Defining pottery use and exploitation of natural products at Clairvaux XIV during the Middle Neolithic

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Introduction

Although organic residue analysis has been applied to archaeological vessels for more than 30 years surprisingly few studies have provided an integrated approach to vessel function (Cramp et al. 2011; Evershed et al. 1997a; Kimpe et al. 2001), partly because of high degree of fragmentation of archaeological ceramic assemblages. Defining pottery function and morphological variability within assemblages is essential to understanding: (i) why pottery was manufactured, (ii) how natural products were processed using such containers, (iii) activities carried out at the site, and (vi) the organisation of prehistoric communities.

The study of organic residues preserved in archaeological pottery provides deep insights into the exploitation of natural resources by past communities (Evershed 2008). The development of chemical analysis of organic residues has demonstrated that animal carcass and dairy products were consumed from the beginning of livestock farming in Anatolia in the Early Neolithic (Evershed et al. 2008). These practices spread from the Near East across Europe with the development of Neolithic (Copley et al. 2005b; Craig et al. 2005, 2011; Cramp et al. 2014a,b; Debono Spiteri 2012; Debono Spiteri et al. 2016; Regert et al. 1999; Salque et al. 2012; Salque et al. 2013). Beeswax, and hence evidence of bee product exploitation and processing, has been detected in a wide range of prehistoric pottery dating to the earliest phases of the Neolithic (Heron et al. 1994; Regert et al. 2001a; Regert et al. 1999; Roffet-Salque et al. 2015). Lipid residues of aquatic resources have been detected at the Mesolithic-Neolithic transition (Craig et al. 2011; Cramp et al. 2014a; Heron et al. 2015), while the evidence for plant processing remains scarce during this period, except at some Mediterranean sites (Debono Spiteri 2012; Šoberl et al. 2014).

Characterising absorbed organic residues extracted from a vessel is only one aspect of understanding its use, as intended and actual function must also be examined (Sigaut 1991). To achieve this, it is important to consider the shape (including capacity, stability, accessibility, and transportability) and physical properties (thermal and mechanical) of the vessel which help to define *intended use* (Rice 1987, pp. 224–226; Skibo 2013, pp. 28–51). Traces of sooting and carbonised deposits, use-wear damage and contents provide information about *actual use* (Rice 1987, pp. 232–236; Skibo 2013, pp. 63–181). In the last few years, some studies have attempted to determine pottery function by combining chemical analysis of vessel contents (characterisation of natural substances, concentration and distribution of lipids, molecular markers of heating) with ceramic morphology (Copley et al. 2005a; Evershed et al. 2003; Heron et al. 2015; Salque et al. 2012; Šoberl et al. 2014), mechanical properties of vessels (Nieuwenhuyse et al. 2015), or thickness of pottery walls (Craig et

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al. 2015). Relatively few studies have integrated the full range of available approaches, i.e. morphological classification, morphometric measurements, chemical characterisation of the content, and use-wear analysis (Fanti et al. 2018; Urem-Kotsou et al. 2002; Vieugué 2010). In most cases, the differential preservation of the molecular and ceramic records has prevented a complete and efficient functional study. Only specific use of particular shapes have been highlighted: sieves for dairy products (Salque et al. 2013), pedestal dishes (Šoberl et al. 2014), and cooking pots (Fanti et al. 2018; Salque et al. 2013) used for carcass product processing, *mortaria* for plant and animal product processing (Cramp et al. 2011), and vessels used as lamps (Copley et al. 2005a; Evershed et al. 1997a; Evershed et al. 1997b; Heron et al. 2013). Understanding the function of a whole ceramic assemblage via integrated analysis thus remains an unrealised goal since extensive and well-preserved ceramic material is needed, including both vessel profiles and well-preserved lipids trapped in the porous walls of ceramic vessels.

The *Néolithique Moyen Bourguignon* (NMB) group settled in eastern France, in the region extending from Belfort to Lyon and from Eastern Burgundy to the Western side of Neuchatel Lake between c. 4000/3900 and 3600 B.C. This culture has mainly been defined based on the characteristics of its ceramic assemblages, which are readily differentiated from the older and contemporaneous sites in the region (Michelsberg, Munzingen, Chasséen, and Cortaillod). Recent excavations at the lacustrine site of Clairvaux aimed at complementing our understanding on the NMB culture using ceramic studies. Thousands of vessels were excavated in this lake dwelling site and preliminary analyses of pottery from this and similar lake sites have shown the preservation of lipids to be quite exceptional (Mirabaud et al. 2007; Regert et al. 1998; Regert et al. 2001b, 2003b; Spangenberg et al. 2006). Vessel shapes were previously reconstructed by refitting sherds (P. Pétrequin et al. 2016). Combining typological and morphometric analyses, macroscopic identification for heating evidence, and organic residue analysis from pottery is thus a unique opportunity to investigate ceramic function and use at Clairvaux XIV and to better understand the NMB culture.

Herein, we report the investigation of pottery function at Clairvaux XIV, using molecular and isotopic analysis of lipid extracts, morphology and volume of the vessels, and evidence of heating.

