Insights into Ceramic Use in Prehistoric Northwest China Obtained from Residue Analysis: A Pilot Study on the Andersson Collection at the Museum of Far Eastern Antiquities, Stockholm

The Andersson Collection housed at the Museum of Far Eastern Antiquities holds finds from the earliest archaeological fieldwork ever conducted in northwest China. Recent years have seen an increased interest in the archaeology of that region, especially prehistoric subsistence practices and their environmental correlates. The Andersson Collection which has lain largely dormant since the 1940s provides a great opportunity for further research on this topic, especially on sites which are no longer accessible for a variety of reasons. As part of a larger project aimed at "re-excavating" these materials from museum storage and answer questions of identity and interaction along the old exchange corridor of the proto-Silk Road, this pilot study explores the potential of using molecular and isotopic characterization of organic residues from Neolithic and Bronze Age ceramic vessels to understand subsistence practices in northwest China.

Organic residue analysis can aid our understanding what kinds of foodstuffs these vessels held and shed light on cooking and eating habits and how these practices change or remain consistent over time and/or space. There is some concern that long-term storage in potentially unsuitable containers, cleaning with harsh chemicals, or restauration methods deemed suitable at the time may have destroyed much of the residue. Indeed, this pilot study finds that there is a considerable amount of contaminants in all samples, however, some residue has been observed and analysed, suggesting that more advanced extraction methods combined with an investigation into museum records to find out about previous cleaning and restauration methods, may help mitigate these issues. A similar approach may also be applied to other legacy collections.

Keywords: Organic residue analysis; Northwest China; Neolithic; Andersson; Painted Pottery; Pottery use; Majiayao; Qijia; Xindian; Kayue

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Introduction

Recent years have seen an increasing amount of research on changes in subsistence practices in prehistoric northwest China, especially in connection with climate changes such as the 4.2 ka BP climatic event.[1] So far, these discussions have largely relied on model building based on changes in site distribution patterns combined with isotope studies and a limited amount of paleobotanical and zooarchaeological work.[2] Residue analyses of the abundant ceramic material from the region can also provide insights into cooking and eating practices, however, so far this avenue of analysis remains underexplored, a lacuna that this study addresses. Rather than sampling material from recent excavations in China, which are difficult or even impossible to export, this research focuses on the large and not fully explored collections of The Museum of Far Eastern Antiquities (MFEA) in Stockholm. In particular, samples were chosen from sites that are by now destroyed or otherwise inaccessible, to complement new fieldwork being conducted in China. The current study thus aims to understand subsistence practices in northwest China through molecular and isotopic characterization of lipids extracted from ceramic vessels from the Andersson collection held at the MFEA. This unique collection holds finds from the earliest archaeological surveys and excavations ever conducted in northwest China. These finds furthermore provided the basis for the culture chronological framework developed by Andersson for the region, a framework which in large parts is used until the present day.[3] As several of the sites that Andersson excavated in the 1920's are now destroyed or severely disturbed, the material held at the MFEA allows for unique insights into the prehistory of northwest China that even very recent excavations may not be able to provide.

While collections such as this are a great source for research, there are some concerns with preservation as organic residues in ceramics have shown to degrade quicker once excavated.[4] Furthermore, given that the finds have been in storage for about a century, a period during which conservation treatments and storage regulations have changed significantly, there may have been further sources of contamination or degradation of the residues. Here, we present the results of a pilot study investigating the preservation of organic residues for the material

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[1] Yitzchak Y., Jaffe, and Anke Hein, "Considering Change with Archaeological Data: Reevaluating Local Variation in the Role of the 4.2 K BP Event in Northwest China," *Holocene* 31, 2 (2021): 169–182. https://doi.org/10.1177%2F0959683620970254 0959683620970254.

[2] Andrew Womack, Yitzchak Jaffe, Jing Zhou, Ling yu Hung, Hui Wang, Shuicheng Li, Pochan Chen, and Rowan Flad, "Mapping Qijiaping: New Work on the Type-Site of the Qijia Culture (2300–1500 B.C.) in Gansu Province, China," *Journal of Field Archaeology* 42, no. 6 (2017): 488–502; Ma, M. M., G. H. Dong, E. Lightfoot, H. Wang, X. Y. Liu, X. Jia, K. R. Zhang, and F. H. Chen, "Stable Isotope Analysis of Human and Faunal Remains in the Western Loess Plateau, Approximately 2000 Cal BC," *Archaeometry* 56, no. SUPPLS1 (2014): 237–55.

