only made a significant contribution in the Gromatuka pottery, whereas salmonids made a much greater contribution at the Lower Amur sites, especially at Goncharka 1 and Gasya, where some vessels may have been used exclusively for processing salmonids (Figure 6).

6. Discussion

This study presents the first organic residue analysis of Late Glacial pottery assemblages from the RFE. The results indicate that early pottery was being used in different ways along different sections of the Amur River – ceramic vessels were used to process aquatic resources at sites on the lower parts of the river (Osipovka Culture), while significant contribution from ruminant animals to the pottery content was observed in the middle reaches (Gromatukha Culture), although this study demonstrate a result from the limited numbers of samples. This is noteworthy as these pottery traditions show clear difference in terms of production techniques as above-mentioned.

The fact that early pottery along the Lower Amur is linked to the processing of aquatic resources is perhaps not particularly surprising given that sites are located along the main banks. Moreover, the Amur River has around 100 freshwater species and several anadromous fish (such as salmon) which migrate from the late spring to early autumn, all of which would have offered abundant resources to prehistoric populations. In addition, Gasya has direct technological evidence for fishing activities, with several net sinker weights recovered from layers that have early pottery (Derevianko and Medvedev 2006: 130). However, it is important to note that bone fishhooks, fibre nets, traps, weirs and baskets would not have survived in the acidic soil conditions. This means that the current results represent the first direct evidence for an association between early pottery and the exploitation of aquatic resources in the Lower Amur River.

Whether exploitation of fish and the development of ceramic cooking containers formed part of a more general pathway towards growing sedentism along the Lower Amur remains unclear. The only sites with evidence of more substantial house pit structure are the Khummi (Lapshina 1999; see also Derevianko and Medvedev 2006) and Goncharka 1 (e.g. Shevkomud and Yanshina 2012), while other sites appear only to have had surface structures. Both would have been adequate for occupations involved at harvesting fish runs, most of which occur anyway in the warmer months. Thus, it seems unlikely that the appearance of the first pottery strictly coincided with a shift towards settled village societies. Pottery could easily have been made, fired and cached at seasonal fish harvesting sites by aggregating populations that were more dispersed at other times of the year.

Whether the presence of seasonal fish harvesting sites triggered a major expansion in early pottery use is also unclear, but appears doubtful. Excavators at all four Lower Amur sites have noted that pottery appears to have been used in only very limited quantities, with only a few tens of sherds recovered from the Initial Neolithic levels. The numbers of sherds is also much lower than at Incipient Jōmon sites in Japan, which broadly date to the same period (e.g. Keally et al. 2003; Kuzmin and Shevkomud 2003), and early pottery in the RFE may perhaps have been used for restricted purposes, such as the preparation of novel or ceremonial foods at annual aggregation sites.

Linking the appearance of early pottery in the RFE with the onset of major climatic and environmental shifts is also difficult. Reconstruction of paleoenvironmental conditions in the RFE indicates: i) the dominance of birch and alder forests at ca. 15,500–13,900 cal BP during the Older Dryas cold phase; ii) light conifer forests with larch groves during the warmer Bølling–Allerød interstadial, ca. 14,900–12,900 cal BP; and iii) the shrub birch and alder formations during the Younger Dryas cold phase, ca. 12,900–11,500 cal BP (Kuzmin 2006b, 2010; see also Klimin et al. 2004). Clearly, early pottery appears at sites that date to both warmer and colder phases (see: Table 1). In contrast, in other parts of East Asia the very earliest pottery seems to appear during some of the coldest climatic conditions in the entire Late Pleistocene (e.g. Kawahata et al. 2017, Meyer et al. 2016), perhaps because aquatic foods may have provided an important alternative to depleted supplies of terrestrial resources.

Interestingly, however, the contrasting use of early pottery in the RFE appears to correlate with different pottery-making traditions. Technological and stylistic analysis has identified three distinct ceramic traditions in East Asia during the Late Glacial: the Lower Amur (Osipovka Culture), the Incipient Jōmon and the Transbaikal/Middle Amur (including the Gromatukha Culture) (Yanshina 2017). While organic residue analysis now appears to show that both Osipovka and the separate Incipient Jōmon pottery tradition both shared a focus on processing aquatic resources (Figure 7), our results from Gromatukha may suggest that pottery from the Transbaikal/Middle Amur tradition, which is found in more continental areas, may have been used in a different ways. This pottery making tradition embraces other early pottery sites like Ust'Karenga in the Transbaikal Region, and further research could clarify whether this was also being used to process ruminants rather than aquatic resources.

