

Earliest evidence for the use of pottery

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Pottery was a hunter-gatherer innovation that first emerged in East Asia between 20,000 and 12,000 calibrated years before present^{1,2} (cal BP), towards the end of the Late Pleistocene epoch, a period of time when humans were adjusting to changing climates and new environments. Ceramic container technologies were one of a range of late glacial adaptations that were pivotal to structuring subsequent cultural trajectories in different regions of the world, but the reasons for their emergence and widespread uptake are poorly understood. The first ceramic containers must have provided prehistoric hunter-gatherers with attractive new strategies for processing and consuming foodstuffs, but virtually nothing is known of how early pots were used. Here we report the chemical analysis of food residues associated with Late Pleistocene pottery, focusing on one of the best-studied prehistoric ceramic sequences in the world, the Japanese Jōmon. We demonstrate that lipids can be recovered reliably from charred surface deposits adhering to pottery dating from about 15,000 to 11,800 cal BP (the Incipient Jōmon period), the oldest pottery so far investigated, and that in most cases these organic compounds are unequivocally derived from processing freshwater and marine organisms. Stable isotope data support the lipid evidence and suggest that most of the 101 charred deposits analysed, from across the major islands of Japan, were derived from high-trophic-level aquatic food. Productive aquatic ecotones were heavily exploited by late glacial foragers³, perhaps providing an initial impetus for investment in ceramic container technology, and paving the way for further intensification of pottery use by hunter-gatherers in the early Holocene epoch. Now that we have shown that it is possible to analyse organic residues from some of the world's earliest ceramic vessels, the subsequent development of this critical technology can be clarified through further widespread testing of hunter-gatherer pottery from later periods.

On the basis of evidence from East Asia, North Africa, the Americas and Northern Europe, it is now widely accepted that pottery production precedes the emergence of farming and should be considered a hunter-gatherer innovation. From radiocarbon dates of an archaeological sequence excavated at Xianrendong Cave, Jiangxi Province, China, it has been proposed that ceramic container technology extends in age to about 20,000–19,000 cal BP¹, some 10,000 yr before the earliest evidence of rice cultivation in that region. Regional case studies^{4,5} have explored the origins, adoption and dispersal of ceramic container technology, dates are being refined and pushed back, and models are emerging that encompass economic, technological, social and environmental factors that both facilitated, and potentially inhibited, pottery innovation in different parts of the world⁶. Yet the actual uses of these early pottery vessels have not been addressed beyond assumed functional roles such as cooking and storage, or speculative associations with available animal and plant resources^{7–9}.

An understanding of how pottery was used is necessary to elucidate both the social and the economic context for its innovation and also to

explain why the technology flourished in some hunter-gatherer groups and not others. Direct evidence of pottery use can be gained through molecular and stable isotope analyses of lipid residues and other organic matter preserved in association with ceramics^{10,11}. Although such investigations have been widely conducted on vessels associated with early agricultural or pastoral communities^{12,13}, the use of earlier hunter-gatherer pottery has been largely ignored or considered only for late forager groups^{14,15}. The main challenge limiting systematic investigations of early hunter-gatherer pottery use is the extent to which molecular evidence is preserved after such long-term exposure to the burial environment. The earliest known pottery use in Eurasia gradually developed, probably independently, in Southern China, the Amur River basin (Russian Far East) and Japan at the end of the Late Pleistocene^{1,5}, necessitating the recovery of organic residues at least 3,000 yr older than has so far been reported¹². The rarity of the very earliest pottery vessels also limits widespread destructive residue analysis. Here we present an investigation of Late Pleistocene pottery use, focusing on the Japanese ceramic sequence known as the Incipient Jōmon, using small subsamples (~20 mg) of charred deposits adhering to the inside and outside of potsherds. We investigated the carbon and nitrogen stable isotope characteristics of the charred deposits using bulk isotope ratio mass spectrometry (IRMS). Where the sample was large enough, lipids were extracted and analysed by gas chromatography mass spectrometry (GCMS) and GC combustion IRMS (GC-C-IRMS) to determine their structural and carbon isotope characteristics^{10,11}.