[3] Johan Gunnar Andersson, "Researches into the Prehistory of the Chinese," *Bulletin of the Museum of Far Eastern Antiquities* 15 (1943): 1–198.

[4] Laura Fanti, Léa Drieu, Arnaud Mazuy, Thierry Blasco, Carlo Lugliè, and Martine Regert, "The Role of Pottery in Middle Neolithic Societies of Western Mediterranean (Sardinia, Italy, 4500-4000 Cal BC) Revealed through an Integrated Morphometric, Use-Wear, Biomolecular and Isotopic Approach," *Journal of Archaeological Science* 93 (May 1, 2018): 110–28.

from seven sites in northwest China dating to the Neolithic and Bronze Age which are part of the Andersson collection.

Background

Archaeology of northwest China

Having been an exchange corridor between China and Central Asia from the Neolithic onwards, northwest China has seen a significant amount of interest in recent years resulting in a number of articles discussing directions and types of long-distance interaction.[5] It was precisely his interest in potential long-distance interactions that brought Andersson to northwest China initially. Having discovered painted pottery in Henan further to the east in 1921, Andersson was told by European archaeologists that there might be a connection with painted wares in Central Asia and Eastern Europe.[6] Andersson thus went West to explore the potential for such a connection, finding both painted and unpainted wares and establishing a first chronological framework.[7] Though the sequence was later corrected by Xia Nai[8] and the connections with eastern European painted pottery traditions were disproven, the culture names and part of the definitions that Andersson established are being used until the present day (Table 1).

Table 1. Chronology according to Andersson and according to recent research

Andersson Chronology	**Current Chronology**		
Kayue Xindian	Xindian 1600-600BC	Kayue 1600-700BC	Siwa 1400-700BC

[5] Guanghui Dong, Yishi Yang, Xinyi Liu, Haiming Li, Yifu Cui, Hui Wang, Guoke Chen, John Dodson, and Fahu Chen. "Prehistoric Trans-Continental Cultural Exchange in the Hexi Corridor, Northwest China." *Holocene* 28, no. 4 (April 1, 2018): 621–28; Jaang, Li, "The Landscape of China's Participation in the Bronze Age Eurasian Network," *Journal of World Prehistory* 28, no. 3 (2015): 179–213; Yang, Y., L. Ren, G. Dong, Y. Cui, R. Liu, G. Chen, H. Wang, S. Wilkin, and F. Chen, "Economic Change in the Prehistoric Hexi Corridor (4800-2200 BP), North-West China," *Archaeometry* 61, no. 4 (August 27, 2019): 957–76; Fu Luowen 傅羅文 [Rowan Flad], "Zaoqi sichouzhilu de jishu biange – Gansu Taohe liuyu de kaogu chengguo 早期絲綢之路上的技術變革——甘肅洮河流域的考古成果 [Technological Change on the Proto-Silk Road: Archaeological Results from the Tao River in Gansu]," in *Zhongguo keji kaogu conglun* 中國科技考古從輪 [*Discussion of the Archaeological Sciences in China*], ed., Yuan Jing [袁靖] (Shanghai: Fudan Daxue Chubanshe 復旦大學出版社, 2019).

[6] Magnus Fiskesjö, and Chen Xingcan, *China before China: Johan Gunnar Andersson, Ding Wenjiang, and the Discovery of China's Prehistory* (Stockholm: Museum of Far Eastern Antiquities, 2004).

[7] Andersson, *Researches into the Prehistory of the Chinese*.

[8] Xia Nai 夏鼐, *Kaoguxue lunwenji* 考古學論文集 [*Collected Essays in Archaeology*] (Beijing: Kexue Chubanshe 科學出版社, 1961).

<table>
<tr><td rowspan="4">Siwa

Majiayao

Yangshao

Qijia</td><td colspan="2"></td><td rowspan="2">Qijia
2300-1500BC</td></tr>
<tr><td rowspan="3">Majiayao
3200-2000BC</td><td>Machang Subphase
2300-2000BC</td></tr>
<tr><td>Banshan Subphase
2650-2300BC</td><td rowspan="2">Caiyuan
2800-2200BC</td></tr>
<tr><td>Majiayao Subphase
3200-2650BC</td></tr>
</table>