6. Conclusions

The use of the Late Glacial pottery that appears around 16,000 years ago in the RFE has long been unclear. This study provides the first direct evidence for early pottery use at sites along the Amur River dated from ca. 16,200 to 10,200 years ago. The results provide several answers but also generates new questions. First, we expected a close general link between early pottery and the processing of aquatic resources, as has already been demonstrated for Japan, Korea and Sakhalin Island. This 'aquatic model' seems applicable in the RFE - but *only* for sites of the Lower Amur Osipovka Culture – where we have confirmed the long-suspected association between the emergence of pottery and what was probably equated to seasonal harvesting of migratory fish. However, the early pottery at Gromatukha on the Middle Amur was being used in a very different way, and the high contribution of ruminants to the pottery content showed a strikingly different pattern of use. Second, these contrasts in use appear to map closely onto the two very

different pottery-making traditions that have been identified in the region (Yanshina 2017), perhaps pointing to greater local variability in the development and early use of pottery in the RFE than has hitherto been appreciated. Potentially, the results from Gromatukha may indicate a contrasting and more 'continental' mode of early pottery use, which does not follow the 'aquatic model' identified in surround regions. More work is needed to establish whether other early pottery sites in surrounding regions of Siberia also follow this alternative pattern.

Acknowledgements

Figure Captions:

Fig. 1. Location of the sites investigated in this study (Osipovka Culture: Goncharka-1, Gasya, Khummi sites; Gromatukha Culture: Gromatukha site)

Fig. 2. Typological differences between pottery of the Osipovka and Gromatukha cultures**.** Shapes and main patterns of the ceramic vessels of the Gromatukha (1-3, 8) and Osipovka cultures (4-9). 1, 3, 5, 7 – Shevkomud and Yanshina 2012: fig. 111-112; 6 – Naganuma et al 2005; 8-9 - Yanshina 2017.

Fig. 3. Pottery from the Osipovka and Gromatukha Cultures form entirely different ceramic traditions. These photographs highlight some of the main differences in pottery fabric, tempers and surface treatments: Gromatukha (8-12), Khummi (13-15), and Goncharka-1 (16-18) sites. Note differences in temper, surface treatment, and zigzag pattern between two ceramic traditions. Type I. Temper: grog (15, 18b), gravel inclusions (12 b, 18a), grass additions (9, 12 a). Type II. Surface treatment: grooves rolling by cord wrapped tool (8-9, 11), haphazard cord impressions (14), combing by hard toothed tool (13, 16). Type III. Zigzag pattern: stepping by cord wrapped tool (8, 10, 11) and rolling by hard toothed tool (17).

Fig. 4. Partial total ion and selected ion chromatograms of extracts from a pottery sherd from Khummi (Osipovka Culture) (Sample KHM01).

A (1): Characteristic distribution of aquatic oil components: total ion chromatogram that showing lipids typical for a heated and degraded aquatic oil, dominated by medium- and long-chain saturated and mono-unsaturated fatty acids (FA) and isoprenoid fatty acids. б,ω-dicarboxylic acids (■) with carbon chain ranges of C_8-C_{13} resolved on a DB-5 chromatography column. **B. Heating of aquatic oils:** Ion chromatogram (*m/z* 105) showing the presence of щ-(o-alkylphenyl) alkanoic acids with 16(✱), 18(+), 20(#) carbon atoms. **C: Isoprenoid fatty acids identified:** Ion chromatogram (*m/z* 101) showing isoprenoid fatty acids, TMTD: 4,8,12-trimethyltridecanoic acid, Pri: pristanic acid and Phy: phytanic acid resolved on a DB-5 chromatography column. **D: Separation of SRR and RRR diastereomers:** Ion chromatogram (*m/z* 101) shows the ratio of phytanic acid diastereomers (SRR and RRR) resolved on a DB-5 chromatography column.

Fig. 5. Plot of the $\Delta^{13}C$ and %SRR of lipids extracted from early pottery from sites on the Amur River; The values are compared to the reference range of aquatic oils and ruminant fats based on authentic samples (Lucquin et al 2016, Lucquin et al 2018) corrected for the recent burning of fossil fuels. Red circles: Khummi, orange: Gasya, brown: Goncharka-1, and blue: Gromatukha. Plots with asterisk meet full criteria of aquatic biomarkers (Evershed et al. 2008).

Fig. 6. Estimated percentage contribution of salmonid and wild ruminant resources using a concentration-dependent mixing model. The model parameters have been previously described (Lucquin et al. 2018). Box plots show model output for individual sample. The boxes represent a 68% credible interval while the whiskers represent a 95% credible interval. The horizontal continuous line indicates the mean while the horizontal discontinuous line indicates the median.

Fig. 7. Comparative plot of the $\delta^{13}C$ values of $C_{16:0}$ and $C_{18:0}$ n-alkanoic acids extracted from pottery from Russian (RFE – this study) and Japanese pottery (refs). A: Samples from Amur river basin, Osipovka culture (black) and Gromatukha culture (red), B: Samples from Incipient Jōmon (Lucquin et al. 2018). Closed symbols represent samples meeting the full criteria for aquatic biomarkers (Evershed et al. 2008). The data is compared with reference ranges for authentic reference lipids from both modern and archaeological material (Lucquin et al. 2016, Shoda et al. 2017, Hansel et al. 2004, Evershed et al. 2008, Ackman and Hooper 1968) plotted at 95% confidence. M: Marine, S: Salmonids, WB: Wild Boar, FW: Freshwater, WR: Wild Ruminant and NU: acorns and nuts.

