



Pottery use across the Neolithic transition in northern Belgium: evidence from isotopic, molecular and microscopic analysis

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Abstract

This study represents the first extensive residue analysis of prehistoric pottery from northern Belgium. It examines pottery use and culinary practices across the Mesolithic-Neolithic transition, from the late 6th to the early 4th millennium cal BC. Residue analyses were performed on more than 200 samples from nine archaeological sites, representing different cultural groups from this transitional phase. This includes the analysis of charred food residues encrusted on the vessel surfaces by elemental analysis-isotope ratio mass spectrometry (EA-IRMS), gas chromatography-mass spectrometry (GC-MS), stereomicroscopic analysis and Scanning Electron Microscopy (SEM), as well as the analysis of absorbed lipids by gas chromatography-combustion-isotope ratio mass spectrometry (GC-C-IRMS). This study provides the first evidence of ruminant dairy fats in Early Neolithic Limburg pottery, supporting the hypothesis that this pottery was produced and used by LBK farmers rather than hunter-gatherer populations. The first indigenous pottery of the Swifterbant culture was frequently used to process freshwater fish (often together with plant foods) and ruminant meat, although several of the studied vessels likely contained mixtures of resources which could also include porcine products. Ruminant dairy is nearly absent from this pottery. Similar results were obtained for pottery of the subsequent Michelsberg culture/Group of Spiere of the late 5th and early 4th millennium cal BC. The limited presence of ruminant dairy fats in this pottery contrasts with the findings for Middle Neolithic pottery from neighbouring regions, providing further evidence for the existence of regional variations in pottery use or culinary practices throughout prehistoric NW Europe. However, our current view of pottery use during the Mesolithic-Neolithic transition in northern Belgium might be biased by the difficulties in distinguishing between wild and domesticated ruminant adipose fats as well as in detecting plant foods through lipid residue analysis.

Keywords NW Europe · Pottery · Hunter-gatherers · Early farmers · Organic residue analysis · Culinary practices

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Introduction

The last decade has seen a significant increase in isotopic and molecular studies on the use of hunter-gatherer pottery in Northern and Eastern Europe (Craig et al. 2011; Courel et al. 2020, 2021; Dolbunova et al. 2022; Lucquin et al. 2023). In these regions, knowledge of pottery production techniques appears to have been largely transferred between hunter-gatherer populations, long before the arrival of Neolithic groups and the appearance of domesticated animals and plants (Jordan and Zvelebil 2009; Gibbs and Jordan 2013). The situation is different in Northwestern Europe. Here, pottery was first introduced by farmers of the *Linearbandkeramik* (LBK) culture, as they settled on the loess soils of the Netherlands, central Belgium (from ca. 5300 cal BC onwards) and later the Paris Basin (from ca. 5100 cal BC onwards). They were followed by farmers of the Blicquy/Villeneuve-Saint-Germain (BVSG) culture in the latter two areas (ca. 4900–4650 cal BC). The LBK sites in Northwestern Europe sometimes also yield the remains of other pottery traditions, including La Hoguette and Limburg pottery, the origin of which is much debated. Many scholars consider it to represent the earliest indigenous hunter-gatherer pottery in the region (e.g. Jeunesse 1987, 2002; Gronenborn 1998; Manen and Maruzié de Keroualin 2003; Kirschnick 2021), while others claim LBK farmers produced it, possibly as some kind of special purpose pottery (Constantin et al. 2010; Gomart 2014).

Small amounts of these initial pottery traditions, and other Neolithic commodities (stone adzes, cereal grains, domesticated animals), are occasionally found outside the loess area, for instance in the sandy lowlands of northern Belgium and the Netherlands, indicating that there was mobility between both regions. These finds are often interpreted as the possible result of contacts between early farmers from the loess and hunter-gatherers from the sandy regions north of the loess, in the late 6th and early 5th millennium cal BC (Raemaekers 1999; Verhart 2000; Louwe Kooijmans 2007; Crombé 2010; Crombé et al. 2015a, 2020). As a result of these contacts, knowledge of pottery production was transferred to indigenous hunter-gatherers in the first quarter of the 5th millennium cal BC, leading to the formation of the Swifterbant (SWB) culture, sites of which are mainly known from the Netherlands and northern Belgium (Raemaekers 2011; Teetaert and Crombé 2021; Dreshaj et al. 2023). Recent studies show clear parallels in pottery technology and lithic industry between the SWB culture of the Scheldt river basin in northern Belgium and the BVSG culture of the central Belgian loess area (Teetaert 2020; Halbrucker et al. 2022; Messiaen et al. 2023), indicating close interactions between these groups. It seems that the local hunter-gatherer populations adopted pottery

production long before agricultural practices. Direct evidence of animal husbandry and crop cultivation among the SWB populations appears later, during the second half of the 5th millennium cal BC, especially at sites in the Netherlands, but the timing and pace of this neolithisation process remains the subject of research and debate (e.g. Crombé et al. 2020, 2022; Raemaekers et al. 2021, 2023; Brusgaard et al. 2022; ten Anscher et al. 2023).

Recent studies also revealed a partial continuity in lithic technology between the SWB culture and the Middle Neolithic Michelsberg culture/Group of Spiere (MK/SP) (Messiaen 2020; Messiaen et al. 2023), which occurred in the Scheldt river basin between ca. 4300/4250–3800 cal BC. Rather than resulting from human migration, its development is thought to be the result of local adaptations through contact with Middle Neolithic cultural complexes in neighbouring areas, most notably northern France and the Paris Basin (Vanmontfort 2001, 2004; Bostyn et al. 2011). These are considered to have been more fully agro-pastoral communities, with a subsistence economy largely based on cereal cultivation and animal husbandry (Vermeersch and Burnez-Lanotte 1998; Vanmontfort et al. 2001/2002; Vanmontfort 2004). However, the relatively high numbers of wild animal bones at some MK/SP sites, and the presence of charred remains of fruits and nuts, indicate that hunting, fishing and gathering still complemented the diet.

Information on pottery use during this long transitional phase in the Low Countries (late 6th to early 4th millennium cal BC) is so far restricted. While recent studies provided valuable insights into the use of SWB pottery from the Netherlands (Raemaekers et al. 2013; Demirci et al. 2020, 2021; Kubiak-Martens and Oudemans 2023), research into the use of SWB pottery from northern Belgium remains limited to a few pilot studies (Craig 2004; Craig et al. 2007; Boudin et al. 2009, 2010; Teetaert et al. 2017). Moreover, there is a significant gap in knowledge regarding pottery use among the early farming communities throughout the entire region. The current paper presents the first comprehensive organic residue analysis of prehistoric pottery from northern Belgium, integrating existing data with the results of recent analyses. This includes the analysis of absorbed lipids by GC-C-IRMS and of residues encrusted on the vessel surfaces (or “food crusts”) by GC-MS, EA-IRMS, stereomicroscopic and SEM analysis. With over 200 samples from nine archaeological sites, the combined results of these isotopic, molecular and microscopic analyses provide new insights into pottery use and culinary practices across the Neolithic transition in northern Belgium.

Materials and methods

Archaeological sites and samples

The pottery included in this study originates from nine archaeological sites situated in the Scheldt river basin in northern Belgium (Fig. 1). Three sites at Doel Deurganckdok (B, J and M) represent the remains of repeated SWB occupations on Pleistocene coversand dunes in the lower Scheldt river floodplain, dated between ca. 4500–4000 cal BC (Crombé 2005; Boudin et al. 2009, 2010; Crombé et al. 2015b). Doel Deurganckdok site C represents the remains of a MK/SP occupation on the same location, possibly between ca. 4000–3800 cal BC based on radiocarbon (^{14}C) dates of food residue encrusted on the pottery from this site (Crombé 2005). Also in the lower Scheldt river basin, the levee sites of Bazel Sluis and Melsele Hof ten Damme yielded the remains of several occupation events, dating from the Early Mesolithic to Middle Neolithic periods (ca. 8th to early 4th millennium cal BC) (van Berg et al. 1992; Crombé et al. 2015a, 2020; Meylemans et al. 2018). Both

sites yielded limited numbers of Early Neolithic pottery traditions, including late LBK and Limburg pottery at Bazel and BVSG pottery at Melsele (Crombé et al. 2015a; Teetaert and Crombé 2022). Likely, this “exotic” pottery originated from the central Belgian loess area, where several clusters of LBK and BVSG settlements are known (Fig. 1). Apart from this limited number of Early Neolithic pottery, the sites at Bazel and Melsele mainly yielded occupation remains of the local SWB and MK/SP cultures. These include thousands of lithic artefacts and potsherds, as well as numerous plant and animal remains. However, at both sites, the remains of these different occupation events became mixed, due to human/animal trampling and bioturbation, making it difficult to link the plant and animal remains to specific occupation events or cultural groups (Crombé et al. 2015a, 2019). As a result, the SWB and MK/SP occupations at Bazel and Melsele cannot be accurately dated within the 5th to early 4th millennium cal BC. Finally, the study includes three more sites with MK/SP occupations (ca. 4250–3800 cal BC), located in the upper Scheldt river basin. These are the wetland site of Oudenaarde Donk Neo 1, and the sites of Menen

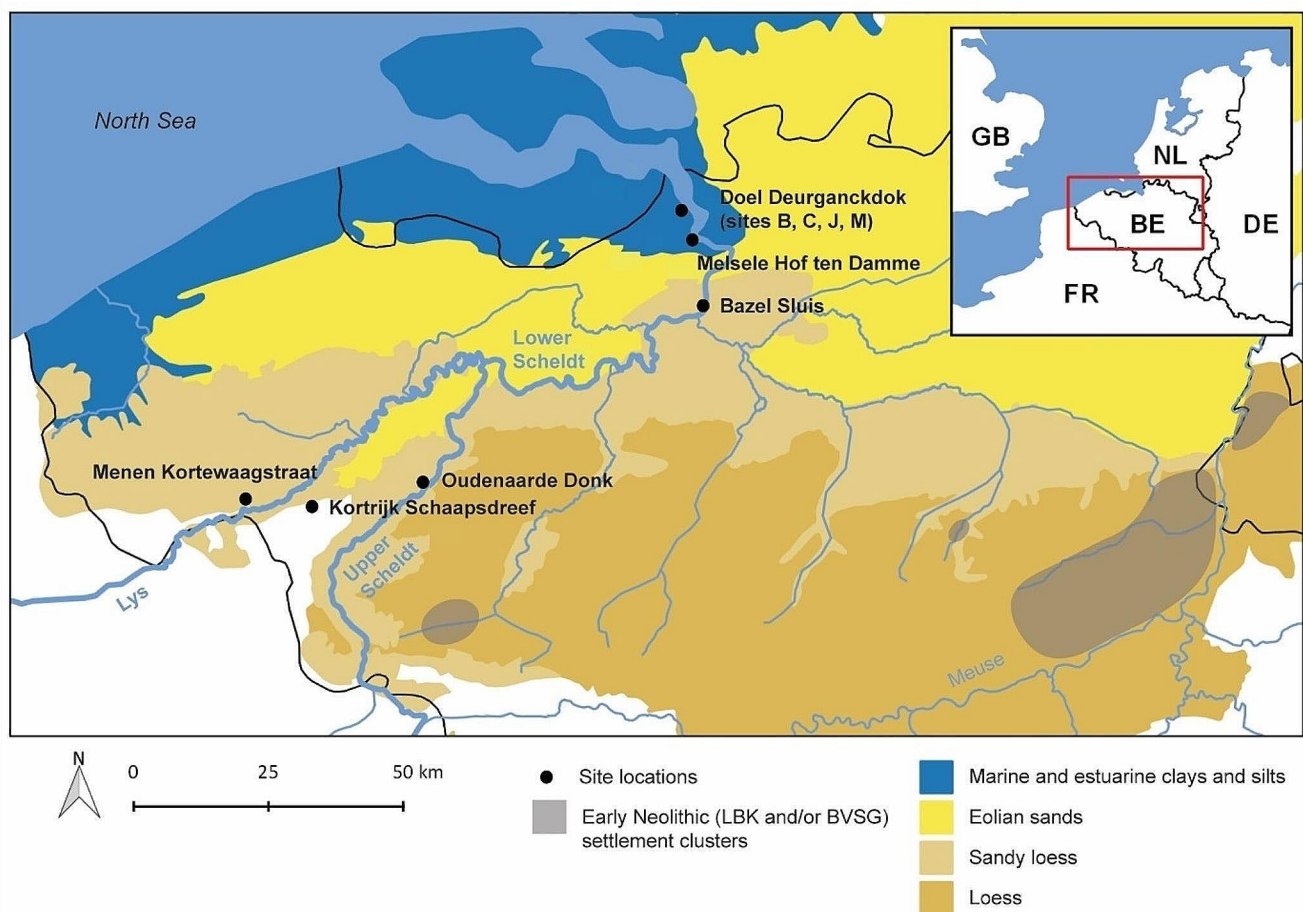


Fig. 1 Location of the nine study sites in the Scheldt river basin, northern Belgium. DMS geographical coordinates of the archaeological sites are provided in Online Resource 1

Kortewaagstraat and Kortrijk Schaapsdreef, both of which are situated on higher grounds some distance away from the Scheldt and Lys rivers (Parent et al. 1987; Verbrugge et al. 2009; Teetaert et al. 2019).

