Article:
Di Muro, Alice, Admiraal, Marjolein, McGrath, Krista M. et al. (2026) Culinary diversity in early modern Brazil revealed by residue analysis of ceramic artefacts. Royal Society Open Science. rsos260385. ISSN: 2054-5703

https://doi.org/10.1098/rsos.260385

ROYAL SOCIETY OPEN SCIENCE

royalsocietypublishing.org/rsos

Research

Cite this article: Di Muro A *et al.* 2026 Culinary diversity in early modern Brazil revealed by residue analysis of ceramic artefacts. *R. Soc. Open Sci.* **13**: 260385.
https://doi.org/10.1098/rsos.260385

Received: 24 February 2026
Accepted: 1 June 2026

Subject Category:
Chemistry

Subject Areas:
analytical chemistry, environmental chemistry, environmental science

Keywords:
organic residue analysis, ceramic artefacts, historical cuisine

Author for correspondence:
Andre Carlo Colonese
e-mails: andrecarlo.colonese@uab.cat

Supplementary material is available online at https://doi.org/10.6084/m9.figshare.c.8526193.

THE ROYAL SOCIETY PUBLISHING

Culinary diversity in early modern Brazil revealed by residue analysis of ceramic artefacts

Alice Di Muro[1,2,3], Marjolein Admiraal[3], Krista M. McGrath[1,2], Thiago Fossile[1,2], Yun Chiang[4,5], Joannes A. A. Dekker[3,4,6], Margherita Cantelli[1,2,7], Nuria Moraleda-Cibrián[1], Joan Villanueva[1], Kerry Sayle[8], Dione DaRocha Bandeira[9,10], Fernanda Mara Borba[10], Maria Cristina Alves[10], Matthew Collins[4], Oliver E. Craig[3], and Andre Carlo Colonese[1,2]

[1]Institute of Environmental Science and Technology (ICTA-UAB), Universitat Autònoma de Barcelona, Bellaterra, Spain
[2]Department of Prehistory, Universitat Autònoma de Barcelona, Bellaterra, Spain
[3]BioArCh, Department of Archaeology, University of York, University of York, UK
[4]Section for Geobiology, Globe Institute, University of Copenhagen, Copenhagen, Denmark
[5]The Nice Institute of Chemistry, Université Côte d'Azur, France
[6]Department of Life Sciences and Systems Biology, University of Turin, Italy
[7]CEPAM, Université Côte d'Azur, CNRS, France
[8]University of Glasgow, Scottish Universities Environmental Research Centre, Scotland, UK
[9]Laboratório de Arqueologia e Patrimônio Arqueológico, Universidade da Região de Joinville (UNIVILLE), Brazil
[10]Museu Arqueológico de Sambaqui de Joinville (MASJ), Brazil

KMM, 0000-0003-0692-1217; TF, 0000-0002-6997-4677; JAAD, 0000-0002-3952-4448; MC, 0000-0003-4226-5501; ACC, 0000-0002-0279-6634

Brazil is a multi-cultural and ethnically diverse country shaped by a complex history of colonization, migration, slavery and economic intensification that has transformed it into one of the world's leading food-producing regions. The roles played by Indigenous, African and European populations, and their descendants, in this trajectory are preserved in the rich archaeological materials they left behind. In this study, we analysed lipids and proteins extracted from ceramic artefacts used by Indigenous, Luso-Brazilian, Afro-descendant communities and recently established German immigrants in Joinville and São Francisco do Sul (southern Brazil), dating to the late nineteenth and early twentieth centuries. Extracted molecules

were characterized using state-of-the-art chromatographic and isotope ratio techniques, including gas chromatography flame ionization detection, gas chromatography mass spectrometry, gas chromatography–combustion–isotope ratio mass spectrometry, elemental analysis–isotope ratio mass spectrometry and liquid chromatography–tandem mass spectrometry. Our results show that users of industrially produced, wheel-thrown wares relied primarily on C_3 plants and terrestrial animal products. By contrast, marine foods played a more prominent role in the cuisine of those using hand-thrown ceramics, associated with African, Afro-descendant and Indigenous groups. These results demonstrate how molecular archaeology can illuminate patterns of food access and distribution among coexisting communities, revealing historical processes that continue to shape contemporary culinary traditions in Brazil.

1. Introduction

Brazil is renowned for its multi-ethnic population and for culinary traditions shaped by centuries of Indigenous, African, European and Asian influences. Historical reconstructions of Brazilian foodways, however, have largely privileged regional commodity production, export economies and broad demographic transformations [1,2], leaving a limited understanding of how food practices evolved at the level of everyday domestic consumption. This gap is particularly striking given the profound historical transformations that have shaped the country since the sixteenth century, including the eradication of Indigenous populations, the introduction of European agricultural systems, successive waves of immigration and, more recently, industrialization and globalization, all of which have influenced regional food habits [2,3]. Nowadays, food consumption in Brazil is shaped by socio-economic and demographic factors [4–6], and is marked by an increasing intake of processed foods [7–9], with major implications for public health and nutrition policies [10]. Understanding how these patterns have evolved over time requires examining dietary practices across much longer temporal scales, an approach that can be achieved through the archaeological record.

The cities of Joinville and São Francisco do Sul, situated in Babitonga Bay in the state of Santa Catarina (southern Brazil), have a unique concentration of archaeological sites produced by Indigenous, European and African peoples who contributed to the rich culinary traditions and foodways of southern Brazil (figure 1). Babitonga Bay, which covers approximately 160 km^2, hosts the largest mangrove remnants in the state and supports high biodiversity. Fishing and shellfish collection, together with plant cultivation and hunting, have been core components of the local economy since pre-European times and throughout much of the European colonial period until the late nineteenth century [11–13]. Significant demographic and economic transformations took place between the late nineteenth and early twentieth centuries, following Brazil's major regime changes—the transition from the Colonial period to the Empire (1822–1889), and subsequently to the First Republic (1889–1930). Political agendas promoting industrialization and nationalization accelerated the shift from predominantly subsistence-based practices to increasingly commercial forms of agriculture and fishing [13,14]. The gradual abolition of slavery (terminated in 1888), together with waves of European immigration and the establishment of settler colonies such as Dona Francisca (present-day Joinville) in 1851, introduced new labour forces and food products. Alongside staple crops, such as manioc, rice, maize, beans and sugarcane, new products were cultivated, livestock husbandry gradually expanded for both meat and secondary products [14,15], commercial fishing intensified [16,17], and food imports from regional and international markets became increasingly common [17].

A diverse range of cooking wares was used at the household level in Babitonga Bay during the late nineteenth and early twentieth centuries [18]. Industrially produced, wheel-thrown, glazed ceramic wares coexisted with locally manufactured, hand-thrown ceramics displaying decorative elements rooted in Indigenous, African and European traditions (figure 2A,D). These technological and stylistic traits can convey information about group identity, socio-economic status and practices, such as the reappropriation of material culture [19,20]. They also offer a window into past culinary practices through the molecular analysis of food residues absorbed into their ceramic matrices. Lipids, in particular, are readily incorporated into ceramic vessels during food processing and have been widely used to reconstruct past foodstuffs [21–24]. Proteins have also been successfully extracted from ceramic vessels, however, less so than lipids, and show strong potential for distinguishing individual components within food mixtures [25–28]. While lipid residue analysis has been used on pre-colonial Indigenous ceramics

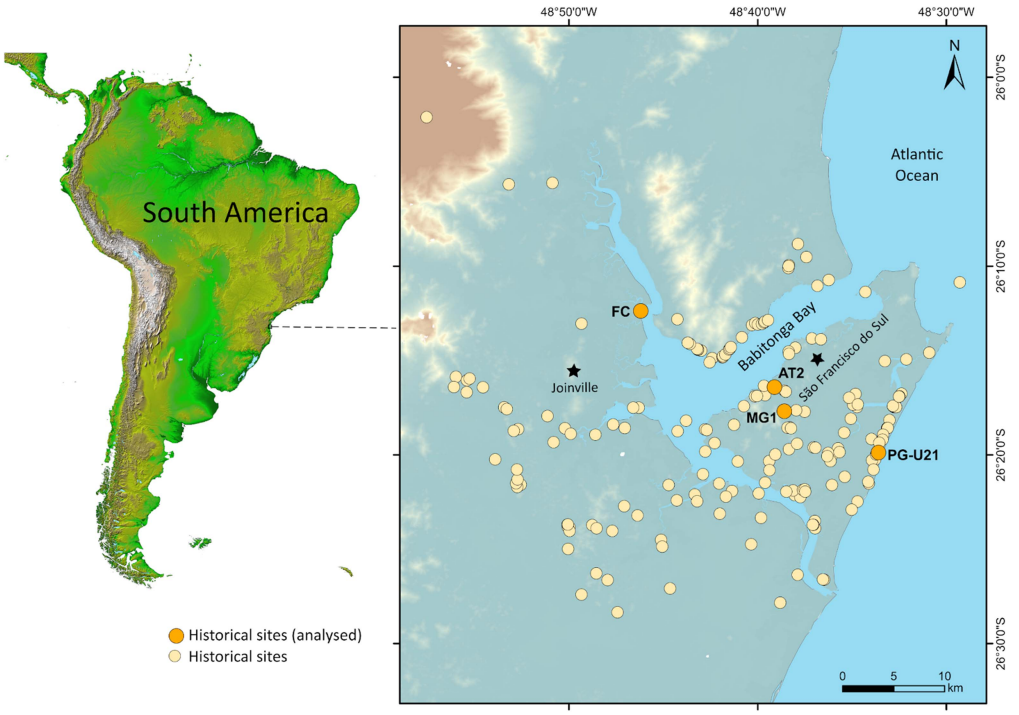

Figure 1. Geographic distribution of historical archaeological sites in Babitonga Bay, including those studied herein: FC, Foz do Cubatão; MG1, Morro Grande 1; AT2, Arroio Tamarina 2; PG-U21, Praia Grande Unidade 21. Stars indicate the two main cities in the region today. Map generated using ArcGIS 10.8 and Inkscape 1.4 with public data from Instituto Brasileiro de Geografia e Estatística (IBGE; https://www.ibge.gov.br/geociencias/downloads-geociencias.html) and [95].

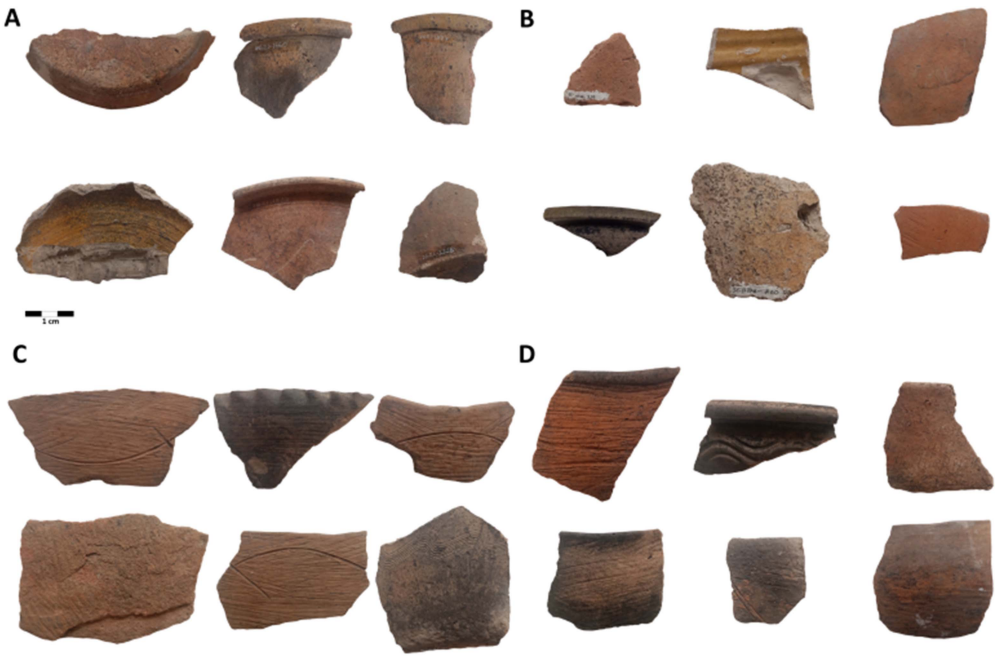

Figure 2. Examples of cooking pot sherds analysed in this study. Sherds of internally glazed and wheel-thrown wares from (A) PG-U21 and (B) AT2. Sherds of locally produced wares made without the use of a wheel (hand-thrown) and with decorations typical of Indigenous, African and European influences from (C) MG1 and (D) FC.

