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Resource Processing, Early Pottery and the Emergence of Kitoi Culture in Cis-Baikal: Insights from Lipid Residue Analysis of an Early Neolithic Ceramic Assemblage from the Gorelyi Les Habitation Site, Eastern Siberia

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Abstract: In the early Holocene, Mesolithic hunter-gatherer communities inhabiting the Cis-Baikal region of Eastern Siberia were participating in a series of important cultural changes. These included the establishment of large cemeteries in the Angara Valley and on the Southwest shores of Lake Baikal, culminating in the formation of the distinctive Early Neolithic Kitoi cultural pattern ca. 7560 cal. BP. Around the same time, the appearance of clay pots in a few Kitoi graves and at some contemporary habitation sites marks the formal transition to the Early Neolithic, which is defined in Russian archaeology by the emergence of pottery (and not the transition to farming). Little is known about how this early pottery was used, and why it was first adopted into the region. This pilot-study presents lipid-residue analysis of a selection of sherds from the oldest and relatively well-dated pottery assemblage in the Cis-Baikal region, which was recovered from the Gorelyi Les habitation site. The results indicate that the pots had been used to process a broad spectrum of food resources, including ruminants, fish and plants, and possibly resin and other by-products derived from pine trees, suggesting that the vessels were being used as general-purpose cooking containers. We conclude that there is scope for a much larger-scale investigation of diversity and change in prehistoric pottery use in Cis-Baikal, and that this research would improve current understandings of the diet, health and subsistence strategies of the Kitoi and other prehistoric populations.

Keywords: Holocene hunter-gatherers; early pottery; Early Neolithic; Cis-Baikal; Siberia; Kitoi Culture; lipid residue analysis.

1. Introduction: hunters in transition

The Cis-Baikal region of Eastern Siberia includes the Angara Valley, the western shores of Lake Baikal, and the upper Lena River. In warmer conditions of the early Holocene, this region was the scene of several major cultural transitions. While there is general continuity in bone and stone tool-making traditions throughout the Mesolithic, the transition from the Early Mesolithic (10,000–8630 cal. BP) to the Late Mesolithic (8630–7560 cal. BP) is marked by the emergence of archaeologically visible mortuary practices, including isolated burials of individuals, and a few small cemeteries (e.g. Pad' Khin'skaia, Pad' Chastye and Ust'-Griaznaia in Angara Valley or Rytvinka in Upper Lena valleys; Weber et al., 2016b). Evidence from these burials and from faunal evidence recovered from habitation sites suggest that aquatic resources were already contributing to Late Mesolithic diets (Weber, submitted; Weber et al., submitted).

At the end of the Late Mesolithic, during a period of further climatic warming and the expansion of forest cover, further changes gathered pace (White and Bush, 2010; Weber, submitted). The small mobile groups of the Mesolithic appear to coalesce into larger social units, as evidenced by the rather sudden formation of large cemeteries, especially along the Angara River and on the shores of Southwest Baikal (Weber, submitted). These complexes contain highly distinctive mortuary traditions, starting with the "Kitoi" (Bazaliiskii, 2010), and have since been subject to comprehensive radiocarbon dating, including corrections for freshwater reservoir effects (Bronk Ramsey et al., 2014; Schulting et al., 2014; 2015; Nomokonova et al., 2015; Weber et al., 2016a; 2016b; Weber et al., submitted). The "classic" Kitoi cemeteries include Shamanka II at the head of the Kultuk Bay, as well as other large burial grounds located at the mouths of the main tributaries of the Angara River, including Kitoi (Kitoi River) and Lokomotiv (Irkut River) or smaller ones such as Ust' Belaia and Galashikha (Belaia River; Fig. 1).

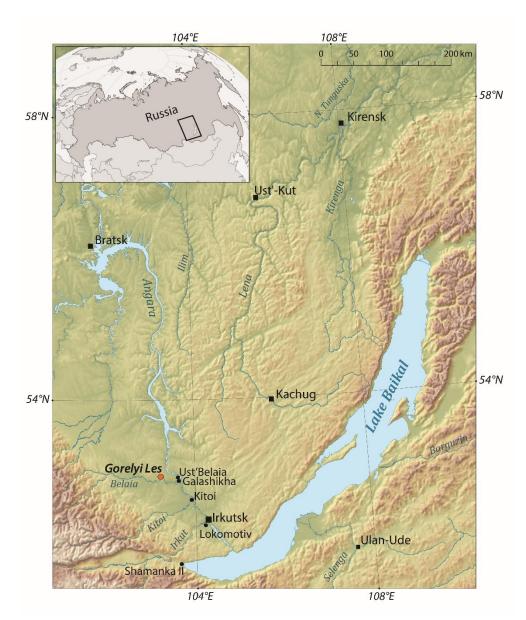


Figure 1. Location map of Cis-Baikal, Eastern Siberia, showing the core areas of the Early Neolithic Kitoi Culture along the Angara River and in SW Baikal. The Kitoi established large cemeteries at the mouths of the main tributaries of the Angara, including, Ust' Belaia (Belaia River), Kitoi (Kitoi River) and Lokomotiv (Irkut River), and also Shamanka II at the head of the Kultuk Bay and Gorelyi Les habitation site located along the Belaia River. Some other Kitoi habitation sites and several small cemeteries are located across the region, predominantly along riverbanks and lake edges (not shown).

The Kitoi mortuary protocol is mainly defined by copious use of red ochre, plus a large number of other diagnostic traits, including distinctive composite fishhooks (Weber, submitted). More generally, analysis of grave goods from Shamanka II indicates that Kitoi populations appear to have acquired several important new technologies, including powerful composite hunting bows, and a range of new fishing implements that supported exploitation of aquatic resources (Weber, submitted). Notable disparities in the quantity and diversity of Kitoi grave goods may point to emerging social differentiation (Weber et al., 2002; Weber and Bettinger, 2010), while the deeper continuity in other bone and lithic artefact types

suggests that it was essentially local Mesolithic populations who were central to these developments (Weber, 1995; Savel'ev, 2001; McKenzie 2009).

It is around this time that clay pots make their first appearance in Cis-Baikal, with isolated finds in a few Kitoi graves, and at some contemporaneous habitation sites. This is an important development, because in Russian archaeology the emergence of pottery among hunter-gatherers is used to define the onset of the Neolithic, unlike in Western Archaeology, where the Neolithic is associated with the transition to agriculture (Chard, 1974: 63–64; McKenzie, 2009; Jordan and Zvelebil 2009). In Cis-Baikal, the shift from the aceramic Mesolithic into the pottery-using Early Neolithic (EN) takes place at around 7560 cal. BP (Weber, submitted). However, this early use of pottery appears to have been relatively limited in scale, and many campsites and Kitoi graves remain essentially aceramic. Very little is known about why pottery was first adopted by the Kitoi Culture, or what kind of resources were processed in the vessels (McKenzie, 2009). Much older evidence of pottery, extending back into the Late Glacial, has been reported from archaeological sites further to the east, including Transbaikal, on the middle and lower sections of the Amur River in the Russian Far East, and in Japan (Kuzmin, 2014). Knowledge of pottery may have dispersed into Cis-Baikal from these areas, though the precise timing and exact routes remain unclear (Jordan et al., 2016; Piezonka et al 2020).