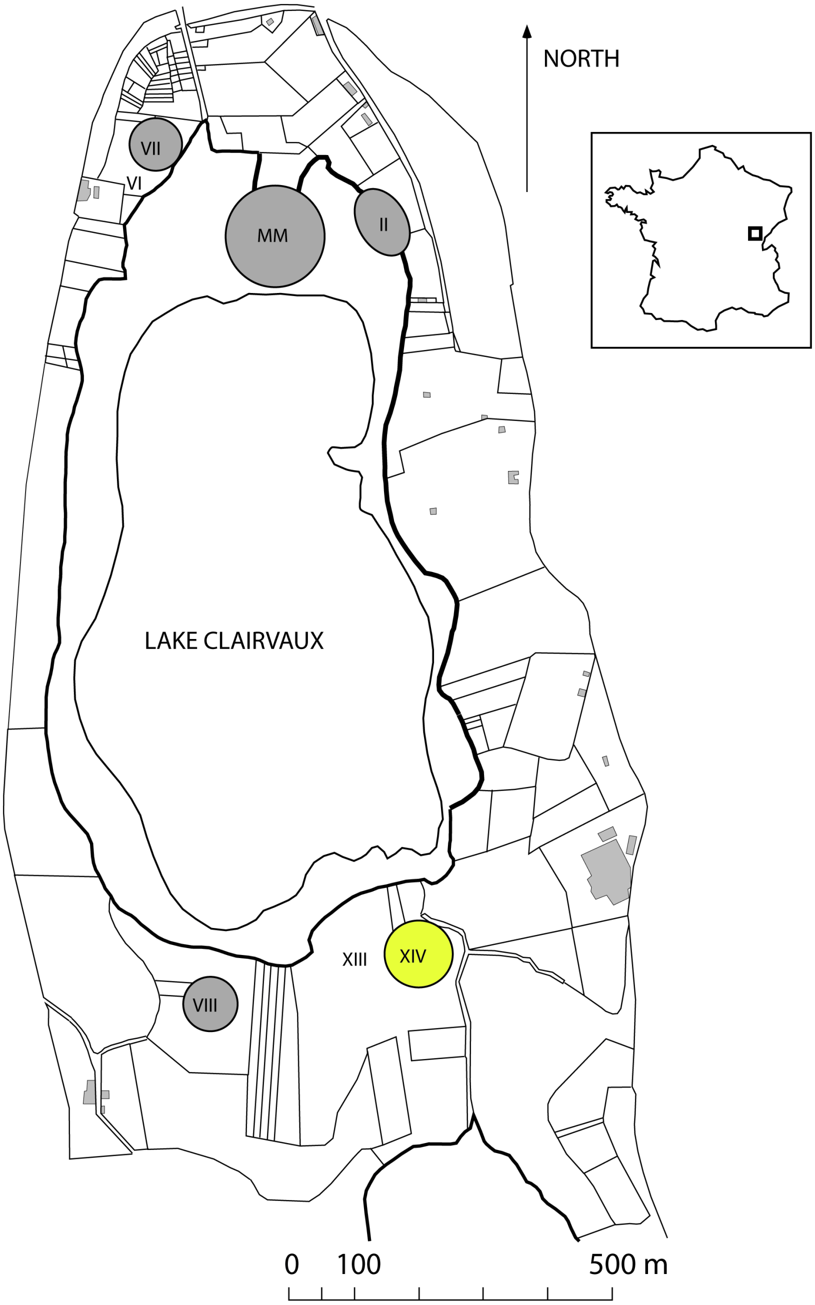

Figure 1: Map of lakes Clairvaux (geographic location in France, and position of the sites around the lake).

Materials and methods

Typological classification, macroscopic observation and morphometric study

In order to fully characterise pottery function at Clairvaux XIV, systemic analyses were performed. A total of 397 out of 440 ceramic vessels (Supplementary information: List of vessels from Clairvaux XIV) recovered from archaeological excavations at the site of Clairvaux XIV were classified in 15 typological categories (Figure 2; P. Pétrequin et al. 2016) and observed visually in order to identify evidence of heating, such as traces of soot and/or charred surface residues (Skibo 2013, p. 63).

Wherever possible, each vessel was characterised using the most relevant criteria applied in morphometric studies: height, maximum diameter and opening diameter (Rice 1987, pp. 224–226; Smith 1988). Volumes were also calculated for all the complete profiles using the application developed by the Archaeological Research Centre of the ULB (Engels et al. 2009). An estimation of minimum volume was made for vessels with incomplete profiles. All these measurements were used to highlight vessel *capacity*, *accessibility,* and *transportability* (Rice 1987, pp. 224–226).

Sampling for lipid analysis

A total of 95 potsherds (Supplementary information: List of vessels from Clairvaux XIV) were selected from the entire assemblage of 397 pottery vessels. Care was taken to ensure this sub-sample reflected the overall

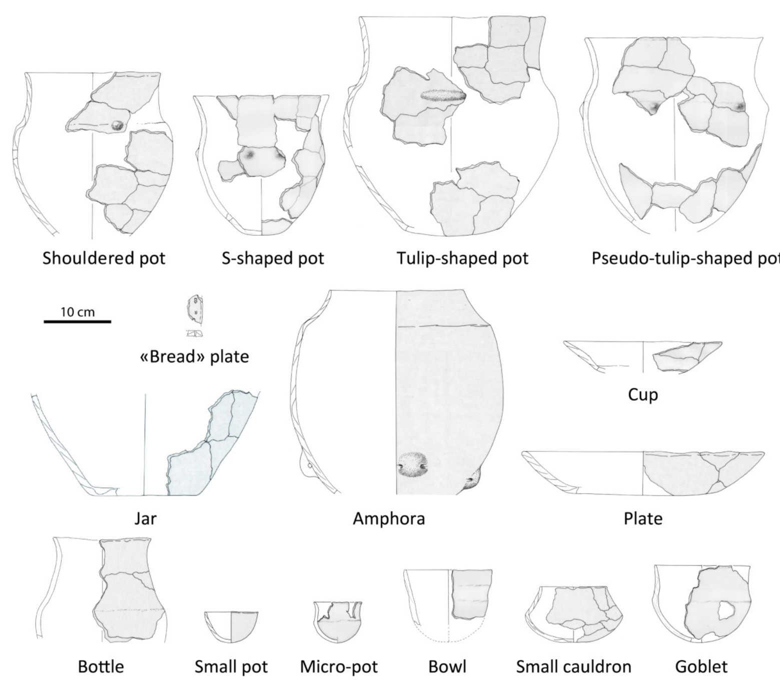

Figure 2: Typological categories from Clairvaux XIV. Drawings A.-M. Pétrequin.

assemblage diversity by taking into account the typology, distribution, dimensions, presence, or absence of charred surface residues on the vessels. A total of 33 thick surface residues were also analysed. For each selected vessel 2 g of potsherd were sampled, preferentially from the body of the vessels, as they have been shown to contain relatively high concentrations of lipids (Charters et al. 1993b, 1997; Evershed 2008). Where possible for shallow containers, such as cups and plates, samples were taken from the sides of vessels.

Extraction of absorbed lipids and molecular and isotopic analyses

Instrumental analyses (HT-GC, HT-GC/MS, and GC-C-IRMS) were performed in three independent laboratories. Details of extraction and derivatisation procedures and instrument operating parameters are available in Supplementary Information.

Results

Macroscopic observation of charring patterns

Macroscopic evidence for heating present on pottery vessels (soot deposits, fireclouds, and carbonised surface residues) have been recorded to understand ceramic use in cooking or other activities involving fire (Charters et al. 1997; Fanti et al. 2018). Two main groups of vessels were established: (i) ceramics with traces and residues suggesting an intense exposition to fire: mainly pots, bottles, jars and amphorae, and (ii) ceramics with no evidence for heating: mainly micro-pots, small pots, small cauldrons, bowls, cups, and goblets (Figure 5b).

Volume measurements

A broad diversity of volume capacities ranging from 0.02 to more than 25 l was observed in the pottery assemblage from Clairvaux XIV. Some typological groups display a narrow capacity range centred on small (cups, small cauldrons, goblets, bowls) and very small volumes (micro-pots, small pots), while in other groups (shouldered pots, S-shape pots, pseudo-tulip-shaped pots, tulip-shaped pots, amphorae) a wide range of volumes (from 0.5 to more than 25 l) is observed (Supplementary information: List of vessels from Clairvaux XIV).

Compositions and concentrations of lipids

Seventy percent of the potsherds yielded more than 5 $\mu g\ g^{-1}$ of adsorbed lipids, with a maximum of 2,055 $\mu g\ g^{-1}$. Surface charred residues contained between 0 and 3,523 $\mu g\ g^{-1}$ of lipids. The analysis of the Total Lipid Extract (TLE) from these potsherds highlighted a wide molecular composition, including intact acyl lipids (triacylglycerols and wax esters) and their degradation products (di- and monoacylglycerols, and fatty acids). Linear

Figure 3: Partial gas chromatograms showing typical trimethylsilylated TLEs from vessels from Clairvaux and containing lipid components characteristic of: (a) dairy products, (b) carcass fat, (c) plant oil, and (d) beeswax.

long chain alcohols and alkanes, sterols, and triterpenes have also been detected. Figure 3 illustrates the diversity of molecular composition by showing typical gas chromatograms of TLE from potsherds from Clairvaux.

Intact acyl lipids were especially well-preserved, with triacylglycerols (TAGs) observed in >30 per cent of the analysed vessels (ranging from 1 per cent to 40 per cent w/w of the TLE). The TAG distributions were characterised by the average carbon number (M) and the dispersion factor (DF; Figure 4) as suggested by S. Mirabaud et al. (2007).

The average carbon number M was calculated using

$$M = \frac{\sum (P_i C_i)}{\sum P_i}$$

where is the relative percentage of each TAG and is the number of atoms of the fatty acid moieties in each TAG.

