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Late Glacial hunter-gatherer pottery in the Russian Far East: Indications of diversity in origins and use

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ABSTRACT

During the Late Glacial, hunter-gatherers began using ceramic cooking containers in three separate geographic regions of East Asia: China, Japan and 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 a series of key sites along the Amur River in the Russian Far East, where early pottery has been recovered from securely-dated contexts that span ca. 16,200 to 10,200 years ago (cal BP). Interpreting how these ceramic vessels were used 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 closely with studies conducted in 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 show a significant contribution of lipids from ruminant animals, indicating that these vessels were being used in different ways. Interestingly, these regional differences in pottery use also map onto contrasting manufacturing techniques, with vessels from the Middle and from the Lower Amur forming distinct pottery-making traditions. These combined insights appear to indicate a greater degree of variability in the development and use of early pottery in East Asia than has hitherto been indicated.

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 centres of early pottery emergence in East Asia. 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, albeit on a rather limited scale, with small numbers of sherds recovered from a series of different sites.

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These early pottery sites are associated with two different archaeological cultures – Osipovka Culture and Gromatukha Culture (Kuzmin, 2002, 2017; Zhushchikhovskaya, 2005; Derevianko and Medvedev, 2006; Shevkomud and Yanshina, 2012; Yanshina, 2017). The sites of Khummi, Gasya and Goncharka 1 are located in the extensive lowlands of the Lower Amur River, and belong to the Osipovka Culture. The site of Gromatukha is located 700 km further westwards, on the west bank of the Zeya River, a large tributary of the Middle Amur, and provides the type site for the Gromatukha Culture (Fig. 1). To date, no other Late Glacial pottery sites have been identified in the extensive region that separates these two archaeological cultures.

While Russian archaeologists have long speculated about the likely economic factors that drove early pottery innovation in the RFE (e.g. Medvedev, 1995; Zhushchikhovskaya, 2005; Kuzmin, 2013), there has been no direct evidence to indicate how early pottery was actually used. This is mainly because of the region's acidic soil conditions which result in very limited preservation of organic materials, rendering detailed palaeo-economic reconstructions impossible. To address this gap in knowledge, our goal was to deploy lipid residue analysis to directly test how early pottery in the RFE was used. The method has already been successfully employed at early pottery sites in Japan (Craig et al., 2013; Lucquin et al., 2016a,Lucquin et al., 2016b; Lucquin et al., 2018), Korea (Shoda et al., 2017) and Sakhalin Island (Gibbs et al., 2017). Emerging results from across all these regions indicate close associations between the earliest appearance of the first ceramic cooking containers and the intensified processing of aquatic resources. Our aim was to test whether the earliest pottery in the RFE was also used to process predominantly aquatic resources, or to cook plants, land animals or mixed foods.

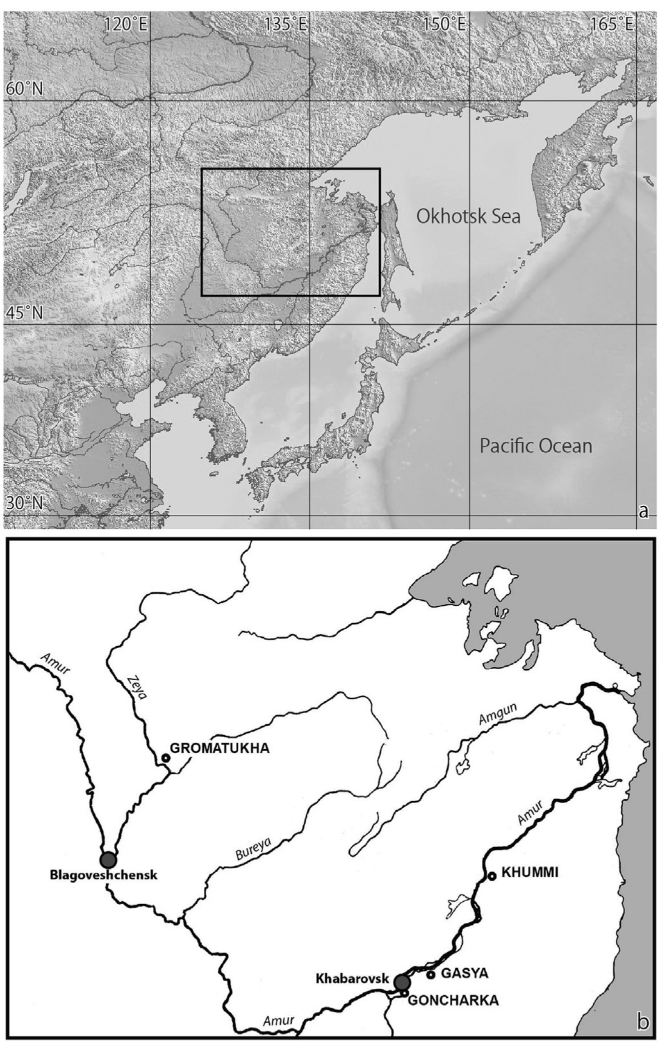

Fig. 1. Locatino of the early pottery site in the RFE. a. Map showing a wider gergraphical area. b. The location of the sites investigated in this study (Osipovka Culture: Goncharka-1, Gasya, Khummi sites; Gromatukha Culture: Gromatukha site) .

2. Regional setting

Unlike the surrounding regions of East Asia, only one Late Palaeolithic site - Golyi Mys 4 - is known from the Lower Amur River (Derevianko et al., 2006: 69–72). This site lacks pottery but yielded 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 and 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 confirmed pre-Holocene pottery assemblages are widely scattered along the Amur River in the RFE. They are now used to define the onset of an 'Initial Neolithic' which is defined by the appearance of pottery and some other Neolithic innovations 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).