Along the Angara River and in Southwest Baikal, two different early pottery styles appear at around the same time: net-impressed wares and cord-impressed "Khaita" wares. While both types have been recorded at habitation sites, only net-impressed vessels have been recovered from Kitoi graves (McKenzie, 2009; Berdnikov and Sokolova, 2014; Weber, submitted). The oldest radiocarbon-dated pottery assemblage from Cis-Baikal was recovered from Layer VI at the Gorelyi Les habitation site, which is situated on the Belaia River that runs into the Angara River (Fig. 1). This pottery-bearing cultural horizon yielded a radiocarbon date on charcoal of 7000 ± 150 BP (7580-8160 cal. BP $\pm 2\sigma$; Veksler, 1989; Kuzmin, 2014), and while the date is derived from a highly-compressed occupation level, it suggests that pottery starts to appear at around the same time that Kitoi Culture was emerging ca. 7560 cal. BP (McKenzie, 2009; Weber, submitted). The Gorelyi Les ceramic assemblage therefore offers important opportunities to investigate how the oldest pottery in the Cis-Baikal region was being used, and also to explore what motivated the emerging Kitoi Culture to adopt clay pots into their subsistence strategies and social life.

2. "Becoming Neolithic": Kitoi motivations for adoption of pottery

The formation of the Early Neolithic Kitoi cultural pattern can be viewed as a rather sudden socio-economic transition, underpinned by a series of important technological innovations (Weber, submitted). In many ways, the gradual intensification of fishing appears to define the trajectory of the Kitoi Culture, and bioarchaeological analysis of skeletal remains indicates that the contribution of fish to Kitoi diets increased steadily over time (Weber et al., 2016b; Weber et al., submitted). The rich fisheries of the Angara River, which remain open throughout the winter months, appear to have encouraged the emergence of a range of either new or more morphologically variable fishing devices, including nets and sinkers, new type of harpoons and leisters, as well as composite fish-hooks (Bazaliiskii, 2010; Weber and Bettinger, 2010; Weber et al., submitted). Nephrite wood-working tools were also in widespread use at this time, and could have been used to construct range of mass capture facilities, including fish weirs, fences and basketry traps (Weber et al., 2002; Bazaliiskii, 2010; Weber and Bettinger, 2010; Weber et al., 2011, Weber, submitted). Intensive fishing — practised either individually or in larger coordinated groups — could also have offered rich and reliable harvests. This surplus could then have been processed, stored and shared out during leaner months. Perhaps not surprisingly, all the main Kitoi cemeteries are located along the major water courses (Fig. 1), and may have reflected regional aggregation sites, where funerary

rites and feasting events may have been combined with cooperative fishing activities. While fishing was important, terrestrial hunting continued to play a significant role in Kitoi groups. The appearance of powerful composite hunting bows would have greatly improved return rates in hunting local game, including moose, red deer, roe deer and boar (Weber, submitted). In contrast, the extent to which the Kitoi exploited local plant and nut resources is less well understood.

Motivations for Kitoi adoption of pottery remain uncertain. In general, clay pots offer an effective means for the slow simmering of resources to extract rich lipids and to combine diverse elements into nourishing stews. However, the fact that pottery comes into use at around the same time that Kitoi groups appear to be intensifying their fishing activities may indicate that the two phenomena are somehow associated with each other. For example, ceramic vessels are ideal for quickly processing fish, and for the slow rendering of oil. Moreover, pottery is a relatively "cheap" container technology in contrast to baskets and boxes because large numbers of pots can potentially be made and then fired together, offering "economies of scale" (Brown, 1989). It is therefore plausible to suggest that opportunities for increasing the harvests of fish could also have encouraged Kitoi groups to adopt and expand their use of pottery, as it could have allowed them to quickly process larger catches, and to render greater volumes of valuable fish oil. This in turn, may have further encouraged the intensification of fishing activities, as growing surpluses could be efficiently processed. More generally, early pottery also tends to be found in close association with fishing equipment across many other parts Eastern Siberia, suggesting that the two have some kind of specific relationship with each other (McKenzie, 2009:189).

Adoption of clay pots may also have been motivated by the desire to stew lean meat or to produce bone grease, which would have enabled groups to extract maximum nutrition from hunted game, especially during leaner seasons (Elston et al., 2011). Extracting bone fats and marrow involves breaking open the long bones with simple hammer stones and anvils, and then the fragments are slowly heated in water to render the grease (Karr et al., 2015). Using direct heating of clay pots over a fire is less time consuming than using hot stones to maintain a constant heat, and would have freed up time for other activities. Seal fat could also have been rendered in the Kultuk Bay area.

The extent to which Kitoi may have processed plant resources in pottery is even less well-understood. Stands of Siberian pine nuts (*Pinus sibirica*) would also have been available to the Kitoi, and can easily be harvested, stored in the cones and consumed without any need for boiling. More usually, they are dry heated, and nut oil can be rendered by cold pressing. Other local plant resources such as berries, lichen and inner bark (e.g. pine, birch or willow; Bogdanova 2016; Shikov et al. 2017; Weber, submitted), which is rich in vitamin A and C and a well-known "starvation food", could also have benefitted from boiling to remove bitter tannins (Lashmanova et al. 2012; Shikov et al. 2017). These plant foods could also have been added to mixed dishes to add flavour or thicken up stews. Finally, pots may also have been used by Kitoi people to produce tree resins, mastics and pitch for the hafting of tools (Connan and Nissenbaum 2003; Croft et al. 2018) or other waterproof boxes, containers or even canoes (Heron and Evershed 1993; Colombini et al. 2005). This process usually involves the slow heating of tree bark in a sealed container (e.g. a pot with a wooden lid); oil can also be thickened into pitch by slow simmering.

Several kinds of Kitoi social dynamics may also have encouraged pottery adoption, such as the use of pots to create rich and nutritious dishes (involving costly-to-produce oils, fats and lipids) that could be prepared and shared out at aggregations, generating social debts, and perhaps leading to seasonal cycles of competitive feasting (Hayden, 2009, 2012). The mortuary record of Kitoi society — which had already acquired the technological means to harvest abundant quantity of fish — also contains abundant evidence for emerging status inequalities (Weber et al., 2002; Weber and Bettinger, 2010; Weber, submitted), and

such feasting events could potentially have served as a central socio-political strategy within these transegalitarian communities (Hayden, 2009, 2012). On the other hand, pottery may simply have been attractive in more routine domestic contexts. It can be left unattended for long periods, generating efficiencies in time-management in hearth-side contexts, and can also be used for producing soft weaning foods (Jordan and Zvelebil, 2009; Hommel, 2012).