According to the current state of research, during the Neolithic, the Northwest was first part of the Yangshao cultural horizon, soon followed by the local variety of painted pottery known as Majiayao with its sub-phases of Majiayao, Banshan, and Machang which were defined by Andersson based on ceramic styles. These ceramic types have been found in large numbers in graves but also at settlement sites associated with agricultural living.[9] The early Bronze Age Qijia cultural phenomenon is likewise defined based on ceramic types, with undecorated fine-ware double-handled vessels being seen as typical for that period. Additionally, metallurgy became more common and there is evidence for agricultural subsistence and domestication of various types of animals, especially pigs.[10] These large-scale archaeological phenomena were followed by a fragmentation into a considerable number of different ceramic traditions, most importantly Xindian, Siwa, and Kayue which are generally believed to have practiced a pastoralist form of subsistence, though this notion has recently been put into question.[11] Given that all of these archaeological cultures are defined based on ceramic types, information on what kinds of foodstuffs these vessels held would thus aid in understanding cooking and eating habits by the various communities during these different periods and thus also contribute to solving the issue of changes and/or continuities in subsistence practices.

Thus far, no such research has been conducted on the materials excavated in recent years from various prehistoric sites in northwest China, and the material held in Stockholm has lain largely dormant since the 1940s. Until the present day, parts of the Andersson collection remain unpublished. The present study is part of a larger project conducted in collaboration between the Museum of Far Eastern Antiquities Stockholm, Stockholm University, and the University of Oxford. The main goal is to "re-excavate" these materials from museum storage and answer questions of identity and interaction along this old exchange corridor, the proto-Silk Road. The

[9] Xie Duanju 謝端琚, *Ganqing diqu shiqian kaogu* 甘肅地區史前考古 [*Prehistoric Archaeology of Gansu and Qinghai*] (Beijing: Wenwu Chubanshe [文物出版社], 2002).

[10] Womack et al., "Mapping Qijiaping: New Work on the Type-Site of the Qijia Culture (2300–1500 B.C.) in Gansu Province, China."

[11] Jaffe, and Hein, "Considering Change with Archaeological Data: Reevaluating Local Variation in the Role of the 4.2 K BP Event in Northwest China."

project uses a combination of macroscopic, microscopic, and chemical techniques to analyse the prehistoric pottery from northwest China held at the museum to learn about relationships between sites during this period of early interaction as reflected in traditions of ceramic production and usage and the transmission of these traditions over time and space. Petrographic analysis shows that production techniques stay relatively consistent from the Neolithic to the Bronze Age in terms of raw materials and tempering choices, however, tempering behaviour changes in the late Bronze Age.[12] While there may be continuity in vessel production, new vessel forms appear in the Bronze Age possibly suggesting a change in vessel function. On this issue, residue analysis can provide further insights.

Food residue analysis in archaeology

Analyses of organic residues absorbed in or encrusted on ceramic vessels have been highly successful in giving insight into vessel technology (e.g. beeswax resins used to impregnate vessel surfaces,[13] agricultural and subsistence practices,[14] human movement,[15] and human-environment interaction).[16] Organic molecules, called biomarkers, can be used as indicators of specific substances (e.g. animal fat, plant oil, beeswax, milk). Much of the published literature on food residues focuses on lipids, as they tend to preserve better in archaeological contexts, and can be indicative of the products processed and stored in ceramic vessels with a varying degree of specificity.[17] Lipids are also common in most foodstuffs, making them ideal proxies for specific research regarding human diet and subsistence in the past.

The study of organic residues has gained traction in recent years due to developments in analytical chemistry, namely, advancements in chromatographic coupled to mass spectrometric instrumentation – allowing for organic components in small quantities to be identified with a

[12] Anke Hein, and Ole Stilborg, "Ceramic Production in Prehistoric Northwest China: Preliminary Findings of New Analyses of Old Material from the Museum of Far Eastern Antiquities, Stockholm," *Journal of Archaeological Science: Reports* 23, no. August 2018 (2019): 104–15.

[13] Mélanie Roffet-Salque, Martine Regert, Richard P. Evershed, Alan K. Outram, Lucy J. E. Cramp, Orestes Decavallas, Julie Dunne, et al., "Widespread Exploitation of the Honeybee by Early Neolithic Farmers," *Nature* 527, no. 7577 (2015): 226–30.

[14] Julie Dunne, Richard P. Evershed, Mélanie Salque, Lucy Cramp, Silvia Bruni, Kathleen Ryan, Stefano Biagetti, and Savino di Lernia. "First Dairying in Green Saharan Africa in the Fifth Millennium BC," *Nature* 486, no. 7403 (2012): 390–94.