2.1. Sites and pottery assemblages of the Osipovka Culture

The Osipovka Culture of the Lower Amur River includes around 70 archaeological sites, of which at least 15 have been excavated. All are located on the banks of the main river channel, and are associated with some of the earliest human occupation of the Lower Amur region so far discovered. The full chronological range of the Osipovka Culture extends from 13,260 ± 100 to 9890 ± 230 yr BP, that is, 16,200-10,700 cal BP (Kuzmin, 2006; 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 artefacts collected from disturbed layers while the second stage provides the majority of data, including the in-situ contexts of stone tools and pottery. To date, the most intensively investigated sites are Gasya, Khummi and Goncharka-1 (Fig. 1).

The **Gasya** site is ca. 80 km downstream from the city of Khabarovsk, and is situated on top of a cliff that is around 13–16 m above the modern Amur river level. This stratified site was excavated between 1975 and 1990 (Derevianko and 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 (Fig. 2). Among them, one vessel was reconstructed as a flat-bottomed container with a volume of ca. 5.5–6 L. It was ca.

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
13,260–12,055	16,200–13,700	Khummi, Gasya, Goncharka-1, layer 4-5	6	Charcoal
11,650–9890	13,600–10,700	Goncharka-1, layer 3	18	Charcoal, Foodcrust

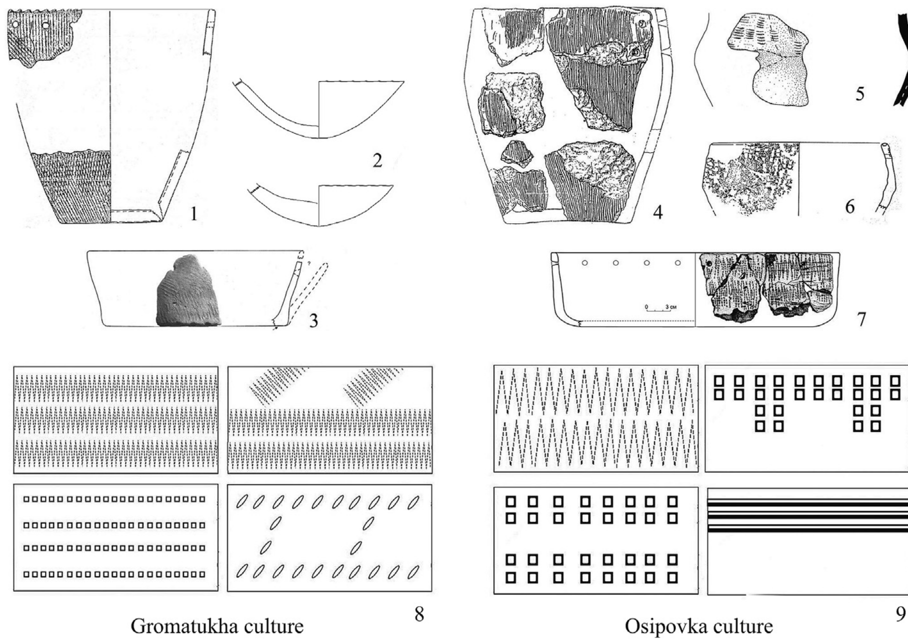

Fig. 2. Summary of key typological differences between pottery of the Osipovka and Gromatukha Cultures. Shapes and main patterns of the ceramic vessels of the Gromatukha (1–3, 8) and Osipovka cultures (4–9). 1, 3, 5, 7 – Shevkomud and Yanshina (2012): Fig. 111–112; 6 – Naganuma et al., (2005); 8–9 -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

25–27 cm high, with walls of around 1.2–1.7 cm in thickness (e.g. Derevianko and Medvedev, 2006; Kuzmin, 2006). 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 and 1997 (Lapshina, 1999). The site is located ca. 20 km upstream from the city of Komsomolsk-on-Amur (Fig. 1), on a high bank around 30 m above the modern level 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 to both the earlier and later stages of the Osipovka Culture, and yield dates spanning a broad chronological range of ca. 13,260-10,375 yr BP, that is, 16,240-11,820 cal BP (Kuzmin, 1997). Only around 40 potsherds of the Osipovka Culture were recovered from this site, despite extensive excavation of the Late Glacial horizons (Fig. 3). This further suggests that the earliest pottery at the site was only being used on a very limited scale. Although it remains challenging to separate out the materials into earlier and later phases according to the site stratigraphy, the design features of the sherds appear to indicate that they predominantly correlate with the oldest stage of Osipovka Culture (Yanshina and 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 main river (Shevkomud and Yanshina, 2012). The lower horizons (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, that is, 15,070-13,750 cal BP. The upper horizon (layer 3B, using the Russian labelling of "3Б") belongs to the late stage of the Osipovka Culture, and has been dated to 11,340–9890 yr BP, thus 13,300-10,650 cal BP (on charcoal samples) and 11,650-10,060 yr BP, that is, 13,590-11,330 cal BP (on foodcrusts) (Kuzmin, 2006; Kuzmin and Shevkomud, 2003; Shevkomud and Kuzmin, 2009; Shevkomud and Yanshina, 2012). Layer 3B also yielded the largest collection of artefacts, as well as evidence of dwelling structures and potentially some ritual activities. The earliest pottery also appears to have been used on a limited scale at this site. Of the more than 2000 potsherds recovered from the two phases of Osipovka Culture phases, only 130 are derived from the oldest layers (4–5) (Shevkomud and Yanshina, 2012).

In general, there appears to be substantial variability within the Osipovka Culture pottery assemblages, although all vessels 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 gravel, dried clay or grog, with plant tempers used infrequently in the early stage. The internal, and occasionally the external surfaces, of the vessels had been scraped with hard comb-like tools. This treatment is a distinctive feature of the pottery in both the early and later stages of Osipovka Culture. In addition, the potsherds from the earlier phase tend to have shallow cord marks on the outer surfaces instead of decoration, while pottery from the later phase is decorated with a comb-like tools for form various patterns (Figs. 2–3) (Yanshina, 2017).