Table 1. Synthesis of site contexts and group attributions.

site	location	chronology	possible group attribution	ceramic manufacture
AT2	São Francisco do Sul	early twentieth century	German	wheel-thrown
PG-U21	São Francisco do Sul	late nineteenth and early twentieth centuries	Luso-Brazilians, African, Afro-descendant and Indigenous	wheel-thrown
MG1	São Francisco do Sul	late nineteenth and early twentieth centuries	Luso-Brazilians, African, Afro-descendant and Indigenous	hand-thrown
FC	Joinville	early twentieth century	Luso-Brazilians, African, Afro-descendant and Indigenous	hand-thrown

from coastal Brazil [12,29–32], neither lipid nor protein analyses have ever been applied to historical ceramic vessels in the country.

Here, we combine lipid and protein analyses of historic ceramic artefacts from Babitonga Bay with information on production (wheel-thrown versus hand-thrown) and stylistic (decoration) features to investigate past food practices in the region. Cooking wares were sampled from four broadly contemporary residential contexts. Three were associated with Luso-Brazilian, Indigenous and African communities (Foz do Cubatão (FC), Morro Grande 1 (MG1) and Praia Grande Unidade 21 (PG-U21)), and one with German residents (Arroio Tamarina 2 (AT2)). Lipids were extracted from over 180 sherds, and proteins from 38 of these. Our results reveal differences in culinary practices possibly linked to ethnicity and social status, patterns that continue to influence local foodways today.

2. Material and methods

2.1. Archaeological samples

Ceramic artefacts were selected from four rural residential sites dated to the late nineteenth and early twentieth centuries: PG-U21, AT2, MG1 and FC. They were chosen based on the quality and extent of excavations, the diversity of ceramic typologies and decorations, and the availability of archaeological contexts containing complementary records, such as artefacts and faunal remains. In this study, we analysed 182 ceramic sherds from four household deposits (supplementary material, SI1; table 1), each spanning at most a few decades. This provides a higher sampling resolution per year than is typically achieved in organic residue analysis (ORA) studies [33]. The location of the four sites is shown in figure 1 along with other known archaeological locations, although not all of the known sites have been excavated or have yielded sufficient material suitable for ORA.

PG-U21 is a coastal site on the island of São Francisco do Sul. Radiocarbon dating and archaeological artefacts constrained the site chronology to the late nineteenth and early twentieth centuries [34]. The presence of domestic milling and combustion facilities reveals that manioc cultivation was an important activity at PG-U21, serving both household and market consumption [34,35]. Faunal remains indicate this group subsisted also on fishing, hunting and raising domestic animals, such as chicken and livestock (cattle and pig) [34]. Sherds for residue analysis were attributed to wheel-thrown, smoothed and glazed cooking wares. They could be linked to small to medium-sized bowls used to process and consume food [35,36]. Overall, the archaeological record suggests that these groups occupied neither a particularly high nor a particularly low socio-economic status [37]. Bones of domestic animals, including cattle, pig and chicken, and of wild fauna were sampled from PG-U21 for stable carbon and nitrogen isotope analysis (see below).

AT2 is a coastal site located on São Francisco do Sul island. The material culture from AT2 included residential structures, glass bottles, porcelain, faience, wire, faunal remains and tile fragments [38]. Historical information indicates the site was occupied by German immigrants in the early twentieth century [36]. Ceramic vessels from the sites were wheel-thrown, with one sample internally glazed, while others were brushed, painted and smoothed.

MG1, also located on São Francisco do Sul island but a few kilometres from the coast, was possibly occupied between the late nineteenth and early twentieth centuries [34]. Manioc cultivation, along with hunting, fishing and raising domestic animals, such as chicken and livestock (cattle and pig), was practised at the site [18,34]. The rich material culture from MG1 includes, among others, ceramics (white and decorated earthenware, porcelain, stoneware and clay pottery, pipes, slingshot pellets and fragments of couscous steamers), metal (nails, coins, buttons, spoons, knives, etc.), glass, faunal remains and lithic material (flakes). Of particular significance is the presence of ceramics and artefacts such as pipes decorated with motifs commonly linked to African, Afro-descendant and Indigenous groups [18]. The cooking wares analysed here were hand-thrown, so produced without the aid of a wheel, suggesting local manufacture. They presented morphological and stylistic features typical of Indigenous, African and European influences [39]. These sherds were mainly identified as medium to large bowls and pans used to process food, and presented decorations with straight, curved or diagonal lines, along with brushed patterns on the body and rims [40,41]. These artefacts express the enduring traditions of enslaved Indigenous and African peoples along with Portuguese descendants, and are probably associated with groups of low socio-economic status. Bones of domestic animals and of wild fauna were also sampled from MG1 for stable carbon and nitrogen isotope analysis (see below).

FC is a site located in the modern city of Joinville, in northwest Babitonga Bay. The presence of glass and other material culture (white and decorated porcelain) suggests that FC was occupied from the first half of the twentieth century. Marine mollusc shells, fish otoliths and bones (catfish and whitemouth croaker), along with fishing implements, attest to the local significance of fishing [41]. Also of particular significance at FC is the presence of artefacts such as pipes decorated with motifs linked to African, Afro-descendant and Indigenous groups [18]. The cooking wares were similar to those from MG1, being hand-thrown and exhibiting Indigenous, African, and European influences [41], and were associated with groups of relatively low socio-economic status [42,43].

2.2. Extraction of archaeological lipids

Drilled ceramic powder (diameter 2–5 mm) from the internal surface of the sherds (approx. 1 g) was lipid-extracted using the methanol (CH_3OH, hereafter MeOH) and sulphuric acid (H_2SO_4) protocol described in [44] (hereafter acid extraction). In short, 4 ml of MeOH and 10 µl of alkane $C_{34:0}$ were added to samples in sterilized glass tubes, then vortexed and ultrasonicated for 15 min and then digested with H_2SO_4 (800 µl). The acidified suspension was heated for 4 h at 70°C, and the supernatant was extracted with *n*-hexane (2 ml × 3). The extract was then dried under a gentle flux of N_2, resuspended with 90 µl isooctane and transferred to autosampler vials containing an internal standard (10 µl of *n*-hexatriacontane, $C_{36:0}$). In addition, 40% of the samples ($n = 75$) were solvent-extracted to investigate the presence of triacylglycerols and wax esters. The ceramic powder (approx. 1 g) was extracted using 4 ml of a dichloromethane : MeOH (2 : 1 v/v) mixture along with 10 µl of alkane $C_{34:0}$ as an internal standard. Samples were sonicated for 15 min at 25°C, then centrifuged (10 min, 3000 r.p.m.). Samples were then divided into aliquots (20% and 80%), dried down under a stream of N_2 and stored at –25°C. The 20% aliquots of the solvent extracts were derivatized using N,O-bis(trimethylsilyl) trifluoroacetamide (BSTFA) with an addition of 1% trimethylchlorosilane, and heated at 70°C for 1 h. The mixture was then dried under a gentle stream of N_2. Dried samples were resuspended with 100 µl of *n*-hexane, vortexed and subsequently transferred to autosampler vials, as previously mentioned for the acid extracts. A subsample of the acid extracts ($n = 35$) with stable carbon isotope values falling within the range of marine and C_4 plants was also derivatized to their trimethylsilylation (TMS) esters using BSTFA, following the protocol described above, to check for the presence of long-chain fatty alcohols derived from plant products. Acid extraction was performed on modern samples of rice (*Oryza sativa* subsp. *indica*, $n = 2$), beans (*Phaseolus vulgaris* Pinto Group, $n = 4$) and manioc flour (*Manihot esculenta*, $n = 2$) in order to assess the isomer distribution of ω-(o-alkylphenyl) alkanoic acids (APAA) [45], which are known to preserve well in burial environments [45,46]. Plants were commercially acquired in a European market. Dried plant samples (65 mg) were mixed with 100 mg of sterile pottery powder (drilled from wheel-thrown replica pottery vessels made with red clay) in sterile glass tubes. The mixture was then covered with deionized water to homogenize the sample within the ceramic matrix. The samples were heated until the water evaporated and then heated to 270°C for 5 h. Samples were then extracted following the same acid extraction protocol described above, and analysed by gas chromatography–flame ionization detection (GC-FID), gas chromatography–mass spectrometry (GC-MS) and gas chromatography–combustion–isotope ratio mass spectrometry (GC-C-IRMS).

2.3. Collagen extraction from bones

Historical accounts indicate that livestock (cattle and pig) and chickens in Babitonga Bay were variably fed with corn (*Zea mays*) and sugarcane (*Saccharum officinarum*) products, both C_4 plants [15]. As a consequence, fatty acids from these food sources may present a wider range of $\delta^{13}C$ values [47], with some overlap with marine resources [12]. We assessed this potential bias through bulk collagen stable carbon isotope analysis of key domestic animals in the region, including cattle ($n = 16$), domestic pig ($n = 7$) and chickens ($n = 20$), along with wild fauna such as armadillo ($n = 3$), deer ($n = 12$) and marine fish ($n = 11$) from the historic sites of MG1 and PG-U21. Bones were manually cleaned, and then samples (113–1120 mg) were demineralized using 0.6 M hydrochloric acid (HCl) at 4°C for several days. Samples were then rinsed with ultrapure water and immersed in 0.05 M sodium hydroxide (NaOH) for 20 min at room temperature. NaOH wash cycles were repeated as needed, typically one or two times, until no further colour change occurred in the solution. Samples were rinsed three times with ultrapure water for 10 min each to ensure the complete removal of NaOH [48,49]. Samples were then gelatinized in 0.001 M HCl at 80°C for 48 h. The supernatant containing collagen was filtered using Polyethylene Ezee filters (9 ml, pore size 60–90 µm, Elkay Laboratories Ltd.), frozen for at least 48 h at –20°C and lyophilized. Samples were then analysed by elemental analysis–isotope ratio mass spectrometry (EA-IRMS).

2.4. Protein extraction and analysis

We undertook the protein analysis of sherds from all four study sites, for a total of 38 samples: FC ($n = 10$ samples), MG1 ($n = 10$ samples), AT2 ($n = 7$ samples) and PG-U21 ($n = 11$ samples). Three extraction blanks were analysed along with the ceramic samples. Proteins were extracted using a deep eutectic solvent-based method [50], based on the protocol published in [51]. In short, the ceramic samples were incubated in a mixture of urea and guanidine hydrochloride at 75°C for 4 h, while shaking. Following this incubation, 0.5 ml of purified water was added and the samples were homogenized thoroughly. An initial desalting step was then performed using C18 StageTips. The StageTips were first conditioned with 150 µl 100% MeOH, then loaded with 150 µl of 80% acetonitrile (ACN) + 0.1% trifluoroacetic acid (TFA), and washed with 150 µl 0.1% TFA. Each sample was then loaded on a StageTip in steps of 150 µl. The StageTips were then washed twice with 150 µl 0.1% TFA, and the proteins were eluted in 50 µl of 50% ACN + 0.1% TFA. The eluted proteins were dried to near completion with a speedvac and resolubilized in 100 µl 50 mM ammonium bicarbonate. The eluted proteins were initially digested with 1 µl of 0.4 µg μl^{-1} of chymotrypsin (Promega) and incubated at 25°C for 18 h. Following the chymotrypsin digestion, samples were digested again using 1 µl of a 0.4 µg μl^{-1} trypsin solution (Promega) at 37°C for 18 h. After digestion, the samples were acidified with 10 µl of 5% TFA. A final cleaning step was performed using EvoTips. The EvoTips were conditioned with 20 µl of 0.1% formic acid (FA) in ACN, and afterwards, the filters of the EvoTips were soaked in isopropanol for 1 min. The EvoTips were then washed with 20 µl of 0.1% FA, and 20 µl of sample was loaded on the EvoTips, followed by two washes with 20 µl of 0.1% FA, and lastly, 100 µl of 0.1% FA was loaded on top of the EvoTips. Samples were then analysed by liquid chromatography–tandem mass spectrometry (LC-MS/MS).