[15] L. J. E. Cramp, R. P. Evershed, M. Lavento, P. Halinen, K. Mannermaa, M. Oinonen, J. Kettunen, M. Perola, P. Onkamo, and V. Heyd, "Neolithic Dairy Farming at the Extreme of Agriculture in Northern Europe," *Proceedings of the Royal Society B: Biological Sciences* 281, no. 1791 (2014): 20140819–20140819.

[16] O. E. Craig, H. Saul, A. Lucquin, Y. Nishida, K. Taché, L. Clarke, A. Thompson, et al., "Earliest Evidence for the Use of Pottery," *Nature* 496, no. 7445 (2013): 351–54; Alexandre Lucquin, Harry K. Robson, Yvette Eley, Shinya Shoda, Dessislava Veltcheva, and Kevin Gibbs, "The Impact of Environmental Change on the Use of Early Pottery by East Asian Hunter-Gatherers," in *Proceedings of the National Academy of Sciences of the United States of America*, vol. 115, nr 31 (2018): 7931-7936https://doi.org/10.1073/pnas.1803782115.

[17] R. P. Evershed, "Organic Residue Analysis in Archaeology: The Archaeological Biomarker Revolution," *Archaeometry* 50, no. 6 (2008): 895–924; Mélanie Roffet-Salque, Julie Dunne, David T. Altoft, Emmanuelle Casanova, Lucy J. E. Cramp, Jessica Smyth, Helen L. Whelton, and Richard P. Evershed, "From the inside out: Upscaling Organic Residue Analyses of Archaeological Ceramics," *Journal of Archaeological Science: Reports* 16 (2017): 627–40.

high degree of specificity.[18] Currently, single compound isotopic analyses to measure $\delta^{13}C$ values are performed on palmitic acid (C16:0) and stearic acid (C18:0) through gas-chromatography-combustion-isotope-ratio-mass-spectrometry (GC-C-IRMS) to identify a wider variety of food resources.[19] GC-C-IRMS has been successfully applied to the archaeological record in various regions to better understand human environment interactions,[20] plant cultivation,[21] and exploitation of secondary products from domestic animals such as milk and cheese.[22]

Material Investigated

The present study explores whether there is adequate lipid preservation in the ceramic collection of the MFEA for further research. To make full use of the Andersson collections and complement the field research that has been and is currently being done in northwest China, this project focuses on sites discovered by Andersson that are not being re-excavated, giving preference to those that are destroyed or otherwise inaccessible to present-day scholarship. We selected seven sites including two with remains dating to the Majiayao (Dashiquan and Siwashan), Qijia (Caojiaping and Zhujiazhai), and Xindian periods (Machangyan and Xiaxihe), and one site with Kayue period remains (Qiayao, now known as Kayue) as only one Kayue-period site has been explored by Andersson (Table 2). Fourteen samples were taken, i.e., two samples from each site, one coarse ware and one fine ware sample to account for difference in residue absorption and retention between different ceramic porosities (Table A1). This is a pilot study conducted on a small number of samples from a limited number of sites ranging in date from the Neolithic to the late Bronze Age and in location from Lanzhou in the south-eastern part of Gansu to Qinghai in the west, chosen to cover a broad chronological range, different ceramic types, and a range of different soil climates leading to differences in preservation. In this way, the study aims to establish the feasibility and prolificacy of conducting residue

[18] Mark Pollard, Carl P. Heron, and Ruth Ann Armitage, *Archaeological Chemistry*. 3rd ed. (Cambridge: Royal Society of Chemistry, 2017); Roffet-Salque et al., "From the inside out: Upscaling Organic Residue Analyses of Archaeological Ceramics."

[19] Vasiliki Papakosta, Rienk H. Smittenberg, Kevin Gibbs, Peter Jordan, and Sven Isaksson, "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 Chrom," *Microchemical Journal, Devoted to the Application of Microtechniques in All Branches of Science* 123 (2015): 196–200; Marisol Correa-Ascencio, and Richard P. Evershed, "High Throughput Screening of Organic Residues in Archaeological Potsherds Using Direct Acidified Methanol Extraction," *Analytical Methods* 6, no. 5 (2014): 1330.

[20] Alexandre Lucquin, Kevin Gibbs, Junzo Uchiyama, Hayley Saul, Mayumi Ajimoto, Yvette Eley, Anita Radini, et al., "Ancient Lipids Document Continuity in the Use of Early Hunter–gatherer Pottery through 9,000 Years of Japanese Prehistory," *Proceedings of the National Academy of Sciences* 113, no. 15 (2016): 3991–96.