2.2. Sites and pottery assemblages of Gromatukha Culture

The Gromatukha Culture has been less well researched. To date,

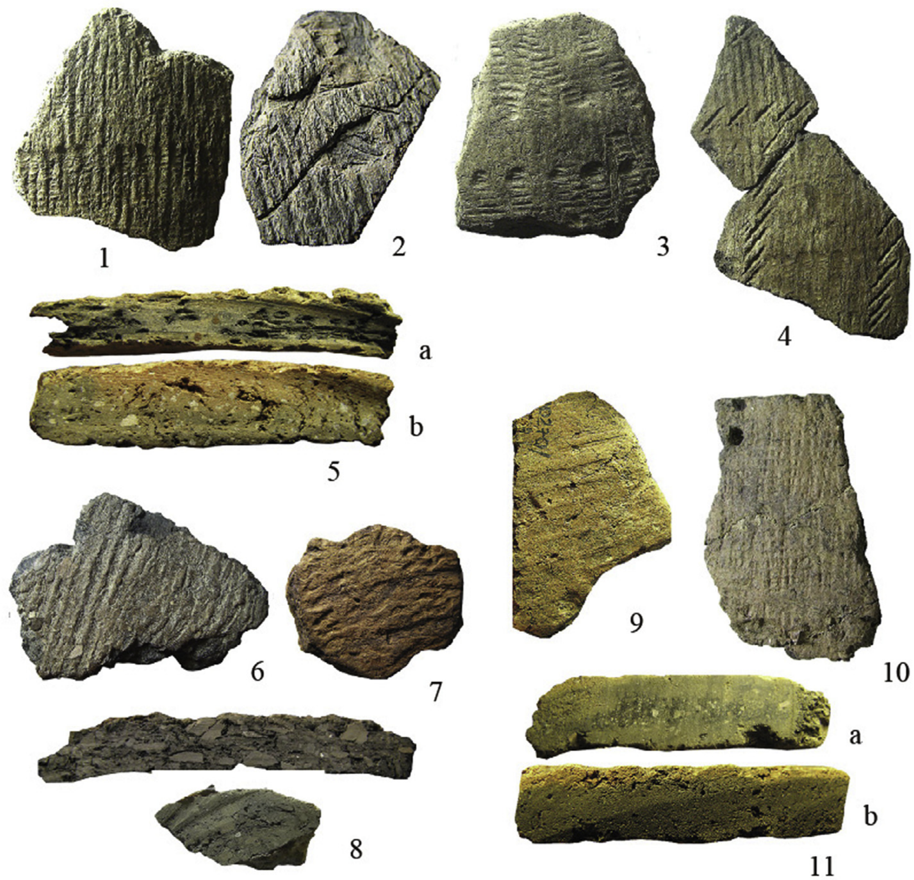

Fig. 3. Pottery from the Osipovka and Gromatukha Cultures form entirely different ceramic traditions. These photographs illustrate some of the main differences in pottery fabric, tempers and surface treatments: Gromatukha (1–5), Khummi (6–8), and Goncharka–1 (9–11) sites. Note differences in temper, surface treatment, and zigzag pattern between two ceramic traditions. Type I. Temper: grog (8, 11b), gravel inclusions (5b, 11a), grass additions (2, 5a). Type II. Surface treatment: grooves rolling by cord wrapped tool (1–2, 4), haphazard cord impressions (7), combing by hard toothed tool (6, 9). Type III. Zigzag pattern: stepping by cord wrapped tool (1, 3–4) and rolling by hard toothed tool (10).

only eight sites have been discovered, and only three have been excavated. These sites are situated in different kinds of landscape to those of the Osipovka Culture. Some sites are situated on the banks of main Amur River, while others are located along the banks of its smaller northern tributaries. The chronology of the Gromatukha Culture overlaps to some extent with the Selemdga Palaeolithic Culture, which is aceramic, suggesting that in the same period, pottery was being used at some sites, but not at others.

The **Gromatukha** site is used to define the Gromatukha Culture and provides almost all the available information for it (Okladnikov and Derevianko, 1977). The site is situated on the high bank of the Zeya River, a tributary joining the middle course of the Amur River (Fig. 1). The main excavations took place in the 1960s (Okladnikov and Derevianko, 1977), and smaller-scale work has been done in the 2000's through to 2010's (Derevianko 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, thus 14,820-11,200 cal BP. Direct ^{14}C dating of pottery using oxygen and oxidation temperature of 400 °C resulted in ages of ca. 13,240–13,310 yr BP, that is, 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 also fall within 12,400–9150 yr BP, thus 15,010-10,190 cal BP (Derevianko et al 2017). Based on pottery analysis (Shevkomud and Yanshina, 2012: 213–230) and parallel ^{14}C dating, two stages of the site occupation can 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 10,060–9150 yr BP or 11,970-10,190 cal BP (Derevianko et al 2017).

In total, 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 and thick walls of ca. 0.7–1.3 cm they are tempered with layers of grass additives, and some have stabbing patterns on the external surfaces. The pottery vessels also have cord marks on their surfaces, with grooves on both internal and external surfaces (Fig. 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, as well as in the cord marks and zigzag patterns (see: Shevkomud and Yanshina, 2012: 207–228; Yanshina, 2017 for more detailed documentation of the assemblage).