List of Tables:

Table 1 List of Sites and Samples analyzed in this study. OD – older dryas. AB –Allerød- Bølling interstadial. YD- Younger Dryas

Table 2. List of Sites and Samples analyzed in this study. OD: older dryas, AB: Allerød-Bølling interstadial, YD: Younger Dryas, EH: Early Holocene.

Table 3. Pottery sherds from Russian Amur region selected for lipid residue analysis. FA (Cx:y) = fatty acids with carbon length x and number of unsaturations, phy = phytanic acid, pri = pristanic acid, TMTD = 4,8,12- trimethyltridecanoic acid. Phy(xx) refers to the ratio of SRR% (Lucquin, Colonese et al. 2016). APAA (Cn) = ω-(o-alkylphenyl) alkanoic acids with carbon length n. tr = trace. DCx = α,ω-dicarboxylic acids with carbon length x. Aquatic oils are interpreted from APAA (C20, 22) with at least one isoprenoid fatty acids (Evershed et al. 2008) while ruminant fats are interpreted from

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Table 1. Calibrated dates of the two stages of the Osipovka culture

[14]C bp	cal BP	Key sites	Number of dates	Material for dating
13260-12055	16200-13700	Khummi, Gasya, Goncharka-1, layer 4-5	6	Charcoal
11650-9890	13600-10700	Goncharka-1, layer 3	18	Charcoal, Foodcrust

Table 2. List of Sites and Samples analyzed in this study. OD: older dryas, AB: Allerød-Bølling interstadial, YD: Younger Dryas, EH: Early Holocene.

Site	Cultural Complex	No. of Sherds	Climate stage	Age / Dates (cal BP).
Khummi	Osipovka	1	OD/AB /YD	16240–11820
Gasya	Osipovka	3	OD/AB	15870-12660
Goncharka 1	Osipovka	19	OD/AB /YD/EH	15070–10650
Gromatukha	Gromatukha	5	OD/AB	15120–13840

Table 3. Pottery sherds from Russian Amur region selected for lipid residue analysis. *FA (Cx:y) = fatty acids with carbon length x and number of unsaturations, phy = phytanic acid, pri = pristanic acid, TMTD = 4,8,12- trimethyltridecanoic acid. Phy(xx) refers to the ratio of SRR% (Lucquin, Colonese et al. 2016). APAA (Cn) = ω-(o-alkylphenyl) alkanoic acids with carbon length n. tr = trace. DCx = α,ω-dicarboxylic acids with carbon length x. Aquatic oils are interpreted from APAA (C20, 22) with at least one isoprenoid fatty acids (Evershed et al. 2008) while ruminant fats are interpreted from the combination of the lower Δ13C value and lower relative amounts of the SRR isomer (<75%) of phytanic acid (Copley et al.. 2003, Lucquin, Colonese et al. 2016).*

Laboratory Code	Site	Lipid conc. ($\mu g\ g^{-1}$)	Major Compounds detected	$C_{16:0}$ $\delta^{13}C$ (‰)	$C_{18:0}$ $\delta^{13}C$ (‰)	$\Delta^{13}C$ ($C_{18:0}$-$C_{16:0}$)	Interpretation
KHM01	Khummi	171	FA($C_{9:0-20:0}$, $C_{18:1}$, $C_{15, 17br}$), DC(C_{7-14}), APAA($C_{16, 18, 20tr}$), phy(89), pri, tmtd, DHA, 7-Oxo-DHA	-28.6	-28.6	0.0	Aquatic
GSH01	Gasya	11	FA($C_{12:0-24:0}$, $C_{16:1,18:1}$), phy(86), pri, *n*-alkane (C_{14-29}), DHA, 7-Oxo-DHA	-28.4	-29.7	-1.3	
GSH02	Gasya	64	FA($C_{10:0-24:0}$, $C_{16:1,18:1}$), phy(84), pri, *n*-alkane (C_{14-29}), DHA, 7-Oxo-DHA	-27.5	-29.4	-1.9	
GSH03	Gasya	72	FA($C_{14:0-20:0}$, C_{17br}), DC(C_{8-9}), phy(92), pri, *n*-alkane (C_{11-29}), DHA, retene, 7-Oxo-DHA	-24.2	-24.1	0.1	
GCK02	Goncharka 1	26	FA($C_{12:0-26:0}$, $C_{18:1}$, C_{17br}), DC(C_9), phy(93), pri, *n*-alkane (C_{15-27}), DHA, 7-Oxo-DHA	-26.2	-27.6	-1.5	
GCK04	Goncharka 1	15	FA($C_{14:0-24:0}$, $C_{16:1,18:1}$, C_{12br}), phy(90), pri, *n*-alkane (C_{13-29}), DHA, retene, 7-Oxo-DHA				
GCK05	Goncharka 1	8	FA($C_{14:0-28:0}$), phy(tr), pri, *n*-alkane (C_{14-29}), DHA, retene, 7-Oxo-DHA				
GCK07	Goncharka 1	18	FA($C_{9:0-20:0}$, C_{17br}), phy(tr), pri, *n*-alkane (C_{13-26}), DHA, 7-Oxo-DHA	-28.6	-29.3	-0.7	
GCK08	Goncharka 1	217	FA($C_{9:0-24:0}$, $C_{15,17br}$), phy(tr), pri, *n*-alkane (C_{12-27}), DHA, retene, 7-Oxo-DHA	-26.9	-27.9	-1.0	
GCK09	Goncharka 1	31	FA($C_{13:0-26:0}$, $C_{18:1}$, $C_{12,17br}$), phy(89), pri, *n*-alkane (C_{14-27}), DHA, retene, 7-Oxo-DHA	-24.4	-24.5	0.0	
GCK10	Goncharka 1	23	FA($C_{9:0-26:0}$), pri, n-alkane (C_{12-29}), DHA, retene, 7-Oxo-DHA	-28.4	-28.1	0.3	
GCK12	Goncharka 1	34	FA($C_{8:0-26:0}$, C_{15br}), pri, tmtd, *n*-alkane (C_{13-27}), DHA, retene, 7-Oxo-DHA				
Amur1	Goncharka 1	462	FA($C_{12:0-20:0}$, $C_{15,17br}$),phy(91), pri, n-alkane (C_{15-20}), DHA	-26.7	-27.5	-0.8	
Amur2	Goncharka 1	113	FA($C_{12:0-20:0}$, C_{15br}), phy(tr), pri, n-alkane (C_{15-25}), DHA, retene, 7-Oxo-DHA	-28.1	-28.7	-0.6	
Amur3	Goncharka 1	144	FA($C_{12:0-22:0}$, C_{17br}), APAA($C_{18,20tr}$), phy(87), pri, n-alkane (C_{15-23}), DHA	-25.5	-26.3	-0.8	Aquatic
Amur4	Goncharka 1	778	FA($C_{14:0-20:0}$, $C_{15,17br}$), phy(91), pri	-24.6	-25.7	-1.1	
Amur5	Goncharka 1	168	FA($C_{12:0-20:0}$, $C_{15,17br}$), phy(84), pri, n-alkane (C_{15-19}), DHA, 7-Oxo-DHA	-25.0	-26.0	-1.0	