[21] Shinya Shoda, Alexandre Lucquin, Chi Ian Sou, Yastami Nishida, Guoping Sun, Hiroshi Kitano, Joon-Ho Son, Shinichi Nakamura, and Oliver E. Craig, "Molecular and Isotopic Evidence for the Processing of Starchy Plants in Early Neolithic Pottery from China," *Scientific Reports* 8, no. 1 (November 19, 2018): 17044.

[22] Dunne et al., "First Dairying in Green Saharan Africa in the Fifth Millennium BC"; Cramp et al., "Neolithic Dairy Farming at the Extreme of Agriculture in Northern Europe"; Jessica Hendy, Andre C. Colonese, Ingmar Franz, Ricardo Fernandes, Roman Fischer, David Orton, Alexandre Lucquin, et al., "Ancient Proteins from Ceramic Vessels at Çatalhöyük West Reveal the Hidden Cuisine of Early Farmers," *Nature Communications* 9, no. 1 (2018): 4064.

analysis on this collection, testing if after nearly a hundred years of storage the sherds in this collection contain sufficient residues and are not compromised too much by the intrusion of modern elements to infer on their original content and usage. Based on the outcomes of the study, recommendations for future research on material from these and other sites presented in the Andersson collection will be made.

Table 2. Sites involved in this study

Site name (English)	Site name (Chinese)	Period	Location
Dashiquan	大石圈	Majiayao	Gansu, Lanzhou
Siwashan	寺洼山遗址	Majiayao	Gansu, Lintao County
Caojiaping	曹家坪遺址	Qijia	Gansu, Heshui County
Zhujiazhai	朱家寨遺址	Qijia	Qinghai, Minhe County
Machangyuan	馬廠塬遺址	Xindian	Qinghai, Minhe County
Xiaxihe	下西河遺址	Xindian	Qinghai, Xining City
Kayue	卡約遺址	Kayue	Qinghai, Xining City

Methodology

For food residue analyses, the organic substance first needs to be extracted from the ceramic matrix. We employed different extraction methods that have been developed over the last few decades.[23] A selection of these results comparing extraction methods can be viewed in table A2. A breakdown of extraction methods used on each sample can be seen in table A4. The extracts were then characterised using analytical techniques, namely gas chromatography coupled with mass spectrometry (GC-MS). Samples which contained a sufficient amount of lipid extracts were then further characterised by GC-C-IRMS (Table A4). The former allows

[23] Papakosta et al., "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 Chrom"; Craig et al., "Earliest Evidence for the Use of Pottery"; Correa-Ascencio, and Evershed, "High Throughput Screening of Organic Residues in Archaeological Potsherds Using Direct Acidified Methanol Extraction."

for the identification and quantification of the lipid residues and biomarkers while the second allows for stable isotope information on these molecules to be acquired.

Results and Discussion

All samples analysed for this study yield low amounts of lipid material mainly consisting of free fatty acids and contaminates including phthalates (Table A3). A fine and coarse ware ceramic was sampled from each site in question to evaluate the effect of ceramic porosity and lipid retention. It was hypothesised that coarse wares would contain higher amounts of organic residues, as the ceramic body is more porous. While overall the course wares have higher lipid yields, this is seemingly more affected by the site location or microenvironment. Preservation overall remains consistent within sites rather than between ware types. This is highlighted by low lipid yields from both fine and coarse ware samples from Caojiaping and much higher yields from both samples from Dashiquan.