2.5. Lipid quantification (gas chromatography-flame ionization detector), molecular characterization (gas chromatography mass spectrometer) and stable carbon isotope analysis (gas chromatography–combustion–isotope ratio mass spectrometry)

Screening and quantification of fatty acid methyl esters and TMS derivatives were performed using an Agilent 7890A GC-FID equipped with a DB-5 high temperature (HT) column (15 m × 0.25 mm internal diameter (i.d.), 0.15 µm film thickness; Agilent). The temperature of the column was kept at 50°C for 2 min and then increased by 6°C every minute until a final temperature of 340°C was reached and held for 19 min. Hydrogen was used as the carrier gas at a constant flow rate of 2 ml min^{-1}. The detector was kept at 340°C with a hydrogen flow of 40 ml min^{-1}. Lipid identification and $\delta^{13}C$ measurements of methyl palmitate ($C_{16:0}$) and methyl stearate ($C_{18:0}$) were simultaneously characterized using a gas chromatograph Trace 1310 (Thermo Fisher, Bremen, Germany) coupled to both a quadrupole mass spectrometer (MS) and an isotope ratio mass spectrometer (IRMS) at the Institute of Environmental Science and Technology, Universitat Autònoma de Barcelona. The MS set-up consisted of a Thermo ISQ7000

quadrupole system, while the IRMS was a Thermo DELTA V Plus coupled to a Conflo IV interface. Combustion of the analytes was achieved using a gas chromatograph (GC)-combustion/high-temperature conversion (HTC) GC-Isolink system (Thermo Fisher, Bremen, Germany).

Samples were diluted with isooctane, and 1 µl of each sample was injected into a DB5-MS fused-silica column (60 m × 0.25 mm i.d., 0.25 µm film thickness, Agilent) using a splitless system (50 s) at 310°C. The GC was programmed from 90°C (1 min) to 320°C at 6°C min^{-1} and held at 320°C for 20 min. Helium was used as the carrier gas at a ramped flow, it was held at 2 ml min^{-1} for 1 min and ramped to –0.02 ml min^{-2} to 1.2 ml min^{-1} and held for 19 min. The mobile phase was split into two flows after the chromatographic column. About 10% of the effluent was directed to the MS system for compound characterization, and the remaining 90% of the mobile phase was directed to an IRMS system for the stable carbon isotope analysis. For compound characterization, the MS detector was programmed in scan mode (70–550 m/z, 0.2 second per scan), with the transfer line temperature set at 300°C, and the source at 260°C.

Conditions for isotopic analysis were the following. During the initial 10 min, the effluent from the GC was diverted away from the combustion reactor (backflush mode). During the solvent-divert period, CO_2 reference gas was automatically introduced into the IRMS in a series of pulses and its $^{13}C/^{12}C$ measured. After the solvent-divert period, the effluent from the GC was allowed to enter the combustion reactor and IRMS. The IRMS automatically measured the ion intensities of m/z 44, 45 and 46 in its three Faraday cups corresponding to $^{12}C^{16}O_2$, $^{13}C^{16}O_2$ and $^{12}C^{16}O^{18}O$, respectively. Isodat 3.0 software automatically computed the $^{13}C/^{12}C$ and of each sample peak, referenced to the standard CO_2 gas and its known $^{13}C/^{12}C$ content. The results were presented in per mil (‰) relative to the Vienna Peedee belemnite (V-PDB) standard. To ensure data comparability, the instrument was calibrated using the Indiana University F8 standard. This standard contains a mixture of eight methylated and ethylated fatty acids of known isotopic compositions. Additionally, quality control standards were intercalated every five or six injections to control the stability of the isotopic measurements over time. Instrumental precision and accuracy were better than ± 0.5‰. All lipid extracts were analysed in duplicate to ensure reproducibility and accuracy. Values were also corrected using mass balance to account for the methylation of the carboxyl group that occurs during acid extraction. Corrections were based on comparisons with a standard mixture of $C_{16:0}$ and $C_{18:0}$ fatty acids of known isotopic composition processed in each batch as a sample.

To investigate the presence of plant and aquatic biomarkers, acid extracts were further analysed on GC-MS (Agilent 7890A GC connected to an Agilent 5975C Inert XL mass-selective detector with a quadrupole mass analyser (Agilent Technologies, Cheadle, Cheshire, UK)) using a more polar column, DB23 (50%-cyanopropyl-methylpolysiloxane, 60 m × 0.250 mm × 0.25 µm; J&W Scientific, Folsom, CA, USA) and operated using the selected ion monitoring approach at BioArCh, University of York. The thermal protocol began at 50°C, holding for 2 min, before undergoing a series of temperature ramps: initially increasing at 10°C min^{-1} up to 100°C, then at 4°C min^{-1} to 140°C, decelerating to 0.5°C min^{-1} until reaching 160°C, and finally surging at 20°C min^{-1} to a peak of 250°C, where it was maintained for 10 min. The analysis specifically targeted four sets of ions, each representing particular compounds: the first set (m/z 74, 87, 213, 270) was indicative of the fragmentation patterns of 4,8,12-trimethyltridecanoic acid (TMTD); the second set (m/z 74, 88, 101, 312) matched with pristanic acid signatures; the third set (m/z 74, 101, 171, 326) was linked to phytanic acid [52] and the fourth set (m/z 74, 105, 262, 290, 318, 346) was identified with APAAs varying from C_{16} to C_{22} in carbon chain length [45,53]. Helium was used as the carrier gas at a consistent flow rate of 2.4 ml min^{-1}. The quantitative assessment of phytanic acid diastereomers was conducted by integrating the ion m/z 101. The $\delta^{13}C_{C16:0}$ and $\delta^{13}C_{C18:0}$ values of the ceramic sherds were compared against modern reference fats and oils for terrestrial animals, plants and aquatic organisms (supplementary material, SI1, dataset 3 [12]). Most reference fats and oils were corrected for the Suess effect. In cases where this had not been done previously, isotopic values were corrected based on the year of collection [54]. The GCMS files have been deposited in Zenodo, under the identifier https://doi.org/10.5281/zenodo.17490448.

2.6. Bulk collagen stable isotope analysis (elemental analysis–isotope ratio mass spectrometer)

Stable isotope analysis of bone collagen was performed at the Scottish Universities Environmental Research Centre, East Kilbride, UK, using an DELTA V Advantage continuous-flow IRMS coupled via a ConfloIV to an IsoLink elemental analyser (Thermo Scientific, Bremen, Germany), as described by Sayle

et al. [55]. Bone collagen (approx. 0.7 mg) was combusted in the presence of oxygen in a single reactor containing tungstic oxide and copper wires at 1020°C to produce N_2, CO_2 and SO_2. A magnesium perchlorate trap was used to eliminate water produced during the combustion process, and the gases were separated in a GC column heated between 70 and 240°C. Helium was used as a carrier gas throughout the procedure. N_2, CO_2 and SO_2 entered the MS via an open split arrangement within the ConfloIV and were analysed against their corresponding reference gases. The International Atomic Energy Agency (IAEA) reference materials USGS40 (L-glutamic acid, δ^{13}C (V-PDB) = –26.39 ± 0.04‰, δ^{15}N (AIR) = –4.52 ± 0.06‰) and USGS41a (L-glutamic acid, δ^{13}C (V-PDB) = 36.55 ± 0.08‰, δ^{15}N (AIR) = 47.55 ± 0.15‰) were used to normalize δ^{15}N and δ^{13}C values. Two in-house standards (GS2, δ^{34}S (V-CDT) = –10.28 ± 0.18‰ and GAS2, δ^{34}S (Vienna-Canyon Diablo Troilite; V-CDT) = 18.56 ± 0.10‰) that are calibrated to the IAEA reference materials IAEA-S-2 (silver sulfide, δ^{34}S (V-CDT) = 22.62 ± 0.08‰) and IAEA-S-3 (silver sulfide, δ^{34}S (V-CDT) = –32.49 ± 0.08‰) were used to normalize δ^{34}S values. Normalization was checked using the well-characterized elemental microanalysis IRMS fish gelatin standard B2215 (δ^{13}C (V-PDB) = –22.92 ± 0.10‰, δ^{15}N (AIR) = 4.26 ± 0.12‰, δ^{34}S (V-CDT) = 1.21 ± 0.24‰) and/or USGS88 (marine collagen, δ^{13}C (V-PDB) = –16.06 ± 0.07‰, δ^{15}N (AIR) = 14.96 ± 0.14‰, δ^{34}S (V-CDT) = 17.10 ± 0.44‰). Precision was determined to be ± 0.2‰ for δ^{15}N, ± 0.1‰ for δ^{13}C and ±0.4‰ for δ^{34}S, and is based on repeated measurements of elemental microanalysis IRMS fish gelatin standard B2215. In total, 19% of samples were analysed in duplicate.

2.7. Protein mass spectrometry data acquisition (liquid chromatography–tandem mass spectrometry)

An EvoSep One nanoflow Ultra Performance Liquid Chromatography (UPLC, EV-1000, EvoSep) with an EvoSep C18 Performance column (EV1109, 8 cm × 150 µm, 1.5 µm) using the 100 samples per day program (from EvoSep One HyStar Driver 2.4.125.0) was used for the chromatographic separation of the protein extracts, after which the samples were introduced into a trapped ion mobility spectrometry (timsTOF) HT MS (Bruker) with a CaptiveSpray ionization source. Compass HyStar (version 6.2, Bruker) was used to acquire positive parallel accumulation-serial fragmentation–data dependent acquisition (PASEF-DDA), electrospray ionization mass spectrometry (ESI-MS) and tandem mass spectrometry (MS2) spectra with the following instrument settings: instrument source: capillary voltage: 1600 V; dry gas: 3 l min^{-1}; dry temperature: 180°C. Mass range: m/z 100–1700. The timsTOF settings were: 1/K0 0.6–1.60 V s cm^{-2}; ramp time: 100 ms; ramp rate: 9.42 Hz. Data-dependent acquisition was performed with 10 PASEF ramps and a total cycle time of 1.17 s. An intensity threshold of 2500 and a target intensity of 20 000 were set with active exclusion applied for 0.4 min post precursor selection. Collision energy was interpolated between 20 eV at 0.6 V s cm^{-2} and 59 eV at 1.6 V s cm^{-2}.

2.8. Protein data analysis

All acquired mass spectra (.mgf files) were analysed using Orthrus (v. 1.0.0) [56,57]. Briefly, the non-enzymatic model provided by *Casanovo* was used for de novo sequencing to accommodate potential, non-specific cleavages caused by protein degradation over time. The entire reviewed Swiss-Prot database (the 2024_06 release) with relevant fish sequences from TrEMBL (n = 635 186 protein sequences) was used for protein shortlisting. For *Sage* database searching, a combined trypsin with chymotrypsin digestion pattern was configured (cleaving at KRFWY), with two missed cleavages. Variable modifications were deamidation (asparagine + glutamine), the oxidation of methionine and hydroxyproline, with a total of three modifications allowed per identification. The MS1 mass tolerance was 7 ppm, and the MS2 mass tolerance was 20 ppm. A joint *Mokapot* model was used to rescore all search outputs to mitigate challenges caused by low abundance and limited peptide-spectrum matches. Results were filtered by a false discovery rate threshold (mokapot q-value ≤ 0.05) and the number of peptides per protein per sample (greater than or equal to 2). Sequences detected in extraction blanks were excluded from taxonomic annotations. UniPept (v. 6.1.2) was used to assign taxa to sequences based on a lowest common ancestor algorithm. The National Center for Biotechnology Information (NCBI) Basic Local Alignment Search Tool (BLAST) was used to cross-check the taxonomic specificity of assigned peptides. Given the limited amount of preserved peptides per protein per sample, authentication was based on the presence of modified peptides. The palaeoproteomic data have been deposited in the ProteomeXchange Consortium via the PRIDE partner repository [58] under the identifier PDX068877.