3. Case-study: Early Neolithic pottery at Gorelyi Les

The Gorelyi Les habitation site is situated on the Belaia River about 50 kilometres from its confluence with the main Angara River (Savel'ev and Ulanov 2018). The site is located on the first terrace above the river, and is surrounded by forest and forest steppe, low hills and open plains (Figs. 1 and 2). At the start of the Kitoi Culture around 7560 cal. BP, this area would already have been experiencing a slow warming trend since around 8630 cal. BP (Weber, submitted), combined with increased precipitation, expansion of forest cover, and thicker, longer-lasting snow cover. The forest expansion would have peaked around 7000–6500 cal. BP (Weber, submitted). The landscapes around Gorelyi Les would have provided diverse game and plant resources, with the river providing transport links and also substantial fishing opportunities.



Figure 2. Photo of Gorelyi Les site (view from northwest) along the Belaia River and surrounded by forest (after Weber, 1997).

3.1 The site: excavation history and chronology

The Gorelyi Les site was first discovered in 1969 and its multi-layered character confirmed in 1970. Excavations were conducted in 1971, 1972 and 1974 by members of the Department of Archaeology of Irkutsk State University, under the leadership of N.A. Savel'ev (Savel'ev et al., 1974, Savel'ev and Ulanov 2018:48). During these earlier seasons, a total of 750 m2 was excavated, producing large archaeological collections (Weber, 1995). Excavations resumed in 1994, 1995 and 1996, this time under the joint supervision of N.A. Savel'ev and A.W. Weber, generating additional archaeological materials (Weber, 1997; Ready, 2008; Kurzybov, 2011). One further excavation season took place in 2002

(Igumnova et al., 2004; McKenzie, 2009). In total, 1000 m2 of the site has now been excavated (Savel'ev and Ulanov 2018:48).

The stratigraphy and chronology of the site have been extensively discussed in previous publications (Weber, 1995; Weber et al., 2002; Ready, 2008; McKenzie, 2009: 186–187; Savel'ev and Ulanov 2018). The consensus is that the site was occupied throughout a period of at least three thousand years, consisting of five distinct occupation phases, of which four have now been radiocarbon dated (Supplementary Material, Table A). The chronology of these periods is: Late Mesolithic (Layer VII; ca. 10,240 to 9300 cal. BP); Early Neolithic (Layer VI; ca. 7950 to 7440 cal. BP); a Middle to Late Neolithic (Layer Vb, compression of MN and LN materials into one Layer; ca. 6320 to 6010 cal. BP); Late Neolithic (Layer Va; ca. 5890 to 5530 cal. BP); the Early Bronze Age (Layer IV; undated).

Pottery sherds have been recovered from some of these phases, though the earlier reports that 16 sherds, probably originating from a single pot, could be securely associated with Late Mesolithic Layer VIIa (ca. 10,240 to 9300 to cal. BP) have since controversial (Weber, 1995). If this interpretation is correct, then these sherds would be far older than the larger pottery assemblage from Early Neolithic Layer VI (ca. 7950 to 7440 cal. years BP). However, most archaeologists, including the original excavators, now conclude that the sherds in the Late Mesolithic context are almost certainly intrusive, and were displaced down to this lower level by the action of burrowing animals, whose traces have been noted in several parts of the site (Weber, 1995; Weber, 1997; McKenzie, 2009: 186; Savel'ev and Ulanov 2018:72).

This means that the pottery from the Early Neolithic Layer VI is now accepted as being the earliest and most-securely dated pottery assemblage in Cis-Baikal, with its date often reported as 7870 cal. BP (Veksler, 1989; Kuzmin, 2014). It has also been suggested that Layer VI potentially forms a habitation site correlate of the earliest phase of the Kitoi mortuary tradition (Weber, 1995; McKenzie, 2009: 186–187), as the occupation spans ca. 7950 to 7440 cal. BP, while the Kitoi tradition also starts at ca. 7560 cal. BP but then extends through to 6660 cal. BP when the dates are corrected for freshwater reservoir effects (Weber, submitted, Weber et al., submitted). While a further suite of radiocarbon dates would be useful to strengthen the chronology of Layer VI, the recovery of a highly-diagnostic Kitoi-type shank from a composite fishhook, combined with early finds of net-impressed pottery in both Kitoi graves and in Layer VI of this habitation site, can all be used to support the argument for general contemporaneity between Layer VI of the habitation site and the start of the Kitoi mortuary tradition.

3.2 The Layer VI Early Neolithic Pottery Assemblage

Excavation efforts at Gorelyi Les have spanned three decades, producing a large pottery assemblage whose general details have recently been summarized (Savel'ev and Ulanov 2018). In total, 2339 "informative" sherds from at least 55 separate pots are associated with the entire Neolithic occupation (2018:51). Of these, 1559 diagnostic sherds from a minimum of 42 pots are associated with the Early Neolithic, with almost all of them recovered from Layer VI (2018:51). A further 599 diagnostic sherds, from at least 11 pots are assigned to the Middle Neolithic (2018:61).

As with other (undated) habitation sites across Cis-Baikal, the Layer VI Gorelyi Les assemblage consists of two distinctive types of pottery styles, which appear to have co-existed in the Early Neolithic (Weber, 1995, 2020; McKenzie, 2009; Savel'ev and Ulanov 2018): (a) net-impressed 1 pottery, which is "mitre-shaped" (the term given for paraboloid-shaped pottery, see Fig 5.2 in McKenzie 2009: 169–170), with an everted rim and decorated with net-impressions (Fig. 3a); (b) "Khaita" pottery (Fig. 3b), which is decorated with cord-impressions, with some examples having herringbone and other geometric motifs incised in the upper half of the vessels (Savel'ev, 1982; McKenzie, 2009; Savel'ev and Ulanov 2018). The

other non-diagnostic sherds recovered from Layer VI cannot be classed into either type, and are usually reported as being "plain-surface pottery" (Weber 1997) or "smooth-walled pottery", generally with an everted rim and bearing no traces of ornamentation (McKenzie, 2009).

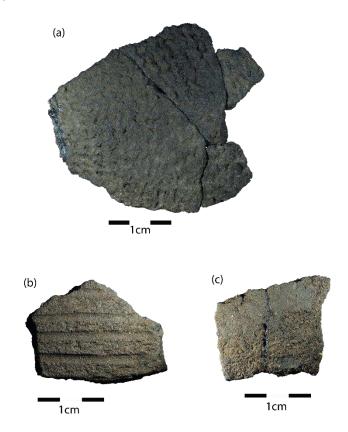


Figure 3. Examples of Early Neolithic pottery types recovered from Layer VI of Gorelyi Les during the 1994–1996 excavations: (a) net-impressed pottery; (b) "Khaita" cord-impressed pottery; (c) "plain-surface pottery" with no traces of diagnostic ornamentation.