Amur6	Goncharka 1	376	FA($C_{16:0-18:0}$), n-alkane (C_{15-19}), DHA	-30.2	-29.8	0.4	
Amur7	Goncharka 1	87	FA($C_{12:0-26:0}$, $C_{15,17br}$), phy(tr), n-alkane (C_{15-19}), DHA, retene, 7-Oxo-DHA	-29.4	-29.4	0.0	
Amur8	Goncharka 1	37	FA($C_{12:0-18:0}$, $C_{15,17br}$), n-alkane (C_{16-18}), DHA	-29.3	-29.3	0.1	
Amur9	Goncharka 1	80	FA($C_{12:0-20:0}$, $C_{15,17br}$), phy(tr), pri, n-alkane (C_{15-21}), DHA	-27.6	-28.5	-0.9	
Amur10	Goncharka 1	44	FA($C_{12:0-22:0}$), phy(87), pri, DHA, 7-Oxo-DHA	-24.6	-23.1	1.5	
Amur11	Goncharka 1	20	FA($C_{16:0-18:0}$), pri, DHA, retene, 7-Oxo-DHA	-29.7	-29.5	0.2	
GMT01	Gromatukha	953	FA($C_{9:0-24:0}$,$C_{18:1}$), phy(62), 7-Oxo-DHA	-28.0	-30.1	-2.0	Ruminant
GMT02	Gromatukha	66	FA($C_{12:0-28:0}$,$C_{16:1-22:1}$, $C_{15,17br}$), DC(C_{9-16}), phy(71), pri, *n*-alkane (C_{15-29}), DHA, 7-Oxo-DHA	-27.6	-29.0	-1.4	Ruminant
GMT03	Gromatukha	6	FA($C_{12:0-28:0}$,$C_{16:1-18:1}$, $C_{15,17br}$), pri, *n*-alkane (C_{14-29}), DHA, 7-Oxo-DHA				
GMT04	Gromatukha	660	FA($C_{10:0-28:0}$,$C_{16:1-18:1}$), APAA(C_{18}), phy(79)	-29.2	-30.4	-1.2	
GMT06	Gromatukha	5009	FA($C_{9:0-22:0}$,$C_{18:1}$), phy(60)	-28.3	-29.9	-1.6	Ruminant

Table S1. Contextual data and dates of the samples from Goncharka-1(GCK, Amur) and Gromatukha (GMT) sites.

Samle ID	Layer	Estimated age, ^{14}C BP	Note
GCK02	layer 3Б	10300	Shevkomud and Yanshina 2012: fig. 85-86
GCK04	layer 3Б	10600-10000	Shevkomud and Yanshina 2012: fig. 90, 1-2
GCK05	layer 3Б	10600-10000	
GCK07	layer 3Б	10600-10000	Shevkomud and Yanshina 2012: fig. 90, 6-7; 91
GCK08	layer 3Б	10600-10000	Shevkomud and Yanshina 2012: fig. 78, 1
GCK09	layer 3Б	10600-10000	
GCK10	layer 3Б	12500-10000	Shevkomud and Yanshina 2012: fig. 99, 4
GCK12	layer 3Б	10600-10000	Shevkomud and Yanshina 2012: fig. 93
Amur1	layer 3Б	10600-10000	
Amur2	layer 3Б	10600-10000	
Amur3	layer 3Б	10600-10000	
Amur4	layer 3Б	10600-10000	
Amur5	layer 3Б	10600-10000	
Amur6	layer 5	12500-10000	
Amur7	layer 5	12500-10000	
Amur8	layer 5	12500-10000	
Amur9	layer 5	12500-10000	
Amur10	layer 3Б	10000-10600	
Amur11	layer 3Б	10000-10600	
GMT01	uknown	12500-10000	
GMT02	uknown	12500-10000	
GMT03	uknown	12500-10000	Shevkomud and Yanshina 2012: fig. 115, 1-3
GMT04	uknown	12500-10000	
GMT06	uknown	12500-10000	