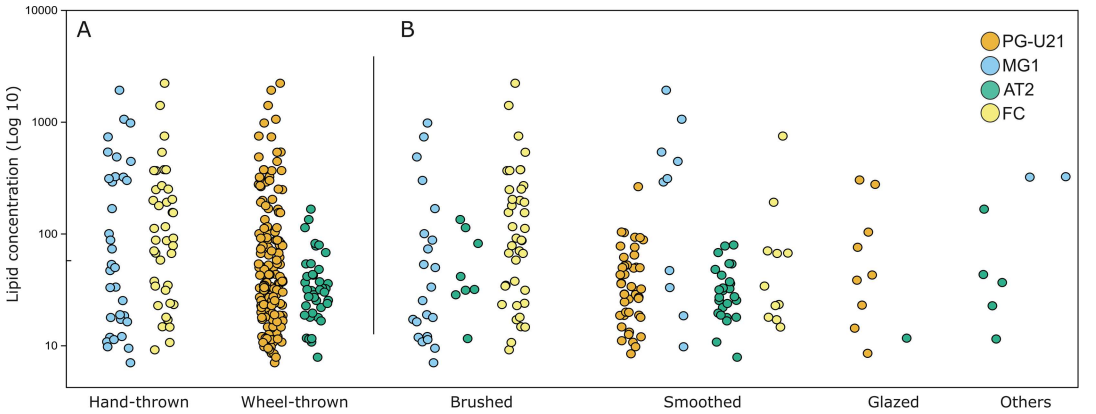

Figure 3. Lipid concentration (log10) according to (A) production type and (B) ceramic decoration. The category 'others' include corrugated and painted vessels, represented by a small number of samples.

3. Results

3.1. Lipid residue analysis

Interpretable amounts of lipids (greater than 5 µg g^{-1} [44,53]) were obtained from 93% (n = 169) of the samples following acidified methanol extraction, ranging from 7 to 2200 µg g^{-1}, with the highest and lowest concentrations found in sherds from FC and PG-U21, respectively (supplementary material, SI1_dataset 1). It is worth noting that the assemblage from FC consisted primarily of rims (75%) and body sherds (23%), whereas PG-U21 was dominated by body sherds (65%) and bases (28%). This variation in fragment distribution may account for some of the observed differences in lipid yields [59,60]. Lipid yields varied significantly by pottery production type ($p = 0.00018$, $U = 4695.5$, Mann–Whitney U test), with hand-thrown vessels from FC and MG1 containing substantially more lipids than wheel-thrown vessels from PG-U21 and AT2 (figure 3A). The only significant difference by decoration was observed at FC, where brushed vessels (n = 29) showed higher lipid concentrations than smoothed vessels (n = 11) (p = 0.028, H = 4.82, Kruskal–Wallis). Our results also indicate that glazed vessels can retain appreciable amounts of lipids, possibly owing to imperfections in the glazing surface [61]. A comparison between glazed (n = 9) and smoothed (n = 45) vessels from PG-U21 revealed no statistically significant differences in lipid concentrations (p = 0.324, U = 245.5, Mann–Whitney U test). Phthalates and petroleum-derived alkanes were found across all sites, probably resulting from post-excavation storage and handling conditions.

Acidified methanol extracts were dominated by saturated fatty acids ($C_{7:0}$ to $C_{26:0}$), of which palmitic ($C_{16:0}$) and stearic ($C_{18:0}$) were the most abundant. In total, 72% (n = 126) of the samples had a palmitic/stearic ratio (P/S = 1 .1 to 4.6) broadly consistent with the presence of aquatic resources (marine and freshwater) and plant oils [62,63]. The remaining samples, with P/S values as low as 0.4, might be consistent with ruminant adipose fats. P/S values, however, must be interpreted with caution, as they may be affected by degradation [64,65]. Myristic acid ($C_{14:0}$) and lauric acid ($C_{12:0}$) were detected in 86% of samples (n = 145 and 146 samples, respectively). While these compounds are present in animal fats, including dairy, pig and poultry [22,66,67], $C_{12:0}$ might also be indicative of plant oils [68,69]. Monounsaturated fatty acids, notably $C_{16:1}$ and $C_{18:1}$, were found in all samples. Polyunsaturated fatty acids ($C_{16:2}$ and $C_{18:2}$) were detected in 41% of samples (n = 69), including 34 samples from PG-U21 (63%), 19 from AT2 (46%), 15 from FC (37%) and only one sample from MG1 (3%). Short- (C_{16}) and long-chain alkanes (C_{22}) were found in two samples from FC. Alkanols were identified in 40% (n = 14) of the acidified methanol extracts following trimethylsilylation. Appreciable amounts of β- and α-amyrin and lupeol were also identified in two samples from FC and one from MG1. These pentacyclic triterpenoids can be found in a variety of plants [70,71], including fruit cuticular waxes [72]. In MG1, α-amyrin was identified in one sample (TRD_1305) exhibiting an n-alkanol distribution dominated by n-dotriacontanol, consistent with C_4 grasses [73,74]. While n-dotriacontanol is present across all panicoid grasses, its relative abundance may serve as a diagnostic indicator for the presence of maize [75].

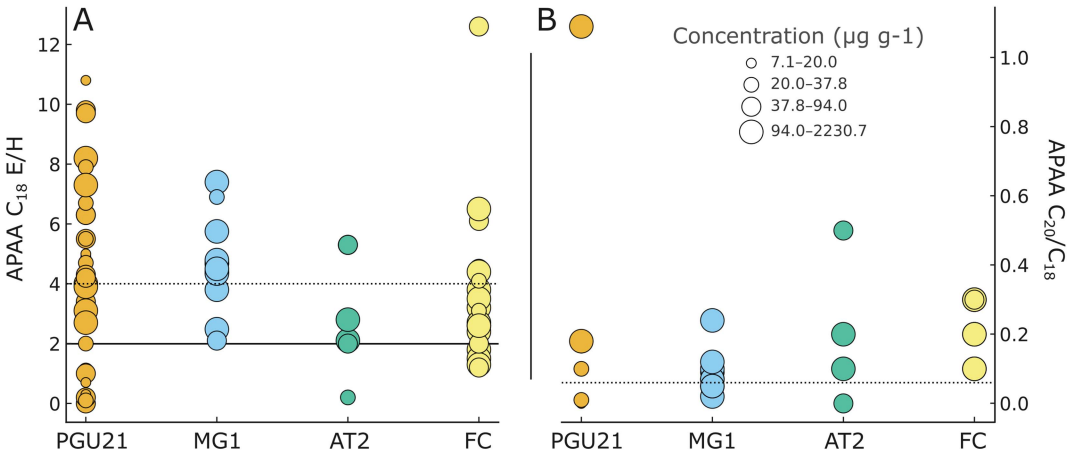

Figure 4. (A) Presence of APAA-C_{18} E/H and (B) APAA-C_{20}/C_{18} plotted against the lipid concentration. Dotted lines indicate thresholds above which cereals/fruits/non-leafy vegetables (greater than 4) and aquatic oils (greater than 0.06) are typically assigned. The continuous line in (A) represents the threshold below which leafy plants, and also beans and manioc, could be tentatively assigned (less than 2).

Degradation products such as short-chain dicarboxylic acids (C_9–C_{11}) were detected in 80% ($n = 121$) of samples. Monomethyl-branched fatty acids ($C_{15:0Br}$ and $C_{17:0Br}$) were detected in 83% ($n = 152$) of the samples. These latter compounds can originate from microbial activity in ruminants [66,76–78]; however, they may also derive from soil bacteria [78,79] or even from non-ruminant animals [80]. Their co-occurrence with other lipid markers of animal fats is consistent with, but not diagnostic of, the processing of ruminant products. Considering the three sites together, triacylglycerols, diacylglycerols and monoacylglycerols were found in 10% ($n = 8$), 21% ($n = 14$) and 67% ($n = 45$) of all solvent-extracted samples ($n = 75$), respectively, but could not be assigned to specific sources [66,76,78]. Trace amounts of oxidation products of coniferous resins (dehydroabietic acid and 7-oxo-dehydroabietic acid) were identified in 80% ($n = 135$) of the samples, in association with animal fats and other organic residues, including aquatic biomarkers.

The heating of animal and plant products is alluded to by the presence of ω-(*o*-alkylphenyl) octadecanoic acids (APAA-C_{18}) in 40% ($n = 70$) of the acidified methanol extracts. This thermal alteration marker was most abundant at PG-U21 ($n = 32$, 59%) and FC ($n = 21$, 53%), with lower frequencies at MG1 ($n = 12$, 35%) and AT2 ($n = 7$, 17%) (figure 4A). These compounds do not occur naturally and are formed from the protracted heating of mono- and poly-unsaturated fatty acids over 200°C [45,46]. Although they are commonly associated with the processing of plant and animal products, they may also form during the application of organic materials for post-firing treatments [80]. The ratio of the E and H isomers (E/H), where E isomers have an alkyl side-chain length of four and H isomers have one, has been used to broadly categorize the source of the fatty acids [45]. For example, E/H ratios of APAA-C_{18} have been used to distinguish broad food categories (e.g. cereals/fruits, leafy vegetables and animal products) under experimental and archaeological conditions [45]. However, E/H is also influenced by temperature, mixing and degradation, and therefore cannot be considered diagnostic on its own, and is better interpreted in combination with other molecular proxies (e.g. APAAs C_{20}/C_{18}) and fatty acid $\delta^{13}C$ values. Ratios of the APAA-C_{18} E/H isomers greater than 4 were detected at all sites, particularly from PG-U21 ($n = 19$, 35%) and MG1 ($n = 9$, 27%), which may be indicative of cereals, including, for example, maize and rice [12,81], which were commonly consumed in the region, but also other non-leafy vegetables and fruits [45]. Lower E/H values (less than 2) could tentatively be linked to other plants also cultivated in the region, such as manioc and beans, which present E/H ranging from 1.0 to 2.7 ($n = 2$) and 0.8 to 1.3 ($n = 4$), respectively (supplementary material, SI1, dataset 2). APAA-C_{18} E/H isomers less than 2 were found in PG-U21 ($n = 7$, 13%) and FC ($n = 4$, 10%), and to a much lesser extent at AT2 ($n = 1$, 2.4%).

Among samples containing APAA-C_{18}, 16% ($n = 27$) presented trace amounts of APAA-C_{20}, which are mostly formed from heating aquatic oils at high temperature, and to a lesser extent, terrestrial plants and ruminants [46,53]. The ratio APAA-C_{20}/C_{18} has been more confidently used to distinguish between terrestrial and aquatic products, with an APAA-C_{20}/C_{18} > 0.06 indicative of aquatic resources [82].