3.3 Subsistence and Site Function

According to the faunal reports and lithic inventories, hunting activities appear to have been central to Gorelyi Les inhabitants' subsistence strategy. Cervidae, including moose (*Alces alces*), red and roe deer (*Cervus elaphus* and *Capreolus capreolus* respectively), were in the Early Neolithic Layer of this site, with a clear preference for roe deer. Traces of butchery such as cut and chop marks mainly on mandible, metatarsal and long bone shafts suggest marrow cracking (Ready, 2008) (Table 1). The site also had isolated finds of hare, bovid and bear remains. Plant remains have not been recovered, although this may rather be related to a recovery bias as no flotation or wet screening was undertaken (Table 1).

Fish bones (e.g. northern pike bones, *Esox lucius*) and a single shank from a composite fishhook were recovered (Weber, 1997; Ready, 2008). While this indicates that fish were present on site, the quantity of remains is very low (n=8), suggesting that it was probably not a major economic activity. In contrast, the faunal assemblage is dominated by larger land animals (Ready, 2008). This may relate simply to preservation and recovery biases, but is more likely to result from differences in site function as large numbers of fish remains were recovered from the Ust' Khaita habitation site, which is located only 2 kilometres upstream from Gorelyi Les (Savel'ev et al. 2001; Kurzybov, 2011).

On balance, it would appear that during the Early Neolithic, the Gorelyi Les habitation site was being used by Kitoi groups either as an active hunting camp or as a "gearing up" station used for readying hunting expeditions which returned to the site to process large game. The Early Neolithic pottery assemblage from Layer VI at Gorelyi Les appears to be one of very few assemblages that can be relatively securely-correlated with the Early Neolithic Kitoi culture in the wider Cis-Baikal region, offering a unique opportunity to explore how these vessels were being used, and also why they were first adopted into Kitoi society.

Period	Lithic assemblage ^{1,3,4}	faunal assemblage (NISP) ²	Pottery assemblage ^{1, 5}	Other ¹
	Total of 1360 lithic artefacts including 65 formal tools.	Total of 10623 faunal remains whose 9507 unidentified.	Pottery appears ca. 7870 cal BP (and appears to be broadly contemporaneous with Kitoi Culture)	Three hearths found: - 50 cm diameter with cobbles and dark charcoal stain One with distinct pit (ca. 20 cm deep, 50 cm
	Processing tools: Blades and microblades (n = 41), burins (n = 4), cores and cores fragments (n = 12), borers (n = 2), scrapers (n=2).	Mammal (n = 1084), artiodactyl (n = 273), cf. Cervidae (n=4), Capreolu pygargus (n = 232), Cerphus elaphus (n = 28), Alces alces (n = 142), rodentia (n = 2), Ursus arctos (n = 4), Bison spp.	72 fragments: -49 net-impressed 1 (this tradition is also found in Kitoi mortuary contexts)	diameter) filled with numerous small bone and charcoal fragments, plus small limestone cobbles on the surface. - One large charcoal stain (1 m diameter) and cobbles.
		Aut Bos spp. (n = 3).	19 smooth-walled4 cord-impressed	A deliberated arrangement of 15 pebbles (3 rows): unknown purpose.
	Hunting gear: Arrowheads $(n = 4)$, points $(n = 6)$.			
		Esox lucius $(n = 8)$		An articulated deer limb.
Layer VI	Fishing tools: Shank of a composite fishhook (n = 1). Culturally significant artefact for Kitoi Mortuary tradition.	Trace of butchery (cut and chop marks, n=168 bones) mainly on mandibles, metatarsals, long bone shafts. Suggests marrow cracking and/or possibly tool production.		
EARLY NEOLITHIC	Identification of the main functions: meat knives, drills, borers, scrapers, chopping (hewing) tools.	Burt bones. Notably all the fish remains were burnt.		
	Large amount and diverse waste of flints suggesting that the flintknapping activity was intensive. Likewise, evidence for local production of tools made with light- and dark-coloured banded grey cherts, red platy chert, quartzite pebbles, argillite, and conglomerates, available on the vicinity of the site. In contrast, tools made with materials such as slate and high-quality chert, also locally available, were manufactured in other areas at Gorelyi Les or in other locations distant from the site.			
Layer VII	Total of 49 lithic artefacts with only 4 formal tools (blades or microblades).	Total of 417 faunal remains whose 404 unidentified. Mammal (n = 9), Rodentia (n = 4).	Pottery sherds reported from this level, but chronology and context are controversial	

Burnt bones.

Table 1. Summary of the artefact and faunal assemblages found at Gorelyi Les site in Layers VII (Late Mesolithic) and VI (Early Neolithic). After: Weber, 1997 (1); Ready, 2008 (2); Kurzybov, 2011 (3); Bazaliiskii, 2003, 2010 (4, 5).

4. Materials

Destructive sampling access to 44 sherds was granted for the purposes of the present pilot-study. These were recovered from secure contexts in the Early Neolithic Layer VI during the well-documented 1994—1996 excavations (Weber 1997, Kurzybov, 2011). These samples consisted of: 11 net-impressed sherds (absorbed residues n = 10, foodcrust sample n = 1); 32 sherds of plain-surface pottery (absorbed residues n = 32); one sherd of cord-impressed Khaita pottery (absorbed residues n = 1). For a full list of samples, see Supplementary Material, Table B).

5. Methods

When pottery is used to cook and store resources lipid residues are frequently absorbed into the ceramic fabric (Evershed, 2008a), or can be burnt onto the vessel surface where they form carbonized food remains (Evershed, 2008a). In certain archaeological conditions, these organic residues can survive for thousands of years (Evershed, 1993; Dunne, 2017). Methods for recovery and analysis of organic residue have seen major development over the last three decades, and are now routinely deployed to study the function of archaeological pottery (Rottländer and Schlichtherle, 1980; Evershed et al., 1990; 1991; 1994; 1997; Heron et al., 1991; Oudemans and Boon, 1991; Charters et al., 1993b; Oudemans, 2007; Evershed, 2008a; 2008b).

Typically, a combination of chemical processes and analytical techniques is employed to recover the lipids and generate insights into vessel use, including GC-C-IRMS (Gas Chromatography-Combustion-Isotope Ratio Mass Spectrometry) to measure stable isotope ratios, and GC-MS (Gas Chromatography-Mass Spectrometry) to analyse the molecular character of the residues and identity lipid biomarkers (Evershed et al., 1990; 1994; 1997; Evershed, 2008a; Correa-Ascencio and Evershed, 2014).