3. Materials

To investigate how the earliest pottery in the RFE had been used we extracted absorbed lipid residues from pottery sherds from the sites of Khummi (n = 1), Gasya (n = 3), Goncharka-1 (n = 19) and Gromatukha (n = 5) (Table 2). All sherds were relatively small (ca. 3–5 cm square) and did not have carbonised deposits on the surface. As far as possible, we selected sherds that could be definitely assigned to specific phases of the two cultures. However, the sherds from Khummi and Gasya have no precise contextual information. Moreover, the ^{14}C dates at both these sites show very wide time

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	16,240–11,820
Gasya	Osipovka	3	OD/AB	15,870–12,660
Goncharka 1	Osipovka	19	OD/AB/YD/EH	15,070–10,650
Gromatukha	Gromatukha	5	OD/AB	15,120–13,840

ranges: 13,260-10,375 yr BP or 16,240-11,820 cal BP in the former, and 12,960-10,875 yr BP or 15,870-12,660 cal BP in the latter. In other words, this means that these particular samples can only be assigned to broad chronological horizons. This is unfortunate, because these two sites yield the oldest dates for the Osipovka culture, as well as the smallest assemblages of potsherds.

The contexts of the samples from Goncharka-1 are summarized in Table S1. The estimated ages of each sample are based on the ^{14}C dates of charcoal, stratigraphy, and the contexts. This indicates that most of the samples (n = 15) belong to horizon 3B, which corresponds to the later stage of the Osipovka culture, while a few are derived from the older stage (n = 4).

Sherds derived from the Gromatukha excavations of 1965–1966 are without information pertaining to grids and layers. However, given their appearance (i.e. the high level of plant additives, the intensive use of external cord impressions, plus other decorative elements), we assume that they will have been associated with the early stage of the Gromatukha culture, which correlates with the Bølling-Allerød period. It should also be noted that ^{14}C dates made directly on the food crusts yield dates that vary from 12,530 to 11,440 yr BP, that is, from 15,117 to 13,136 cal BP, although later dates cannot be excluded at this point (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). In short, methanol was added to the drilled ceramic powder (4 ml of methanol to 1 g of sample) which was then sonicated for 15 min. Concentrated sulfuric acid (800 µl) was added to acidify the samples which were then sealed and heated at 70 °C for 4 h. After cooling to room temperature, lipids were extracted with *n*-hexane (3 × 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 × 2 ml). 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 h). The resulting total lipid extract (TLE) was dried under N_2. The extracts were dissolved in *n*-hexane before analysis by GC.

4.2. Gas Chromatography - Flame Ionization Detector (GC–FID)

General screening and quantification of the lipid extracts was undertaken by GC-FID. 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 × 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 5 µg 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. 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 ionization 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 et al., 2016a,Lucquin et al., 2016b) 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) correspond to fragmentation of 4,8,12-trimethyltridecanoic acid (TMTD), 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 ω-(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 Agilent 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 × 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

All samples yielded interpretable amounts of lipids (i.e. > 5 μg g^{-1} sherd) with a mean value of 346 μg g^{-1} and maximum value of 5009 μg 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 Fig. 4.

5.1. Identification of aquatic derived lipids

Previous organic residue studies of Late Glacial pottery in Japan have indicated that vessels were predominantly used for the processing of aquatic resources (Craig et al., 2013; Lucquin et al., 2016a,Lucquin et al., 2016b; Lucquin et al., 2018). However, in the current study, 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), was 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 $\delta^{13}C$ 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 et al., 2016a,Lucquin et al., 2016b). 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 et al., 2016a,Lucquin et al., 2016b; 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 et al., 2016a,Lucquin et al., 2016b).

The results indicate clear differences in the %SRR between pottery from the different sections of the Amur River (Fig. 5). The samples from Gromatukha on the Middle Amur have a lower %SRR (mean 68.0%, median 66.6%), suggesting that ruminant products processed in these vessels. In contrast, in results for Khummi, Gasya and Goncharka-1 on the Lower Amur these products were either absent or found at much lower frequencies, suggested that processing of ruminants was either minimal or absent (mean 88.3%, median 87.3%).

5.3. Investigating the mixing of resources

Results summarized so far suggest that early pottery was predominantly used for salmonid processing in Osipovka Culture and ruminant processing in Gromatukha Culture. To clarify the extent to which other kinds of plants, freshwater fish or wild non-ruminants may also have been used, 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 et al., 2016a, 2016b). This model investigates the proportion of lipids derived from plants (acorns and chestnuts), freshwater fish, wild boar, wild ruminants and salmonids. It assumes that the vessels were used for multiple cooking events, and so relies on the average and standard error of number of individual measurements made on authentic reference fats. This approach provides a more accurate overview of how a particular pottery vessel was used because 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 generates percentage values in terms of % lipid contribution by weight of total lipid. (Table S1), the contributions of salmonids and wild ruminants are shown in Fig. 6.

Results from the RFE samples indicate that ruminant fats had only made a significant contribution to the use of pottery from Gromatuka, whereas salmonids made a much greater contribution to pottery use at Osipovka Culture sites on the Lower Amur, especially at Goncharka 1 and Gasya, where some vessels may have been used exclusively for processing salmonids (Fig. 6).

6. Discussion

This study presents the first organic residue analysis of Late Glacial pottery assemblages from the RFE. The combined results indicate clear spatial patterning in early pottery use - vessels were

Table 3
Pottery sherds from early pottery sites in the RFE selected for lipid residue analysis. FA (Cx:y) = fatty acids with carbon length x and number of unsaturations y, phy = phytanic acid, pri = pristanic acid, TMTD = 4,8,12- trimethyltridecanoic acid. Phy (xx) refers to the ratio of SRR% (Lucquin et al., 2016a, Lucquin et al., 2016b). 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 ($C_{20, 22}$) with at least one isoprenoid fatty acids (Evershed et al., 2008) while ruminant fats are interpreted from the combination of the lower $\Delta^{13}C$ value and lower relative amounts of the SRR isomer (<75%) of phytanic acid (Copley et al., 2003, Lucquin et al., 2016a).