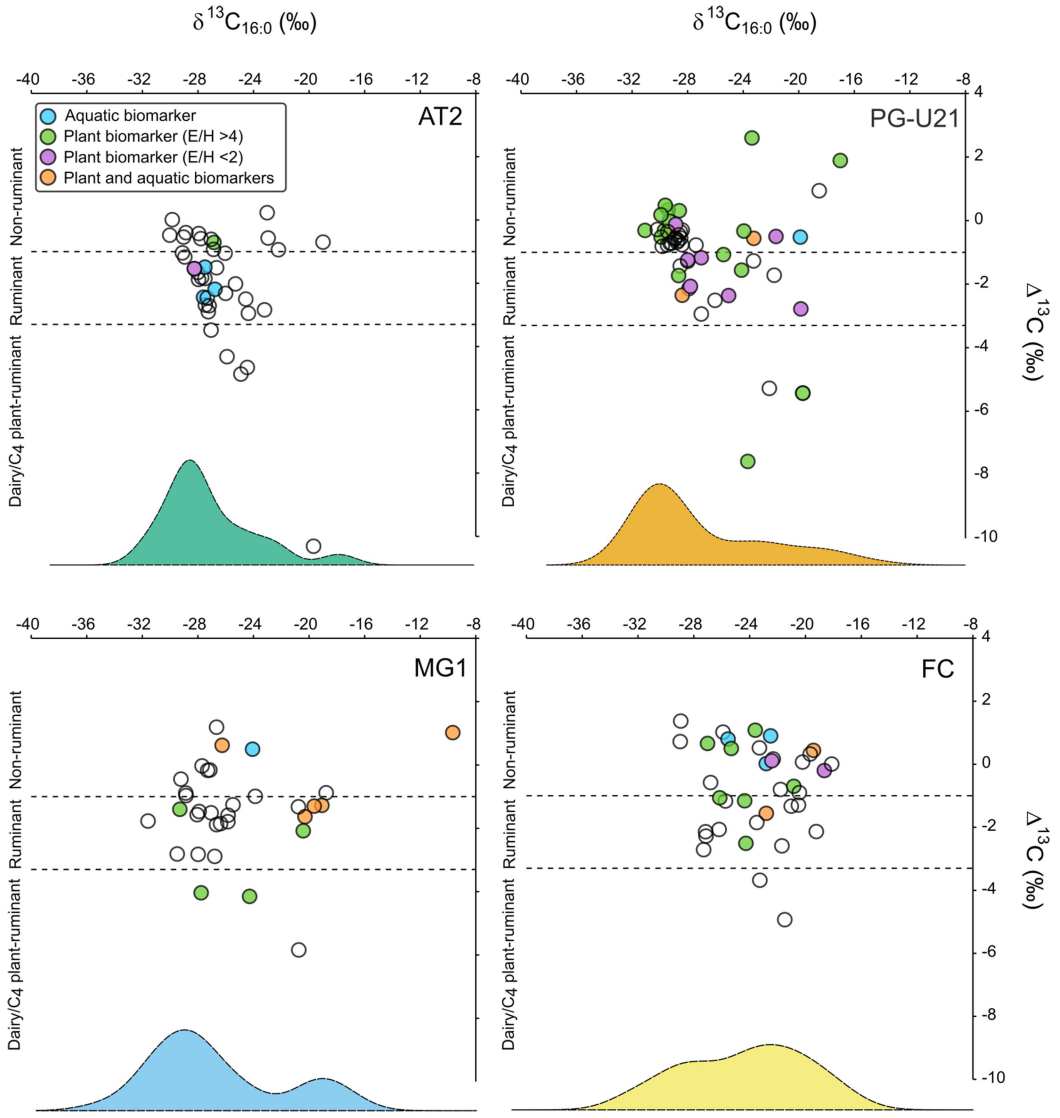

Figure 5. $\Delta^{13}C_{18:0\text{-}16:0}$ values against $\delta^{13}C_{16:0}$. Plant biomarkers are represented by APAA-C_{18} E/H > 4 and < 2; aquatic biomarkers are represented by APAA-C_{20}/C_{18} > 0.06. Kernel distribution of $\delta^{13}C_{16:0}$ values shows increased proportion of C_4 plant/marine lipids from AT2 to FC.

APAA-C_{20}/C_{18} > 0.06 was detected in 11.2% (n = 19) of the samples, the majority of which were from MG1 (n = 6, 17.6%) and FC (n = 6, 15.0%), followed by PG-U21 (n = 4, 7.4%) and AT2 (n = 3, 7.3%) (figure 4B). The presence of isoprenoid acids TMTD, pristanic acid and SRR diastereomer of phytanic acid greater than 75.5% provide further support for aquatic resource processing in the vessels [52]. SRR > 75.5% was observed at FC (n = 9, 22.5%), PG-U21 (n = 12, 22.2%) and MG1 (n = 7, 20.6%), but to a much lesser extent at AT2 (n = 1, 2.4%). In conclusion, if we consider APAA-C_{20}/C_{18} > 0.06 as the most conservative aquatic biomarker in our record, AT2, along with PG-U21, is one of the sites with the lowest frequency of aquatic products. If we expand the aquatic biomarkers to include SRR > 75%, AT2 still features as the site with the lowest frequency of fish processing (n = 4, 10%), followed by PG-U21 (n = 15, 28%), MG1 (n = 11, 32%) and FC (n = 14, 35%).

3.2. Stable isotope analysis

Differences between sites are evident by plotting $\delta^{13}C_{16:0}$ against $\Delta^{13}C_{18:0\text{–}16:0}$ (figure 5). The narrower range of $\delta^{13}C_{16:0}$ values at AT2 is consistent with fats and oils from C_3 plants and C_3-fed animals. A few samples from AT2 (n = 5, 12.2%), MG1 (n = 3, 11.8%), PG-U21 (n = 3, 7.4%) and FC (n = 2, 5%) plot

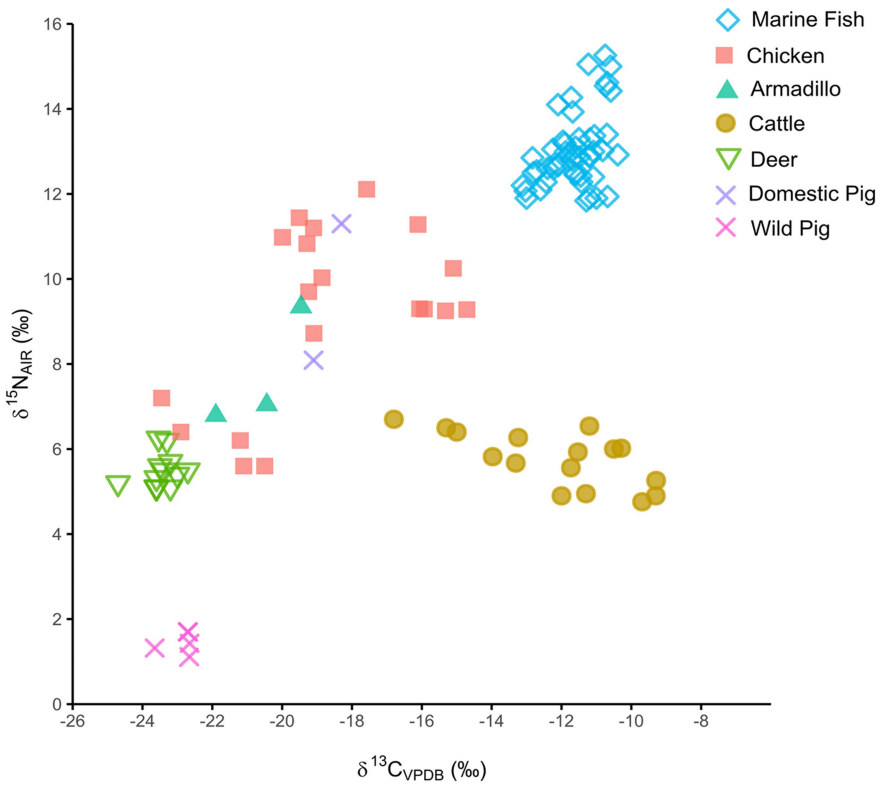

Figure 6. $\delta^{13}C$ and $\delta^{15}N$ values of faunal remains from historic sites MG1 and PG-U21.

within the ruminant dairy region ($\Delta^{13}C_{18:0\text{–}16:0} < -3.3‰$), consistent with previously published data from animals with mixed C_3–C_4 diets [83]; alternatively, these are explained by a mixture of ruminant carcass fat and maize kernel oil, as previously modelled for ceramic vessels in these coastal areas during pre-contact times [12]. By contrast, the spread of $\delta^{13}C_{16:0}$ and $\Delta^{13}C_{18:0\text{–}16:0}$ values in MG1 and FC indicates that these wares were mostly used for processing mixed terrestrial and marine/C_4 plant resources. Samples with high $\delta^{13}C_{16:0}$ and $\delta^{13}C_{18:0}$ values that contained *n*-alkanols did not exhibit distributions characteristic of C_4 grasses. Moreover, their relatively high lipid concentrations could be indicative of terrestrial or aquatic animal fats and oils mixed with plants. Resolving the isotopic equifinality of marine resources and C_4 plants in ceramic vessels, therefore, remains complex. This is further exacerbated by the overlapping $\delta^{13}C$ values between brackish-marine fish and some terrestrial animals.

The bulk collagen $\delta^{13}C$ and $\delta^{15}N$ values of cattle (*Bos taurus*) samples from PG-U21 and MG1 reflect diets dominated by C_4 plants, possibly maize, as is historically documented in the region [13,15,84]. Their $\delta^{13}C$ values are similar to those measured in brackish-marine fish (*Pogonias courbina, Micropogonias furnieri* and *Conodon nobilis*) from MG1 and PG-U21, which in turn originated from benthic microalgae and marine phytoplankton [85]. Collagen $\delta^{13}C$ and $\delta^{15}N$ values of chickens (*Gallus gallus*) and domestic pigs (*Sus scrofa domesticus*) show variable intake of marine fats and proteins. By contrast, the $\delta^{13}C$ and $\delta^{15}N$ values of native deer (*Mazama* sp.) and pig (Tayassuidae) from MG1 are consistent with C_3 plant-based diets. Armadillos (Dasypodidae) from PG-U21 had $\delta^{13}C$ and $\delta^{15}N$ values reflecting an omnivore diet (supplementary material, SI1, dataset 4; figure 6). As a consequence, some of the high $\delta^{13}C_{16:0}$ and $\delta^{13}C_{18:0}$ values in ceramic vessels may also be explained by the processing of these animals.

3.3. Protein residue analysis

In an attempt to obtain greater taxonomic resolution, 38 ceramic samples were randomly selected for proteomics analysis from all sites (supplementary material, SI2, dataset 1). Despite the relatively recent age of the pottery and the successful recovery of lipids, the preservation of proteins was much more limited. Although a range of aquatic, plant, mammalian and microbial peptides were initially identified, the majority of these are probably modern contaminants. After filtering out all peptides assigned to common contaminants, three proteins could be securely identified, meeting an acceptable threshold of having at

Table 2. Average lipid concentration and the relative frequency of biomarkers. The heating of fats and oils at high temperatures (APAA-C_{18}), the heating of plants (APAA-C_{18} E/H isomers > 4 and < 2) and the processing of aquatic oils and fats (APAA-C_{20}/C_{18} > 0.06). The presence of marine products is alluded to by SRR > 75%. $\Delta^{13}C_{18:0-16:0}$ values below −3.3‰ indicate either the processing of C_3/C_4 dairy products or the mixing of ruminant fats with C_4 plants.

site	lipid yield ($\mu g\ g^{-1}$)	APAA-C_{18} (%)	E/H > 4 and < 2 (%)	APAA-C_{20}/C_{18} > 0.06 (%)	SRR > 75% (%)	$\Delta^{13}C_{18:0-16:0}$ < −3.3‰ (%)
AT2	41.15 ± 33.73	17.1	7.3	7.3	2.4	12.2
PG-U21	54.46 ± 62.17	59.3	46.3	7.4	22.2	7.4
MG1	235.32 ± 412.80	35.3	26.5	17.6	20.6	11.8
FC	252.42 ± 341.07	52.5	27.5	15.0	22.5	5

least two distinct and specific peptides [86]. These included the identification of two fish proteins belonging to the Sciaenidae family (drums and croakers) from one vessel from FC (TRD-487) and one from PG-U21 (TRD-1516), and peptides corresponding to chicken vitellogenin, a component of egg yolk, in a vessel from PG-U21 (TRD-1498) (supplementary material, SI2,datasets 2 and 3). Interestingly, none of the vessels with fish-derived proteins contained APAAs indicative of aquatic resources, perhaps as the latter are formed by protracted heating, which is detrimental to protein survival. Therefore, marine proteins would only be preserved in vessels subjected to mild or no heating, while APAAs would only be formed in vessels heated to high temperatures (over 200°C) for prolonged periods. The isotope values for sample TRD-1516 (PG-U21), which contains fish proteins, suggest a mixture of terrestrial and aquatic oils. The SRR is also high (approx. 70%), a level typically observed in marine organisms [52].