To sample the absorbed residues, several millimetres of the outer sherd surface were first mechanically removed using a modelling drill in order to eliminate exogenous residue. About 1 g of clay powder was then drilled from the interior portion of the larger and medium sherds, while the very small sherds were crushed to a fine powder. The single foodcrust sample was also crushed. An established one-step acidified methanol protocol was used to extract lipids from these powdered samples (Craig et al., 2013; Papakosta et al., 2015). This involves adding methanol to the powdered samples in the following proportions: potsherd: 1 g/4 ml; foodcrust: 10-20 mg/1 ml, plus an internal standard (*n*-tetratriacontane: 10 μg) to verify that extraction is running correctly. The mixtures were then sonicated for 15 minutes and acidified with concentrated sulphuric acid (800 μl and 200 μl, respectively), and finally heated for 4 hours at 70 °C. After the samples were left to cool, lipids were extracted with *n*-hexane (3 x 2 ml) and centrifugation (3000 rpm, 5 minutes), and finally reduced and concentrated under an Argon flow. Finally, an additional internal standard was added (*n*-hexatriacontane: 10 μg) to quantify the yielded lipids.

All the lipid extracts were then analysed by GC-MS and GC-C-IRMS to generate molecular and carbon isotope values for the two most abundant fatty acids, hexadecanoic and octadecanoic acid ($C_{16:0}$ and $C_{18:0}$ respectively). Ten of the extracts were also methylsilylated using BSTFA (N, O-bis [trimethylsilyl] trifluorocetamide) in order to better detect dihydroxy fatty acids and alkanols. The single foodcrust sample was also analysed by Elemental Analysis-Isotope Ratio Mass Spectrometry (EA-IRMS) to provide stable

nitrogen (δ^{15} N) and carbon (δ^{13} C) isotope values. Instrument settings were selected following established methodologies (Lucquin et al., 2016a; Shoda et al., 2017; Bondetti et al., 2020) and are also available in the Supplementary Material.

6. Results

6.1 Survival of Absorbed Lipids and Contamination

Both the absorbed residues and single foodcrust sample exhibited satisfactory levels of lipid preservation (potsherds >5 $\mu g.g^{-1}$; foodcrusts >100 $\mu g.g^{-1}$; Evershed, 2008a; Lucquin et al., 2018). The average lipid concentrations of 80 and 459 $\mu g.g^{-1}$ for the absorbed samples and the foodcrust sample respectively, were not especially high, although this is broadly similar to levels reported in other recent studies of early Holocene pottery in other regions (Lucquin et al., 2016a; Gibbs et al., 2017; Oras et al., 2017; Shoda et al., 2017). However, this suggests that organic matter tends to be preserved in this particular context.

Many of the samples had high levels of contamination, especially phthalates, which were identified in all the absorbed samples, but not in the single foodcrust sample. This is probably a result of the sherds being stored in plastic. Many had also been labelled with a combination of varnish, correction-fluid and ink, and some had also been mended with glue. These contaminants often dominated the chromatogram, forming major peaks (Fig. 4). In some cases, this can be problematic, as potentially informative biomarkers may sometimes be hidden by contaminant signals.

6.2. Molecular Characterization

Figure 4 illustrates a typical gas chromatogram of organic residue extracted from pottery samples. The lipid profile of the acid/methanol extracts shows the presence of saturated fatty acids ranged from C_6 to C_{32} , mainly dominated by palmitic acid ($C_{16:0}$), unsaturated fatty acids with mainly even numbers of carbons ranging from $C_{14:1}$ to $C_{24:1}$, and branched fatty acids ranging from C_{13} to C_{26} (Fig. 4; Supplementary Material, Table B). In addition, dicarboxylic acids, which ranged from C_6 to C_{26} , were present in almost all samples, plus hydroxy acids, ranging from C_{22} and C_{24} , were detected in some samples. Both tend to indicate the cooking of plant and animal resources as they are typical oxidation products of unsaturated fatty acids, although they can also result from different degradation processes occurring during burial (Regert et al., 1998; Copley et al., 2005; Baeten et al., 2013).

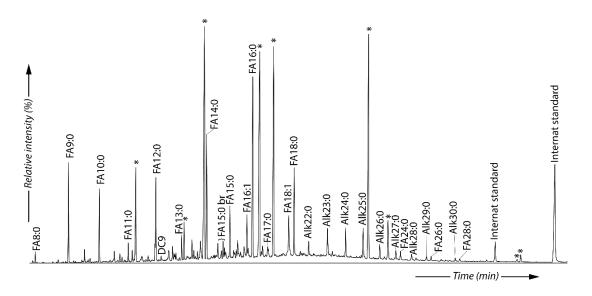


Figure 4. A typical total ion current (TIC) chromatogram of lipid residues from the Gorelyi Les pottery (sample: GL 94.00158). This shows the presence of saturated fatty acids (FA), diacids (DC), branched (br), long-chain unsaturated fatty acids, mid- and long-chain alkanes (Alk). Several contaminants are also present (see *), including phthalates.

Eight of the absorbed samples met established criteria for identifying the processing of aquatic resources (Hansel et al., 2004; Cramp and Evershed, 2014; Lucquin et al., 2016b). These samples contained either long chain (\geq C20) ω-(o-alkylphenyl) alkanoic acids (APAAs), along with at least one isoprenoid acid (e.g. 4,8,12-trimethyltridecanoic acid (TMTD), phytanic acid, pristanic acid) or a phytanic's diastereomers ratio (SRR%) above 75.5% (Fig. 5; Supplementary Material, Table B).

The former (APAAs) are formed by the heating of mono and polyunsaturated fatty acids (M/PUFAs) at a temperature of at least 200°C, from 1 hour of heating (Bondetti et al., forthcoming). Moreover, M/PUFAs with carbon length superior or equal to 20 are only present in significant concentrations in aquatic animals, and so the detection of APAAs with \geq C20 is highly consistent with the processing of freshwater and/or marine organisms (Evershed, 2008b; Baeten et al., 2013; Cramp and Evershed, 2014).

Although the isoprenoid fatty acid TMTD is mainly formed by aquatic organisms, both phytanic and pristanic acid, which are synthesized from phytol, are present in both aquatic and ruminant resources (Ackman and Hooper, 1968; Cramp and Evershed, 2014; Heron and Craig, 2015). However, the co-occurrence of these isoprenoid acids with APAAs \geqslant C₂₀ confirms that they are derived from aquatic rather than terrestrial resources. In addition, the ratio of the two natural diastereomers of phytanic acid, 3S,7R,11R,15-phytanic acid (SRR), which is usually predominant in aquatic organisms, and 3R,7R,11R,15-phytanic acid (RRR) can also be calculated. Therefore higher % contributions of SRRs is characteristic of the processing of aquatic resources (Lucquin et al., 2016b).