The Early Neolithic pottery found at sites in the lower Scheldt river basin is usually highly fragmented and vessel shapes are difficult to reconstruct (Fig. 2a, b). Among the selected pottery from Bazel Sluis are fragments of grog-tempered LBK pottery with a decoration of rows of comb impressions organised in a band (Fig. 2a), typical of the late LBK phase in this area (Modderman 1970), and of bone- and grog-tempered Limburg pottery with a decoration of grooves (Fig. 2b). The latter could represent large bowls

with thickened rims, i.e. the most characteristic shape of Limburg pottery (e.g. Ilett and Constantin 2010). In addition, the selection includes several sherds of undecorated bone-tempered pottery from Bazel Sluis that could either represent Limburg, BVSG or LBK pottery (Table 1; Online Resource 1).

The SWB pottery from the Scheldt river basin is characterised by closed vessel shapes, mainly beakers (Fig. 2c, d), with straight or everted rims and round, conical or pointed bases. Less frequent shapes are large or small open bowls (Fig. 2e, f), open beakers and buckets (Teetaert 2020). Decoration is limited. When it occurs, it usually consists of a row of fingertip or spatula impressions on top of the rim.

Fig. 2 Illustrations of a selection of pottery included in this study. Top: Late LBK (a) and Limburg pottery (b) from Bazel Sluis. It is noted that body sherds of LBK pottery were selected for this study, not the rim sherds shown in this image. Central: SWB beakers (c, d) and bowls (e, f) from Bazel Sluis and Doel sites B and J. Below: MK/SP beakers (g-i) from Bazel Sluis (technical drawings from Teetaert 2020)

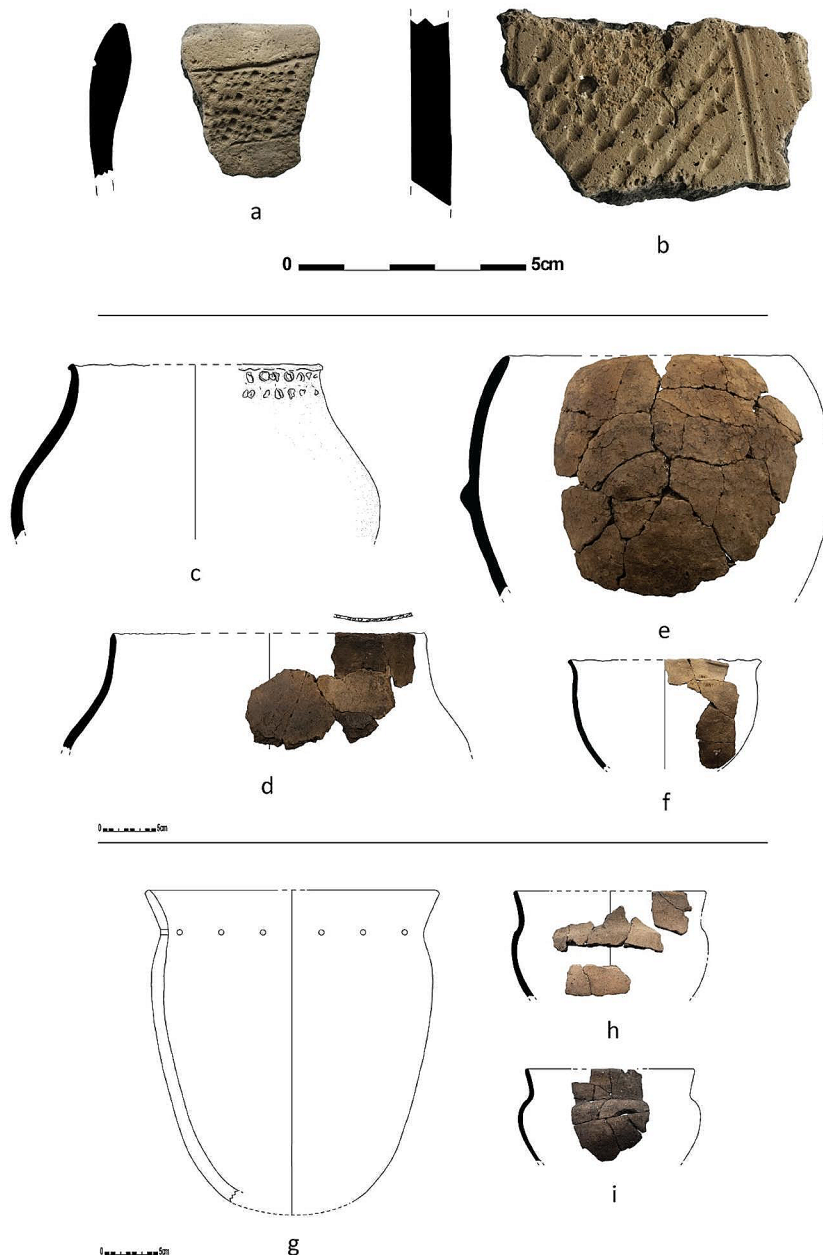


Table 1 Overview of the types of analysis and corresponding number of samples for each cultural group or period included in this study. Information on the different types of analysis performed for each individual sample is listed in Online Resource 1

Archaeological site Cultural group/period	GC-C- IRMS (absorbed lipids)	EA- IRMS (food crusts)	GC-MS (food crusts)	Microscopy/ SEM (food crusts)
Doel Deurganckdok site B				
<i>SWB</i>	10	29	3	-
Doel Deurganckdok site C				
<i>MK/SP</i>	5	4	-	-
Doel Deurganckdok site J				
<i>SWB</i>	5	5	2	5/-
Doel Deurganckdok site M				
<i>SWB</i>	10	2	3	-
Bazel Sluis				
<i>Late LBK</i>	2	-	-	-
<i>Limburg pottery</i>	3	-	-	-
<i>Early Neolithic</i>	5	-	-	-
<i>SWB</i>	5	18	7	55/2
<i>MK/SP</i>	14	4	2	-
Melsele Hof ten Damme				
<i>SWB</i>	3	-	-	-
<i>MK/SP</i>	2	-	-	-
Kortrijk Schaapsdreef				
<i>MK/SP</i>	5	2	-	-
Menen Kortewaagstraat				
<i>MK/SP</i>	2	-	-	-
Oudenaarde Donk Neo 1				
<i>MK/SP</i>	6	4	2	-
Totals	77	68	19	60/2

Rarely, decoration is found on the neck or neck-shoulder transition, consisting of one or two rows of fingertip impressions (Fig. 2c) or a row of perforations. Grog is the dominant temper (> 90%), followed by fine plant material and sand. Pottery from all five currently known SWB sites in the Scheldt river basin was included in this study (Table 1; Online Resource 1). It includes both fragments of closed beakers and of a few open bowls and buckets. However, in most cases it concerns body sherds for which the original vessel shape can no longer be reconstructed.

Finally, the Middle Neolithic MK/SP pottery from the Scheldt river basin is typically tempered with flint, often in combination with fine plant material or sometimes grog. The repertoire consists of a wide array of vessel shapes, including mostly bottles, bottle-shaped vessels or jars, and beakers, sometimes with slightly carinated profiles

(Fig. 2g-i) Decoration is again very limited. Based mainly on the morphological characteristics of the pottery, a number of regional, stylistic groups have been identified for the Middle Neolithic of the Scheldt river basin, including the Belgian Michelsberg culture and the Group of Spiere (Vanmontfort 2001, 2004; Bostyn et al. 2011). However, for many sites with highly fragmented pottery, the attribution to one or the other group is difficult. Hence, the general term MK/SP is used throughout this paper. MK/SP pottery from six sites is included in this study (Table 1; Online Resource 1). It includes both fragments of beakers and bottle-shaped vessels, but again, in many cases it concerns body sherds for which the vessel shape can no longer be reconstructed.

In total, this study includes the analysis of 204 samples (127 food crust samples and 77 potsherds) from nine archaeological sites, with a main focus on the SWB and MK/SP pottery from the Scheldt river basin (Online Resource 1). It is noted that we combine the results of different analyses performed at different moments in time. EA-IRMS analysis was previously performed and published for 44 food crust samples of SWB and MK/SP pottery from several sites in the Scheldt river basin (Craig 2004; Craig et al. 2007; Boudin et al. 2009, 2010; Teetaert et al. 2017). In 2019, additional microscopic, EA-IRMS and GC-MS analysis was undertaken for 83 food crust samples of SWB and MK/SP pottery from this region. Finally, in 2021, within the framework of PhD research at Ghent University (Vannoorenberghe 2022), 77 sherds of Early Neolithic, SWB and MK/SP pottery were selected for the analysis of absorbed lipids by GC-C-IRMS. Molecular (GC-MS) analysis was not performed for these samples. As a result, in most cases, one type of analysis was performed for each sample. However, for some of the food crust samples, different types of analysis were combined. Online Resource 1 provides a clear overview of the 204 samples from the different sites, and of the analyses performed for each of these samples. A summary, including the types of analysis and corresponding number of samples for each cultural group or period, is provided in Table 1.

Microscopic and SEM analysis of food crusts

Microscopic analysis was performed on 55 sherds of SWB pottery from Bazel Sluis and five sherds from Doel Deurganckdok site J with exceptional preservation of charred residues on the inner surfaces, with crusts up to 5 mm thick. Prior to sampling for GC-MS and EA-IRMS, these crusts were visually analysed using an Olympus SZX7 stereomicroscope at 8x – 56x magnification for determinable plant and animal remains. Images were captured with an Olympus SC100 camera. In addition, the residues on two body sherds of SWB pottery from Bazel Sluis were analysed by SEM. This analysis took place at a later stage of the research, after

the residues had already been detached from the potsherds. The samples of isolated charred organic matter were first examined using a Leica microscope with incident light at 6x – 50x magnification. For the SEM analysis, eight fragments of crust from one sherd and six fragments of crust from the other sherd were selected, to ensure maximum coverage and account for potential variations in the composition of the crusts. Selected portions of the residues were mounted on SEM stubs using carbon cement. The samples were sputter-coated with a thin layer (ca. 20 µm) of platinum-palladium to achieve improved contrast during SEM photography. SEM analysis was carried out at the Naturalis Biodiversity Center in Leiden, the Netherlands, using a JEOL JSM-6480 L scanning electron microscope. Images were captured at x25 – x650 magnification.