Incipient Jōmon pottery is thought to have been invented in Japan ~16,000 cal BP², and became established across the major islands of the Japanese archipelago during the last 4,000 yr of the Late Pleistocene¹⁶. Much is known about the glacial foragers who developed this pottery. Their sites have been well dated¹⁷ and the pottery sequence has been intensively studied⁷: three major phases of typological development have been identified, each broadly mapping onto different climate phases of the final Pleistocene (Fig. 1). Japanese Late Pleistocene pottery containers were produced in only very small quantities (Fig. 1b) and their estimated volumes were also relatively low. Therefore, pottery may have initially been reserved for specialized subsistence activities or ritual use, rather than being a widespread technology integral to everyday culinary practices. Nevertheless, this long-lived nascent phase, which in some regions endured severe climatic fluctuations, led to an extremely rapid expansion in pottery production in the early Holocene, possibly in response to a warming climate, demographic expansion and intensification of resource exploitation.

Charred surface deposits were sampled from 101 Incipient Jōmon vessels from 13 sites, dating from 15,300–11,200 cal BP (Supplementary Table 1) and encompassing a wide range of different environments across the archipelago (Fig. 1a). Potsherds from different phases (II and III; Fig. 1b) broadly corresponding to the Bølling–Allerød interstadial and the Younger Dryas stadial were included. Variations in vessel shapes are known to exist in this period^{7,16} but could not be discerned

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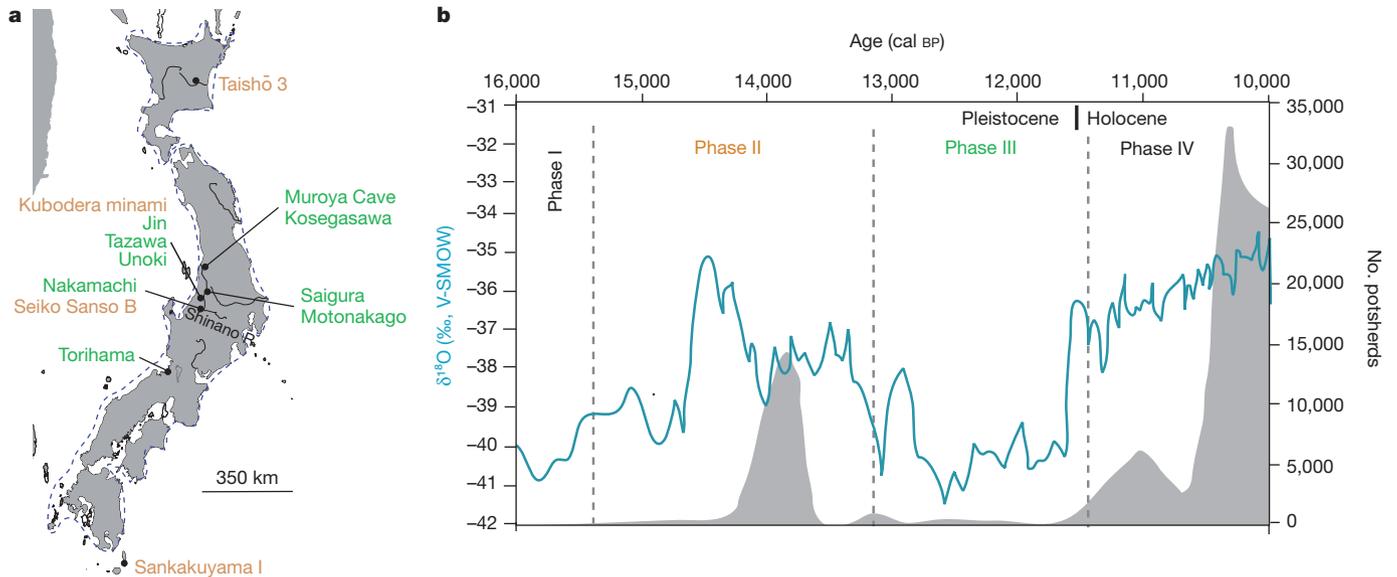


Figure 1 | Locations of sites sampled and temporal changes in early pottery production against Greenland ice-core oxygen isotope record. **a**, Potsherds were selected from assemblages at different sites, dating to chrono-typological phase II, that is, linear-relief wares (orange), and phase III, that is, punctate-marked, nail-impressed and cord-marked wares (green). At Torihama, pottery from both phases was sampled. The coastline corresponding to the Younger