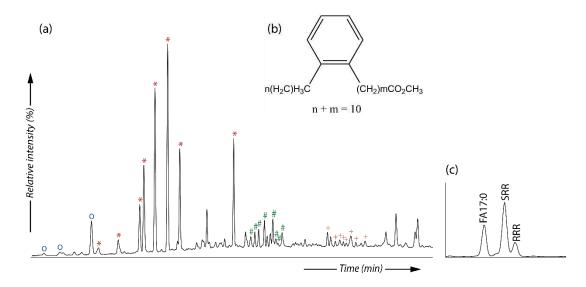


Figure 5. Molecular evidence for the processing of aquatic resources of the foodcrust sample GL 94.00557 run on the DB-23 column: (a) a partial summed mass chromatogram (m/z 105) showing the presence of ω -(o-alkylphenyl) alkanoic acids with 16 (O), 18 (*), 20 (#) or 22 (+) carbon atoms; (b) chemical structure of APAAs (from Hansel et al., 2004; (c) a partial summed mass chromatogram (m/z 101) revealing the diastereomers of phytanic acid (SRR and RRR).

There was tentative evidence that plant leaves had been processed. Substantial quantities of diverse midand long-chain alkanes (C_{14} – C_{33}) were detected in all the samples, except for the single foodcrust sample (Fig. 4; Supplementary Material, Table B). One of the main sources of alkanes is wax covering the leaf and the stem surface of plants (Turunen et al. 1997; Oros et al. 1999, Bush and McInerney, 2013; Horiuchi et al., 2015). In 26 of the 44 samples these compounds were found in combination with a symmetrical ketone (16-Hentriacontanone). Moreover, in all the tri-methylsilylated samples (n = 10) diverse mid and long chain n-alkanols (C_{12} – C_{28}) were also detected (Supplementary Material, Table B). Although the combined presence of all these compounds meets the established criteria for identification of plant-derived waxes (Baeten et al., 2013), the n-alkane distributions did not show a more typical profile associated with the processing of leafy plants, usually displaying a predominance of odd-carbon chain n-alkanes (Diefendorf et al., 2011; Bush and McInerney, 2013; Horiuchi et al., 2015; Dunne et al., 2016). Such profiles could be interpreted as a petroleum contamination (Whelton et al., 2018) but are more likely the result of thermal alteration (March et al., 2014; Wang et al., 2017; Jambrina-Enríquez et al., 2018).

Such profiles commonly display a predominance of alkanes with an odd number of carbon atoms with the prevalence of one or two of them (Eglinton and Hamilton, 1963; 1967; Bush and McInerney, 2013). Furthermore, the 16-hentriacontanone compound could also be generated by the pyrolysis of animal fats alike the non-symmetrical ketones also present in 19 pots (Evershed et al. 1995; Raven et al. 1997; Baeten et al. 2013). Finally, *n*-alkanes and *n*-alkanols are common lipid components of soils (van Bergen et al., 1998), and their presence in the pottery could be related to the burial context. Further analysis of soils from the archaeological site could clarify this issue but remain beyond the scope of the current paper.

In contrast, the samples contained clearer evidence for the use of coniferous tree resources, possibly in relation to firewood, or potentially in the form of food, flavourings or medicine, or in relation to production of glues and sealants. Use of tree resources is suggested by the presence of various diterpenes. A total of 38 samples (86%) yielded totarol, 7α -hydroxy (Supplementary Material, Table B). This

diterpene (totarol) and its various derivatives have been identified in numerous coniferous resin plants, including Cupressaceae (e.g. juniper) (Bendall and Cambie, 1995), and especially, although in low amount, in Pinacea such as spruce (Alwehaibi et al., 2016), which was a major component of the early Holocene forests in the Cis-Baikal region (Demske et al., 2005; White and Bush, 2010).

Furthermore, diterpenes such as methyl dehydroabietate (DHA) and 7-oxo-dehydroabietate (7-oxo-DHA) were also identified in 35 (80%) of the pottery samples. They are indicative of altered Pinaceae resins and wood (Regert, 2004; Mitkidou et al., 2008; Modugno and Ribechini, 2009; Jerković et al., 2011), although they are also produced by bacteria found in various environments (Costa et al., 2016) and would require analyses of soil from the site to confirm or rule out possible exogenous contamination. These biomarkers may have resulted from smoke from campfires if pine was being used as a fuel (Simoneit et al., 2000; Simoneit, 2002). However, the absence of other smoke indicators such as polyaromatic hydrocarbons (PAHs) makes this less likely.

A possible scenario is that resources gathered from pine trees were deliberately added to the pots as part of local cooking practices. Pine nuts and the inner bark are important elements in the diet of many Siberian indigenous peoples (Okladnikov, 1950; 1955; Rushforth, 1987; Shikov et al., 2017). Both are full of nutrients and can easily be harvested and stored for later use. While pine nuts require minimum processing, the bark could have been stewed to remove unpleasant flavours or to remove tannins or added to stews and casseroles as a source of fibre or starch, to add flavour and variety, or to thicken up soups. Finally, pine needles also contain resins and therefore such diterpenoids (Turunen et al., 1997), and may have been added to dishes as they contain abundant vitamin C and are rich in flavour, or the needles may have been used to brew hot tea or medicinal infusions (Cumo, 2015).

These results may also suggest that the pots were used to produce other useful non-food substances, including pine resin or tar, which could have been used for hafting, gluing and sealing various stone tools, bone or organic tools (Croft et al., 2018). Pine resin may also have been used for the waterproofing and repairing and dug-out canoes, and to seal birch-bark boats or other household containers, in turn, requiring significant quantities to be produced (Connan and Nissenbaum, 2003). However, the overall number of these biomarkers remains low, making it unlikely that the pots were purely used to process resin. Another scenario is that the resin could have been used to seal new pots after firing or to repair cracked vessels by boring holes, stitching up the fracture and then sealing up the repairs (Charters et al., 1993a; Jerković et al., 2011; Colombini et al., 2005).

6.3. Stable Isotope Analysis

Further insights about pottery use were generated by undertaking single-compounds isotope analysis of the two main fatty acids: palmitic ($C_{16:0}$) and stearic ($C_{18:0}$). Due to the good lipid preservation, all 44 samples were analysed by Gas Chromatography-Combustion-Isotope Ratio Mass Spectrometry (GC-CIRMS) (Supplementary Material, Table B). Furthermore, the foodcrust sample was also subjected to bulk isotope analysis to determine its stable nitrogen (δ^{15} N) and carbon (δ^{13} C) isotope values using Elemental Analysis-Isotope Ratio Mass Spectrometry (EA-IRMS).