Table S2. Estimated percentage contribution (average and standard deviation) of resources using a concentration-dependent mixing model.

| Sample	Site	Salmonids organisms	Wild ruminant	Wild boar	Freshwater organisms	Plants (acorns and chestnuts)
KHM01	Khummi	27.1 ±16.9	6.5 ±5.8	24.4 ±17.3	29.6 ±19.9	12.4 ±9.2
GSH01	Gasya	27.5 ±16.8	14.2 ±9.2	17.6 ±13.9	30.2 ±20.5	10.5 ±8.6
GSH02	Gasya	40.0 ±17.9	12.3 ±8.3	18.0 ±14.7	22.7 ±17.6	7.0 ±6.3
GSH03	Gasya	89.8 ±5.5	1.0 ±1.1	2.6 ±2.6	4.2 ±4.3	2.4 ±2.4
GCK02	Goncharka 1	65.8 ±14.4	4.1 ±3.7	12.9 ±11.0	12.6 ±11.4	4.6 ±4.4
GCK09	Goncharka 1	89 ±5.7	1.2 ±1.2	2.9 ±3.1	4.4 ±4.3	2.5 ±2.5
Amur1	Goncharka 1	59.8 ±16.1	4.2 ±4.0	15.1 ±13.0	15.4 ±13.4	5.5 ±5.0
Amur3	Goncharka 1	79.3 ±9.1	2.5 ±2.5	6.5 ±6.3	7.8 ±7.1	3.8 ±3.6
Amur4	Goncharka 1	85.9 ±6.8	1.7 ±1.7	4.3 ±4.2	5.4 ±5.2	2.7 ±2.7
Amur5	Goncharka 1	82.9 ±8.2	2.1 ±2.2	4.9 ±4.7	7.0 ±6.7	3.1 ±3.0
Amur10	Goncharka 1	90.3 ±5.3	0.9 ±0.9	2.3 ±2.4	3.9 ±4.1	2.6 ±2.6