Laboratory Code	Site	Lipid conc. (μg 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 ($C_{9:0\text{-}20:0}$, $C_{18:1}$, $C_{15, 17br}$), DC($C_{7\text{-}14}$), APAA ($C_{16, 18, 20tr}$), phy (89), pri, tmtd, DHA, 7-Oxo-DHA	−28.6	−28.6	0.0	Aquatic
GSH01	Gasya	11	FA ($C_{12:0\text{-}24:0}$, $C_{16:1,18:1}$), phy (86), pri, *n*-alkane ($C_{14\text{-}29}$), DHA, 7-Oxo-DHA	−28.4	−29.7	−1.3	
GSH02	Gasya	64	FA ($C_{10:0\text{-}24:0}$, $C_{16:1,18:1}$), phy (84), pri, *n*-alkane ($C_{14\text{-}29}$), DHA, 7-Oxo-DHA	−27.5	−29.4	−1.9	
GSH03	Gasya	72	FA ($C_{14:0\text{-}20:0}$, C_{17br}), DC($C_{8\text{-}9}$), phy (92), pri, *n*-alkane ($C_{11\text{-}29}$), DHA, retene, 7-Oxo-DHA	−24.2	−24.1	0.1	
GCK02	Goncharka 1	26	FA ($C_{12:0\text{-}26:0}$,$C_{18:1}$, C_{17br}), DC(C_9), phy (93), pri, *n*-alkane ($C_{15\text{-}27}$), DHA, 7-Oxo-DHA	−26.2	−27.6	−1.5	
GCK04	Goncharka 1	15	FA ($C_{14:0\text{-}24:0}$,$C_{16:1,18:1}$, C_{12br}), phy (90), pri, *n*-alkane ($C_{13\text{-}29}$), DHA, retene, 7-Oxo-DHA				
GCK05	Goncharka 1	8	FA ($C_{14:0\text{-}28:0}$), phy (tr), pri, *n*-alkane ($C_{14\text{-}29}$), DHA, retene, 7-Oxo-DHA				
GCK07	Goncharka 1	18	FA ($C_{9:0\text{-}20:0}$, C_{17br}), phy (tr), pri, *n*-alkane ($C_{13\text{-}26}$), DHA, 7-Oxo-DHA	−28.6	−29.3	−0.7	
GCK08	Goncharka 1	217	FA ($C_{9:0\text{-}24:0}$, $C_{15,17br}$), phy (tr), pri, *n*-alkane ($C_{12\text{-}27}$), DHA, retene, 7-Oxo-DHA	−26.9	−27.9	−1.0	
GCK09	Goncharka 1	31	FA ($C_{13:0\text{-}26:0}$,$C_{18:1}$, $C_{12,17br}$), phy (89), pri, *n*-alkane ($C_{14\text{-}27}$), DHA, retene, 7-Oxo-DHA	−24.4	−24.5	0.0	
GCK10	Goncharka 1	23	FA ($C_{9:0\text{-}26:0}$), pri, n-alkane ($C_{12\text{-}29}$), DHA, retene, 7-Oxo-DHA	−28.4	−28.1	0.3	
GCK12	Goncharka 1	34	FA ($C_{8:0\text{-}26:0}$, C_{15br}), pri, tmtd, *n*-alkane ($C_{13\text{-}27}$), DHA, retene, 7-Oxo-DHA				
Amur 1	Goncharka 1	462	FA ($C_{12:0\text{-}20:0}$,$C_{15,17br}$),phy (91), pri, n-alkane ($C_{15\text{-}20}$), DHA	−26.7	−27.5	−0.8	
Amur 2	Goncharka 1	113	FA ($C_{12:0\text{-}20:0}$,C_{15br}), phy (tr), pri, n-alkane ($C_{15\text{-}25}$), DHA, retene, 7-Oxo-DHA	−28.1	−28.7	−0.6	
Amur3	Goncharka 1	144	FA ($C_{12:0\text{-}22:0}$,C_{17br}), APAA ($C_{18,20tr}$), phy (87), pri, n-alkane ($C_{15\text{-}23}$), DHA	−25.5	−26.3	−0.8	Aquatic
Amur4	Goncharka 1	778	FA ($C_{14:0\text{-}20:0}$, $C_{15,17br}$), phy (91), pri	−24.6	−25.7	−1.1	
Amur 5	Goncharka 1	168	FA ($C_{12:0\text{-}20:0}$, $C_{15,17br}$), phy (84), pri, n-alkane ($C_{15\text{-}19}$), DHA, 7-Oxo-DHA	−25.0	−26.0	−1.0	
Amur 6	Goncharka 1	376	FA ($C_{16:0\text{-}18:0}$), n-alkane ($C_{15\text{-}19}$), DHA	−30.2	−29.8	0.4	
Amur 7	Goncharka 1	87	FA ($C_{12:0\text{-}26:0}$, $C_{15,17br}$), phy (tr), n-alkane ($C_{15\text{-}19}$), DHA, retene, 7-Oxo-DHA	−29.4	−29.4	0.0	
Amur 8	Goncharka 1	37	FA ($C_{12:0\text{-}18:0}$, $C_{15,17br}$), n-alkane ($C_{16\text{-}18}$), DHA	−29.3	−29.3	0.1	
Amur 9	Goncharka 1	80	FA ($C_{12:0\text{-}20:0}$, $C_{15,17br}$), phy (tr), pri, n-alkane ($C_{15\text{-}21}$), DHA	−27.6	−28.5	−0.9	
Amur 10	Goncharka 1	44	FA ($C_{12:0\text{-}22:0}$), phy (87), pri, DHA, 7-Oxo-DHA	−24.6	−23.1	1.5	
Amur 11	Goncharka 1	20	FA ($C_{16:0\text{-}18:0}$), pri, DHA, retene, 7-Oxo-DHA	−29.7	−29.5	0.2	
GMT01	Gromatukha	953	FA ($C_{9:0\text{-}24:0}$,$C_{18:1}$), phy (62), 7-Oxo-DHA	−28.0	−30.1	−2.0	Ruminant
GMT02	Gromatukha	66	FA ($C_{12:0\text{-}28:0}$,$C_{16:1\text{-}22:1}$, $C_{15,17br}$), DC($C_{9\text{-}16}$), phy (71), pri, *n*-alkane ($C_{15\text{-}29}$), DHA, 7-Oxo-DHA	−27.6	−29.0	−1.4	Ruminant
GMT03	Gromatukha	6	FA ($C_{12:0\text{-}28:0}$,$C_{16:1\text{-}18:1}$, $C_{15,17br}$), pri, *n*-alkane ($C_{14\text{-}29}$), DHA, 7-Oxo-DHA				
GMT04	Gromatukha	660	FA ($C_{10:0\text{-}28:0}$,$C_{16:1\text{-}18:1}$), APAA (C_{18}), phy (79)	−29.2	−30.4	−1.2	
GMT06	Gromatukha	5009	FA ($C_{9:0\text{-}22:0}$,$C_{18:1}$), phy (60)	−28.3	−29.9	−1.6	Ruminant