Dryas stadal (~ 65 m below present-day sea level²²) is shown. **b**, The Greenland Ice Sheet Project 2 ice-core $\delta^{18}\text{O}$ values (blue) generally reflect temperature, with lower $\delta^{18}\text{O}$ values corresponding to lower temperatures. Potsherd quantities (grey) are as previously reported^{7,16}, calculated from 52 site reports and chronologically assigned by their typology and by 118 associated ^{14}C dates.

because of the fragmentary nature of the assemblages. Bulk carbon ($\delta^{13}\text{C}$) and nitrogen ($\delta^{15}\text{N}$) isotope ratio values obtained from each charred deposit were variable across the whole sample and between individual sites (Fig. 2a). Despite this variability, more than 75% of the samples had $\delta^{15}\text{N}$ values greater than 9‰ (median, 10.8‰) consistent with the processing of high-trophic-level aquatic (that is, marine or freshwater) products, as has been observed experimentally (Fig. 2a) and on pottery from different archaeological sites associated with the exploitation of fish and marine mammals^{14,18,19}. To confirm this interpretation, lipids were extracted from 57 charred interior deposits and four exterior deposits from Incipient Jōmon vessels and analysed by

GCMS (Table 1). Seventeen samples from the Torihama site, Western Honshu, and one from the Taishō 3 site, Hokkaido, produced distinctive lipid profiles typically consisting of medium- and long-chain saturated ($\text{C}_{14}\text{--}\text{C}_{24}$) and mono-unsaturated ($\text{C}_{16:1}\text{--}\text{C}_{22:1}$) fatty acids, isoprenoid fatty acids and long-chain ($\text{C}_{18}\text{--}\text{C}_{22}$) ω -(*o*-alkylphenyl) fatty acids (Fig. 3 and Supplementary Table 2). These profiles are typical of degraded aquatic oils, meeting the established criteria for their identification in archaeology²⁰ and providing compelling evidence that freshwater and/or marine foods were processed in these vessels. Such lipid profiles were not observed in the four exterior surface deposits and are highly unlikely to derive from the depositional

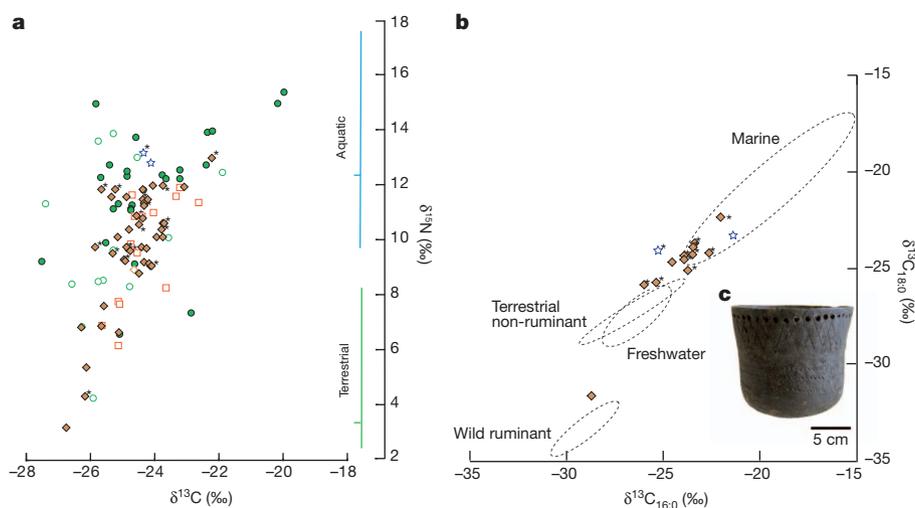


Figure 2 | Bulk carbon and nitrogen stable isotope ratios and fatty acid stable carbon isotope ratios from Incipient Jōmon pottery. **a**, Bulk stable isotope values are discriminated according to site location and period. Torihama, orange diamonds; Shinano river sites, green circles; Sankakuyama I, red squares; Taishō 3, blue stars. Filled symbols indicate phase III and open symbols indicate phase II. The blue and green vertical bars show 95% confidence intervals and the median stable nitrogen isotope value from experimentally charred aquatic (blue, $n = 9$) and terrestrial (green, $n = 8$)

organisms (Supplementary Table 3). **b**, Plot of the $\delta^{13}\text{C}$ values of $\text{C}_{16:0}$ and $\text{C}_{18:0}$ fatty acids from charred deposits on pottery from Torihama and Taishō 3. These are compared with reference fats from wild terrestrial non-ruminant ($n = 5$), wild ruminant ($n = 10$), freshwater ($n = 9$) and marine ($n = 17$) organisms (Supplementary Table 4). Data are plotted with 68% confidence intervals (using STATISTICA v.7). Samples that produced aquatic biomarkers are marked with asterisks. **c**, Photograph of a reconstructed Incipient Jōmon vessel from Torihama.