EA-IRMS analysis of food crusts

Sixty-eight food crust samples were analysed by EA-IRMS for their stable isotope measurements ($\delta^{13}\text{C}$, $\delta^{15}\text{N}$, atomic C: N) (Online Resource 2). Only residues from the inner vessel surfaces were sampled. A proportion of the stable isotope data included in this study ($n=44$) were previously published by Craig (2004), Craig et al. (2007), Boudin et al. (2009, 2010) and Teetaert et al. (2017) (Online Resource 2). For those samples, we refer to the methods section of the respective publications. For the 24 samples analysed in this study, EA-IRMS analysis was performed at the Department of Earth and Environment Sciences of KU Leuven. All residues were pre-treated with the acid-alkali-acid (AAA) method (De Vries and Barendsen 1954; Taylor 1997). $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ analyses were performed in duplicate on a Thermo Flash EA/HT elemental analyser, coupled to a Thermo V Advantage IRMS through a ConFlo IV Interface (Thermo Fischer Scientific, Bremen, Germany). Normalisation was undertaken using IAEA-N1 (accepted $\delta^{15}\text{N}=+0.43\pm0.07\%$ vs. AIR), IAEA-C6 (accepted $\delta^{13}\text{C}=-10.80\pm0.47\%$ vs. V-PDB) and acetanilide as standard of quantification and as internal quality assurance. Analytical precision was 0.25‰ for both $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$, based on multiple measurements of the standard acetanilide. EA-IRMS provides information on the total carbon and nitrogen isotopic compositions of the residues, which can be useful in broadly discriminating between aquatic (marine and freshwater) and terrestrial animal/plant products (Craig et al. 2007; Philippsen and Meadows 2014; Heron and Craig 2015). However, as the $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ values of the food residue may have been altered by the cooking process, by degradation or diagenetic change in the burial environment, they should be treated with caution and are best interpreted in relation to evidence from more specific chemical and molecular analyses (Craig et al. 2007, 2020). In this study,

the bulk isotope values of the food crusts are discussed against the results of GC-MS analyses of food crusts and GC-C-IRMS analyses of absorbed lipids originating from the same types of pottery.

GC-MS analysis of food crusts

GC-MS analysis of 19 food crust samples (Online Resource 3) was performed at the Department of Organic and Macromolecular Chemistry of Ghent University. Sample sizes varied between 5 mg and 169 mg, with an average of 130 mg and a median of 102 mg across the entire sample set. All samples were crushed into a vial, to which 10 mL of dichloromethane/methanol (1:1) was added to dissolve the organic compounds. The samples were put in an ultrasonic bath followed by centrifugation (2000 rpm for 10 min), prior to recovery of the liquid fraction. This procedure was repeated once and the liquid fraction was combined. The DCM/MeOH was evaporated after which the compounds were re-dissolved in hexane, filtered and transferred to smaller vials. Heptadecane (1 ppm) was added as an internal standard. Derivatisation was achieved using BSTFA with 1% TMCS. All samples (with the internal standard included) were dissolved in 1 or 0.1 mL of hexane in GC vials. In both cases, the internal standard was always kept at 1 ppm of the total volume. A representative sample chromatogram is shown in Online Resource 3. All analyses were conducted on an Agilent 7890a GC system coupled to an Agilent 5975c MSD. The GC was equipped with an Agilent HP-5MS (30 m x 0.25 mm ID, 0.25 µm). One microliter of sample was injected through a split/splitless injector in splitless mode. The start temperature of the oven was set at 40 °C followed by a 10 °C/min temperature ramp up to 150 °C, followed without delay by a further increase at 4 °C/min up to 320 °C. The latter temperature was held for 20 min prior to the cooling cycle. The column flow was either 1 mL of He per minute or alternatively 1.2 mL with hydrogen as the carrier gas.

GC-C-IRMS analysis of absorbed lipids

A total of 77 sherds were selected for compound-specific $\delta^{13}\text{C}$ analysis of palmitic ($\text{C}_{16:0}$) and stearic ($\text{C}_{18:0}$) acids absorbed into the ceramic matrix by GC-C-IRMS (Online Resource 4). GC-C-IRMS analysis was performed at the Isotope bioscience laboratory (ISOFS) of the Department of Green Chemistry and Technology at Ghent University. The complete lipid extraction and analysis parameters as well as the obtained precision and accuracy are to be found in Online Resource 5. Briefly, a small part of each potsherd was broken off and the pottery surface was removed using a power tool with miniature tungsten carbide tip (Dremel)

to remove superficial contaminants, after which the sample was pulverized by hand using an agate mortar and pestle. The absorbed palmitic and stearic fatty acids were extracted from about 2 g of powdered ceramic and directly methylated in a procedure modified from established one-step acidified methanol protocols (Correa-Ascencio and Evershed 2014; Papakosta et al. 2015). The fraction obtained was further purified over 10% AgNO₃-impregnated silica gel following a procedure adopted from Papakosta et al. (2019). Where necessary, as observed after a preliminary measurement, elemental sulfur was removed from the samples by adding about 200 mg of high-purity activated copper turnings to a 0.5 mL hexane solution of the extract and letting it react for 24 h at room temperature. After preliminary assessment using GC-FID (Trace GC ultra, Interscience), the solvent (*n*-hexane) volume and injection volume were adapted to ensure an injection of ca. 40 µg of the C_{16:0} and C_{18:0} fatty acid methyl esters (FAMES) on the GC-C-IRMS (Trace-GC 1310 coupled to a GC-ISOLINK II coupled to Delta V Advantage through a ConFlo IV interface, all Thermo Fischer Scientific, Bremen, Germany). Injection was done on a programmable temperature vaporizer inlet kept at 50 °C during inception after which it was heated to 220 °C and kept in splitless mode for 0.75 min. Chromatographic separation was done on a (5%-Phenyl)-methylpolysiloxane GC column (DB-5MS, 30 m x 0.25 mm x 0.5 µm, Agilent) kept at 50 °C for 1.5 min, heated to 200 °C at 25 °C·min⁻¹ after which the heating rate was reduced to 3 °C·min⁻¹ to 245 °C. Finally, the column was baked out for 4 min at 310 °C. A representative sample chromatogram is shown in Online Resource 3. All samples were injected at least in duplicate, deviations in δ¹³C were always smaller than 0.2‰. Normalization on the Vienna Pee Dee Belemnite (VPDB)-LSVEC scale was done using the F8-4 FAME and FAEE mix of Arndt Schimmelmann. Quality assurance (QA) was done using a mix of 3 FA (C16, C18 and C19) derivatised together with every sample batch. Reproducibility of the QA mix was always better than 0.2‰. To identify the origin of the extracted fatty acids, the δ¹³C values of the C_{16:0} and C_{18:0} FAMES obtained from the samples were compared to those of modern authentic reference animals (after correction for the Suess Effect). A dataset of δ¹³C values for modern authentic reference animals fed with a paleo diet, as published by Courel et al. (2020), was used as dietary endmember signature. This reference data has been broadly grouped into five potential food sources (freshwater, marine, porcine, ruminant adipose, ruminant dairy). Furthermore, the difference in δ¹³C values between stearic and palmitic fatty acid (Δ¹³C) is considered a robust criterion for separation between non-ruminant adipose fats (Δ¹³C > ca. -2‰), ruminant adipose fats (Δ¹³C between ca. -1 and -5‰)

and ruminant dairy fats (Δ¹³C < -3.1‰) (Copley et al. 2003; Craig et al. 2012; Cramp and Evershed 2014).

Results and interpretations

Early Neolithic (LBK and Limburg) pottery

GC-C-IRMS analysis of absorbed lipids

Absorbed lipids from 10 sherds of Early Neolithic pottery from the site of Bazel Sluis were analysed by GC-C-IRMS (Online Resource 4). It includes two sherds of late LBK pottery which, based on their fabric and decoration, could belong to the same vessel, and three sherds of Limburg pottery, all from different vessels. Another five sherds belong to five vessels that cannot be assigned to a specific cultural group or pottery group, but can be assigned to the Early Neolithic period based on distinct typo-technological characteristics, e.g. the use of bone temper (Crombé et al. 2015a; Teetaert 2020). They either represent undecorated parts of Limburg, LBK or BVSG pottery.

All samples yielded enough palmitic and stearic FAMES for ¹³C/¹²C ratio determination. The δ¹³C values obtained for C_{16:0} and C_{18:0} for each sample are listed in Online Resource 4. The first LBK sample plots within the reference ranges of both freshwater organisms and ruminant adipose fats (Fig. 3), but close to the ranges of porcine and marine fats. It has a Δ¹³C value (difference between δ¹³C_{18:0} and δ¹³C_{16:0}) of -0.8‰. Typically, Δ¹³C values > -1‰ are interpreted as an indication of the presence of non-ruminant fats (Copley et al. 2003; Evershed et al. 2008a). The slightly elevated Δ¹³C value for this LBK vessel could point to the processing of aquatic resources. However, the mixing of different food sources, including terrestrial non-ruminant (e.g. porcine) fats, could also explain these isotopic values. The second LBK sample plots within the ranges of ruminant adipose and dairy fats and has a Δ¹³C value of -2.8‰. Δ¹³C values between -1 and -3.1‰ are widely regarded as indicative of ruminant adipose fats (Copley et al. 2003; Evershed et al. 2008a, 2022; Craig et al. 2012) and it is likely that ruminant meat was processed in this vessel. Despite strong similarities in fabric and decoration, the divergent isotopic values for both LBK samples indicate that they represent two different vessels.

The three Limburg samples show divergent values as well. A first sample plots within the ranges of both freshwater organisms and ruminant adipose fats, but close to the ranges of porcine and marine fats. It has a Δ¹³C value of -0.6‰, which could be explained by the processing of aquatic resources but also by the mixing of different food sources. The second sample plots within the ranges of

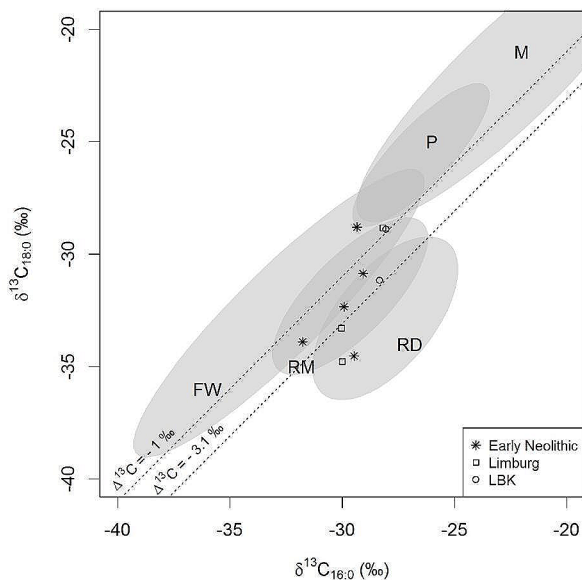


Fig. 3 $\delta^{13}\text{C}$ values of $\text{C}_{16:0}$ and $\text{C}_{18:0}$ fatty acids from the Early Neolithic pottery included in this study. 95% confidence ellipses indicate the range of $\delta^{13}\text{C}_{16:0}$ and $\delta^{13}\text{C}_{18:0}$ values from modern authentic reference animals, corrected for the Suess Effect (Courel et al. 2020). M=marine, FW=freshwater, P=porcine, RM=ruminant adipose, RD=ruminant dairy. The diagonal lines represent $\Delta^{13}\text{C}$ values of -1 and -3.1‰

ruminant adipose and dairy fats and has a $\Delta^{13}\text{C}$ value of -3.3‰. In recent studies, animal fats extracted from pottery are considered to contain dairy lipids if their $\Delta^{13}\text{C}$ value is below the threshold of -3.1‰ (Evershed et al. 2022; Lucquin et al. 2023). However, Craig et al. (2012) showed that wild ruminant adipose or carcass fats may yield a wide range of $\Delta^{13}\text{C}$ values between -2.7 and -4.3‰. Therefore, this Limburg vessel likely contained ruminant dairy, yet the presence of wild ruminant carcass fats cannot be excluded. The third sample of Limburg pottery, on the other hand, has a $\Delta^{13}\text{C}$ value of -4.8‰, leaving no doubt about the presence of ruminant dairy in this vessel.