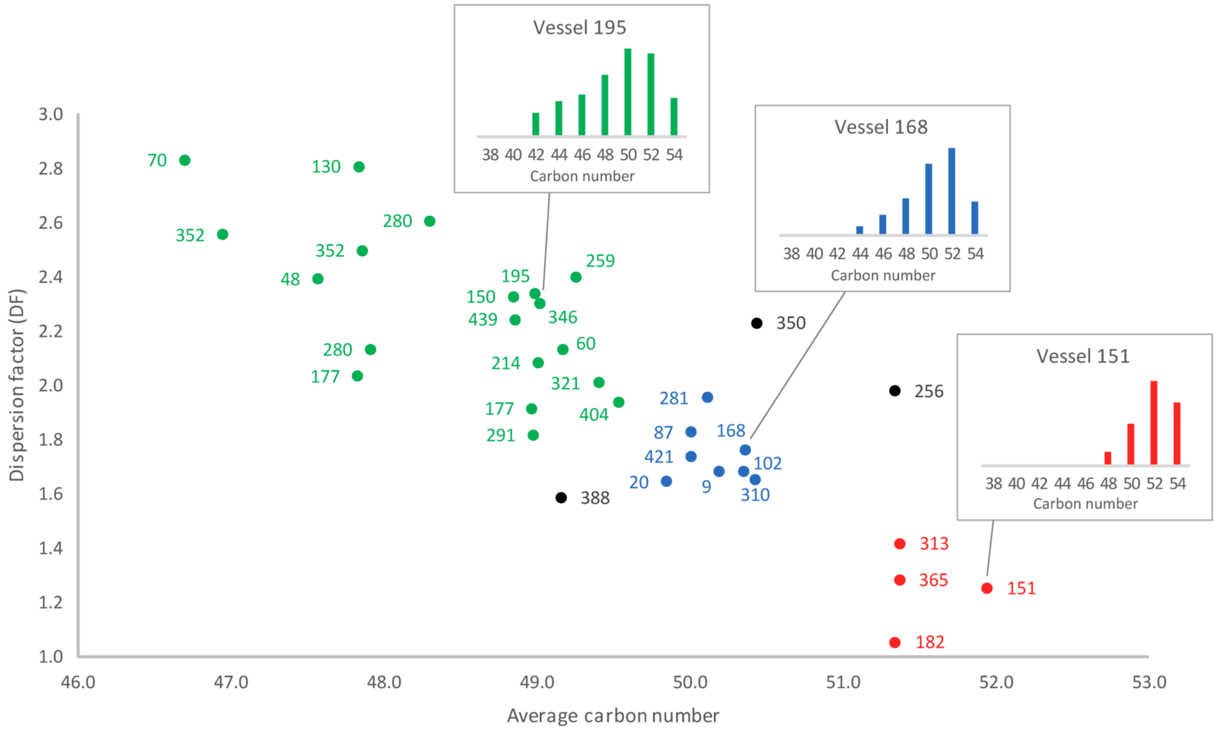

Figure 4: Distribution of archaeological samples based on HT-GC analysis of the TAG distributions: wide distribution (T_{40} to T_{54}) in green, intermediate distribution (T_{44} to T_{54}) in blue and narrow distribution (T_{46} or T_{48} to T_{54}) in red. Samples with unusual TAG distributions are plotted in black. The numbers correspond to the archaeological reference number of the vessels. The inserts are typical TAG distribution for the three groups.

The dispersion factor DF was calculated as follows:

$$DF = \frac{\sqrt{\sum \left((C_i - M)^2 \, C_i P_i\right)}}{\sum P_i}$$

The identification of three main categories made by S. Mirabaud et al. (2007) was confirmed, with wide (T_{40} to T_{54}), intermediate (T_{44} to T_{54}) and narrow (T_{46} or T_{48} to T_{54}) distributions of TAGs. These distributions have been shown to correlate with the origin of the fat.

Subcutaneous animal fats and dairy products

Several criteria were used to distinguish the sources of animal products, including: fatty acid, sterol and TAG compositions, and the carbon isotopic values of the $C_{16:0}$ and $C_{18:0}$ fatty acids. The presence of odd-numbered fatty acids ($C_{15:0}$ and $C_{17:0}$, linear and branched isomers), biosynthesised by bacteria in the rumen of ruminant animals, is indicative of ruminant products (Evershed et al. 1997a; Mottram et al. 1999). Degraded dairy products were characterised based on the fatty acid carbon isotope proxy: $\Delta^{13}C = \delta^{13}C_{18:0} - \delta^{13}C_{16:0}$, (Copley et al. 2003), a large distribution of TAGs between $T_{40\text{-}42}$ and T_{54} (Dudd and Evershed 1998), and the presence of cholesterol. Dairy fats were identified based on their wide TAG distribution including the presence of odd-carbon-numbered fatty acids and

$\Delta^{13}C$<-3.1 per mille, in 12 vessels (vessels 60, 70, 130, 150, 195, 214, 259, 321, 346, 350, 352 and 439; Figure 5). The extracts of 5 of the vessels displayed an intermediate TAG distribution (vessels 87, 102, 168, 185 and 421) and 4 vessels contained only traces or no TAGs (vessels 31, 107, 133 and 383; Figure 5), but were identified as dairy fats based on their fatty acid carbon isotope compositions. Dairy products were identified in a further 6 other vessels (vessels 48, 177, 280, 291, 388 and 404) based on their TAG profiles. ESI MSxMS analyses confirmed and clarified the identification of dairy products in 9 of these vessels (vessels 48, 60, 87, 130, 150, 168, 195, 214, 350; Mirabaud, et al. 2007). The $\delta^{13}C$ values of $C_{16:0}$ and $C_{18:0}$ fatty acids (Figure 5) allowed the identification of mixtures of dairy and non-ruminant adipose fats (vessel 215) and ruminant adipose and dairy fats, or deer adipose fats (vessels 151 and 256; (Craig et al. 2012)). Hence, dairy products were identified in 26 ceramics, >25 per cent of the vessels investigated.

Degraded animal adipose fats were characterised by a narrow TAG distribution, with ranges between T_{46} and T_{54} (Dudd and Evershed 1998), $\Delta^{13}C$ values ranging between 0.3 and -3.1 per mille for ruminant adipose fats and >0.3 per mille for non-ruminant adipose fats (Copley et al. 2003), and the presence of cholesterol. Mixtures of ruminant and non-ruminant adipose fats were detected based on their $\delta^{13}C$ values (vessels 30, 46, 180, 188, 313, 388 and 440; Figure 5). Animal adipose fats were identified based on a narrow TAG distribution in 7 vessels (vessels 1, 2, 9, 12, 20, 31 and 185). In vessel 182, the atypical TAG profile largely dominated by T_{52} and the $\delta^{13}C_{16:0}$ and $\delta^{13}C_{18:0}$ values indicate a non-ruminant adipose fat origin (Figure 5; Evershed et al. 1997a). Animal adipose fats were thus identified in 15 vessels (16 per cent of the analysed vessels), one of them was characterised as originating from non-ruminant adipose fats while mixtures of ruminant and non-ruminant adipose fats were observed in 7 vessels.

Four samples could not be analysed by GC-C-IRMS, mainly due to mixing with waxy substances, and contained no TAGs to identify securely the natural origin of the fats (vessels 13, 365, 416 and 432). The high concentration of fatty acids, dominated by stearic acid ($C_{18:0}$) and the presence of cholesterol allows their characterisation as animal fats.