Table 1 | Summary of GCMS results of charred deposits on Incipient Jōmon pottery

Site	Region	Phase	Associated dates* ($\times 10^3$ cal BP)	No. samples analysed	No. samples yielding lipid†	No. samples yielding aquatic biomarkers
Kubodera minami	Central Honshu	II	15.3–14.2	4	0	0
Taishō 3	Hokkaido	II	15.0–14.0	2	2	1
Seiko Sanso B	Central Honshu	II	14.1–13.9	1	1	0
Sankakuyama I	Kyushu	II	13.6–13.4	13	0	0
Torihamama	Western Honshu	III	12.1–11.8	35	32	17
Muroya Cave	Central Honshu	III	NA	1	0	0
Tazawa	Central Honshu	III	NA	1	0	0

Further details are available in Supplementary Table 2.

*Range corresponds to ± 2 s.d.

†Greater than $0.1 \mu\text{g mg}^{-1}$ of food crust.

NA, not available.

environment because the ω -(*o*-alkylphenyl) alkanolic acids are formed only at high temperatures ($>270^\circ\text{C}$) from the protracted heating of polyunsaturated fatty acids²⁰. The presence of one of these biomarkers in two exterior deposits (Supplementary Table 2) is most probably attributable to overspill of the vessel's contents.

Further information regarding the source of the organic residues was obtained by measuring the stable carbon isotope ratio of two medium-chain-length saturated fatty acids ($C_{16:0}$ and $C_{18:0}$) preserved in sufficient quantities in the charred surface deposits adhering to 12 of the Torihamama vessels and two of those from Taishō 3. Eleven samples, including nine with characteristic aquatic biomarkers, yielded either $C_{16:0}$ or $C_{18:0}$ fatty acids noticeably enriched in ^{13}C (that is, $\delta^{13}\text{C} \geq -25\text{‰}$). These $\delta^{13}\text{C}$ values are more consistent with authentic marine products than with freshwater or terrestrial animals (Fig. 2b). Two samples from Torihamama, both with aquatic biomarkers, yield $\delta^{13}\text{C}$ values closer to those of lipid extracts from modern pottery vessels used to prepare freshwater as well as non-ruminant animals from Japan (Fig. 2b), but their use cannot be resolved more specifically. In contrast, one Torihamama sample (T66), which was depleted in ^{15}N ($\delta^{15}\text{N} = 6.8\text{‰}$), contained fatty acids with an isotope composition comparable to wild ruminant fats²¹ (Fig. 2b). Aquatic biomarkers were absent from this sample and the lipid profile was instead dominated by saturated fatty acids (C_{14} – C_{18}) that are more typical of degraded terrestrial animal fat, indicating a very different use history of this particular pottery vessel.

Despite some variation in pottery use at Torihamama, the majority of the samples, and all but one with aquatic biomarkers, had relatively high bulk $\delta^{13}\text{C}$ ($> -26\text{‰}$) and $\delta^{15}\text{N}$ ($> 9\text{‰}$) values, the latter consistent with charred deposits formed experimentally from authentic marine organisms (Supplementary Table 3) and both comparable to those associated with Northern European hunter-gatherer pottery found at coastal sites¹⁴. Both the bulk and the compound-specific isotope results from the earlier site of Taishō 3 ($\sim 15,000$ – $14,000$ cal BP) are also consistent with the processing of marine products in pottery vessels (Fig. 2). Therefore, we suggest that the preparation of aquatic resources, particularly marine foodstuffs, was the dominant mode of pottery use at these sites even though neither site is situated directly on the coast (Fig. 1a). At Torihamama, the nearby exposed littoral shelf conceivably provided easy access to a range of marine mammals and fish during the Late Pleistocene, even considering the slightly lower sea level during that period²² (Fig. 1a). An alternative explanation is that migratory fish, such as salmon, were caught much closer to each site as they travelled upriver from marine feeding grounds.