Finally, three samples of the non-specified Early Neolithic pottery plot within the ranges of freshwater organisms and ruminant adipose fats and have a $\Delta^{13}\text{C}$ value between -1 and -3.1‰ (Online Resource 4). It is likely that at least some ruminant meat was processed in these vessels. Another sample plots within the range of ruminant dairy fats. With a $\Delta^{13}\text{C}$ value of -5.1‰, it is clear that this vessel contained dairy products. The fifth sample of Early Neolithic pottery ($\Delta^{13}\text{C}$ value = 0.6‰) plots within the freshwater range, but close to the ranges of porcine, marine and ruminant fats. Again, the isotopic values could reflect a mixture of different food sources.

To conclude, the isotopic values of several of these Early Neolithic vessels could reflect mixtures of food sources,

including aquatic (freshwater), ruminant and terrestrial non-ruminant fats, possibly as a result of subsequent cooking events. Without molecular data to support the isotopic data, we cannot provide more information about the lipid sources in these vessels. It is however clear that ruminant meat was processed in at least one late LBK vessel and three vessels of the non-specified Early Neolithic pottery. In addition, one or two Limburg vessels and one other Early Neolithic vessel contained ruminant dairy products.

Swifterbant culture pottery

Pottery use among the SWB populations of the Scheldt river basin was studied by both the analysis of food crusts and absorbed lipids, providing complementary information about the materials processed. While food crusts mainly inform us about the final content(s) of a vessel, absorbed residues represent an accumulation of cooking events and a mix of ingredients related to the vessel's entire period of use (Miller et al. 2020). It is however stressed that the analyses of food crusts on one hand and of absorbed lipids on the other hand were not performed on the same potsherds, but on different sherds from the same sites and archaeological contexts, i.e. representing the same occupation events.

Microscopic and SEM analysis of food crusts

Microscopic analysis was performed on 55 sherds of SWB pottery from Bazel Sluis and five sherds from Doel Deurganckdok site J with exceptional preservation of charred residues on the inner surfaces, with crusts up to 5 mm thick. These residues were examined using a stereomicroscope and, in seven cases, this was combined with GC-MS and EA-IRMS analysis (Online Resource 3). Microscopic analysis allowed the identification of fish remains (fin rays, other fish bones, and scales) in the residues of nine sherds from Bazel Sluis and one sherd from Doel site J (Fig. 4a-d). This includes two samples from Bazel Sluis for which aquatic biomarkers were detected by GC-MS (cf. infra). Plant remains were observed in the residues of five sherds from Bazel Sluis (Fig. 4e-f), in one case together with fish remains. Although the plant species could not be identified based on the stereomicroscopic images, SEM analysis of two of these food crust samples confirmed these observations and provided information on the origin of some of the plant material. Again, numerous fish remains were observed in both residue samples (Fig. 5a-b). In addition, both residues contained multiple fragments of herbaceous stems or leaves (Fig. 5c-h). Where the epidermal surface remained intact, it showed a distinctive configuration of elongated, rectangular cells with blunt or tapering ends (Fig. 5d, f). This particular pattern of epidermal cells is diagnostic of

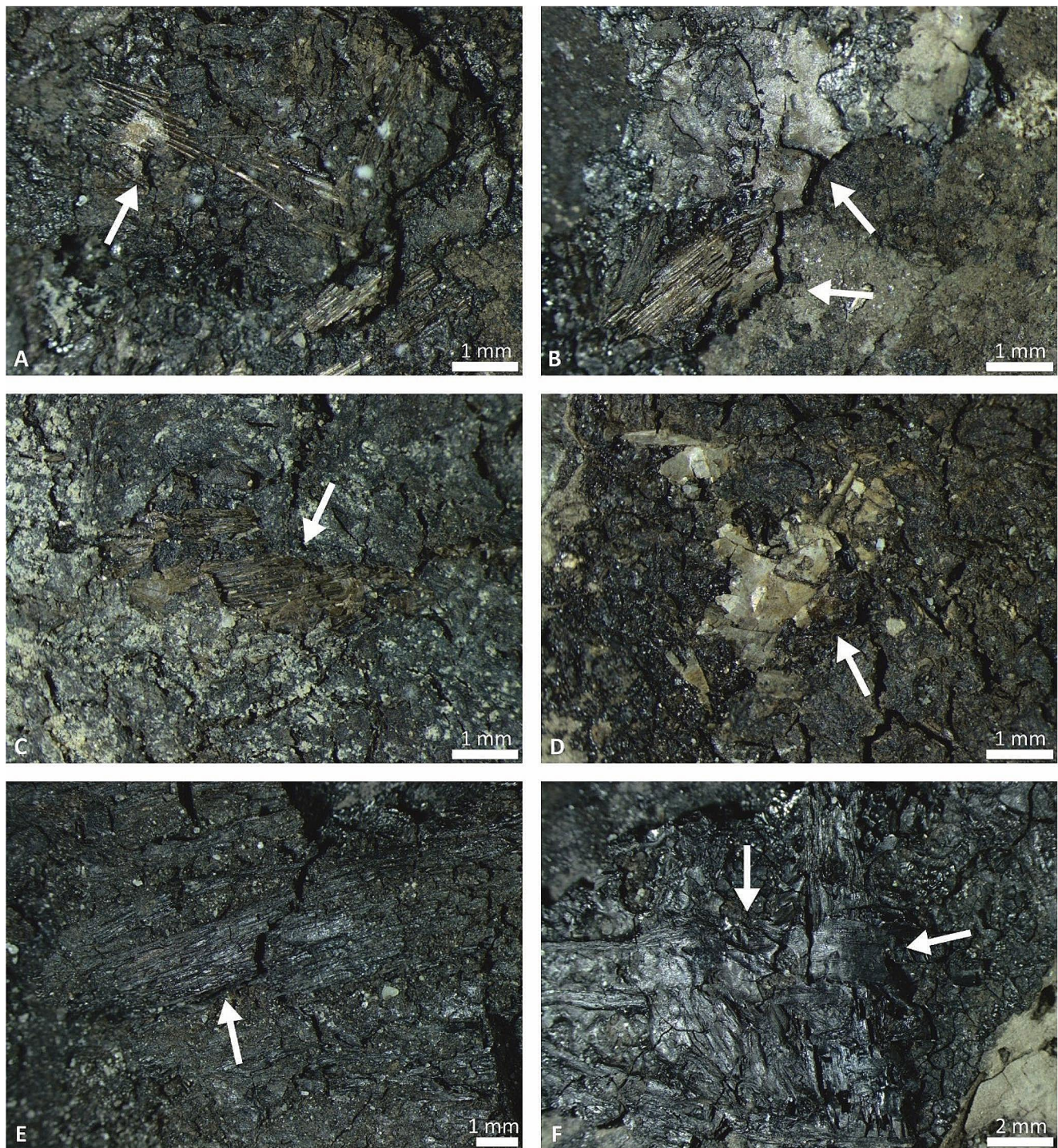


Fig. 4 Stereomicroscope images of fish (a–d) and plant remains (e–f) embedded in the residue of SWB pottery from the site of Bazel Sluis, northern Belgium (images by D. Teetaert, Ghent University)

Allium species and, in this case, must be derived from one of the wild onions, leek, chives or garlic species. The presence of a significant amount of parenchyma cells accompanied by vascular tissue, noted in some of the herbaceous stem fragments (Fig. 5g–h), suggests that these are fleshy or succulent stems or leaves resembling those in wild garlic

(*Allium vineale*), wild chives (*Allium schoenoprasum*) or sand leek (*Allium scorodoprasum*).

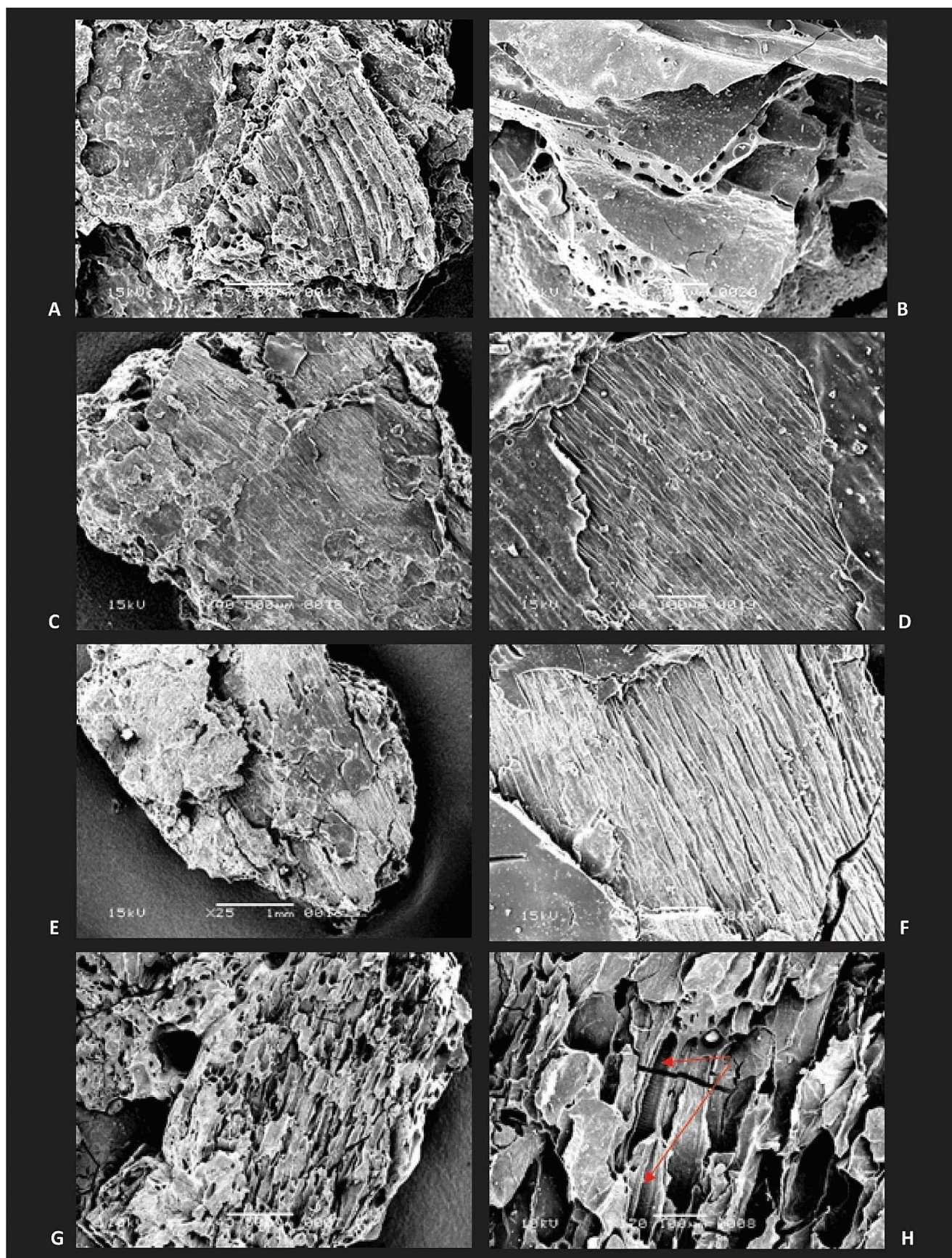


Fig. 5 SEM images of fish (a–b) and plant remains (c–h) embedded in a residue of SWB pottery from the site of Bazel Sluis, northern Belgium. These remains include: fish scale (a) and fish bone (b); herbaceous plant fragments (c, e), with epidermis showing long cells with blunt or tapering ends (d, f); herbaceous stem fragments with parenchymatous tissue (g), and, marked by red arrows, details of xylem elements of vascular tissue surrounded by parenchyma cells (h) (images by L. Kubiak-Martens, BIAx).