used to process aquatic resources at sites of the Osipovka Culture on the Lower Amur, while processing of ruminant animals made a significant contribution to the residues in pottery at Gromatukha on the Middle Amur.

The fact that early pottery along the Lower Amur is linked to the processing of aquatic resources is perhaps not particularly surprising, given that these sites are all located along the main river channel. Today, the Amur River has an enormous wealth of aquatic resources, with over 100 freshwater species, plus several anadromous fish species (such as salmon), which migrate upriver to spawn, starting in the late spring through to the early autumn.

These abundant resources would have attracted prehistoric hunter-gatherers and perhaps stimulated the development of new harvesting techniques. For example, 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). While bone fishhooks and harpoons, fibre fish nets, woven traps, weirs and baskets could and probably were also being used, their remains will not be preserved in the acidic soils of the RFE. This lack of further technological evidence means that the current results are even more significant because for the first time they constitute direct evidence for a close association between the exploitation of aquatic resources and the emergence of early pottery along the Lower Amur River.

It is unclear whether increasing exploitation of fish and the development of ceramic cooking containers formed part of a more general move towards sedentism. Given that these sites only appear to have had ephemeral surface structures, it seems unlikely that the earliest appearance of pottery was somehow associated with a rapid transition to fully sedentary village-based societies. Instead, vessels could easily have been made and cached at seasonally fishing sites by aggregating populations that were moving around the landscape for the rest of the year.

Whether the targeting of fish runs at seasonal harvesting sites triggered a sudden expansion of pottery use also appears doubtful. The excavators at all four Lower Amur sites have noted that the earliest pottery is 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). This could indicate that early pottery in the RFE may have been used for more restricted purposes, such as the preparation of novel or ceremonial foods at annual aggregation sites.

Linking the earliest appearance of pottery in the RFE to the onset of major climatic and environmental shifts is also difficult.

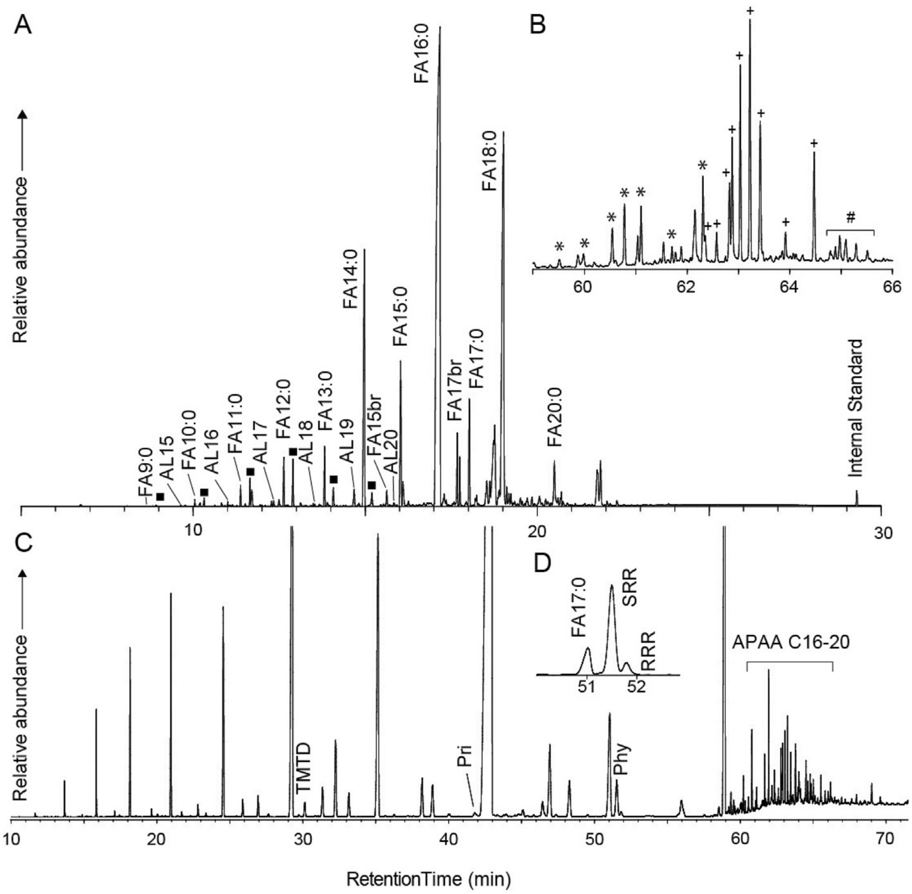

Fig. 4. Partial total ion and selected ion chromatograms of extracts from a pottery sherd from Khummi (Osipovka Culture) (Sample KHM01). A. Total ion chromatogram showing lipids typical for a heated and degraded aquatic oil, dominated by medium- and long-chain saturated and mono-unsaturated fatty acids (FA) and isoprenoid fatty acids. α,ω-dicarboxylic acids (■) with carbon chain ranges of C_8–C_{13} resolved on a DB-5 chromatography column. B. Ion chromatogram (*m/z* 105) showing the presence of ω-(o-alkylphenyl) alkanoic acids with 16(*), 18(+), 20(#) carbon atoms. C. 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: Ion chromatogram (*m/z* 101) shows the ratio of phytanic acid diastereomers (SRR and RRR) resolved on a DB-5 chromatography column.