GMT01	Gromatukha	18.9 ±14	31.7 ±14.7	23.7 ±16.7	18.1 ±15.9	7.7 ±6.7
GMT02	Gromatukha	34.6 ±17.8	13.9 ±9.7	24.3 ±18.3	20.1 ±16.4	7.1 ±6.3
GMT04	Gromatukha	21.5 ±13.8	22.5 ±11.4	15.0 ±12.0	26.0 ±18.2	14.9 ±10.9
GMT06	Gromatukha	15.1 ±12.5	31.4 ±14.7	26.9 ±17.5	17.9 ±15.9	8.7 ±7.5
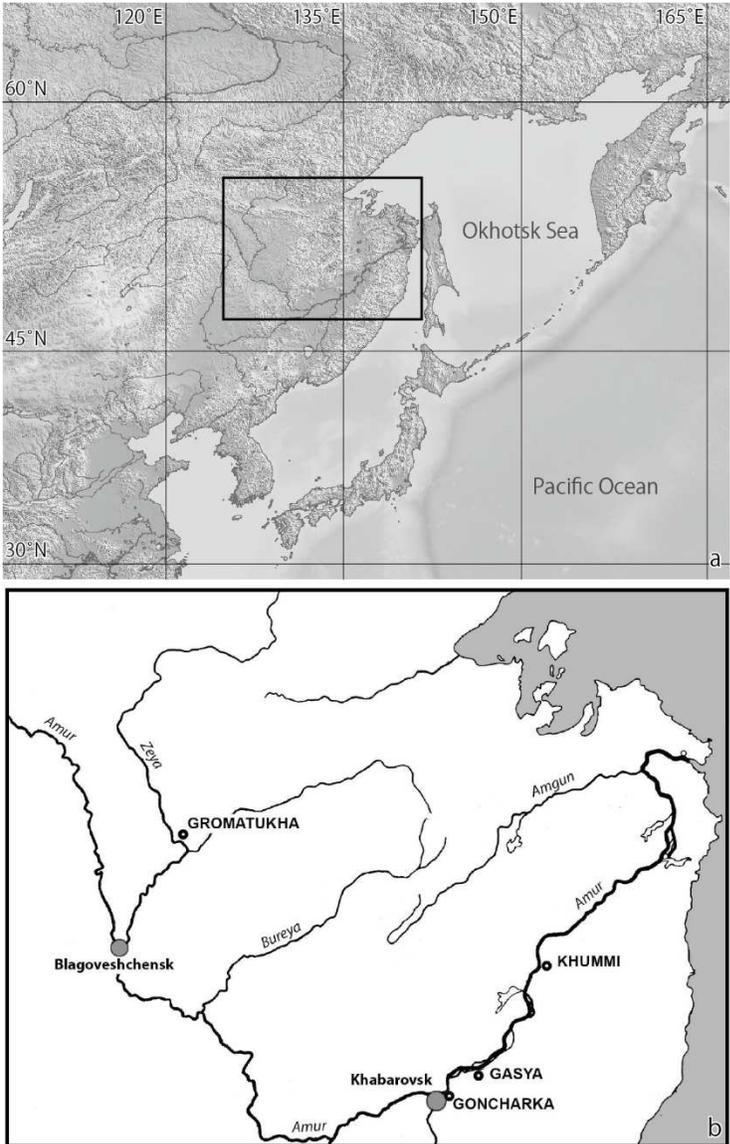
Fig. 1. a. The geographical area of this study. b. Location of the sites investigated in this study. The area corresponds with the square in a. (Osipovka Culture: Goncharka-1, Gasya, Khummi sites; Gromatukha Culture: Gromatukha site)
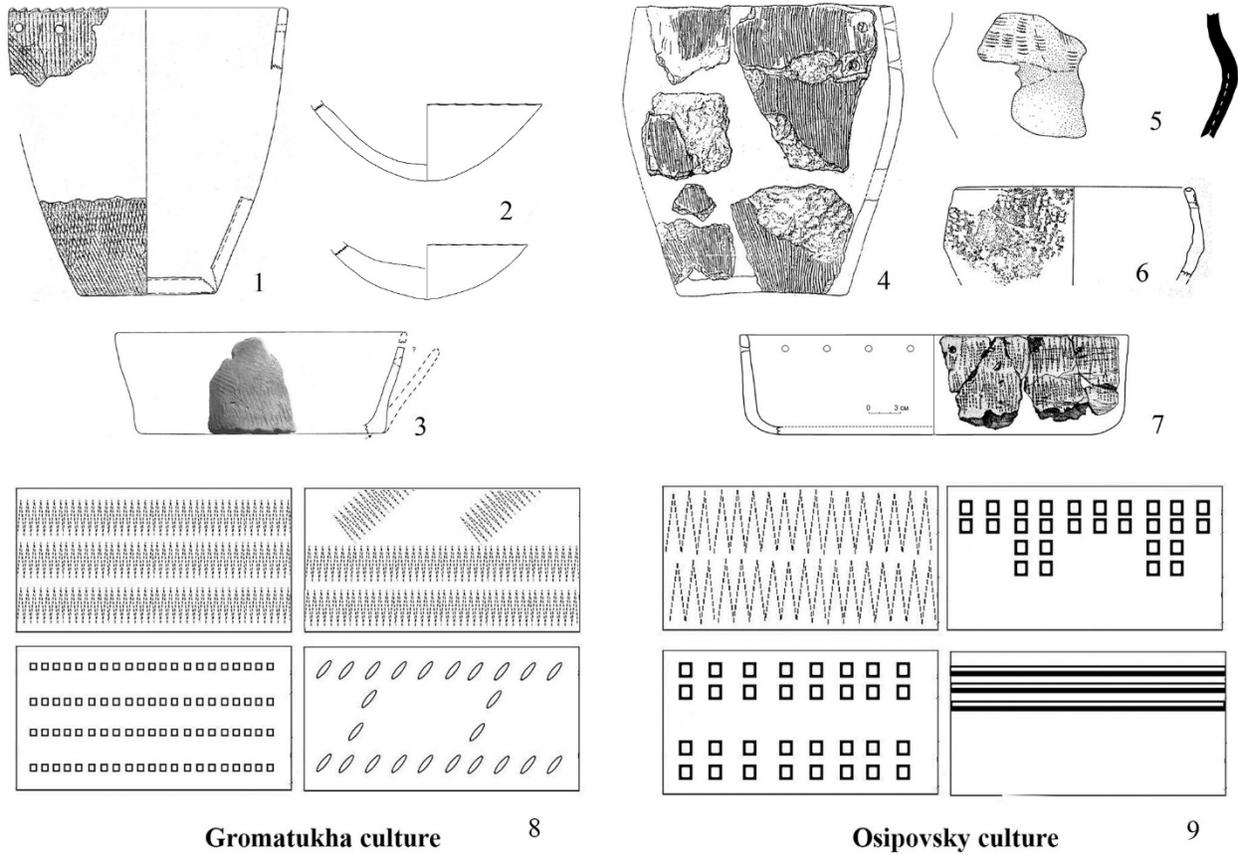
Fig. 2. Typological differences between pottery of the Osipovka and Gromatukha cultures**.** Shapes and main patterns of the ceramic vessels of the Gromatukha (1-3, 8) and Osipovka cultures (4-9). 1, 3, 5, 7 – Shevkomud and Yanshina 2012: fig. 111-112; 6 – Naganuma et al 2005; 8-9 - Yanshina 2017.