GC-MS analysis of food crusts

Food crusts of 15 sherds of SWB pottery from Bazel Sluis and Doel sites B, J and M were analysed by GC-MS. An overview of the biomarkers identified for each sample and an example chromatogram are provided in Online Resource 3. Cholesterol and its derivatives (cholesta-3,5-dien-7-one and cholesta-4,6-dien-3-one) were detected in nine samples, indicating the presence of degraded animal fats (Evershed 1993; Heron et al. 2016; Oras et al. 2017). Eight samples from the four different sites contained both ω -(*o*-alkylphenyl)alkanoic acids (APAAs), with 18 to 20 carbon atoms, and isoprenoid fatty acids, including TMTD (4,8,12-trimethyltridecanoic acid), phytanic acid (3,7,11,15-tetramethylhexadecanoic acid) and, in some cases, pristanic acid (2,6,10,14-tetramethylpentadecanoic acid). These compounds, noted in various studies in vessels used to process freshwater and marine resources, are considered strong indicators for the processing of aquatic lipids when found together (Hansel et al. 2004; Craig et al. 2007; Evershed et al. 2008b; Cramp and Evershed 2014; Farrell et al. 2014; Heron and Craig 2015; Hart et al. 2018). In two of these samples from the site of Bazel Sluis, fish remains were visually identified in the food crusts using a stereomicroscope (Online Resource 3). In addition, five of the eight samples containing APAAs and isoprenoid fatty acids also contain traces of the mono-unsaturated fatty acids $C_{17:1}$ and $C_{19:1}$. The co-occurrence of the latter compounds has been linked to certain species of fish (e.g. carp, catfish) and sea food (e.g. shrimp, crab, sponges) (Baeten et al. 2013; and the references cited therein). Based on these results, it seems that aquatic resources were processed in several of these SWB vessels. However, C_{18} APAAs can also be formed through the heating of terrestrial adipose fats (Heron and Evershed 1993; Evershed et al. 2008b; Bondetti et al. 2021), while phytanic and pristanic acids are found in both aquatic and ruminant animals (Cramp and Evershed 2014; Heron and Craig 2015). As such, the studied residues could contain mixtures of different products.

Plant lipids are often difficult to detect in pottery, especially when mixed with animal fats (Steele et al. 2010; Craig et al. 2020). Yet various samples here at hand show multiple biomarkers that can be related to plant components. Among the common biomarkers for plant lipids in our samples are long-chain *n*-alkanes and alkanols (Dunne et

al. 2016; Poulain et al. 2016; Whelton et al. 2018), which were observed in three samples from Bazel Sluis and Doel sites B and J. Other well-known plant degradation products are short-chain fatty acids and diacids, such as azelaic acid (Dunne et al. 2016). Azelaic acid was identified in three samples from Doel sites B and M, while short-chain fatty acids ($C_{8:0}$ to $C_{14:0}$) were identified in almost all of the samples (Online Resource 3). Short-chain fatty acids with a carbon-count lower than 8 were detected in several samples. In addition, β -sitosterol, one of the major plant sterols (Evershed 1993; Heron et al. 2010), was identified in two samples from Doel sites J and M, and has previously been identified in two other food crust samples from Doel site J (Craig 2004). Nonacosane was detected in one sample from Bazel Sluis. This long-chain aliphatic compound is an important constituent of epicuticular leaf waxes of higher plants, e.g. *Brassicaceae* (cabbage family) (Evershed et al. 1991; Dunne et al. 2019). Finally, several samples included aldehydes, such as syringaldehyde and *p*-hydroxybenzaldehyde, which are lignin-derived compounds related to plant biomass degradation (Simoneit et al. 1993; Poulain et al. 2016). We note that five vessels from the four sites that have multiple plant biomarkers also yielded aquatic biomarkers (Online Resource 3). Together with the results of the microscopic analysis, this reinforces the idea that mixtures of different food sources, in particular fish and plant components, were processed together in several of the SWB vessels.

EA-IRMS analysis of food crusts

Bulk carbon and nitrogen isotope values were measured for 54 food crust samples of SWB pottery from Bazel Sluis and Doel sites B, J and M. The obtained values are listed in Online Resource 2. Most samples ($n=47$) have lower $\delta^{13}C$ values (-25.8 to -30.5‰) and $\delta^{15}N$ values between +5 and +13‰, with most $\delta^{15}N$ values between +6 and +10‰ (Fig. 6A). This is consistent with the values for freshwater resources mentioned in several studies (e.g. Craig et al. 2007; Philippsen and Meadows 2014; Lucquin et al. 2016; Piezonka et al. 2016; Kunikita et al. 2017). Eight of these samples were included in the GC-MS analysis and aquatic biomarkers were identified in four of them. Moreover, in three of these samples from Bazel Sluis, fish remains were observed using a stereomicroscope (Online Resource 3). Nevertheless, mixing of terrestrial animal or plant products cannot be ruled out or accurately quantified using this approach (Lucquin et al. 2016). The remaining samples ($n=7$) have relatively lower $\delta^{13}C$ values (-26.3 to -28.6‰) and low $\delta^{15}N$ values (< +5‰). Residues depleted in ^{15}N are more likely to derive from terrestrial plant or animal sources than from aquatic sources (Craig et al. 2007; Lucquin et al. 2016). Terrestrial animal products are probably

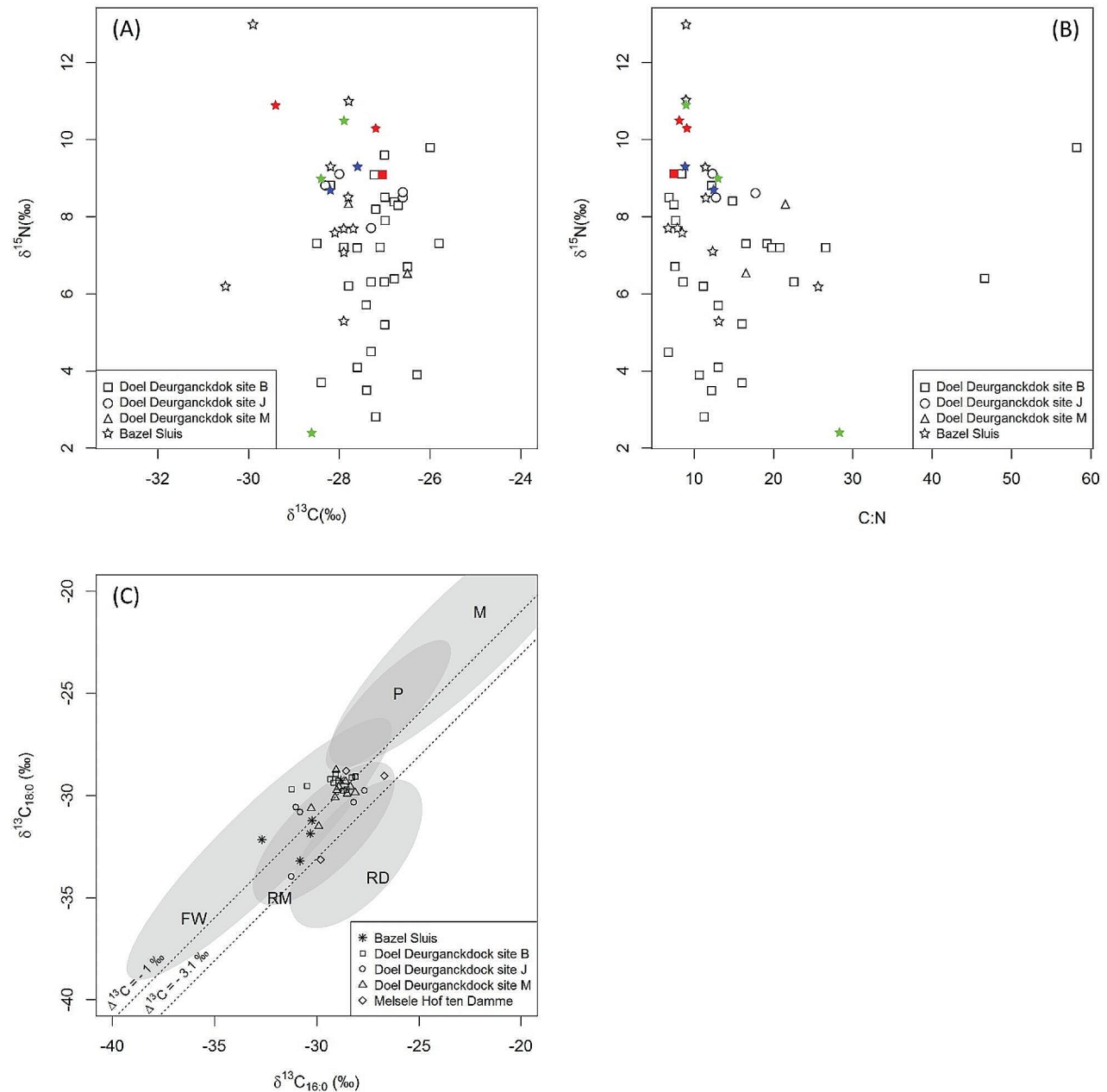


Fig. 6 (A) $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ bulk stable isotope values obtained from 54 food crust samples of SWB pottery from four sites in the Scheldt river basin, northern Belgium, and (B) $\delta^{15}\text{N}$ bulk stable isotope values against C: N ratio from the same food crust samples (the C: N ratio is missing for five samples). Coloured symbols refer to samples for which aquatic (blue), plant (green) or aquatic and plant components (red) were detected by GC-MS and microscopic analysis (Online Resource 3). (C) $\delta^{13}\text{C}$ values of $\text{C}_{16:0}$ and $\text{C}_{18:0}$ fatty acids extracted

from 33 samples of SWB pottery from the same sites, with addition of the site of Melsele Hof ten Damme. 95% confidence ellipses indicate the range of $\delta^{13}\text{C}_{16:0}$ and $\delta^{13}\text{C}_{18:0}$ values from modern authentic reference animals corrected for the Suess Effect (Courel et al. 2020). M=marine, FW=freshwater, P=porcine, RM=ruminant adipose, RD=ruminant dairy. The diagonal lines represent $\Delta^{13}\text{C}$ values of -1 and -3.1‰

the main component of six of these residues, all from Doel site B, based on their relatively low atomic C: N ratios. Only one sample from Bazel Sluis combines a low $\delta^{15}\text{N}$ value (+2.4‰) with a relatively high C: N ratio (28.3) (Fig. 6B),

indicating a likely contribution of terrestrial plant products to this residue (Yoshida et al. 2013; Lucquin et al. 2016, 2023; Oras et al. 2017; Robson et al. 2019; Bondetti et al. 2020). This sample was included in the GC-MS analysis

and contained short-chain fatty acids, which possibly derive from plant material. It is however noted that the $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ values of bulk organic matter should be interpreted with caution, as they may have been altered by the cooking process, by degradation or diagenetic change in the burial environment (Craig et al. 2007; Heron and Craig 2015). For instance, two samples from Doel site B have high C:N ratios (46.6 and 58.2) combined with relatively high $\delta^{15}\text{N}$ values (6.4 and 9.8‰), which might indicate microbial degradation in the soil (e.g. Soldatova et al. 2024).