Aquatic resources

ω-(*o*-Alkylphenyl)alkanoic acids (APAAs) resulting from alkali isomerisation of polyunsaturated fatty acids occurring during heating were detected in eleven vessels (vessels 30, 46, 130, 195, 214, 215, 256, 313, 388, 421, 259). As C_{20} and C_{22} polyunsaturated acids rarely exist in plant oils but are well distributed in marine and freshwater fats, C_{20} and C_{22} APAAs are thus considered as being biomarkers of aquatic product processing (Evershed et al. 2008; Hansel et al. 2004). C_{20} APAAs were identified in low concentrations in eight vessels (vessels 30, 46, 130, 195, 214, 215, 313, 421; Figure 6), but no C_{22} APAAs were identified. The weak presence of C_{20} APAAs points to the processing of aquatic resources although the evidence is relatively scarce. Since the carbon isotopic composition of the fatty acids from these extracts are not characteristic of freshwater organism products (Cramp and Evershed 2014; Outram et al. 2009), mixing with terrestrial fats is very likely. In fact, due to the low concentration of fatty

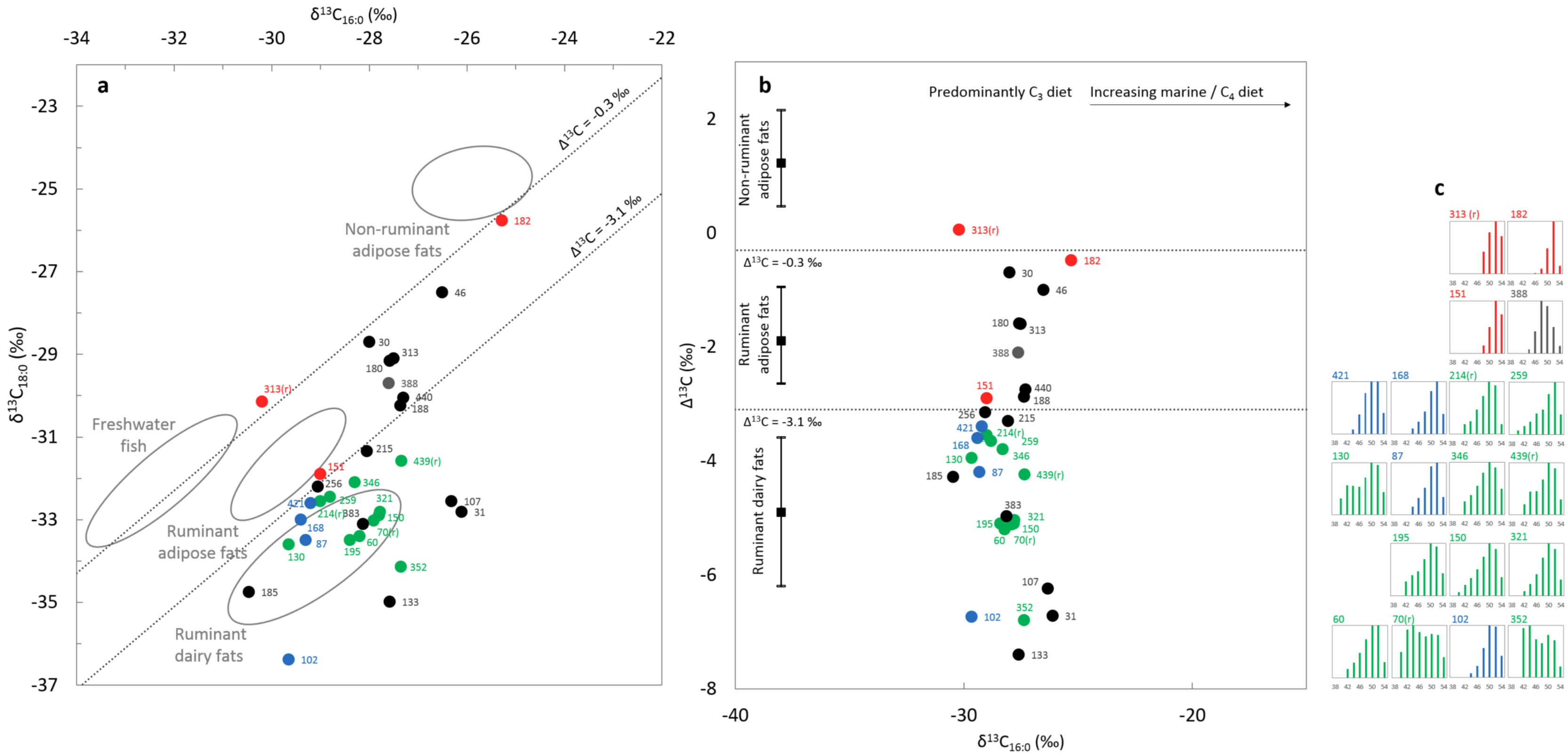

Figure 5: (a) δ^{13}C values for the C16:0 and C18:0 fatty acids of TLEs from Clairvaux XIV (the numbers correspond to the archaeological reference numbers of the vessels, (r) indicates a carbonised surface residue). Ellipses correspond to the δ^{13}C values of reference animal fats obtained from animals raised on a strict C3 diet in Britain (Copley, et al. 2003), and freshwater fish from Kazakhstan (Outram et al., 2009); (b) δ^{13}C values (δ^{13}C18:0 - δ^{13}C16:0) of the same samples from Clairvaux XIV plotted against their δ^{13}C16:0 values; (c) TAG distribution of the corresponding samples. Red and grey plots: narrow distribution of TAGs (T46-48 to T54), blue plots: medium distribution (T44 to T54), green plots: large distribution (T40-42 to T54; Figure 4 for TAG distribution groups).

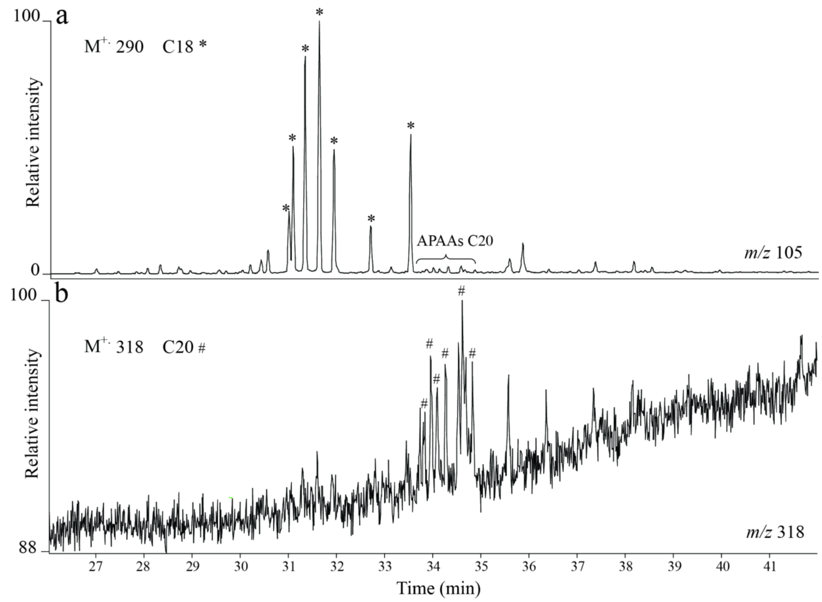

Figure 6: Mass chromatograms of (a) *m/z* 105 and (b) *m/z* 318 for vessel 214 (carbonised surface residue), showing C_{18} and C_{20} APAAs.