Reconstruction of palaeoenvironmental 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., 2017), perhaps because aquatic foods may have provided an important alternative to depleted supplies of terrestrial resources during these periods.

The results from Gromatukha site on the Middle Amur are particularly intriguing because they suggest that terrestrial hunting and the processing of ruminants may also have generated situations that encouraged emergence of pottery, at least in some regions. Investment in ceramic cooking technologies may have been an attractive option to hunting groups in the Late Glacial, who may have been keen extract maximum nutrition from carcasses by using pottery to render bone grease, especially during lean seasons, and possibly in relation to climatic downturns (Elston et al., 2011).

Finally, it is important to note that these divergent patterns of pottery use in the Middle and the Lower Amur appear to map directly onto two distinct pottery-making traditions – clearly, different local communities in the RFE were making and also using their pottery in contrasting traditions (Yanshina, 2017). These results can be combined with large-scale comparative technological and stylistic analysis of early pottery assemblages across Late Glacial East Asia, which confirm the existence of three separate pottery-making cultures: (a) the Lower Amur (Osipovka Culture); (b) the Incipient Jōmon of Japan; plus (c), a third culture which embraces Transbaikal and the Middle Amur (including the Gromatukha Culture) (Yanshina, 2017).

Our current results suggest that pottery assemblages (a) and (b) (i.e. Osipovka Culture on the Lower Amur, plus the Incipient Jōmon of Japan) share a common focus on early use of pottery to process aquatic resources (Fig. 7). In contrast, the results from Gromatukha may suggest that culture (c) (i.e. Gromatukha on the Middle Amur, plus the series of late Glacial sites like Ust' Karenga located in the adjacent Transbaikal region), may have a more 'continental' flavour. Here, mobile hunting may have played a more central role in subsistence, and the need to render bone grease and other ruminant resources, especially during seasonal shortfalls, may have encouraged local investment in ceramic processing technologies.

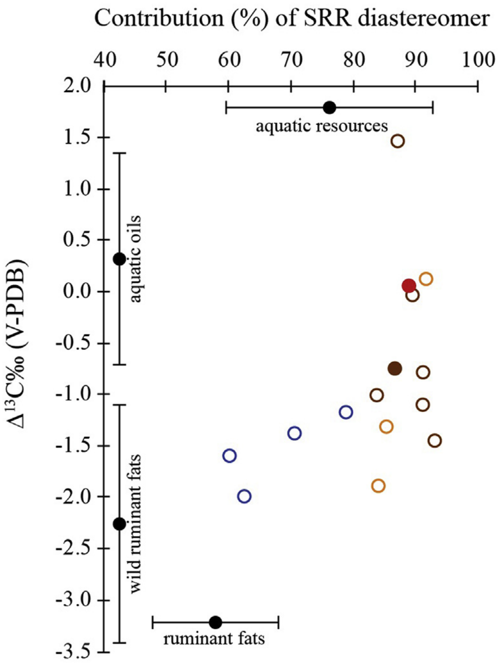

Fig. 5. Plot of the Δ^{13}C and %SRR of lipids extracted from early pottery from sites on the Amur River. The values are compared to the reference range of aquatic oils and ruminant fats based on authentic samples (Lucquin et al., 2016a, 2016b 2018) corrected for the recent burning of fossil fuels. Red circles: Khummi, orange: Gasya, brown: Goncharka-1, and blue: Gromatukha. Closed symbols meet full criteria of aquatic biomarkers (Evershed et al., 2008). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