4. Discussion

The late nineteenth and early twentieth centuries represented a key turning point for food production and consumption habits in southern Brazil, marked by economic and demographic shifts driven by nationalistic political agendas, industrialization and the rise of a middle class. In Babitonga Bay, Luso-Brazilians, African and Afro-descendants, Indigenous and recently established European immigrants intensified agricultural production in an area previously dominated by small-scale, household agriculture and fishing. Through subsistence and incipient market-oriented production, these groups cultivated manioc, rice, beans, maize, sugarcane, peanuts and raised domestic animals (cattle, pigs and chicken), along with fishing and hunting wildlife [15,34,87]. In this context, food-derived molecules preserved in ceramic artefacts provide valuable insight into how broad food categories were distributed and consumed among different groups in the region (table 2).

Our results indicate that residents of AT2 predominantly used wheel-thrown ceramic vessels to process/store terrestrial C_3 plants and animals, consistent with historical records of agriculture and livestock production among recently established German immigrants. Many samples show low carbon isotope values of fatty acids, which are plausibly linked to the processing of C_3 plants. Nevertheless, heating markers were identified in only 17% of the samples, suggesting that many vessels were either used for cooking at low temperatures or not used for cooking at all, but rather for serving or storing food and liquids. This is also supported by the generally low lipid concentrations in these vessels. Among all the sites, AT2 exhibited the highest relative proportion of samples (12%) with stable carbon isotope values suggestive of the storage or processing of dairy products in these vessels. Conversely, it yielded the lowest number of samples associated with the processing of aquatic resources, suggesting that fish may have been only occasionally cooked in a limited number of vessels.

Lipid analysis revealed that C_3 and C_4 plants (e.g. maize), and animals, including marine products, were part of the cuisine of residents at PG-U21, who also used wheel-thrown cooking wares. Nearly 50% of the samples contained plant biomarkers associated with cereals (e.g. maize and rice), non-leafy vegetables and fruits, possibly including beans and manioc. Manioc cultivation and processing, in particular, is documented at PG-U21 by the presence of domestic milling and combustion facilities. Zooarchaeological data from PG-U21 show the presence of cattle and sheep, which were probably used

for traction and meat, as dairy products appear to be poorly represented in the analysed vessels. Fish, including shorthead drum (*Larimus breviceps*), whitemouth croaker (*M. furnieri*) and black drum (*P. courbina*), chickens and native mammals, such as armadillo and pig, contributed substantially to the overall economy [34], a pattern further supported by protein evidence indicating the cooking of fish and chicken in some pots. As demonstrated here, food proteins can survive within ceramic matrices even in hot and humid environments, although their preservation was limited despite the relatively recent age of these contexts. Compared with lipids, protein survival and subsequent extraction from ceramic matrices are poorly understood [88–90], and the greatest levels of success tend to derive from specific environmental (cold) or contextual (dairying and limescale) circumstances, which enhance the chances of protein preservation, and when actual food crusts, the solidified and often charred residues of food items, are present [88]. Protein identification was further hampered owing to the incomplete representation of protein sequences available in public databases (particularly for regionally specific plant and animal species), the high presence of conserved peptides across different species, and extensive degradation.

Hand-thrown vessels from MG1 and particularly FC were characterized by a high lipid content, the presence of both plant and aquatic biomarkers and the general tendency toward ^{13}C-enriched fatty acids. This suggests that aquatic animals (e.g. fish and shellfish) and C_4 plants/animals played a particularly important role in both sites. For MG1, the results are supported by faunal analyses, which identified fish, chicken and livestock (with C_4 diet), along with a broader range of native species (deer, lowland paca, armadillo, opossum, dolphin and whale) being exploited at the site [34]. For FC, shellfish remains, fish otoliths and bones (catfish and whitemouth croaker), along with fishing implements, also support our conclusions that fishing was locally important [41].

The widespread occurrence of oxidation products from coniferous resins suggests resins were intentionally applied as waterproofing or sealing agents by these communities [91]. Although coniferous trees producing abietane diterpenoids were introduced to southern Brazil in the second half of the nineteenth century, their large-scale expansion occurred primarily from the 1950s onward [92]; thus, it is possible that such resins were secured mostly through long-distance markets. It is also improbable that these compounds derived from wood used as fuel [93], as exotic coniferous trees were unlikely to have been sufficiently abundant to serve as a significant source of firewood. Nevertheless, because these compounds can also be produced by cyanobacteria from marine and estuarine environments [94], a post-depositional origin cannot be entirely ruled out.

Our results are supported by historical and archaeological sources that emphasize the importance of crops (e.g. manioc and maize), along with the consumption of fish, domesticates and wild animals in late nineteenth and early twentieth century Babitonga Bay [15,34]. At the same time, they reveal previously undocumented contrasts in food practices, expressed in the variable proportions of C_3 plants and marine resources processed in ceramic vessels. C_3 plants appeared to have been more prominent among groups using wheel-thrown wares, who may have had more reliable access to livestock and cultivated crops within a market-oriented economy (AT2 and PG-U21).

Nevertheless, the extent to which aquatic resources were processed at PG-U21 remains ambiguous and heavily contingent upon the criteria used for aquatic identification. Maintaining a conservative approach (APAA-$C_{20}/C_{18} > 0.06$) suggests that aquatic products did not dominate the culinary practices involving ceramic wares, even though fish and shellfish were the most abundant faunal remains at the site. When expanding the aquatic biomarker toolkit (SRR > 75%), however, the aquatic context at PG-U21 becomes similar to that of MG1 and FC. It is possible that the fish remains at PG-U21 reflect catches destined for local and external markets [34]. Moreover, ceramic vessels only partially capture the full diversity of household food preparation. Alternative cooking methods, such as grilling or the use of non-ceramic utensils (e.g. metalware), may have been common. Arguably, this consideration extends beyond PG-U21 and should be expected across all sites in the region.

The processing of marine foods in ceramic vessels appears to have been more central to the culinary practices of groups who produced or acquired hand-thrown wares displaying Indigenous, African and European stylistic elements. Fish and shellfish may have been more accessible to lower-status groups than livestock products and certain cultivated crops, which were more directly governed by restrictive private property regimes and a formal market economy. Conversely, a higher reliance on coastal habitats characterized by relatively open access would have allowed these households to bypass the market to meet their subsistence needs. In this light, the exploitation of coastal resources could have served as a critical strategy for buffering food insecurity among poorer households within the social and economic inequalities of early modern Brazil.

5. Conclusion

Cuisine and food habits in Brazil have changed dramatically over recent decades. However, the extent of these transformations remains difficult to assess owing to limited information on domestic food consumption prior to the accelerated globalization of food systems in the twentieth and twenty-first centuries. Here, we contribute to addressing this gap by analysing lipids and proteins extracted from late nineteenth and early twentieth century cooking wares from Babitonga Bay, southern Brazil. These artefacts were recovered from rural residential sites associated with Luso-Brazilian, Indigenous and Afro-descendant communities (FC, MG1 and PG-U21), as well as German immigrants (AT2). Our results reveal considerable variability in processed resources. Notably, hand-thrown vessels with Indigenous, African and European influences contained higher lipid concentrations and comparatively greater proportions of C_4 plant and aquatic products than wheel-thrown wares. These contrasts are most evident when comparing ceramics from MG1 and FC, probably used by African or Afro-descendant groups, with those from AT2 associated with German residents who appear to have relied more heavily on C_3 plants. We acknowledge that other forms of food processing existed in the past and that our study captures only those practices associated with the use of ceramic cooking wares. Nevertheless, within the sphere of ceramic use, distinct culinary practices and food habits appear to have coexisted in Babitonga Bay in the late nineteenth and early twentieth centuries. Our results suggest that access to and use of food resources may have been structured by ethnicity and social status. Perhaps not surprisingly, these same factors continue to shape food habits in Brazil today, potentially reflecting deeper historical legacies.

Ethics. Permits for exporting archaeological ceramic sherds for organic residue analysis and faunal remains for bone collagen isotope analysis were obtained from the Instituto do Patrimônio Histórico e Artístico Nacional (IPHAN, protocols 01510.000608/2021-80, 01512.000594/2020-01, 01510.000612/2020-67 and 01510.000422/2022-10).

Data accessibility. All data generated or analysed during this study are included in this published article (and its supplementary information files) and available in the following publicly accessible digital repository: the GC-MS files have been deposited in Zenodo (open to the public), under the identifier [96]. The palaeoproteomic data has been deposited to the ProteomeXchange Consortium via the PRIDE partner repository under the identifier PDX068877 and can be accessed using the following link: https://doi.org/10.6019/PXD068877.

Supplementary material is available online [97].

Declaration of AI use. AI was partially used for improving readability and language of the text, and to assist in debugging and refining R scripts.

Authors' contributions. A.D.M.: conceptualization, data curation, formal analysis, investigation, methodology, writing—original draft, writing—review and editing; M.A.: conceptualization, formal analysis, investigation, writing—review and editing; K.M.M.: data curation, formal analysis, methodology, writing—original draft, writing—review and editing; T.F.: data curation, formal analysis, investigation, writing—review and editing; Y.C.: data curation, formal analysis, methodology, writing—original draft, writing—review and editing; J.A.A.D.: conceptualization, formal analysis, investigation, methodology, writing—original draft, writing—review and editing; M.C.: formal analysis, writing—review and editing; N.M.-C.: formal analysis; J.V.: formal analysis; K.S.: formal analysis, writing—review and editing; D.D.B.: data curation, investigation, writing—review and editing; F.M.B.: data curation, investigation, writing—review and editing; M.C.A.: formal analysis, investigation, writing—review and editing; M.C.: conceptualization, data curation, formal analysis, funding acquisition, investigation, methodology, project administration, supervision, writing—original draft, writing—review and editing; O.E.C.: conceptualization, data curation, formal analysis, funding acquisition, investigation, methodology, project administration, supervision, writing—original draft, writing—review and editing; A.C.C.: conceptualization, data curation, formal analysis, funding acquisition, investigation, methodology, project administration, supervision, writing—original draft, writing—review and editing.

All authors gave final approval for publication and agreed to be held accountable for the work performed therein.

Conflict of interest declaration. We declare we have no competing interests.

Funding. This work was funded by the European Union's Horizon 2020 research and innovation programme under grant agreement ChemArch no. 956351. This work was also funded by the ERC Consolidator project TRADITION, which has received funding from the European Research Council (ERC) under the European Union's Horizon 2020 research and innovation programme under grant agreement no. 817911. This work contributes to EarlyFoods, which has received funding from the Agència de Gestió d'Ajuts Universitaris i de Recerca de Catalunya (SGR-Cat 2021, 00527). This work contributes to ICTA-UAB 'María de Maeztu' Programme for Units of Excellence of the Spanish Ministry of Science, Innovation and Universities (CEX2024-001506-M funded by MICIU/AEI/10.13039/501100011033).

References

1. da Câmara Cascudo L. 1967 *História da alimentação no Brasil. Brasiliana, 323, Cardapio Indígena, Dieta Africana, Ementa Portuguésa, Primeiro Volume*. São Paulo, Companhia Editora Nacional.