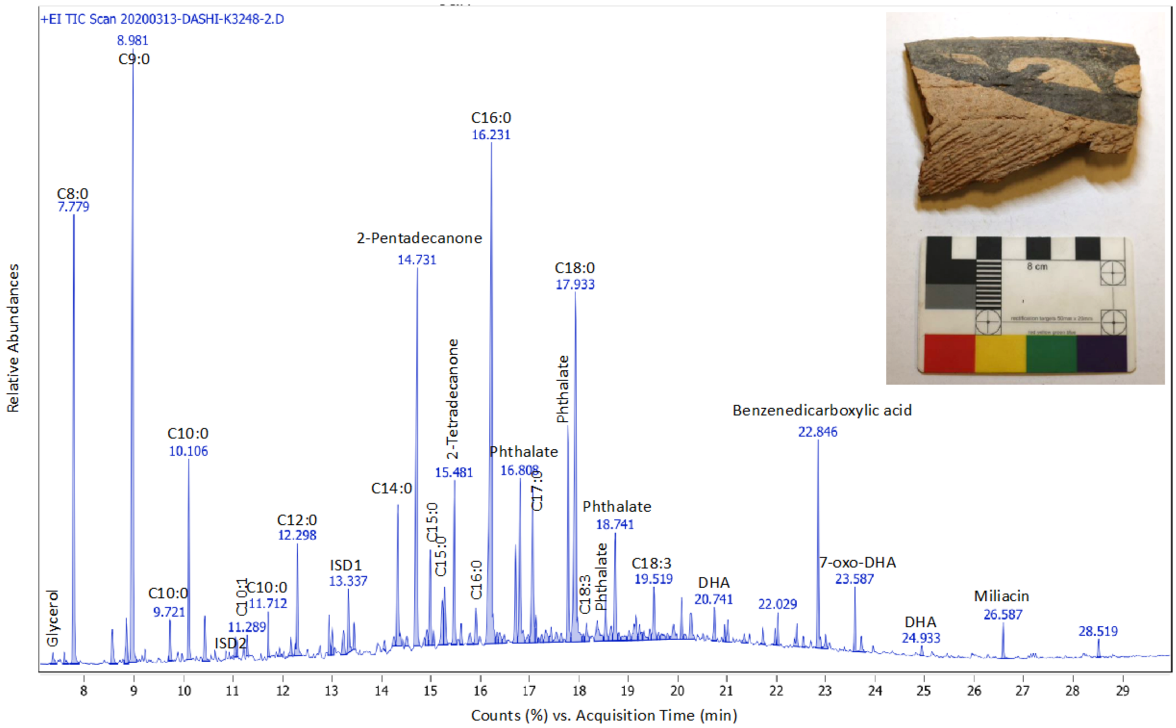

Figure 1. Example of chromatogram obtained by gas chromatography / mass spectrometry on the sample Dashi-K3248-002 from Dashiquan.

Free fatty acids make up the majority of compounds identified in the residue samples (Fig. 1). Palmitic (C16:0) and stearic acids (C18:0) are present and most abundant in the majority of samples (n=9) albeit in low quantities (<5µg/g). These compounds are the most abundant naturally occurring fatty acids, particularly in plants and animals.[24] Nine samples contain palmitic and stearic acid in abundances just at or slightly above the threshold for compound-specific isotopic analysis by GC-C-IRMS which further allows for the distinction of different

[24] Evershed, "Organic Residue Analysis in Archaeology: The Archaeological Biomarker Revolution."

food commodities and mixtures of more than one food commodities. Samples analysed showed biomarker evidence for C_4 plant processing which was not reflected in stable isotope analysis. Results mainly exhibit depleted $\delta^{13}C_{C16:0}$ and $\delta^{13}C_{C16:0}$ values (-30.4 to -27.05; Table 3) consistent with food commodities including C_3 plants, terrestrial animals (non-ruminant/wild ruminant) or freshwater fish. However, these results cannot be further distinguished using this approach alone.[25] For the wild ruminant, non-ruminant, and freshwater species, the use of the $\Delta^{13}C$ ($\delta^{13}C_{18:0}$–$\delta^{13}C_{16:0}$) proxy, which emphasizes differences in metabolic physiologies, may provide more distinction.[26] Results cluster between -1 to 2.5‰ $\Delta^{13}C$ which falls in the range established for non-ruminant and freshwater aquatic resources (Fig. 2). This is consistent with the faunal assemblages at sites from this time period, which mainly include pig and freshwater fish remains.[27] Isoprenoid alkanoic acids and long-chain (C18-C20) ω-(o-alkylphenyl) alkanoic acids (APAAs) were not identified in lipid extracts, and these results do not satisfy the full molecular criteria for aquatic products in archaeological pottery.[28]

Table 3. Summary of isotopic data of lipid residue analysis of ceramics from the Andersson collection

Sample Number	$\delta^{13}C_{16:0}$	$\delta^{13}C_{18:0}$	$\Delta^{13}C$
MCY-K2360-143	-29.041	-30.438	-1.397
DASHI-K3248-002	-30.4325	-29.21	1.2225
XIA-K2165-5	-29.8415	-29.942	-0.1005
XIA-K2165-3	-23.7195	-27.0955	-3.376
QY-2019-001	-28.813	-29.3015	-0.4885
QY-2019-002	-27.329	-27.6845	-0.3555
MCY-2019-001	-29.291	-28.9075	0.3835

[25] Lucquin et al., "Ancient Lipids Document Continuity in the Use of Early Hunter–gatherer Pottery through 9,000 Years of Japanese Prehistory."