GC-C-IRMS analysis of absorbed lipids

Absorbed lipids from 33 sherds of SWB pottery were analysed by GC-C-IRMS. The sherds were selected from all five currently known SWB sites in the Scheldt river basin: Doel Deurganckdok sites B, J and M, Bazel Sluis and Melsele Hof ten Damme. All samples yielded sufficient palmitic and stearic FAMES for $^{13}\text{C}/^{12}\text{C}$ ratio determination. The $\delta^{13}\text{C}$ values obtained from the $\text{C}_{16:0}$ and $\text{C}_{18:0}$ FAMES for each sample are listed in Online Resource 4.

Three samples from Bazel Sluis and Doel site B, with $\Delta^{13}\text{C}$ values above -1‰, plot within the freshwater range and outside the ranges of ruminant and terrestrial non-ruminant adipose fats (Fig. 6C). It is very likely that freshwater fish was processed in these vessels. Another three samples from Doel sites J and M, with $\Delta^{13}\text{C}$ values above -1‰, plot within the overlapping ranges of freshwater and ruminant adipose fats. In our view, freshwater fish is also a likely component of these residues, possibly mixed with other resources.

Three more samples from Bazel and two from Doel sites J and M plot within the ranges of freshwater and ruminant adipose fats. With $\Delta^{13}\text{C}$ values between -1 and -3.1‰, it is likely that ruminant meat was processed in these vessels (Copley et al. 2003; Evershed et al. 2008a, 2022; Craig et al. 2012), although mixing with other, e.g. aquatic resources cannot be excluded. As mentioned by Cramp et al. (2019), the relatively low content of $\text{C}_{18:0}$ in aquatic fats makes that they are readily masked isotopically by mixing with terrestrial animal fats and could therefore plot within the ruminant ranges. Further, only one SWB vessel from Melsele has a $\Delta^{13}\text{C}$ value of -3.3‰ and probably contained ruminant dairy (Evershed et al. 2022; Lucquin et al. 2023), although wild ruminant carcass fats may yield similar isotopic values (Craig et al. 2012).

Most SWB samples however cluster at the intersection of the reference ranges of freshwater, ruminant and terrestrial non-ruminant fats. Their isotopic values can reflect a wide range of mixtures of different food sources. Without molecular data, this is difficult to determine. The $\Delta^{13}\text{C}$ values do provide some indications of possible lipid sources in

these vessels. Half of the samples ($n = 11$) have $\Delta^{13}\text{C}$ values above -1‰, which is more indicative of aquatic and terrestrial non-ruminant fats. Based on the results of the food crust analyses of SWB pottery from the same sites and contexts (cf. supra), it seems plausible that freshwater fish was processed in several of these vessels. Of course, this does not exclude the presence of porcine fats. The other samples ($n = 10$) have $\Delta^{13}\text{C}$ values between -1 and -3.1‰, which indicate that ruminant adipose fats were present in these vessels, possibly mixed with other animal fats.

To conclude, GC-C-IRMS analysis of absorbed lipids indicates that freshwater and ruminant adipose fats were processed in several SWB vessels from Bazel Sluis and the Doel sites. Only one vessel from Melsele likely contained ruminant dairy. However, most of the vessels analysed might have contained mixtures of freshwater, ruminant or terrestrial non-ruminant fats, likely as a result of successive cooking events. We lack molecular data to provide more detailed information about the lipid sources in these vessels. Additional information is available from the analyses of food crusts of pottery from the same sites and archaeological contexts (with exception of the site of Melsele Hof ten Damme for which food crusts are absent), offering insight into the final meal(s) prepared in these vessels. GC-MS and EA-IRMS analysis mainly indicate the processing of aquatic, in particular freshwater resources in SWB pottery from Bazel Sluis and the Doel sites. This is corroborated by the observation of fish bones and scales in food crusts from Bazel Sluis and Doel site J by stereomicroscopic and SEM analysis. The co-occurrence of plant and aquatic biomarkers in five food crusts from the four sites indicates that fish and plant components were sometimes processed together. This is also evidenced by the observation of fish remains and fragments of herbaceous stems or leaves of *Allium* species in two food crust samples from Bazel Sluis. Finally, the processing of terrestrial animal fats in a portion of the SWB pottery is not to be excluded based on the GC-MS and EA-IRMS results.

Michelsberg culture/Group of Spiere pottery

For the Middle Neolithic pottery from the Scheldt river basin, information on pottery use was obtained from the analysis of food crusts and absorbed lipids. As for the SWB pottery, the analyses of food crusts on one hand and of absorbed lipids on the other hand were performed on different potsherds coming from the same sites and archaeological contexts.

GC-MS analysis of food crusts

GC-MS analysis was undertaken on two food crust samples of MK/SP pottery from Bazel Sluis and two from Oudenaarde Donk Neo 1 (Online Resource 3). Cholesterol was detected in all samples. Both samples from Oudenaarde contain APAAs, and in one sample these APAAs are detected together with isoprenoid fatty acids (TMTD, phytanic acid) and the mono-unsaturated fatty acids $C_{17:1}$ and $C_{19:1}$, showing that aquatic resources were likely processed in these vessels. This is in line with the results of previous analyses of two other food crust samples of MK/SP pottery from Oudenaarde Donk, which yielded clear indications for the processing of aquatic resources, most likely freshwater fish (Craig 2004; Craig et al. 2007). However, while no plant-related compounds were identified in our study, Craig (2004) also found β -sitosterol in both their samples, indicating that the final meal(s) prepared in these vessels at least involved both aquatic (freshwater) and plant products. One of the samples from Bazel Sluis also has aquatic biomarkers in the form of APAAs, isoprenoid fatty acids (TMTD, phytanic acid, and possibly pristanic acid) and the mono-unsaturated fatty acids $C_{17:1}$ and $C_{19:1}$. In addition, long-chain *n*-alkanes indicate a plant contribution to this residue (Online Resource 3). The second sample from Bazel did not yield any clear biomarkers other than cholesterol.

EA-IRMS analysis of food crusts

Bulk carbon and nitrogen isotope values were measured for 14 food crust samples of MK/SP pottery from Doel site C, Bazel Sluis, Oudenaarde Donk Neo 1 and Kortrijk Schaapsdreef. The obtained values are listed in Online Resource 2. Eight samples have lower $\delta^{13}C$ values (-26.1 to -27.9‰) and higher $\delta^{15}N$ values (+5.8 to +12.2‰), indicating the likely presence of freshwater fats in these residues (Craig et al. 2007; Philippsen and Meadows 2014; Lucquin et al. 2016; Piezonka et al. 2016; Kunikita et al. 2017). This includes all four samples from Oudenaarde, three of four samples from Bazel, and one of two samples from Kortrijk (Fig. 7A). One sample from Doel site C has a $\delta^{13}C$ value of -24.8‰ and a $\delta^{15}N$ value of +11.2‰. Similar values have been noted for pottery originating from coastal sites, with evidence for the processing of marine organisms (Craig et al. 2007, 2011; Taché and Craig 2015).

Finally, three samples from Doel site C, one from Bazel and one from Kortrijk have relatively lower $\delta^{13}C$ values (-26.3 to -32.5‰) in combination with low $\delta^{15}N$ values (+2.3 to +5‰), which is in line with the expected values for terrestrial animal or plant resources (Craig et al. 2007; Lucquin et al. 2016). In each of these cases, the low $\delta^{15}N$ value is combined with a relatively high atomic C: N ratio

(values between 25.4 and 37.5) (Fig. 7B), indicating a likely contribution of terrestrial plant products to these residues (Yoshida et al. 2013; Lucquin et al. 2016; Oras et al. 2017; Robson et al. 2019).

GC-C-IRMS analysis of absorbed lipids

Absorbed lipids from 34 sherds of Middle Neolithic MK/SP pottery were analysed by GC-C-IRMS. The sherds were selected from six sites: Doel Deurganckdok site C, Bazel Sluis and Melsele Hof ten Damme, located in the lower Scheldt river basin, and Oudenaarde Donk Neo 1, Kortrijk Schaapsdreef and Menen Kortewagstraat, located in the upper Scheldt river basin (Fig. 1). All samples yielded sufficient palmitic and stearic FAMES for $^{13}C/^{12}C$ ratio determination. The $\delta^{13}C$ values obtained from the $C_{16:0}$ and $C_{18:0}$ FAMES for each sample are listed in Online Resource 4.

Most samples ($n=20$) plot within the freshwater range (Fig. 7C) and have a $\Delta^{13}C$ value above -1‰. Four of these, from Bazel Sluis, plot outside the ranges of terrestrial animal fats, indicating that these vessels were probably mainly used to process freshwater fish. Another 16 samples plot within the overlapping ranges of freshwater and ruminant adipose fats and, in several cases, close to the ranges of marine and porcine fats. Without molecular data, it is again difficult to determine the lipid sources in these vessels. They may have been used to process a variety of resources, including aquatic organisms, ruminant and terrestrial non-ruminant meat. Further, two samples from Bazel and one from Melsele plot within the overlapping ranges of freshwater, marine and porcine fats. This shows that marine and/or porcine food could have been processed in a portion of the MK/SP pottery. This is further corroborated by one vessel from Doel site C, for which the isotopic values plot entirely within the range of marine and porcine fats.

About a third of the samples ($n=10$) have a $\Delta^{13}C$ value between -1 and -3.1‰, indicating the presence of ruminant adipose fats. However, many of these plot within the overlapping ranges of freshwater and ruminant fats. Given the bias against aquatic fats when mixed with terrestrial animal fats (Cramp et al. 2019), it is certainly possible that some of these vessels contained freshwater resources. Mixing with terrestrial non-ruminant fats is also not excluded for these samples. It is noted that, with the exception of two vessels from Bazel Sluis, all vessels with clear indications of ruminant adipose fats originate from the upper Scheldt river sites at Oudenaarde ($n=4$), Kortrijk ($n=3$) and Menen ($n=1$).

Finally, three vessels from the sites of Bazel, Kortrijk and Oudenaarde plot within the range of ruminant dairy fats and have $\Delta^{13}C$ values between -4.4 and -4.9‰, which meets the widely accepted criteria for ruminant dairy (Craig et al. 2012; Evershed et al. 2022; Lucquin et al. 2023).