More generally, considering the 13 Incipient Jōmon sites, the distribution of $\delta^{15}\text{N}$ values in charred deposits associated with pots that date from the warmer Bølling–Allerød interstadial, when there was greater deciduous forest cover²³ and larger numbers of large mammals, is not significantly different (Mann–Whitney U test) from that of the Younger Dryas stadial for sites on Honshu ($U = 392$, $P = 0.21$, $n_{\text{Bølling–Allerød}} = 14$, $n_{\text{Younger Dryas}} = 71$) or overall ($U = 893$, $P = 0.20$, $n_{\text{Bølling–Allerød}} = 30$, $n_{\text{Younger Dryas}} = 71$). From both the bulk stable isotope data and the more specific product identifications based on available lipid data, we suggest that aquatic products were the most frequently processed products in Incipient Jōmon pottery, through the

fluctuating climate and across a range of environments, from Hokkaido in the north to Kyushu in the south. Whether ceramic vessels were integral to the processing of aquatic resources or, as is suggested by their rarity, were used only occasionally, perhaps ceremonially or as a prestige technology²⁴, remains debatable. The appearance of pottery at Late Pleistocene sites located on narrowing coastal plains and along rivers where there was an abundance of terrestrial game, aquatic resources and fresh water²⁵ might have led to decreased mobility permitting a greater investment in pottery production for either purpose. The long 'experimental' phase of pottery use in the late glacial contrasts

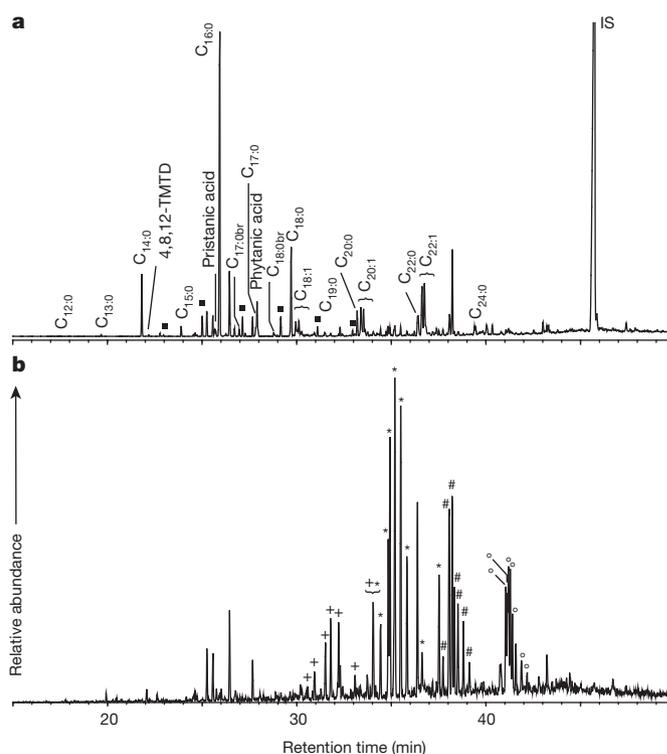


Figure 3 | Partial gas chromatograms of a lipid extract from a charred deposit adhering to a Torihamama potsherd ($\sim 12,000$ cal BP). **a**, The total ion chromatogram is characteristic of degraded aquatic oil and is dominated by medium- and long-chain saturated and mono-unsaturated fatty acids and isoprenoid fatty acids, pristanic, phytanic and 4,8,12-TMTD (4,8,12-trimethyltridecanoic acid). α,ω -dicarboxylic acids (■) with carbon chain ranges of C_8 – C_{13} were also observed and are the likely oxidation products from the complex mixture of long-chain precursor unsaturated fatty acids^{29,30}, derived from freshwater or marine oils, or both. $C_{n,x}$ fatty acids with carbon length n and number of unsaturations x ; br, branched-chain acids; IS, internal standard (*n*-hexatriacontane). **b**, The summed m/z 91, m/z 105 (mass-to-charge ratio) ion chromatogram shows the presence of ω -(*o*-alkylphenyl) alkanolic acids with 16 (+), 18 (*), 20 (#) or 22 (o) carbon atoms. The presence of the third and fourth components, thermally produced from C_{20} and C_{22} unsaturated fatty acids, and the distribution of their isomers confirm that this residue (sample T124) is derived from an aquatic organism³⁰.