acids in freshwater fish compared to mammals, the isotopic signal from these fatty acids can be easily masked by the presence of fat-rich animal foodstuffs (Gunstone 2007). Furthermore, unusually high concentrations of $C_{20:1}$ fatty acids in two samples (vessels 180 and 188) could be linked to freshwater products (Heron et al. 2015), but no APAAs were detected in these samples. Five of the vessels containing APAAs up to C_{20} (vessels 21, 130, 195, 214 and 421) displayed $\Delta^{13}C$ values below -3.1 per mille (Figure 5) and are thus identified as dairy products. One sample (188) was interpreted as a mixture between ruminant and non-ruminant fats based on its carbon isotopic composition ($\Delta^{13}C$ value close to -3.1 per mille). The molecular and isotopic signal for freshwater product processing is thus very low in the ceramic vessels and it could be linked to an occasional processing of freshwater products in pottery vessels, swamped by the use of ceramic containers for cooking terrestrial animal products at Clairvaux.

Plant oils

Plant oils were identified by the presence of $C_{16:0}$ fatty acids in much higher concentration than $C_{18:0}$ fatty acids together with high proportion of $C_{18:1}$ (vessels 36, 133, 188, 216, 259, 267, 425). Further evidence for plant oil was obtained through the identification in some vessels of degradation markers of unsaturated acids (Copley et al. 2005b). Hydroxy- and dihydroxy- carboxylic acids ($C_{18:0}$ and $C_{20:0}$; vessels 133 and 188), diacids ($C_{8:0}$, $C_{9:0}$, $C_{10:0}$, $C_{11:0}$; vessels 36, 216, 267, 404, 425) and C_{18} APAAs without C20 APAAs (vessels 256, 259, 388, 421) were detected in a total of 11 vessels. Series of long-chain alkanes with odd-over-even chain lengths predominance (C_{25}-C_{31}) and a distribution maximising at C_{29} or C_{31} and alcohols (C_{24}-C_{30}) were detected in some TLEs (vessels 9 and 383). 16-Hentriacontanone was present in one extract. Wax ester profile (W_{42}-W_{46}) in which the acyl moiety is composed of both palmitic and stearic acids confirmed that vessel 9 had contained waxy plant-derived material (Ribechini et al. 2008). Unfortunately, the source of oily and waxy plant products was not determined as no specific biomarkers exist for the plants identified in the archaeobotanical record from Clairvaux XIV, such as cereals, peas, acorn, hazelnut, linseed, or poppy seed (Schaal and P. Pétrequin 2016).

Beeswax

A total of 10 vessels contained beeswax, which was easily identified through palmitic wax ester characteristic profiles accompanied by series of long-chain alkanes with an odd-over-even carbon number predominance, fatty acids, and alcohols (Heron et al. 1994; Regert et al. 2001a; Roffet-Salque et al. 2015). Degraded beeswax was identified mixed with fatty material, adipose, or dairy products in four vessels (vessels 2, 45, 177, and 280). Alteration of the characteristic molecular assemblage of beeswax may be due to natural degradation or to heat-treatment (Evershed et al. 2003, 1997b; Heron et al. 1994; Regert et al. 1998, 2001a). Exceptionally well-preserved biomarkers of beeswax were identified in several vessels (vessels 1, 12, 20, 190, 281, 310, 324 and 432), indicating that the material was mildly or not heated at all (Regert et al. 2001a), especially when, in addition, the corresponding ceramic vessel did not display any macroscopic evidence for heating.

Birch bark tar

Biomarkers of birch bark tar (lupenone, betulin, allo-betul-2-ene, lupa-2,20(29)lupadien-28-ol; Charters et al. 1993a; Regert et al. 2003a) were identified in only two ceramic sherds (vessels 31 and 416). Birch bark tar was mixed with animal adipose or dairy fats and could have been used to repair or decorate vessels consequently used to process animal products (Charters et al. 1993a; Dudd and Evershed 1999; Mirabaud et al. 2016).

Discussion

Preservation of lipids

Preservation of lipids was expected to be very good due to degradation at the site being limited by the lack of oxygen in the anoxic burial environment. The concentrations of TLE from Clairvaux pottery, together with TAG preservation, are similar to those obtained from other sherds from submerged marine and lacustrine contexts (Craig et al. 2007; Craig et al. 2011; Regert et al. 1999; Spangenberg et al. 2008), but also to more classical contexts.

TAG preservation can also be considered as a marker of vessel use since repetitive episodes of heating of fat-rich products lead to TAG hydrolysis (Choe and Min 2007; Evershed 2008). It thus suggests that TLE with low percentages of TAGs are likely to have been heated repeatedly, even though this point needs to be tested experimentally. Good preservation of TAGs was mainly observed in cups, small cauldrons, small pots, goblets, and microvases (half of these vessels contained between 5 per cent and 40 per cent of TAGs in the TLE). Most of the vessels from this group also presented clear evidence for heating (sooting marks or visible residues).