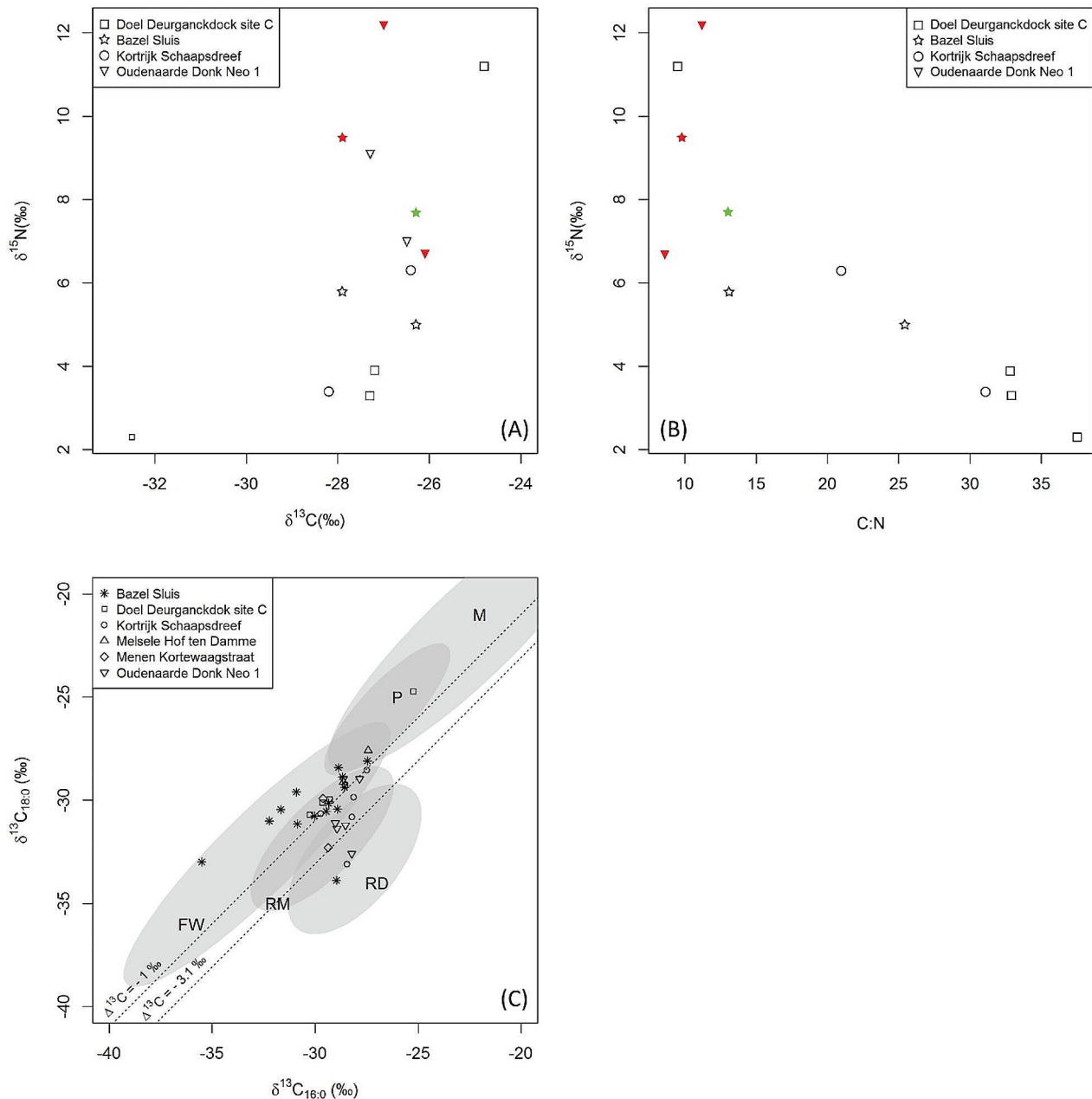


Fig. 7 (A) $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ bulk stable isotope values obtained from 14 food crust samples of MK/SP pottery from four sites in the Scheldt river basin, northern Belgium, and (B) $\delta^{15}\text{N}$ bulk stable isotope values against C: N ratio from the same food crust samples (the C: N ratio is missing for two samples). Coloured symbols refer to samples for which plant (green) or aquatic and plant components (red) were detected by GC-MS and microscopic analysis (Online Resource 3). (C) $\delta^{13}\text{C}$ values of $\text{C}_{16:0}$ and $\text{C}_{18:0}$ fatty acids extracted from 34 samples

To conclude, GC-C-IRMS analysis of absorbed lipids indicates that several of the studied MK/SP vessels were used to process freshwater and ruminant products. It is hereby noted that ruminant adipose fats are mainly observed

of MK/SP pottery from the same sites, with addition of the sites of Melsele Hof ten Damme and Menen Kortewagstraat. 95% confidence ellipses indicate the range of $\delta^{13}\text{C}_{16:0}$ and $\delta^{13}\text{C}_{18:0}$ values from modern authentic reference animals corrected for the Suess Effect (Courel et al. 2020). M = marine, FW = freshwater, P = porcine, RM = ruminant adipose, RD = ruminant dairy. The diagonal lines represent $\Delta^{13}\text{C}$ values of -1 and -3.1‰

in pottery from the upper Scheldt river sites. Contrary, the samples plotting within the freshwater range and yielding $\Delta^{13}\text{C}$ values above -1‰ are largely originating from the lower Scheldt river sites. Only three vessels from Bazel,

Oudenaarde and Kortrijk yield clear indications for ruminant dairy fats. It is likely that marine or porcine food was also processed in a portion of the MK/SP pottery, in particular in pottery from the lower Scheldt river sites (Bazel, Melsele and Doel site C). However, due to a lack of molecular data for the extracted lipids, we cannot provide more information about the lipid sources in these vessels. Most of the studied vessels likely contained mixtures of different food sources, including aquatic, ruminant and/or terrestrial non-ruminant adipose fats. GC-MS and EA-IRMS analysis of pottery food crusts from Doel site C, Bazel, Oudenaarde and Kortrijk provide more information about the final cooking events in some vessels from the same archaeological contexts. These analyses indicate the presence of plant material in some residues from all four sites, in a few cases together with aquatic resources. Several vessels from Bazel and Oudenaarde, and one vessel from Kortrijk, were likely used to process freshwater resources. On the other hand, and in line with the GC-C-IRMS results, EA-IRMS analysis of one food crust sample from Doel site C indicates the processing of marine resources.

Discussion

Early Neolithic pottery (late 6th to early 5th millennium cal BC)

The oldest pottery studied here includes two late LBK vessels, three Limburg vessels and five undefined, bone-tempered vessels that could either represent undecorated LBK, Limburg or BVSG pottery, all from the site of Bazel Sluis. Ruminant adipose fats and ruminant dairy fats seem to be present in several of these vessels. This is not surprising, as the LBK and BVSG cultures had subsistence economies based on cereal cultivation and animal husbandry, with cattle predominating over sheep/goat and pig (Bedault and Hachem 2008; Bedault 2009; Salavert 2011; Arbogast and Jeunesse 2013; Bakels 2014; Gillis et al. 2017; Hamon et al. 2021). Of particular interest, however, is the clear evidence for dairy products in one or two Limburg vessels. The origin of Limburg pottery is heavily debated. Many researchers consider it to have been produced by hunter-gatherers during the late 6th millennium cal BC, who had contact with or were partially assimilated into LBK farming communities (Jeunesse 1987, 2002; van Berg 1990; Manen 1997; Gronenborn 1998; Manen and Mazurié de Keroualin 2003; Crombé 2009; van Willigen 2018; Kirschneck 2021). Other researchers consider it to be part of the LBK pottery production, possibly used for special, communal purposes (Constantin et al. 2010; Blouet et al. 2013; Gomart 2014). This debate, important within the framework of forager-early farmer relations

in Western Europe, has mainly focused on the find contexts and the typological and technological aspects of these pottery traditions. Evidence from residue analysis is largely lacking. The current study provides the first evidence of dairy products in Limburg pottery¹, here found outside of a LBK context in the sand region of northern Belgium. The presence of dairy does not entirely exclude the possibility that this pottery was produced or used by hunter-gatherers; it could have been produced by Mesolithic hunter-gatherers who were assimilated into LBK communities or it could indicate exchange of dairy products between farmers and foragers, as was recently also suggested for dairy fats in Ertebølle pottery from the western Baltic region, predating the appearance of domesticated animals in that region (Lucquin et al. 2023). However, the dairy evidence is much more in line with the hypothesis that Limburg pottery was produced and used by LBK farmers. This idea is strengthened by the observation, based on petrographic and geochemical analysis, that the late LBK vessels and some of the Limburg vessels from Bazel Sluis have similar fabrics, mineralogical and chemical compositions, indicating the same provenance area, most likely situated in the loess area of central Belgium (Teetaert 2020; Vannoorenberghe et al. 2022). The pottery may have been left at the site by LBK farmers in the context of transhumance or explorations outside their core area, or it may have been obtained by hunter-gatherers through contact with LBK communities or through collection from abandoned LBK settlements during their annual migrations towards the Belgian loess area in search for raw lithic materials among others (Crombé et al. 2015a; Mesiaen et al. 2023).

Further, at least three vessels, i.e. one late LBK, one Limburg and one undefined Early Neolithic vessel, may have been used to process freshwater resources. However, without molecular data to support the isotopic data, this remains uncertain. So far, residue analyses of LBK pottery from different areas within Europe provided little evidence of aquatic food sources (Salque et al. 2012; Roffet-Salque and Evershed 2015; Matlova et al. 2017; Courel et al. 2020; Cubas et al. 2020), be it with some exceptions (e.g. Robson et al. 2021; Casanova et al. 2022). For Northwestern Europe, aquatic markers were recently observed in some LBK vessels from sites in the Netherlands and the Paris Basin (Casanova et al. 2022), showing that this pottery was at least infrequently used to process aquatic resources.

Swifterbant culture pottery (5th millennium cal BC)

The isotopic, molecular and microscopic data show that freshwater resources were frequently processed in SWB

¹ Ongoing research of Limburg pottery from the Paris Basin shows similar results (Gomart L., pers. comm.).

pottery from the Scheldt river basin. This is in line with the archaeozoological evidence. Especially at the Doel sites (ca. 4500–4000 cal BC), thousands of burned fish bones have been found. The vast majority of these bones belong to freshwater species, mainly of the carp family (*Cyprinidae*), such as roach, rudd and bream, and to a lesser extent pike, perch and eel (Van Neer et al. 2005; Neer et al. 2013). The fact that these bones mainly belong to small and medium-sized fish indicates the use of passive fishing methods, such as fish traps. Marine fish, like sturgeon (Doel site B), thin-lipped mullet, shad and three-spined stickleback (Doel site M) are also represented in smaller numbers. These species are known to seasonally migrate into coastal estuaries and river basins to spawn (=anadromous fish). Although it seems likely that anadromous fish were processed in the same way as freshwater fish, no marine fats were detected in the SWB pottery. These results are consistent with recent lipid analyses (GC-MS and GC-C-IRMS) of SWB pottery from the Netherlands, which showed that this pottery was primarily used to process freshwater fish throughout the entire 5th millennium BC (ca. 5000–3800 cal BC) (Demirci et al. 2020, 2021). The frequent occurrence of freshwater resources in SWB pottery has important implications for radiocarbon dating, as it increases the uncertainties of ^{14}C dates on food residue due to the freshwater reservoir effect (FRE). About 30 food crust samples of SWB pottery from the Doel sites and Bazel Sluis have previously been dated, and almost all of these dates are affected by a FRE (Boudin et al. 2009, 2010; Teetaert et al. 2017).

The current study also provides evidence for the processing of ruminant fats in SWB pottery from the Scheldt river basin. Many of the samples analysed by GC-C-IRMS have indications of ruminant adipose fats, probably often mixed with other resources. To better understand the neolithisation process in the Belgian lowlands, in particular with regard to the timing of the introduction of domesticates and the beginning of local animal husbandry, it would be interesting to know whether these adipose fats are from wild or domesticated ruminants, or both. However, this distinction cannot be made based on the lipid analyses alone. The numerous mammal bones collected from the Doel sites do not provide a conclusive answer either. These bones are severely burned and fragmented, so that only a minor part could be identified (Van Neer et al. 2005; Neer et al. 2013). Although only wild animals (mainly red deer, roe deer and wild boar) are represented among the few species identified, the presence of domesticated animals cannot be fully excluded. This assumption is based on the presence at Doel sites B and M of exceptional numbers of charcoal and charred seeds of ivy (*Hedera helix*) and mistletoe (*Viscum album*), tentatively interpreted as indirect evidence for animal husbandry (Bastiaens et al. 2005; Deforce et al. 2013). It is unlikely that

ivy or mistletoe were collected as firewood, as both taxa yield only small wood volumes and ivy charcoal is nearly absent. It is more likely that the leaves and twigs of these evergreens were used as fodder for animals during winter, when other plant foods were scarce. Mistletoe leaves have recently been found in a pig coprolite from the Swifterbant S3 site, in the Netherlands, indicating their use as animal fodder (Kubiak-Martens and van der Linden 2022). The use of mistletoe as leaf fodder has also been evidenced at several other Neolithic sites in Europe (Deforce et al. 2013; and the references cited therein). In any case, the recent discovery of sheep/goat bones at the site of Bazel Sluis, the oldest specimen of which dates to ca. 4600 cal BC (Crombé et al. 2020, 2022), confirms the presence of domesticated animals in Belgian SWB contexts. Cattle might also have been present at this site as early as ca. 4800/4600 cal BC, but here the evidence is less conclusive (Crombé et al. 2022).