2. Fish WR. 1978 Changing food use patterns in Brazil. *Luso-Braz. Rev.* **15**, 69–89.
3. Dean W. 1997 *With broadax and firebrand: the destruction of the Brazilian Atlantic forest*. Berkeley, CA: University of California Press.
4. Silva Júnior AE, de Oliveira ADS, Praxedes DRS, da Costa Paula DT, de Lima Macena M, de Menezes Toledo Florêncio TM, Clemente APG, Bueno NB. 2023 Social and racial disparities in food consumption among Brazilian college students: a nationwide study. *J. Racial Ethn. Health Disparities* **10**, 2630–2640. (doi:10.1007/s40615-022-01441-0)
5. Costa JC, da Silva de Jesus AC, de Jesus JGL, Madruga MF, Souza TN, Louzada MLDC. 2023 Differences in food consumption of the Brazilian population by race/skin color in 2017-2018. *Rev. Saude Publica* **57**, 4. (doi:10.11606/s1518-8787.2023057004000)
6. Oths KS, Carolo A, Dos Santos JE. 2003 Social status and food preference in southern Brazil. *Ecol. Food Nutr.* **42**, 303–324. (doi:10.1080/03670240390247797)
7. Bezerra IN, Sichieri R. 2011 Household food diversity and nutritional status among adults in Brazil. *Int. J. Behav. Nutr. Phys. Act.* **8**, 22. (doi:10.1186/1479-5868-8-22)
8. Cattafesta M, Petarli GB, da Luz TC, Zandonade E, de Paula Alves Bezerra OM, Salaroli LB. 2020 Dietary patterns of Brazilian farmers and their relation with sociodemographic, labor, and lifestyle conditions. *Nutr. J.* **19**, 23. (doi:10.1186/s12937-020-00542-y)
9. Levy-Costa RB, Sichieri R, Pontes N dos S, Monteiro CA. 2005 Household food availability in Brazil: distribution and trends 1974-2003. *Rev. Saude Publica* **39**, 530–540. (doi:10.1590/S0034-89102005000400003)
10. Monteiro Dos Santos JE, Crispim SP, Murphy J, de Camargo Cancela M. 2021 Health, lifestyle and sociodemographic characteristics are associated with Brazilian dietary patterns: Brazilian National Health Survey. *PLoS One* **16**, e0247078. (doi:10.1371/journal.pone.0247078)
11. Toso A *et al.* 2021 Fishing intensification as response to Late Holocene socio-ecological instability in southeastern South America. *Sci. Rep.* **11**, 23506. (doi:10.1038/s41598-021-02888-7)
12. Admiraal M *et al.* 2023 Chemical analysis of pottery reveals the transition from a maritime to a plant-based economy in pre-colonial coastal Brazil. *Sci. Rep.* **13**, 16771. (doi:10.1038/s41598-023-42662-5)
13. Cabral OR. 1937 *Santa Catharina: História - evolução. Série 5, 80, Brasiliana. Bibliotheca Pedagogica Brasileira*. São Paulo, Brazil: Companhia Editora Nacional.
14. Noble AG. 1967 Geographical aspects of the agriculture of Santa Catarina state, Brazil. *Ohio J. Sci.* **67**, 257–273.
15. Saint-Hilaire A de. 1936 *Viagem à Província de Santa Catarina 1820 (voyage dans la Province de Sainte-Catherine). Série 5, 58, Brasiliana. Bibliotheca Pedagogica Brasileira*. São Paulo, Brazil: Companhia Editora Nacional.
16. Sandoval Gallardo S, Fossile T, Herbst DF, Begossi A, Silva LG, Colonese AC. 2021 150 years of anthropogenic impact on coastal and ocean ecosystems in Brazil revealed by historical newspapers. *Ocean Coast. Manag.* **209**, 105662. (doi:10.1016/j.ocecoaman.2021.105662)
17. Herbst DF, Rampon J, Baleeiro B, Silva LG, Fossile T, Colonese AC. 2023 180 years of marine animal diversity as perceived by public media in southern Brazil. *PLoS One* **18**, e0284024. (doi:10.1371/journal.pone.0284024)
18. Borba FM. 2013 Arqueologia da escravidão numa vila litorânea: vestígios negros em fazendas oitocentistas de São Francisco do Sul (Santa Catarina). *Rev. Confluências Cult.* **2**, 71. (doi:10.21726/rccult.v2i2.31)
19. Symanski LCP. 2010 Cerâmicas, identidades escravas e crioulização nos engenhos de Chapada dos Guimarães (MT). *Hist. Unisinos* **14**, 294–310. (doi:10.4013/htu.2010.143.06)
20. Symanski L, de Souza MAT. 2007 O registro arqueológico dos grupos escravos: questões de visibilidade e preservação. *Estud. Demogr.* **33**, 215–243.
21. Craig OE. 2021 Prehistoric fermentation, delayed-return economies, and the adoption of pottery technology. *Curr. Anthropol.* **62**, S233–S241. (doi:10.1086/716610)
22. Evershed RP. 2008 Organic residue analysis in archaeology: the archaeological biomarker revolution. *Archaeometry* **50**, 895–924. (doi:10.1111/j.1475-4754.2008.00446.x)
23. Cramp LJE, Evershed RP. 2014 Reconstructing aquatic resource exploitation in human prehistory using lipid biomarkers and stable isotopes. In *Treatise on geochemistry*, pp. 319–339. Amsterdam, The Netherlands: Elsevier.
24. Dudd SN, Evershed RP. 1998 Direct demonstration of milk as an element of archaeological economies. *Science* **282**, 1478–1481. (doi:10.1126/science.282.5393.1478)
25. Buckley M, Melton ND, Montgomery J. 2013 Proteomics analysis of ancient food vessel stitching reveals >4000-year-old milk protein: proteomics analysis of ancient food vessel stitching. *Rapid Commun. Mass Spectrom.* **27**, 531–538. (doi:10.1002/rcm.6481)
26. Chowdhury MP, Campbell S, Buckley M. 2021 Proteomic analysis of archaeological ceramics from Tell Khaiber, southern Iraq. *J. Archaeol. Sci.* **132**, 105414. (doi:10.1016/j.jas.2021.105414)
27. Hendy J *et al.* 2018 Ancient proteins from ceramic vessels at Çatalhöyük West reveal the hidden cuisine of early farmers. *Nat. Commun.* **9**, 4064. (doi:10.1038/s41467-018-06335-6)
28. Solazzo C, Fitzhugh WW, Rolando C, Tokarski C. 2008 Identification of protein remains in archaeological potsherds by proteomics. *Anal. Chem.* **80**, 4590–4597. (doi:10.1021/ac800515v)
29. Colonese AC *et al.* 2014 Long-term resilience of late holocene coastal subsistence system in southeastern South America. *PLoS ONE* **9**, e93854. (doi:10.1371/journal.pone.0093854)
30. Hansel FA, Evershed RP. 2009 Formation of dihydroxy acids from Z-monounsaturated alkenoic acids and their use as biomarkers for the processing of marine commodities in archaeological pottery vessels. *Tetrahedron Lett.* **50**, 5562–5564. (doi:10.1016/j.tetlet.2009.06.114)

31. Lantos I, Chaile C, Careaga VP, de Salazar L, Maier MS. 2024 Organic residue analysis in Latin American archaeology: past, present, and future perspectives. *Archaeometry* **67**, 503—519. (doi:10.1111/arcm.12978)
32. Admiraal M, Colonese AC, Milheira RG, Di Muro A, Talbot HM, Lucquin A, Craig OE. 2025 Feasting on fish: specialized function of pre-colonial pottery of the Cerritos mound builders of southern Brazil. *PLoS ONE* **20**, e0311192. (doi:10.1371/journal.pone.0311192)
33. Prévost C, Drieu L, Pasqualini A, Regert M. 2023 The ARchaeological Organic residues Literature Database (AROLD): construction of a tool for reviewing and querying published lipid data in organic residue analysis. *Archaeometry* **65**, 1125–1143. (doi:10.1111/arcm.12869)
34. Fossile T *et al.* 2023 Long-term perspective on fishing and mammal defaunation in the Atlantic forest coast of Brazil using archaeological faunal remains. *Trop. Conserv. Sci.* **16**, 19400829231218419. (doi:10.1177/19400829231218419)
35. Alves MC, Oliveira MSC. 2002 Salvamento Arqueológico do Sítio Histórico Praia Grande - Unidade 21, em São Francisco do Sul - SC.
36. de Farias DSE. 2012 Programa de educação patrimonial e valorização arqueológica na área de implantação do centro de distribuição e no terminal marítimo Mar Azul, município de São Francisco do Sul, SC.
37. Alves MC. 2017 Farinheiros e pescadores do interior da Ilha de São Francisco do Sul (SC). In *Patrimônio Cultural de São Francisco do Sul a partir da pesquisa em Arqueologia Histórica* (eds D da Rocha Bandeira, FM Borba, MC Alves), pp. 101–121. Joinville, Brazil: Editora Univille.
38. Borba FM. 2017 Vestígios materiais da presença Africana e Afrodescendente em São Francisco do Sul. In *Patrimônio Cultural de São Francisco do Sul a partir da pesquisa em Arqueologia Histórica* (eds D da Rocha Bandeira, FM Borba, MC Alves), pp. 70–100. Joinville, Brazil: Editora Univille.
39. Chmyz I *et al.* 1976 Terminologia arqueológica brasileira para a cerâmica. *Cadernos de arqueologia* **1**, 119–148.
40. Silva OP da. 2001 Salvamento Arqueológico dos Sítios Morro Grande 1, Morro Grande 2 e Morro Grande 3. *Relatório de Pesquisa. Florianópolis, Vega do Sul.*
41. Borba FM. 2014 A cultura material das populações africanas e afrodescendentes em coleções arqueológicas da baía Babitonga (Santa Catarina): usos e práticas negras no passado. Joinville, Brazil: Universidade da Região de Joinville. http://snh2015.anpuh.org/resources/anais/39/1433956611_ARQUIVO_SNH2015TextoCompleto-FernandaMaraBorba.pdf.
42. da Rocha Bandeira D, Alves MC. 2017 O patrimônio arqueológico histórico de São Francisco do Sul: contribuição à memória com base na cultura material. In *Patrimônio Cultural de São Francisco do Sul a partir da pesquisa em Arqueologia Histórica* (eds D da Rocha Bandeira, FM Borba, MC Alves), pp. 7–22. Joinville, Brazil: Editora Univille.
43. da Rocha Bandeira D. 2001 *Pesquisa de salvamento arqueológico do sítio histórico Foz do Cubatão*. Joinville, Brazil: Relatório Final.
44. Craig OE *et al.* 2013 Earliest evidence for the use of pottery. *Nature* **496**, 351–354. (doi:10.1038/nature12109)
45. Bondetti M, Scott E, Courel B, Lucquin A, Shoda S, Lundy J, Labra-Odde C, Drieu L, Craig OE. 2021 Investigating the formation and diagnostic value of ω-(o-alkylphenyl)alkanoic acids in ancient pottery. *Archaeometry* **63**, 594–608. (doi:10.1111/arcm.12631)
46. Hansel FA, Copley MS, Madureira LAS, Evershed RP. 2004 Thermally produced ω-(o-alkylphenyl)alkanoic acids provide evidence for the processing of marine products in archaeological pottery vessels. *Tetrahedron Lett.* **45**, 2999–3002. (doi:10.1016/j.tetlet.2004.01.111)
47. Dunne J, Evershed RP, Salque M, Cramp L, Bruni S, Ryan K, Biagetti S, di Lernia S. 2012 First dairying in green Saharan Africa in the fifth millennium BC. *Nature* **486**, 390–394. (doi:10.1038/nature11186)
48. Ambrose SH. 1990 Preparation and characterization of bone and tooth collagen for isotopic analysis. *J. Archaeol. Sci.* **17**, 431–451. (doi:10.1016/0305-4403(90)90007-R)
49. Szpak P, Metcalfe JZ, Macdonald RA. 2017 Best practices for calibrating and reporting stable isotope measurements in archaeology. *J. Archaeol. Sci.: Reports* **13**, 609–616.
50. Dekker J, Ramsøe M. 2025 DES foodcrust protein extraction protocol. *protocols.io* (doi:10.17504/protocols.io.j8nlk9r6wv5r/v1)
51. Chowdhury M, Makarewicz C, Piezonka H, Buckley M. 2022 Novel deep eutectic solvent-based protein extraction method for pottery residues and archeological implications. *J. Proteome Res.* **21**, 2619–2634. (doi:10.1021/acs.jproteome.2c00340)
52. Lucquin A, Colonese AC, Farrell TFG, Craig OE. 2016 Utilising phytanic acid diastereomers for the characterisation of archaeological lipid residues in pottery samples. *Tetrahedron Lett.* **57**, 703–707. (doi:10.1016/j.tetlet.2016.01.011)
53. Evershed RP, Copley MS, Dickson L, Hansel FA. 2008 Experimental evidence for the processing of marine animal products and other commodities containing polyunsaturated fatty acids in pottery vessels. *Archaeometry* **50**, 101–113. (doi:10.1111/j.1475-4754.2007.00368.x)
54. Hellevang H, Aagaard P. 2015 Constraints on natural global atmospheric CO_2 fluxes from 1860 to 2010 using a simplified explicit forward model. *Sci. Rep.* **5**, 17352. (doi:10.1038/srep17352)
55. Sayle KL, Brodie CR, Cook GT, Hamilton WD. 2019 Sequential measurement of $\delta^{15}N$, $\delta^{13}C$ and $\delta^{34}S$ values in archaeological bone collagen at the Scottish Universities Environmental Research Centre (SUERC): a new analytical frontier. *Rapid Commun. Mass Spectrom.* **33**, 1258–1266. (doi:10.1002/rcm.8462)
56. Chiang Y, Nair BAB, Ramsoe MEE, Ravnsborg T, Jensen ON, Collins MJ. 2024 Anubis: a multi-level authentication scale for ancient proteins using random forest classification. *bioRxiv*. (doi:10.1101/2024.11.15.623824)
57. Chiang Y, Collins MJ. 2024 Orthrus: an AI-powered, cloud-ready, and open-source hybrid approach for metaproteomics. *bioRxiv*. (doi:10.1101/2024.11.15.623814)
58. Perez-Riverol Y *et al.* 2025 The PRIDE database at 20 years: 2025 update. *Nucleic Acids Res.* **53**, D543–D553. (doi:10.1093/nar/gkae1011)
59. Evershed RP. 2008 Experimental approaches to the interpretation of absorbed organic residues in archaeological ceramics. *World Archaeol.* **40**, 26–47. (doi:10.1080/00438240801889373)