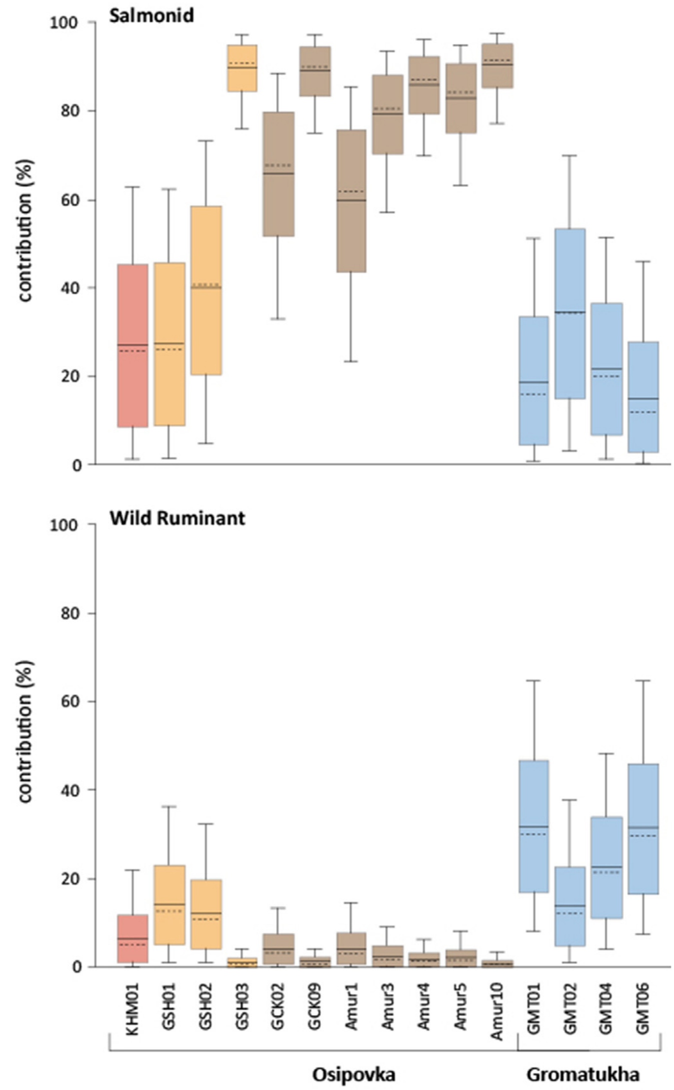

Fig. 6. Estimated percentage contribution of salmonid and wild ruminant resources using a concentration-dependent mixing model. The model parameters have been previously described (Lucquin et al., 2018). Box plots show model output for individual sample. The boxes represent a 68% credible interval while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

Further research is needed to clarify the extent of such large-scale patterning in early pottery usage.

7. Conclusions

Early pottery starts to appear at a number of sites along the Amur River between ca. 16,200 to 10,200 cal BP. Exactly how this pottery was actually used has long been the focus of intensive debate. Our study provides the first direct evidence of early pottery use in the Late Glacial of the RFE, and clarifies which kinds of resources were processed. The results confirm some expectations.

First, we predicted that the early pottery was used for intensified processing of aquatic resources, as this relationship had already been established in Late Glacial Japan, and in early Holocene Korea and Sakhalin Island. This 'aquatic model' also appears to explain early pottery use in the RFE during the late Glacial, but only for sites of Osipovka Culture of the Lower Amur, where pottery appears to have emerged in relation to seasonal harvesting of migratory fish. In contrast, early pottery use at the Gromatukha site on the Middle Amur did not meet these expectations, with the high contribution of ruminant fats indicating a strikingly different pattern of use.

Second, the contrasting patterns of pottery usage map directly onto two distinctive pottery-making traditions (Yanshina, 2017). Together, these combined insights may suggest greater variability in the emergence of early pottery than has hitherto been appreciated, with different traditions emerging in different locations and perhaps for very different reasons. In particular, the unexpected results from Gromatukha may indicate an alternative and perhaps more 'continental' trajectory of early pottery use, which emerged in parallel with the 'aquatic' trajectory already identified in surrounding regions.

While these preliminary insights are intriguing, much more work on lipids, pottery-making traditions, lithic technologies and other palaeoeconomic and palaeoenvironmental datasets needs to be undertaken across East Asia before we can fully apprehend what led local populations to start investing in pottery technology during the Late Glacial, as well as the contributions that this important new cooking innovation made to the evolution of prehistoric lifeways.

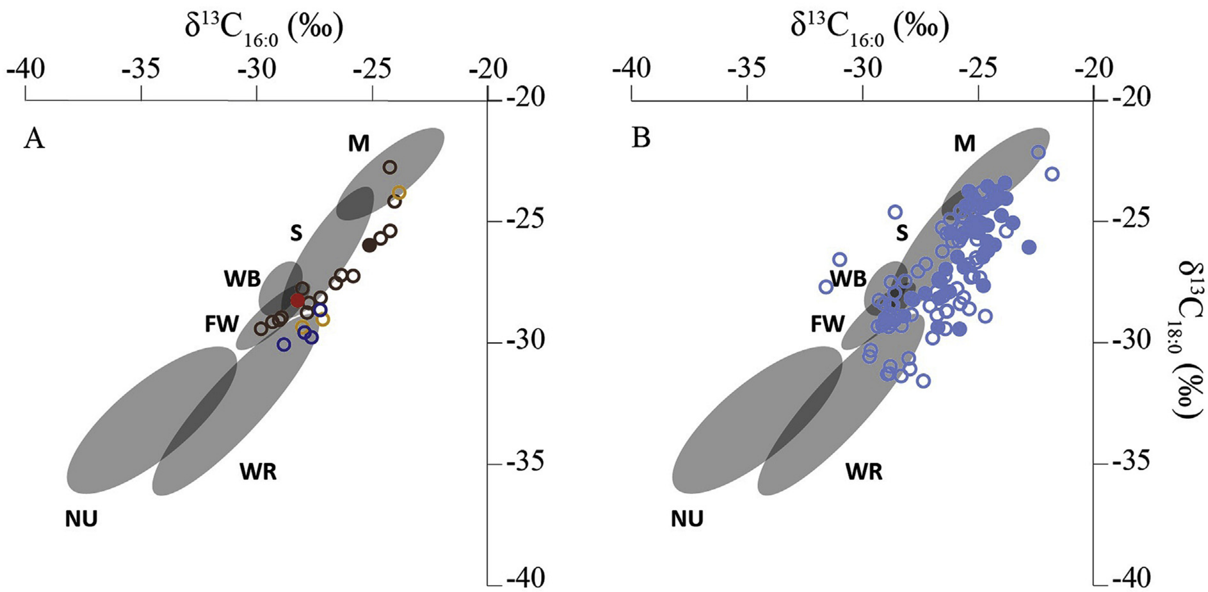

Fig. 7. Comparative plot of the δ^{13}C values of $C_{16:0}$ and $C_{18:0}$ n-alkanoic acids extracted from pottery from Russian (RFE – this study) and Japanese pottery (Lucquin et al. 2018). A: Samples from Amur River basin. Red circles: Khummi, orange: Gasya, brown: Goncharka-1, and blue: Gromatukha. 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 are compared with reference ranges for authentic reference lipids from both modern and archaeological material (Lucquin et al., 2016b; 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. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

Declaration of competing interest

The authors declare that there is no conflict of interest.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.quascirev.2019.106124.

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