The results are presented in Figure 6a, with $\delta^{13}C_{16:0}$ values plotted against $\Delta^{13}C$ ($\delta^{13}C_{18:0} - \delta^{13}C_{16:0}$). $\Delta^{13}C$ values provided a means of distinguishing lipids derived from ruminant adipose, dairy fats and other non-ruminant sources, including other terrestrial and also aquatic sources (Evershed et al., 1999; Craig et al., 2012; 2013; Colonese et al., 2015; Taché and Craig, 2015; Lucquin et al., 2016a).

In total, 25 samples (57%) had Δ^{13} C values of < -1.1 which are commonly taken to infer the presence of ruminant fats (Copley et al., 2003; Šoberl et al., 2008; Craig et al., 2012; Salque et al., 2012). Interestingly, a negative correlation could be observed between the δ^{13} C calculuses samples with progressively more negative Δ^{13} C produced fatty acids enriched in 13 C. Enrichment in 13 C is observed in the tissues of ruminant animals that feed on C₄ plants (Dunne et al. 2016). However, given that C₄ plants are absent in this region (Lam, 1994; Katzenberg et al., 2010), and that ruminants recovered from modern and archaeological contexts exhibit carbon isotope values that are consistent with a diet consisting exclusively of C₃ plants (Fig. 6a) (Weber et al., 2002; Katzenberg et al., 2012), it is difficult to explain this pattern adequately. Potentially, the negative correlation between δ^{13} C calculuses could be a result of resources from different sources being mixed in the pottery during cooking activities.

Interestingly, samples with aquatic biomarkers, usually exhibiting Δ^{13} C value > -1.1%, here mainly plot in the ruminant area (Δ^{13} C < -1.1) but with relatively enriched δ^{13} C_{16:0}. Likewise, the δ^{15} N isotopic value generated for the single foodcrust sample clearly point to the processing of aquatic resources (δ^{15} N = 10.64 %) (Weber et al., 2011; Craig et al., 2013; Choy et al., 2016) while its Δ^{13} C values again fall within the range expected for ruminant fats (Fig. 6a). This suggests a mixture between fish, with a more positive δ^{13} C value, i.e. > -27 %, and ruminant fats. Collagen of modern fish, notably Salmonidae (e.g. lenok and arctic grayling), found in the rivers of Angara region have carbon isotope values relatively high (δ^{13} C mean_{salmonidae} = -23.1 ± 0.8% after a correction for the collagen to lipid offset of -7%; Weber et al., 2011; Fernandes et al. 2015; Supplementary Material, Table C) in keeping with enriched values observed in the pots, although more archaeological fish would require to be analysed to confirm this trend.

To explore this hypothesis further, tests were conducted to ascertain how $\Delta^{13}C$ values respond to a range of simulated mixing scenarios where aquatic foods, including freshwater and migratory fish, are mixed either with C_3 plants (e.g. fruits, nuts and grass), non-ruminant or ruminant commodities taking into account differences in the amount of C_{16} and C_{18} fatty acids in these sources. The theoretical mixing model presented in figure 6 shows that when even a modest amount (ca. >10%) of ruminant fats are mixed with fish oils, the $\Delta^{13}C$ values can shift to below -1% (Fig. 6b). While, in contrast, when fish fats are mixed with plants or non-ruminant fats, these do not shift the $\Delta^{13}C$ values within the range observed in the pottery i.e. < -1.1%. (Fig. 6c and d). Therefore, the interpretation that the pots containing aquatic biomarkers were used for processing a mixture of fish and ruminant resources, either together or through successive uses is supported.

In total, a further 19 samples (43%) had Δ^{13} C values consistent with non-ruminant products, which could include aquatic and terrestrial non-ruminant animals. Interestingly, very few of these samples contained aquatic biomarkers (n = 2). Therefore, pots displaying such signals, instead suggest a use oriented toward the processing of terrestrial non-ruminant fats. Another alternative, which cannot be ruled out, is the treatment of plants or tree resources in these pots (see above).

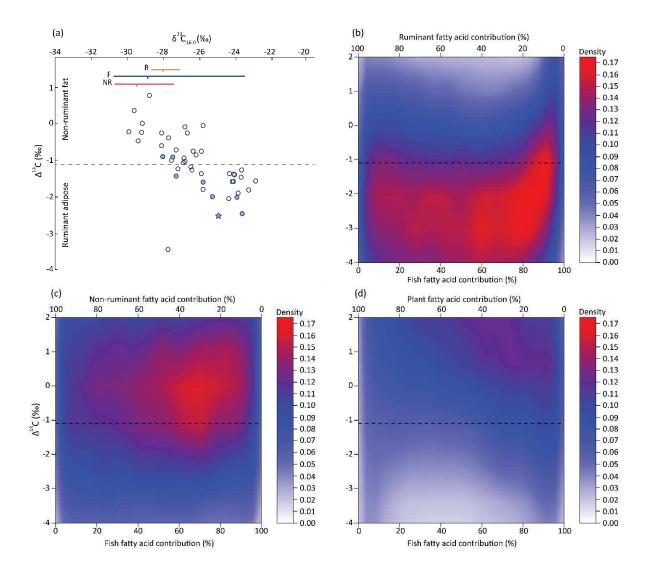


Figure 6. Lipid stable isotope characteristics from the all Gorelyi Les samples compared with theoretical mixes of animal fats, plant oils (C_3 types) and fish oils. (a) Scatter plot of $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) against $\delta^{13}C_{16:0}$ of Gorelyi Les samples. The foodcrust sample is represented by a star. Samples containing aquatic biomarkers are filled. The data are compared with the median and ranges (1σ) of bone collagen of archaeological ruminant (R), modern fish (F) and non-ruminant animals (NR) from the Baikal region, after being corrected for the collagen to tissue offset (Fernandes et al. 2015), plotted on the x-axis only (Weber et al., 2002; 2011; Katzenberg et al., 2012; Supplementary Material, Table C). Kernel density estimates of $\Delta^{13}C$ values obtained by theoretical mixing of authentic reference lipids from modern tissues of fish with (b) ruminant, (c) non-ruminant adipose fats and (d) plant oils (Supplementary Material, Table D). Isotopic data were obtained from published (Dudd, 1999; Spangenberg et al., 2010; Craig et al., 2012; Choy et al., 2016) and unpublished data and using fatty acid concentration values obtained from the USDA database (https://fdc.nal.usda.gov/). Color is required for this figure in print.

6.4. Summary of Results

Overall, these combined results appear to indicate that: (a) the Early Neolithic pottery samples from Gorelyi Les had been used to process a wide spectrum of resources, ranging from fish and ruminants, to pine tree resources, possibly including pine needles, inner bark or pine resin; (b) there was some diversity

in patterns of pottery use, with approximately half of the pots containing molecular signals consistent with the processing of non-ruminant products (probably a broad array of terrestrial plant and animal products), while the other half had been used to process ruminants, most probably in combination with aquatic resources. Finally (c), the limited range of materials we had access to did not enable us to clarify whether each of the three different pottery types — i.e. net-impressed (n=11), plain-surface pottery (n=32), cord-impressed Khaita pottery (n = 1) — had been used to process specific sets of resources, although this would be an interesting theme for follow-up research.