[26] Oliver E. Craig, M. Forster, S. H. Andersen, E. Koch, P. Crombé, N. J. Milner, B. Stern, G. N. Bailey, and C. P. Heron, "Molecular and Isotopic Demonstration of the Processing of Aquatic Products in Northern European Prehistoric Pottery," *Archaeometry* 49, no. 1 (2007): 135–52; Lucquin et al., "Ancient Lipids Document Continuity in the Use of Early Hunter–gatherer Pottery through 9,000 Years of Japanese Prehistory"; Lucy Cramp, and Richard P. Evershed, "Reconstructing Aquatic Resource Exploitation in Human Prehistory Using Lipid Biomarkers and Stable Isotopes," in *Treatise on Geochemistry. Archaeology and Anthropology*, ed., H. D. Holland, and K. K. Turekian (Oxford: Elsevier, 2014), 319–339.

[27] Andrew Womack, "Crafting Community: Exploring Identity and Interaction through Ceramics in Late Neolithic and Early Bronze Age Northwestern China" (Ph.D. diss., Yale University, 2017).

[28] R. P. Evershed, M. S. Copley, L. Dickson, and F. A. Hansel, "Experimental Evidence for the Processing of Marine Animal Products and Other Commodities Containing Polyunsaturated Fatty Acids in Pottery Vessels," *Archaeometry* 50, no. 1 (2008): 101–13.

ZJZ-2019-0001	-29.3695	-27.054	2.3155
ZJZ-K2055-226	-27.928	-27.727	0.201

The low lipid yields, the skewed ratio between palmitic and stearic acids, combined with the high amount of contaminants and lack of additional biomarkers draws scepticism about the origin of such lipid residues and they may not all derive from the use of the vessel in antiquity. Solvent extracts show high amounts of phthalates and other contaminants, and this may have been a contributing factor in inconsistent isotopic data. There is a possibility that extracted lipid material is derived from cleaning or conservation techniques which may have been routine in the past. While there are no published notes about such treatment on the sherds in question, such treatments are noted for whole vessels of the same collection.[29] In addition, as this is an exploratory pilot study, the sample size is small compared to the size of the collection; as only two samples - one coarse and one fine - were taken and they cannot be representative for the pottery of the sites as a whole. There is a possibility some ceramics from additional contexts in this collection may yield more promising results. Additional samples may also benefit from purification using $AgNO_3$-impregnated silica gel packed in pipette columns to isolate the FAMEs from contamination products for IRMS analysis.[30] Both avenues of exploration should be explored in follow-up studies.

[29] Nils Palmgren, "Kansu mortuary urns of the Pan Shan and Ma Chang groups, China. Geological survey," *Palaeontologia sinica*, ser. d. vol. III. fasc. I. (Peiping [Peking]: Geological survey of China, 1934); Andersson, *Researches into the Prehistory of the Chinese*.

[30] Vasiliki Papakosta, Ester Oras, and Sven Isaksson. "Early Pottery Use across the Baltic--a Comparative Lipid Residue Study on Ertebølle and Narva Ceramics from Coastal Hunter-Gatherer Sites in Southern Scandinavia, Northern Germany and Estonia," *Journal of Archaeological Science: Reports* 24 (2019): 142–51.