60. Drieu L, Regert M, Mazuy A, Vieugué J, Bocoum H, Mayor A. 2022 Relationships between lipid profiles and use of ethnographic pottery: an exploratory study. *J. Archaeol. Method Theory* **29**, 1294–1322. (doi:10.1007/s10816-021-09547-1)
61. Pecci A, Degl'Innocenti E, Giorgi G, Cau Ontiveros MÁ, Cantini F, Solanes Potrony E, Alós C, Miriello D. 2016 Organic residue analysis of experimental, medieval, and post-medieval glazed ceramics. *Archaeol. Anthropol. Sci.* **8**, 879–890. (doi:10.1007/s12520-015-0262-3)
62. Papakosta V, Smittenberg RH, Gibbs K, Jordan P, Isaksson S. 2015 Extraction and derivatization of absorbed lipid residues from very small and very old samples of ceramic potsherds for molecular analysis by gas chromatography–mass spectrometry (GC–MS) and single compound stable carbon isotope analysis by gas chromatography–combustion–isotope ratio mass spectrometry (GC–C–IRMS). *Microchem. J.* **123**, 196–200.
63. Craig OE, Forster M, Andersen SH, Koch E, Crombé P, Milner NJ, Stern B, Bailey GN, Heron CP. 2007 Molecular and isotopic demonstration of the processing of aquatic products in northern European prehistoric pottery. *Archaeometry* **49**, 135–152. (doi:10.1111/j.1475-4754.2007.00292.x)
64. Kimpe K, Jacobs PA, Waelkens M. 2002 Mass spectrometric methods prove the use of beeswax and ruminant fat in late Roman cooking pots. *J. Chromatogr. A* **968**, 151–160. (doi:10.1016/S0021-9673(02)00825-7)
65. Rosiak A, Kałużna-Czaplińska J, Gątarek P. 2020 Analytical interpretation of organic residues from ceramics as a source of knowledge about our ancestors. *Crit. Rev. Anal. Chem.* **50**, 189–195. (doi:10.1080/10408347.2019.1602821)
66. Regert M. 2011 Analytical strategies for discriminating archeological fatty substances from animal origin. *Mass Spectrom. Rev.* **30**, 177–220. (doi:10.1002/mas.20271)
67. Colonese AC *et al.* 2017 The identification of poultry processing in archaeological ceramic vessels using in-situ isotope references for organic residue analysis. *J. Archaeol. Sci.* **78**, 179–192. (doi:10.1016/j.jas.2016.12.006)
68. Gunstone F. 2009 *The chemistry of oils and fats: sources, composition, properties and uses*. Chichester, UK: Wiley-Blackwell.
69. Coimbra MC, Jorge N. 2012 Fatty acids and bioactive compounds of the pulps and kernels of Brazilian palm species, guariroba (*Syagrus oleraces*), jerivá (*Syagrus romanzoffiana*) and macaúba (*Acrocomia aculeata*): fatty acids and bioactive compounds of Brazilian palm species. *J. Sci. Food Agric.* **92**, 679–684. (doi:10.1002/jsfa.4630)
70. Mahato SB, Nandy AK, Roy G. 1992 Triterpenoids. *Phytochemistry* **31**, 2199–2149. (doi:10.1016/0031-9422(92)83257-Y)
71. Moses T, Papadopoulou KK, Osbourn A. 2014 Metabolic and functional diversity of saponins, biosynthetic intermediates and semi-synthetic derivatives. *Crit. Rev. Biochem. Mol. Biol.* **49**, 439–462. (doi:10.3109/10409238.2014.953628)
72. Szakiel A, Pączkowski C, Pensec F, Bertsch C. 2012 Fruit cuticular waxes as a source of biologically active triterpenoids. *Phytochem. Rev.* **11**, 263–284. (doi:10.1007/s11101-012-9241-9)
73. Rommerskirchen F, Plader A, Eglinton G, Chikaraishi Y, Rullkötter J. 2006 Chemotaxonomic significance of distribution and stable carbon isotopic composition of long-chain alkanes and alkan-1-ols in C_4 grass waxes. *Org. Geochem.* **37**, 1303–1332. (doi:10.1016/j.orggeochem.2005.12.013)
74. Bianchi G, Avato PFS. 1984 Surface waxes from grain, leaves, and husks of maize (*Zea mays* L.). *Cereal Chem.* **61**, 45–47.
75. Reber EA, Evershed RP. 2004 Identification of maize in absorbed organic residues: a cautionary tale. *J. Archaeol. Sci.* **31**, 399–410. (doi:10.1016/j.jas.2003.09.008)
76. Copley MS, Bland HA, Rose P, Horton M, Evershed RP. 2005 Gas chromatographic, mass spectrometric and stable carbon isotopic investigations of organic residues of plant oils and animal fats employed as illuminants in archaeological lamps from Egypt. *Analyst* **130**, 860–871. (doi:10.1039/b500403a)
77. Regert M, Bland HA, Dudd SN, Bergen PFV, Evershed RP. 1998 Free and bound fatty acid oxidation products in archaeological ceramic vessels. *Proc. R. Soc. Lond. B Biol. Sci.* **265**, 2027–2032. (doi:10.1098/rspb.1998.0536)
78. Dudd SN, Regert M, Evershed RP. 1998 Assessing microbial lipid contributions during laboratory degradations of fats and oils and pure triacylglycerols absorbed in ceramic potsherds. *Org. Geochem.* **29**, 1345–1354. (doi:10.1016/S0146-6380(98)00093-X)
79. Kühn J, Schweitzer K, Ruess L. 2019 Diversity and specificity of lipid patterns in basal soil food web resources. *PLoS ONE* **14**, e0221102.
80. Lundy J, Haas J, Öhlinger B, Drieu L. 2025 Beyond the surface: untangling molecular complexity in organic residue analysis of coated archaeological ceramics. *R. Soc. Open Sci.* **12**, 250639. (doi:10.1098/rsos.250639)
81. Lundy J *et al.* 2024 Culinary continuity in central Japan across the transition to agriculture. *Archaeol. Anthropol. Sci.* **16**, 97. (doi:10.1007/s12520-024-01992-9)
82. Bondetti M. 2021 Is there an 'Aquatic' neolithic? New insights from organic residue analysis of early Holocene pottery from European Russia and Siberia. (doi:10.33612/DISS.157185365)
83. Roffet-Salque M, Lee MRF, Timpson A, Evershed RP. 2017 Impact of modern cattle feeding practices on milk fatty acid stable carbon isotope compositions emphasise the need for caution in selecting reference animal tissues and products for archaeological investigations. *Archaeol. Anthropol. Sci.* **9**, 1343–1348. (doi:10.1007/s12520-016-0357-5)
84. Pereira C da C. 2004 *História de São Francisco do Sul*. Florianopólis, Brazil: Editora UFSC.
85. Fossile T *et al.* 2024 The historical ecology of subsistence and early commercial fisheries in mangrove systems in Brazil. *J. Archaeol. Sci.* **166**, 105986. (doi:10.1016/j.jas.2024.105986)
86. Hendy J, Welker F, Demarchi B, Speller C, Warinner C, Collins MJ. 2018 A guide to ancient protein studies. *Nat. Ecol. Evol.* **2**, 791–799. (doi:10.1038/s41559-018-0510-x)
87. Silva PPE. 2019 *Farinha, feijão e carne-seca: um tripé culinário no Brasil colonial*. Sao Paulo, Brazil: Editora Senac São Paulo.

88. Evans M, Hagan R, Boyd OJ, Bondetti M, Craig OE, Collins MJ, Hendy J. 2024 The impact of cooking and burial on proteins: a characterisation of experimental foodcrusts and ceramics. *R. Soc. Open Sci.* **11**, 240610. (doi:10.1098/rsos.240610)
89. Hendy J. 2021 Ancient protein analysis in archaeology. *Sci. Adv.* **7**, eabb9314. (doi:10.1126/sciadv.abb9314)
90. Barker A, Dombrosky J, Venables B, Wolverton S. 2018 Taphonomy and negative results: an integrated approach to ceramic-bound protein residue analysis. *J. Archaeol. Sci.* **94**, 32–43. (doi:10.1016/j.jas.2018.03.004)
91. Reber EA, Hart JP. 2008 Pine resins and pottery sealing: analysis of absorbed and visible pottery residues from central New York state. *Archaeometry* **50**, 999–1017. (doi:10.1111/j.1475-4754.2008.00387.x)
92. Bechara FC, Reis A, Bourscheid K, Vieira NK, Trentin BE. 2013 Reproductive biology and early establishment of *Pinus elliottii* var. *elliottii* in Brazilian sandy coastal plain vegetation: implications for biological invasion. *Sci. Agric. (Piracicaba, Braz.)* **70**, 88–92. (doi:10.1590/S0103-90162013000200005)
93. Reber EA, Kerr MT, Whelton HL, Evershed RP. 2019 Lipid residues from low-fired pottery. *Archaeometry* **61**, 131–144. (doi:10.1111/arcm.12403)
94. Costa MS *et al.* 2016 The conifer biomarkers dehydroabietic and abietic acids are widespread in cyanobacteria. *Sci. Rep.* **6**, 23436. (doi:10.1038/srep23436)
95. Miranda EE. 2025 Brasil em relevo. *Campinas: Embrapa Monitoramento por Satélite*. http://www.relevobr.cnpm.embrapa.br (accessed 17 February 2025).
96. Di Muro A *et al.* 2025 Culinary diversity in early modern Brazil revealed by residue analysis of ceramic artefacts. (Version 1). Zenodo. (doi:10.5281/zenodo.17490448)
97. Di Muro A *et al.* 2026 Supplementary material from: Culinary diversity in early modern Brazil revealed by residue analysis of ceramic artefacts. Figshare. (doi:10.6084/m9.figshare.c.8526193)