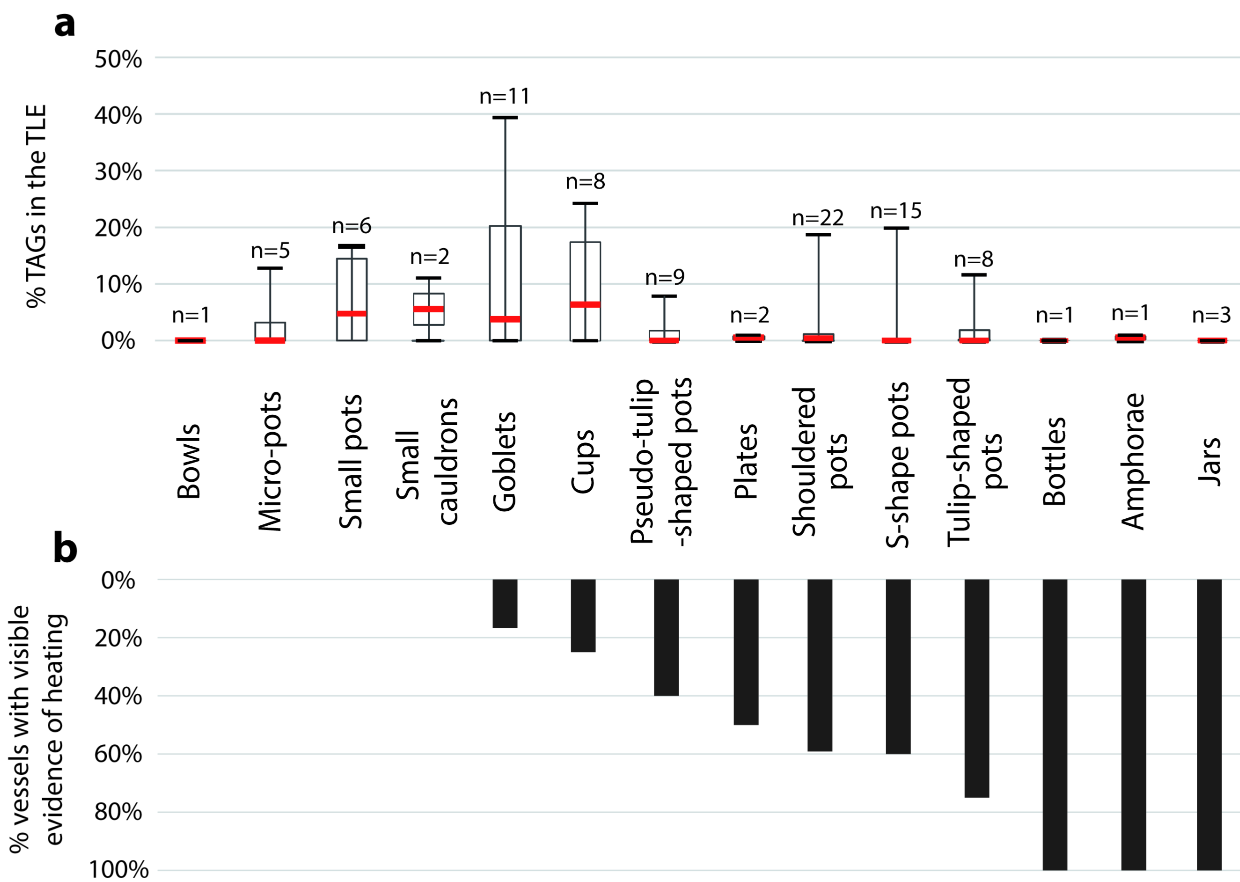

Figure 7: (a) Box plot of TAG percentage in TLEs from absorbed residues (minimum and maximum, median, 1st and 3rd quartiles); (b) percentage of vessels analysed for lipid residues presenting visible evidence for heating (trace of soot deposits or carbonised surface residues) for different pottery typological groups. Please note a correlation between well-preserved TAG distribution and lack of evidence for heating.

Three quarters of the larges pots, bottles, plates, jars, and amphorae contained <1 per cent of TAGs (Figure 7a). Low TAG preservation in these groups correlates well with visible evidence for heating, except for the bowls where both low abundance of TAGs and no evidence for heating were observed.

Functional categories

This work addresses the question of ceramic function by bringing together, morphological shapes, morphometric measurements (volumetric capacity, rim diameter) with evidence of heating (traces of sooting, carbonised surface residues) and organic residue identification for vessel contents. We focused on the most numerous typological categories of ceramics including: shouldered pots, S-shape pots, pseudo-tulip-shaped pots, tulip-shaped pots, small pots, micro-pots, cups, and goblets, which together represent around 80 per cent of the ceramic assemblage. Three main uses of the vessels were identified, with specific subcategories of use defined based mainly on the volume of ceramic vessels (Figure 8).

Pots of less than 1 l capacity (vessels 102, 130, 195, 251, and 352), whether they are shouldered, S-shape, pseudo- or tulip-shaped, displayed charac-

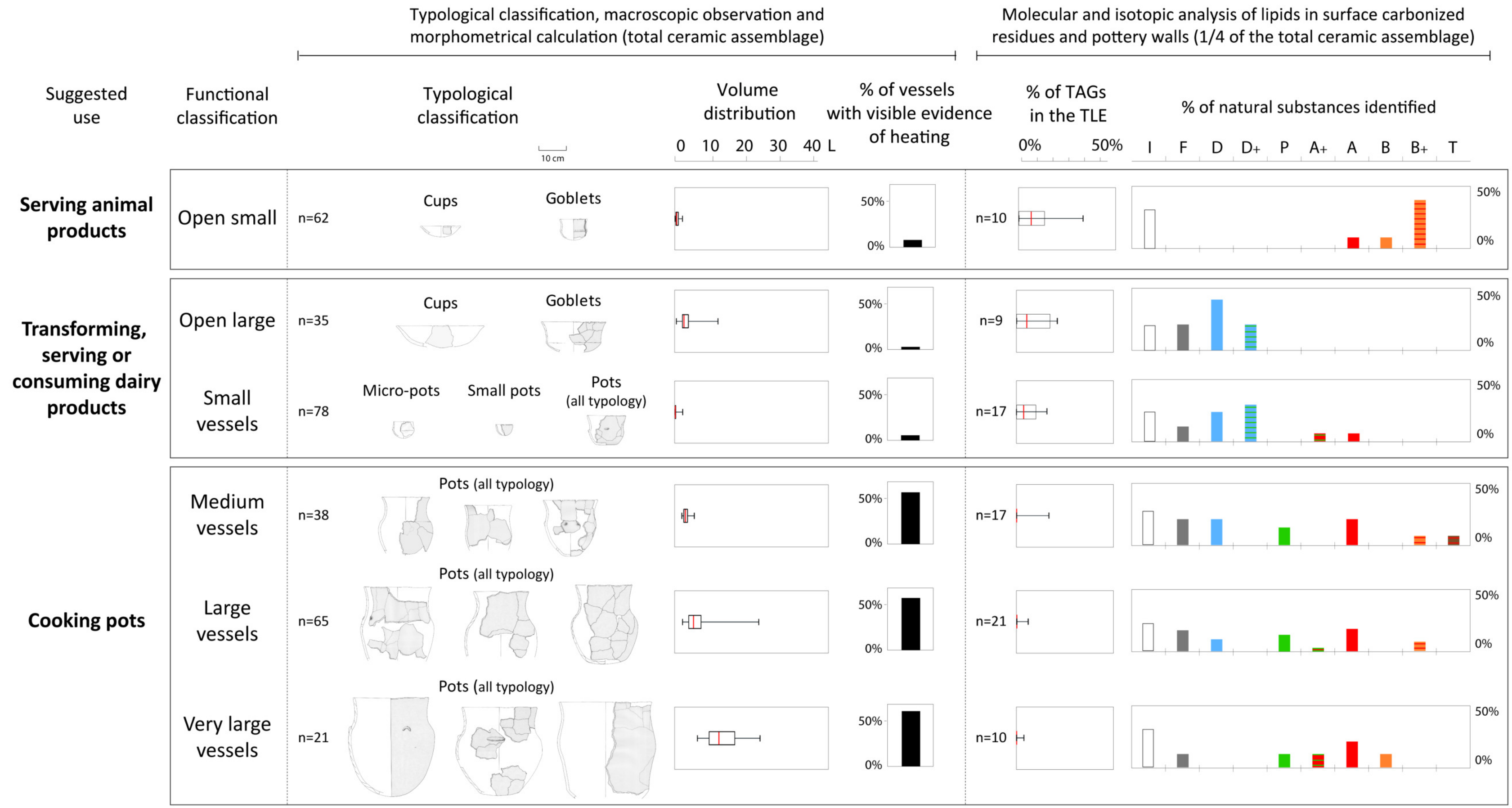

Figure 8: Classification of vessels from Clairvaux XIV and results from the integrated approach (typology, volume calculation, macroscopic observations, and lipid analysis); I: not interpretable; F: fatty products; D: dairy products; D+: dairy products, mixed with plant or freshwater products; P: plant products; A+: Animal adipose fat, mixed with plant or freshwater products; A: ruminant, non-ruminant, deer carcass products, and mixtures of animal carcass products; B: beeswax; B+: beeswax mixed with animal carcass or dairy products; T: birch bark tar mixed with animal carcass or dairy products.

teristics in common with small pots (vessels 150, 269, 273, 346, 382, and 385) and micro-pots (vessels 13, 81, 87, 133, 400). These vessels mainly contained dairy products, either pure or mixed with plant or aquatic oils. Almost 90 per cent of them did not display any traces of heating and contained well-preserved TAG profiles (half of them contained between 4 and 20 per cent of TAGs in the TLE). Furthermore, all these vessels had small or very small capacities: they could easily have been manipulated and handled with only one hand, and thus were likely intended for individual use (Henrickson and McDonald 1983; Smith 1988; Tsirtsoni 2001). Hence, the analyses revealed a new group of small and very small capacities vessels (Figure 8: Small vessels) that were specifically used for dairy products processing, serving or consumption. Other commodities were sometimes mixed with dairy products, maybe to prepare fermented products, such as yoghurt or cheese or to flavour dairy products. The large number of this type of ceramic vessels at Clairvaux XIV (10 per cent of the total assemblage) and other NMB sites, is seen on several sites from the Cortaillod culture, although they are rarely found on contemporaneous sites of the Michelsberg and Chassey cultures (P. Pétrequin et al. 2016). The use of small pots to process or serve milk has also been seen at sites from the British Neolithic (Copley et al. 2005a).