In the Lower Rhine-Meuse area of the Netherlands, the processing of ruminant food (either wild and/or domesticated) becomes an important part of SWB pottery use around the middle of the 5th millennium cal BC (Demirci et al. 2021; Raemaekers et al. 2021). However, it seems to be replaced by the processing of porcine food (wild boar/pig) by the late 5th millennium cal BC (ibid.). For the Scheldt river basin, the importance of porcine food processing in the SWB pottery remains difficult to assess. Although none of the samples analysed by GC-C-IRMS clearly plot within the range of porcine fats, many of the vessels from the different sites might have contained porcine fats mixed with other resources. After all, suids are represented among the animal bones at the SWB sites in this region, for instance at Doel site B and Bazel Sluis (Van Neer et al. 2005; Crombé et al. 2020). Future research including both GC-MS and GC-C-IRMS of absorbed lipids should provide more conclusive information on this matter.

Dairy seems almost completely absent from the SWB pottery. For the Netherlands, out of 111 samples from seven different sites, Demirci et al. (2021) could only detect ruminant dairy fats in one or possibly two vessels from the Lower Rhine-Meuse area, dated to the final quarter of the 5th millennium cal BC. This low number raised the question whether dairy was locally produced by the SWB communities, meaning that these one or two vessels are an underrepresentation of a wider use of dairy products, or if dairy products were only obtained through interactions with neighbouring farming communities of the late 5th millennium cal BC (ibid.). The current study shows that dairy products are equally limited in the SWB pottery from the Scheldt river basin. Only a single vessel from the site of Melsele, which, unfortunately, cannot be dated precisely within the 5th millennium cal BC, possibly contained ruminant dairy fats. This speaks against an underrepresentation

of a wider use of dairy products within the SWB culture. After all, a considerable number of SWB vessels from 12 sites in three different regions have already been analysed, with almost no indications for dairy products. This could indicate that ruminants were mainly raised by the SWB culture populations for their meat and only to a lesser extent for their milk. Of course, it remains possible that they obtained dairy products through exchange with farming communities, as has recently also been suggested for the Ertebølle culture in the western Baltic region (Lucquin et al. 2023).

Finally, various samples from the Doel and Bazel sites contain evidence for plant processing, often in combination with aquatic biomarkers. The archaeobotanical data from the Doel sites shows that the SWB people gathered a variety of seeds and fruits of edible plants from the alluvial hardwood forest environment, including hazelnuts, acorns, crab apples, sloe plums, and hawthorn, dogwood and guelder rose berries (Bastiaens et al. 2005; Deforce et al. 2014). Most of these can be consumed without preparation, but some, like acorns, must have been cooked or roasted first. For now, we lack information about which plants were processed in the SWB pottery. The only insights are provided by SEM analysis of a small number of food crusts from Bazel Sluis, which contained the remains of wild chives, leek or garlic as well as numerous fish remains. As food crusts probably represent the last meal(s) prepared in a vessel (Miller et al. 2020), it is likely that the stems and/or leaves of these plants were prepared together with the fish. The same hypothesis was proposed for the SWB cooking practices at the Swifterbant S3 and S4 sites in the Netherlands, where fish was often prepared together with cereals but also with the addition of green plants (Raemaekers et al. 2013; Kubiak-Martens and van der Linden 2022). Although remains of wild chives, leek or garlic were not observed in the food crusts from these sites, evidence for *Allium* consumption at the S3 site was recently shown by the analysis of human coprolites (Kubiak-Martens and van der Linden 2022). So, it seems that in both SWB regions, fish was often cooked together with plant components, and that *Allium*, possibly various species, were well-known and valued green and root vegetables for the SWB populations. Contrary to the Swifterbant S3 site in the Netherlands, where SEM analysis revealed chaff of emmer (*Triticum dicoccum*) embedded in the food crusts (Raemaekers et al. 2013), there are currently no indications for the presence of cereals in the SWB pottery from the Scheldt river basin. However, we must keep in mind that plant lipids, including those from cereals, are difficult to detect through lipid residue analysis alone, especially when they have become mixed with animal fats (Steele et al. 2010; Colonese et al. 2017; Hamman and Cramp 2018; Craig et al. 2020). For example, while cereal remains could be observed in the food crusts from the Swifterbant S3 site

by SEM analysis, lipid analysis of pottery from that same site yielded no plant biomarkers (Demirci et al. 2020). Therefore, we should be cautious in interpreting the absence of cereal biomarkers in SWB pottery from the Scheldt river basin.

Michelsberg culture/Group of Spiere pottery (late 5th to early 4th millennium cal BC)

Although the MK/SP populations of the Scheldt river basin are generally considered to be agro-pastoral communities, with a subsistence based mainly on crop cultivation and animal husbandry (Vermeersch and Burnez-Lanotte 1998; Vanmontfort et al. 2001/2002; Vanmontfort 2004), the current study shows that aquatic resources remained an important addition to their diet. Freshwater fats are present in the pottery of at least three, but possibly all six sites under study. In addition, some of the pottery from Doel site C may have contained marine fats, which, based on the fish bones collected from the nearby SWB sites, could be of anadromous fish. This continued exploitation of aquatic resources beyond the arrival of domesticated plants and animals is also well documented for Neolithic groups in other parts of Northern Europe (Lucquin et al. 2023). The results of the GC-C-IRMS analysis seem to indicate that the processing of aquatic resources was more important in the MK/SP pottery from the lower Scheldt river basin, i.e. in the same area where the SWB occupations are located. This might be explained by the environmental situation. In the lower Scheldt river basin, both SWB and MK/SP occupations took place during a long period of tidal flooding, indirectly resulting from the rising sea level in the adjacent North Sea basin (Verhegge et al. 2014; Crombé et al. 2015b; Storme et al. 2020). This estuarine environment may have facilitated fishing, especially in the shallow, tidal freshwater creeks along the occupied sand dunes. The MK/SP sites in the upper Scheldt basin, on the other hand, are more often situated further away from the river, on hill tops (e.g. Kortrijk Schaapsdreef) or along dry banks of the former Late Glacial floodplains (e.g. Menen Kortewagstraat), making freshwater fish less readily available.

In contrast, the processing of ruminant adipose fats is more often observed in pottery from the upper Scheldt river than lower Scheldt river sites. Based on the low percentage (< 10%) of wild fauna at most MK/SP sites (Arbogast 1993; Vanmontfort 2004), we may assume that these adipose fats are mainly from domesticated animals, most likely cattle. However, at the wetland site of Oudenaarde Donk, wild game represents up to 39% of the faunal remains, with red deer as most important species (Parent et al. 1987). As such, it cannot be excluded that part of the ruminant adipose fats in this pottery still originates from wild animals. Ruminant

dairy fats are only detected in three out of 34 samples (9%) analysed by GC-C-IRMS. This is surprising and in contrast with the evidence for Middle Neolithic pottery from neighbouring areas. For instance, lipid analyses of Michelsberg pottery from the Lower Rhine area yielded ruminant dairy fats in 69% of the cases (Lucquin et al. 2023). As for the SWB culture, this might indicate that the MK/SP populations of the Scheldt river basin mainly raised ruminants for their meat.

Several of the MK/SP vessels analysed by GC-C-IRMS may have contained porcine fats, intermixed with other resources. However, without molecular data for the same samples, this is difficult to assess. More research combining GC-MS and GC-C-IRMS analysis of absorbed lipids is necessary to better understand the importance of porcine food in this pottery. Finally, plant biomarkers are rather scarce, but this might be due to the difficulties in detecting plant foods through lipid residue analysis.

Based on the current results, pottery use among the SWB and MK/SP populations of the Scheldt river basin shows some interesting similarities. In both cases, pottery seems to have been used frequently to process freshwater fish and ruminant meat, while ruminant dairy is nearly absent. This might reflect shared culinary practices. Together with earlier observations of continuity between both groups, such as similarities in the lithic technology (Messiaen et al. 2023) or the shared use of antler beam mattocks (Crombé et al. 2018), this could be an argument in favour of a local development of the MK/SP culture. However, additional residue analyses are necessary for both the SWB and MK/SP pottery from northern Belgium, especially with regard to the processing of porcine and plant foods, to study the evolutions in pottery use.

Conclusions

This study represents the first extensive organic residue analysis of prehistoric pottery from northern Belgium, including over 200 samples from nine archaeological sites. It provided new insights into the local use of pottery, from its first appearance in the late 6th millennium cal BC until the development of fully agrarian communities in the late 5th to early 4th millennium cal BC. The most significant observation for the Early Neolithic pottery is the presence of ruminant dairy fats in Limburg pottery found outside of the loess region. This reinforces the hypothesis that Limburg pottery was produced and used by LBK farmers rather than by hunter-gatherer populations. The current study especially provides insight into the use of the first indigenous pottery of northern Belgium, of the SWB culture, the local production of which started at the latest around 4600 cal BC.

This pottery was frequently used to process freshwater fish (together with plant foods) and ruminant meat, although several of the studied vessels may have contained mixtures of resources also including terrestrial non-ruminant (porcine) meat. Ruminant dairy is however nearly absent from this pottery. Interestingly, these culinary practices seem to have largely persisted during the subsequent MK/SP occupations of the Scheldt river basin, dated to the late 5th to early 4th millennium cal BC. Again, the pottery was frequently used to process aquatic resources as well as ruminant meat, while the processing of ruminant dairy is remarkably limited even in the pottery of these fully agrarian communities. This apparent continuity in pottery use, next to earlier observations of continuity between both groups, seems to support the hypothesis that the Middle Neolithic MK/SP culture of the Scheldt river basin developed from local populations, rather than resulting from human migrations from neighbouring areas. However, our current view on local pottery use might be biased by the difficulties in distinguishing between wild and domesticated ruminant fats and in detecting plant foods through lipid residue analysis. After all, as the transition between both cultural groups coincided with an increasing reliance on domesticated animal and plant resources, one might expect changes in pottery use. Future research should therefore include more SEM analysis of food crusts, alongside lipid and protein analyses, to address these gaps in our knowledge. There is also need for combined GC-MS and GC-C-IRMS analyses of absorbed lipids, which is missing from the current study, to gain insight into the importance of porcine food processing in the SWB and MK/SP pottery. Finally, the near absence of dairy products in the MK/SP pottery from the Scheldt river basin is in contrast with the Middle Neolithic pottery from neighbouring areas, such as the Lower Rhine area. This supports the existence of regional variations in pottery use or culinary practices throughout prehistoric Northwestern Europe, once again highlighting the importance of and need for regional studies.

The results of this study also have implications for future radiocarbon dating of pottery residues. The frequent processing of freshwater resources increases the risk of a FRE on ^{14}C dates, as demonstrated by previous studies of SWB pottery from the Scheldt river basin. However, it is becoming clear that this risk also exists for the pottery of early agrarian communities (MK/SP and possibly LBK), at least within this region. Therefore, direct dates of pottery should be accompanied by residue analysis, to exclude the possible sampling of aquatic products. In addition, we should continue to search for other ways to directly date pottery, for instance through the extraction and ^{14}C dating of plant temper, which is present in a considerable part of the prehistoric pottery from Northwestern Europe. The identification,

extraction and ^{14}C dating of plant temper materials preserved in pottery from Belgium, northern France and the Netherlands is the subject of ongoing research (ORG-ID project, funded by BELSPO).

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Declarations

Competing interests The authors declare no competing interests.

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