Fig. 3. Pottery from the Osipovka and Gromatukha Cultures form entirely different ceramic traditions. These photographs highlight some of the main differences in pottery fabric, tempers and surface treatments: Gromatukha (8-12), Khummi (13-15), and Goncharka-1 (16-18) sites. Note differences in temper, surface treatment, and zigzag pattern between two ceramic traditions. Type I. Temper: grog (15, 18b), gravel inclusions (12 b, 18a), grass additions (9, 12 a). Type II. Surface treatment: grooves rolling by cord wrapped tool (8-9, 11), haphazard cord impressions (14), combing by hard toothed tool (13, 16). Type III. Zigzag pattern: stepping by cord wrapped tool (8, 10, 11) and rolling by hard toothed tool (17).
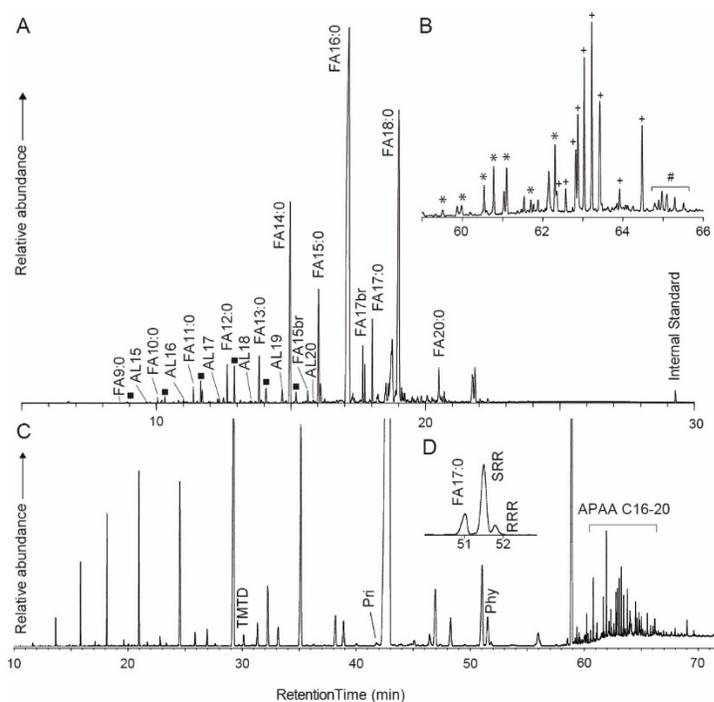
Fig. 4. Partial total ion and selected ion chromatograms of extracts from a pottery sherd from Khummi (Osipovka Culture) (Sample KHM01).