Cups and goblets are both shallow vessels allowing easy *access* to the contents (Rice 1987, pp. 225–226), exhibit similar characteristics, i.e. a wide range of substances was identified in those vessels and they were rarely exposed to fire: traces of soot or visible carbonised residues were rarely detectable on their walls (only 11 per cent of vessels displayed evidence of heating) and hydrolysable lipids (TAGs and esters) were usually well-preserved (the TLE of 50 per cent of these vessels contained more than 10 per cent of TAGs).

Understanding the use of cups and goblets with large rim diameters (vessels 37, 60, 225, 321, 357 and 421) is challenging (Figure 8: Open large) as the TLE from their walls seem to be similar to those from small vessels (dairy products, pure or mixed with plant-derived products, see above). Cups and goblets may have been used to serve dairy products mixed with low-fat commodities (e.g. cereals, fruits) or were used to process dairy products, possibly being used in association with small vessels, for example to collect the liquid whey from the draining of cheese curds (Gouin 1997; Salque et al. 2013).

Cups and goblets with small diameters (<20 cm; vessels 1, 12, 20, 213, 2801, 310, 320, 324, 388, and 391) mainly contained beeswax mixed with animal fats (Figure 8: Open small). Since these products seem to have rarely been heated, beeswax was probably used to waterproof these vessels, which were then used to serve or consume animal-based commodities (Copley et al. 2005a; Šoberl et al. 2014).

Large pots seem to have been regularly exposed to fire: 69 per cent of the pots (shouldered, S-shape, pseudo- or tulip-shaped) with more than 1 l capacities display visible evidence for heating. Furthermore, bases of these vessels are often difficult to reconstruct because of extensive flaking and alteration, probably linked to extended exposure to fire (P. Pétrequin and A.-M. Pétrequin 2016). When considering the molecular assemblage extracted from the sherds, TAGs are rarely detected in these vessels (77 per cent of them have no TAGs in the TLE) and, when present, beeswax molecular profiles are often highly altered (vessels 2, 45 and 117). The alteration of the *n*-alkane profile, hydrolysis of TAGs, and esters can happen during repeated episodes of heating involving water (Choe and Min 2007; Regert et al. 2001b), such as cooking soups and broths or frying water-rich commodities. The shape of these vessels would have been perfectly be suited to extensive boiling as their relatively closed rim prevents excessive evaporation but allows access to the content and facilitates release of steam, preventing the vessel content from boiling over (Henrickson and McDonald 1983; Skibo 2013, pp. 33–34). The high number of large pots excavated at the site (30 per cent of the total assemblage) could be explained by the frequent turnover of these cooking pots weakened by thermal shocks (P. Pétrequin and A.-M. Pétrequin 2016). Since some pots can reach more than 20 l capacities (Figure 8: Very large vessels), it would have been difficult for one person to move the vessel on and off the fire. Such large pots were certainly used to cook commodities for a group rather than for individuals. This wide range of capacities may be interpreted as fulfilling a variety of uses, ranging from family use (medium to large vessels) to community ceremonial use (large to very large vessels), as seen in ethnoarchaeological examples (Skibo 2013, p. 77). A broad diversity of natural substances appears in the residues of these medium to very large vessels, including: dairy fats, animal adipose fats, plant oils, and beeswax, either pure or mixed, with some slight variability depending on the volume (Figure 8: Medium vessels, Large vessels, and Very large vessels). The presence of several substances in the same vessel could be seen as intentional mixing or successive episodes of use. These vessels were thus identified as cooking pots, probably intensively used to cook a wide variety of commodities, as seen in other European Neolithic contexts (Fanti et al. 2018; Salque et al. 2013; Šoberl et al. 2014).

Pure beeswax with a molecular distribution very close to that of fresh beeswax was extracted from a very large vessel (vessel 190, more than 20 l). This pot exhibited no evidence of heating on its outer surface; and thus could have thus been waterproofed with beeswax and used as a storage vessel for water or water-based commodities, or used for storing beeswax itself.

Exploitation of natural products at Clairvaux XIV

Archaeozoological remains from Clairvaux XIV suggest that domestic ruminants have been exploited both for meat and milk (Arbogast 2016). This is reflected in the lipids preserved in pottery vessels through widespread occurrence of ruminant carcass and dairy fats identified using the fatty acid stable isotope compositions and TAG distributions. In contrast, non-ruminant fats were detected in few vessels, although boar and pig skeletal remains are abundant in the faunal assemblage. According to the very high number of bone remains of wild animals and domestic non-ruminants compared to the domestic ruminants, the formers appear to have been the major source of meat in Clairvaux in most of the occupation phases, even when considering meat yield (Lyman 1979; White 1953). Non-ruminant animals could have been processed without pottery, roasted over a fire or heated on stones, as evidenced by the presence of several burnt bones at the site, especially amongst the porcine assemblage (Arbogast 2016).

Molecular and isotopic analyses confirmed the importance of milk exploitation at Clairvaux, supporting herd reconstruction based on the kill-off profiles for domesticates (Arbogast 2016). Dairy products were detected in 25 per cent of the vessels, with specific vessels being dedicated to the processing and consumption, i.e. small vessels and open large vessels. Dairy products thus seem to have been of particular importance for the Neolithic inhabitants of Clairvaux, reflecting trends seen at other Neolithic sites (Copley et al. 2005b; Craig et al. 2015; Salque et al. 2013). Dairy products appear to have been exploited since the beginnings of animal domestication (Debono Spiteri et al. 2016; Evershed et al. 2008) and their consumption has continued over millennia across the European Neolithic, probably because of their nutritional qualities but also because of their relatively non-perishable properties allowing storage through the winter period (Copley et al. 2005a). Milk could have been consumed fresh or mixed and cooked with other commodities (vessels 214, 256, 280) or transformed in other dairy products, such as yoghurt, butter, or cheese. Mixing milk with plant extracts could have been used as an alternative to rennet in cheese production (Egito et al. 2007; Macedo et al. 1993). Plant products could have also been used for flavouring or increasing the shelf-life of dairy products (Guarrera et al. 2005).