sharply with its sudden ubiquity in the Early Holocene of Japan (Fig. 1b) and other regions of East Asia⁷, when use intensified and became integrated into everyday practice, triggering further long-range dispersals of the new technology into other regions⁶. Although it is tempting to link this change in pottery abundance directly to resource intensification, increased sedentism and the warmer climate, more complex demographic and social factors could also be posited²⁶. Our study, showing that organic residues can be reliably obtained from Late Pleistocene pottery, will make it possible to clarify, through further widespread analysis, the drivers of the continued development of pottery in the Early Holocene of East Asia and its emergence at other innovation centres.

METHODS SUMMARY

Subsamples of charred surface deposits (10–30 mg) were removed with a sterile scalpel from 101 potsherds from 13 different sites (Supplementary Table 3 and Fig. 1a). The chronological phase of each sample was established by its typology, stratigraphic position and ¹⁴C dating of associated materials (Supplementary Table 1). In most cases these agree but, where available, radiocarbon dates of materials directly associated with the pottery analysed were favoured to assign a chronological phase using the published date ranges⁷. Lipids were extracted by alkali saponification with NaOH in methanol²⁷ (5% (w/v), 2 h, 70 °C) or extracted and methylated in one-step with acidified methanol²⁸, or both. For the second process, methanol (1 ml) was added to homogenized charred deposits (10–30 mg) and the mixture was sonicated for 15 min and then acidified with concentrated sulphuric acid (200 µl). The acidified suspension was heated in sealed tubes for 4 h at 70 °C and then cooled, and lipids were extracted with *n*-hexane (3 × 2 ml) and directly analysed by GCMS and GC-C-IRMS using standard protocols^{18,20,21}. Saponified extracts were cooled, neutral lipids were removed (*n*-hexane, 3 × 2 ml), the extracts were acidified with HCl, and the acid fraction was extracted (*n*-hexane, 3 × 2 ml) and methylated to produce fatty acid methyl esters (FAMES) using BF₃-methanol complex (14% (w/v), 200 µl, 1 h, 70 °C). For GC-C-IRMS, instrument precision on repeated measurements was ±0.3‰ (s.e.m.) and the accuracy determined from FAME and *n*-alkane isotope standards was ±0.5‰ (s.e.m.). Modern samples and experimental pots were extracted using exactly the same procedure or as previously reported^{21,27}, or both. In addition, crushed surface residues (~1 mg) were analysed by elemental analysis IRMS as previously reported¹⁸. Samples yielding less than 1% N were discarded and instrument precision on repeated measurements was ±0.2‰ (s.e.m.). δ¹³C, δ¹⁵N or δ¹⁸O = [(R_{sample}/R_{standard} - 1)] × 1,000, where R = ¹³C/¹²C, ¹⁵N/¹⁴N or ¹⁸O/¹⁶O. The standard for δ¹³C is Vienna PeeDee Belemnite (V-PDB), the standard for δ¹⁵N is air N₂ and the standard for ¹⁸O is Vienna Standard Mean Ocean Water (V-SMOW).

Received 31 January; accepted 20 March 2013.

Published online 10 April 2013.

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Supplementary Information is available in the online version of the paper.

Acknowledgements We thank the Leverhulme trust (F/00 152/AM) and the Japanese Society for the Promotion of Science (PE 11560) for their support. We are grateful to K. Adachi, K. Higashi, Y. Kasai, H. Kato, K. Nagahama, H. Oguma, T. Tsuchiya, T. Watanabe and T. Yamahara for providing access to samples.

Author Contributions O.E.C., H.S., Y.N., S.I. and P.J. planned the project. O.E.C. wrote the paper with assistance from all other authors. P.J., H.S., Y.N. and K.G. carried out sampling with assistance of M.A. and J.J., who provided contextual data. O.E.C., H.S., A.L., K.T., D.A. and A.T. carried out the lipid analysis. C.P.H. and L.C. carried out the bulk stable isotope analyses. All authors commented on the manuscript.

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