A (1): Characteristic distribution of aquatic oil components: total ion chromatogram that showing lipids typical for a heated and degraded aquatic oil, dominated by medium- and long-chain saturated and mono-unsaturated fatty acids (FA) and isoprenoid fatty acids. б,ω-dicarboxylic acids (■) with carbon chain ranges of C_8-C_{13} resolved on a DB-5 chromatography column. **B. Heating of aquatic oils:** Ion chromatogram (m/z 105) showing the presence of щ-(o-alkylphenyl) alkanoic acids with 16($\ast$), 18(+), 20(#) carbon atoms. **C: Isoprenoid fatty acids identified:** Ion chromatogram (m/z 101) showing isoprenoid fatty acids, TMTD: 4,8,12-trimethyltridecanoic acid, Pri: pristanic acid and Phy: phytanic acid resolved on a DB-5 chromatography column. **D: Separation of SRR and RRR diastereomers:** Ion chromatogram (m/z 101) shows the ratio of phytanic acid diastereomers (SRR and RRR) resolved on a DB-5 chromatography column.
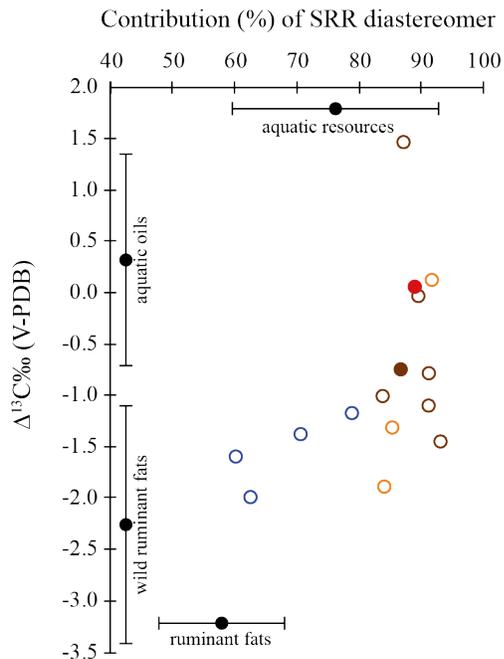
Fig. 5. Comparative plot of the δ^{13}C values of C$_{16:0}$ and C$_{18:0}$ n-alkanoic acids extracted from pottery from Russian (RFE – this study) and Japanese pottery (refs). A: Samples from Amur river basin, Osipovka culture (black) and Gromatukha culture (red), B: Samples from Incipient Jōmon (Lucquin et al. 2018). Closed symbols represent samples meeting the full criteria for aquatic biomarkers (Evershed et al. 2008). The data is compared with reference ranges for authentic reference lipids from both modern and archaeological material (Lucquin et al. 2016, Shoda et al. 2017, Hansel et al. 2004, Evershed et al. 2008, Ackman and Hooper 1968) plotted at 95% confidence. M: Marine, S: Salmonids, WB: Wild Boar, FW: Freshwater, WR: Wild Ruminant and NU: acorns and nuts.
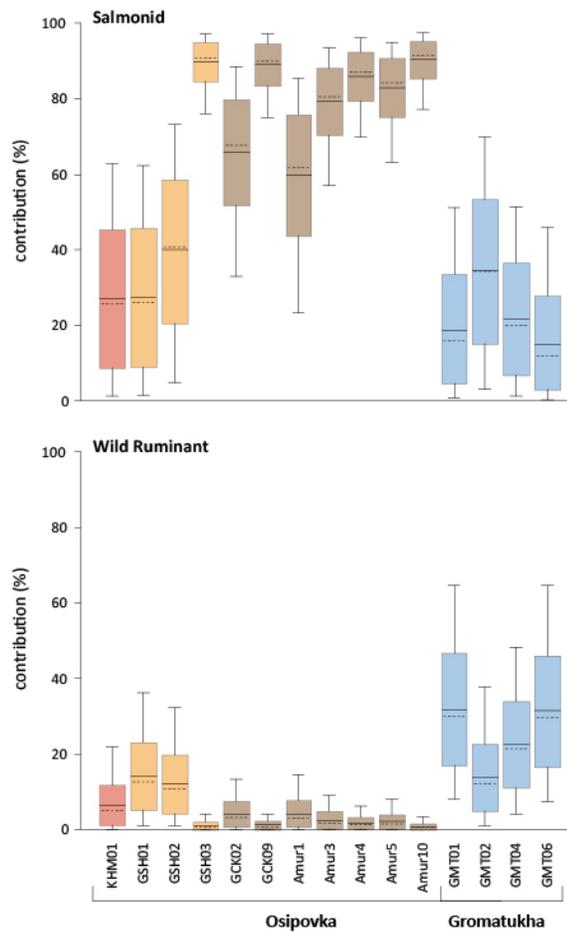
Fig. 6. Estimated percentage contribution of salmonid and wild ruminant resources using a concentration-dependent mixing model. The model parameters have been previously described (Lucquin et al. 2018). Box plots show model output for individual sample. The boxes represent a 68% credible interval while the whiskers represent a 95% credible interval. The horizontal continuous line indicates the mean while the horizontal discontinuous line indicates the median.
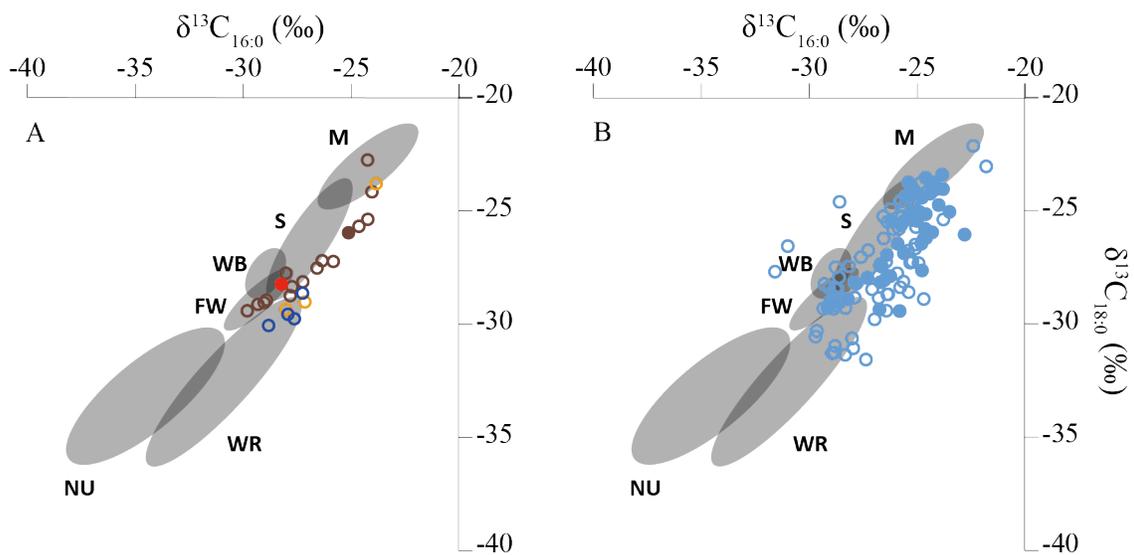
Fig. 7. Plot of the Δ¹³C and %SRR of lipids extracted from early pottery from sites on the Amur River; The values are compared to the reference range of aquatic oils and ruminant fats based on authentic samples (Lucquin et al 2016, Lucquin et al 2018) corrected for the recent burning of fossil fuels. Red circles: Khummi, orange: Gasya, brown: Goncharka-1, and blue: Gromatukha. Plots with asterisk meet full criteria of aquatic biomarkers (Evershed et al. 2008).