Plant oils and waxes were detected in several ceramic vessels, but their molecular distributions are insufficiently diagnostic to determine their natural origins. However, carpological studies revealed that cereals (barley, wheat, durum wheat, einkorn wheat, and emmer wheat), peas, acorns, hazelnuts, linseed, and poppy seeds were available at Clairvaux XIV (Schaal and P. Pétrequin 2016). Exploitation of flax for textile-production can be ruled out since no flax yarn or linen textile has been identified among the well-preserved ropes and woven materials (Schaal and P. Pétrequin 2016). Flax seeds were probably used as a source of vegetable oil, which is extracted by boiling ground seeds (Ertuğ 2000; Marinval 2005, p. 25) as seen on the contemporary sites of the Pfyn culture (Maier and Schlichtherle 2011). Vegetable oil could also have been produced from poppy seeds and hazelnuts. Boiling could have been used to remove toxic tannins from acorns for human consumption with lipid residues being deposited in the walls of vessels as a by-product of this process (Aurenche 1997; Deforce et al. 2009; Saul et al. 2012). Plant oils at Clairvaux were mainly identified in large and very large vessels exhibiting visible evidence for heating (vessels 36, 216, 267 and 425). Such vessels would indeed be particularly suitable for boiling large quantities of seeds. Plant products were occasionally identified together with animal fats (vessels 9, 67, 188 and 440).

Aquatic biomarkers were scarce in the ceramics from Clairvaux XIV, which is surprising considering the lacustrine location of the settlement. Evidence for fishing is seen at contemporaneous sites around the lake Clairvaux based on the presence of fish vertebra and the remains of fishing nets, however, the overall interpretation is that it was an occasional activity, with fishing occurring preferentially on the river. As with many foodstuffs, fish may have been prepared without the use of ceramics, being consumed raw, salted, smoked, or dried. The discovery of the eggs from fish parasites in human faeces from the site, would support the consumption of raw or undercooked fish (Dommelier-Espejo and P. Pétrequin 2016).

Beeswax occurs extensively in the extracts of the Clairvaux XIV pottery vessels, sometimes mixed with animal carcass or dairy fats presenting carbonised surface residues (vessels 2, 45, 177, 280). Residues of beeswax could indicate that honey was used as part of a recipe involving various foodstuffs, as it has also been identified in late Neolithic vessels from Chalain 3 (Regert et al. 1999). In several vessels (vessels 1, 12, 20, 190, 281, 310, 324 and 432), the molecular signal from beeswax is very well preserved, probably because the material had been mildly heated or not heated at all. When detected alone, we can suggest that the ceramics were waterproofed with beeswax or that this product was stored in the vessels. Beehive products are known to have been exploited at least from the beginning of Neolithic since beeswax is attested in numerous ceramic vessels (Roffet-Salque et al. 2015), in particular in contemporaneous archaeological sites in Germany (Heron et al. 1994) and France (Regert et al. 1999). Beeswax was probably obtained from wild beehives in forests surrounding the lake of Clairvaux. Those forests were also highly exploited for other products such as wood (Duffraisse 2016), wild fruits (Schaal and P. Pétrequin 2016), and wild game (Arbogast 2016). Besides a good knowledge of the forest ecosystem, collecting bee products required specific harvesting strategies and skills from the villagers (Mirabaud et al. 2016).

Conclusions

This study of the ceramic assemblage from Clairvaux XIV with both traditional ceramic analysis and organic residue perspective highlights the complex use of pottery vessels during the first half of the IVth millennium BC. Combining macroscopic observation, typological classification, morphometric measurements, and analysis of preserved lipids, using molecular and isotopic approaches, has provided new insights into the society living at Clairvaux XIV.

We have identified a wide range of vessel uses among the pottery assemblage: utilitarian pots used for cooking or processing a wide variety of animal and plant products, small shallow vessels for serving or consumption, and dedicated vessels for milk and/or dairy product processing or consumption. Several other studies of Neolithic ceramic vessels have focused on cooking pots (Debono Spiteri 2012; Dudd and Evershed 1999; Fanti et al. 2018; Šoberl et al. 2014; Vieugué 2010). One study demonstrated the specific use of some vessels for dairy products in comparison to cooking pots (Salque et al. 2013), while in the British Neolithic dairy products were mainly processed in carinated bowls (Copley et al. 2005c; Cramp et al. 2014b; Smyth and Evershed 2016). Most the vessels investigated were thus clearly related to food and culinary or consumption activities.

The diversity of vessel functions seems to have remained constant within the early phases of the settlement, which indicates that the economic activities remained the same through time at Clairvaux XIV (Smith 1988). During the last phases (5 and 6), the percentage of vessels dedicated to dairy products slightly decreases (around 30 per cent of the whole ceramic assemblage during phases 1 to 4, but falling to 22 per cent during phases 5 and 6), reflecting the decline in the importance of small ruminants in the faunal assemblage and the increase of game before the abandonment of the village (Arbogast 2016).

Archaeobotanical (Schaal and P. Pétrequin 2016) and archaeozoological (Arbogast 2016) studies have already demonstrated that agriculture and herding were complemented by forest exploitation (fruit collection, hunting). Our approach completes our understanding of the complex resource management practices at Clairvaux XIV. This study revealed that non-ruminant domestic and wild animals were likely prepared and consumed without using ceramics, a practice highlighted at other Neolithic sites (Craig et al. 2015; Mukherjee et al. 2008; Šoberl et al. 2014). Fishing was an occasional activity at Clairvaux, and as in most Neolithic sites (Copley et al. 2005a; Cramp et al. 2014a; Debono Spiteri 2012; Fanti et al. 2018; Smyth and Evershed 2016; Spangenberg et al. 2006, 2008), aquatic products were rarely processed in ceramics. Dairy products are present at high abundance in the ceramic assemblage, sometimes mixed with plant or aquatic products. Milk was mainly obtained from small ruminants and seems to be of particular importance for the Neolithic people living at Clairvaux XIV. Chemical analysis also revealed that bee products were exploited at the site. Because of the broad diversity of natural products identified in the ceramic vessels, Clairvaux XIV stands apart from other Neolithic sites: only few sites in Italy (Debono Spiteri 2012), Slovenia (Šoberl et al. 2014) and England (Copley et al. 2005a) have revealed the processing of such a wide range of commodities in ceramic vessels. Several complementary hypotheses can account for the wide range of natural products detected in these vessels, particularly the exceptional preservation of organic matter in lacustrine context, the range of potshapes investigated in this study and the diversity and the complementarity of the exploited ecosystems.

Studying the ceramic assemblage from Clairvaux XIV has highlighted the potential of integrating archaeological and chemical analyses in large-scale study of pottery assemblages to unravel their use. Pottery use, activities carried out at the settlements, and evolution of food practices can be investigated using this interdisciplinary approach. Further integrated studies of pottery vessels from other sites from NMB and neighbouring cultures such as Michelsberg, Munzingen, Chasséen, Pfyn, and Cortaillod now need to be carried out in order to understand if these practices were common in the Middle Neolithic sites or if they were specific to Clairvaux XIV. It would be of particular interest to study ceramic vessels from La Motte-aux-Magnins V (Middle Neolithic, around Lake Clairvaux) where clearer evidence of fishing activities has been found. The status of milk and milk products during this period should also be investigated: dairy fats have already been identified in four vessels at Chalain 4 (Late Neolithic, 3040–2950 cal. BC) and sieves appear in this region at sites from the Late Neolithic (Clairvaux III and Chalain) and the Early Bronze Age (La Motte-aux-Magnins; P. Pétrequin 1986). Investigating vessel use of small and very small vessels and cups could bring information concerning the specific use of these vessels in the Cortaillod culture (P. Pétrequin et al. 2016).

Supplementary Information

For this contribution, research data are freely available online at https://doi.org/10.11588/data/5NSDVM.

Files:

S1: Devices and parameters. (docx)
S2: List of vessels from Clairvaux XIV. (xlsx)
S3: Results from chemical analysis. (xlsx)

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