7. Discussion: emergence of Kitoi culture and adoption of early pottery

Although Kitoi groups were adopting a wide array of new technological innovations after ca. 7500 cal. BP, including powerful hunting bows and new kinds of fishing equipment (Weber, submitted), it appears that exploitation of aquatic resources was probably the only branch of the economy amenable to intensification. In turn, the increasing economic surplus may also have supported larger social groups and the emergence of status differentiation (Weber, submitted). Other factors encouraging an increasing dietary focus on aquatic resources (Weber et al., submitted) may have included the expansion and infilling of the boreal forest from about 7500–7000 cal. BP, which could have depressed local deer populations (Weber, submitted). Early pottery — signalling the onset of the Early Neolithic — also makes its first appearance in Cis-Baikal precisely within this dynamic social and ecological context (McKenzie, 2009: 198), and it appears plausible to suggest that adoption was somehow linked to the growing reliance on aquatic resources. Moreover, the earliest well-dated pottery assemblage in Cis-Baikal is from the riverside Gorelyi Les habitation site.

Interestingly, however, the results of this pilot-study of the Early Neolithic pottery assemblage fail to demonstrate that the early pottery was used for the specialised processing of aquatic resources. This may be related to the site's geographic location, which is some 50 km from the richest fisheries on the Angara River that are available throughout the year (Weber, submitted). In addition, the faunal and tool-kit evidence from Gorelyi Les suggest that it was more general-purpose site, perhaps serving as a stopping off point for groups moving up and down the Belaia River. Very few fish bones have been recovered here, whereas the abundant ruminant bones suggest a closer link to hunting activities. In contrast, excavations of the contemporaneous Early Neolithic occupation levels at the Ust' Khaita site located further upstream did produce abundant evidence of fishing activities (Savel'ev et al., 2001; Kurzybov, 2011). This suggests that the two sites may have had different functions, and that pottery may have been used differently at each location, a question warranting further comparative research. However, despite the clear evidence of hunting activities at Gorelyi Les (Ready, 2008), there is also no indication in these results that the pottery was used for specialised processing of ruminant fats, or that they were part of the technology for operating a "grease station" at the site (Elston et al 2011; Karr et al 2015).

Overall, the results pointed to a much more generalised use of early pottery at Gorelyi Les, which may reflect the incorporation of general-purpose cooking pots into a flexible and broad-based subsistence strategy. In managing routine tasks around the hearth, these new kinds of containers may simply have offered practical advantages over the hot-stone boiling of foods in baskets and boxes, allowing the slow simmering of mixed foods and preparation of nutritious mixed dishes, while freeing up time for performance of other domestic tasks (Weber, submitted). At the same time, there are hints at particular culinary practices, with a combination of fish and ruminant products being cooked in some pots, and more mixed dishes including non-ruminant animals cooked in others. While these patterns are difficult to interpret, there are no indications that the pottery was used for ritual activity, or to prepare or serve exotic or lavish foods at group feasting events (Hayden, 2009).

The highly generalized pattern of early pottery use at Gorelyi Les contrasts strongly with the growing number of studies from across northern Eurasia that all point to a close and persistent association between early hunter-gatherer pottery and the specialised processing of aquatic resources (Craig et al., 2007; Craig et al., 2013; Lucquin et al., 2016a; Gibbs et al., 2017; Kunikita et al., 2017; Oras et al., 2017; Shoda et al., 2017, 2020; Lucquin et al., 2018). However, more variable patterns of pottery use are now starting to emerge in the northern part of European Russia, where Early Neolithic hunter-gatherers initially make generalized use of early pottery, and only later use it for the more specialised processing of fish during the Middle Neolithic (e.g. Bondetti et al., 2020). A similar pattern may also emerge in Cis-Baikal. However, it may be useful to note that the dates of the Early Neolithic pottery from Gorelyi Les appear to fall at the start of the Kitoi cultural sequence, and that the dietary importance of aquatic resources increased only in later parts of the sequence (Weber et al., submitted).

8. Conclusion and outlook

The Late Mesolithic communities of Cis-Baikal were undergoing major changes, which culminated with the emergence of the distinctive Kitoi mortuary pattern at around 7560 cal. BP. At around the same time, the Kitoi were also "becoming Neolithic" through their initial adoption of clay pots. This study has aimed to understand how this early pottery was being used, and to explore why it may have been adopted. Our initial expectation was that the pottery would have been adopted for the specialised processing of aquatic resources, as small-scale use of clay cooking vessels appears at around the same time that the Kitoi were intensifying fishing activities, and embarking on a gradual transition towards a more aquatic-based diet. Despite these expectations, the results indicated that the earliest use of pottery in Cis-Baikal involved more generalised patterns of use, with the vessels employed to prepare a wide range of foods, which perhaps mirrors the broad and more flexible foraging strategies that existed at the start of the Kitoi cultural pattern. Of course, patterns of pottery use may have shifted as fishing started to play a more substantial role in the Kitoi diet. Ironically, the combined impacts of improved fishing technologies — alongside new kinds of powerful hunting bows — may have had a far more substantial and transformative effect on early Kitoi subsistence and social life than the initial adoption of the pottery traditions that actually defines them as "being" Early Neolithic.

Beyond these more specific conclusions, this pilot study has demonstrated that recovery, analysis and interpretation of lipid residues from archaeological pottery assemblages is viable at Cis-Baikal archaeological sites. Clearly much more work still needs to be done on a wider selection of Kitoi pottery assemblages from different sites and micro-regions. Some of the most interesting patterns may well emerge via comparative analysis of residues from the net-impressed pottery that has been recovered from both Kitoi graves and from contemporary habitation sites. Combined with this, it would be useful to start to source the clays used in pottery manufacture in order to test whether pots were being moved around the landscape, or just made, used and cached at campsites or other hunting or fishing stations.

Finally, it is also important to bear in mind that the scale and stylistic diversity of pottery use undergoes significant expansion in the later periods of Cis-Baikal prehistory (McKenzie, 2009), and that in parallel with this, the function of pottery may also have evolved and diversified over time. To address these issues, comparative lipid-residue analysis of multiple assemblages could be used to generate better understanding of spatiotemporal variability in food processing and consumption activities. In turn, these insights would greatly complement the large bioarchaeological datasets that document the diet, health and mobility of individuals from the Kitoi and Cis-Baikal's subsequent Late Neolithic and Early Bronze Age mortuary traditions (i.e. the Isakovo, Serovo, and Glazkovo).

Mixing Model

The mixing model was performed using R studio (version 3.5.1). Further information is provided in the Supplementary Material, Table D.

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