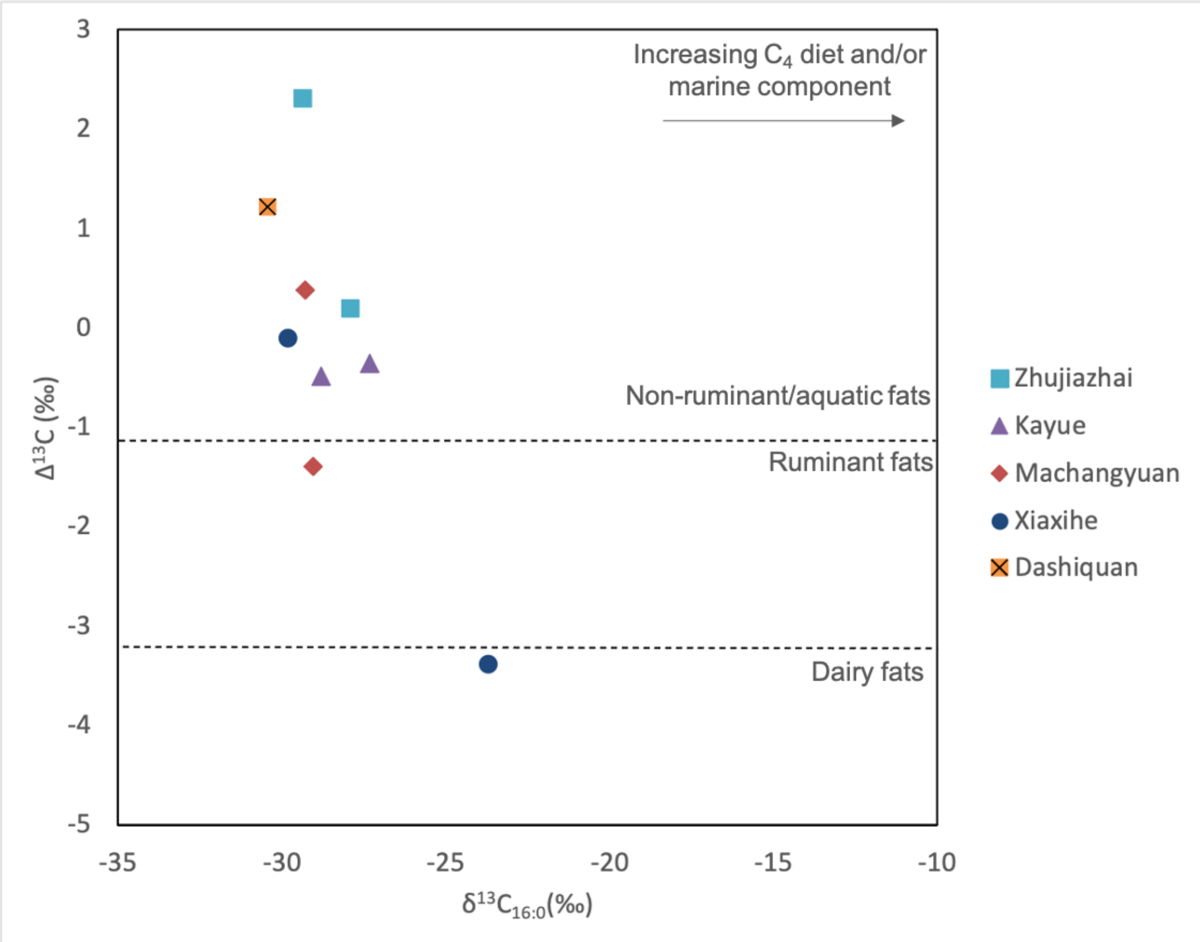

Figure 2. Plot of $\Delta^{13}C$ ($\delta^{13}C_{C18:0}$ - $\delta^{13}C_{C16:0}$) values against $\delta^{13}C_{C16:0}$ values of Late Neolithic and Early Bronze Age pottery from northwest China from the Andersson Collection. This plot allows for the distinction of different animal fats.[31]

In the analysis, a few notable biomarkers emerge. The molecule miliacin (Fig. 3) was identified in the residues of four samples: Dashi-K3248-002 and Dashi-K3241-038, from Dashiquan, K2165-3 from Xiaxihe, and CJP-2019-029 from Caojiaping (Fig. 1). This compound can be used as a biomarker for millet.[32] It has previously been identified in ceramics from South Korea dating to the Late Bronze Age (800–500 cal BC), ceramics from Poland dating from the Early to Middle Bronze Age, and samples from Early Celtic contexts in western France.[33] During the Neolithic, millet was the dominant crop produced in northwest China. By 7,000 BP, settlements

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[31] Cramp and Evershed, "Reconstructing Aquatic Resource Exploitation in Human Prehistory Using Lipid Biomarkers and Stable Isotopes."

[32] Carl Heron, Shinya Shoda, Adrià Breu Barcons, Janusz Czebreszuk, Yvette Eley, Marise Gorton, Wiebke Kirleis, et al. "First Molecular and Isotopic Evidence of Millet Processing in Prehistoric Pottery Vessels," *Scientific Reports* 6, no. June (2016): 1–9.

[33] Maxime Rageot, Angela Mötsch, Birgit Schorer, David Bardel, Alexandra Winkler, Federica Sacchetti, Bruno Chaume, et al., "New Insights into Early Celtic Consumption Practices: Organic Residue Analyses of Local and Imported Pottery from Vix-Mont Lassois," *PloS One* 14, no. 6 (2019): 1–19.

with domesticated broomcorn millet emerge in the western Loess Plateau at Dadiwan.[34] Isotopic evidence from human and animal bones in this region show that millet was a dominant dietary staple until 2000 cal BC.[35]

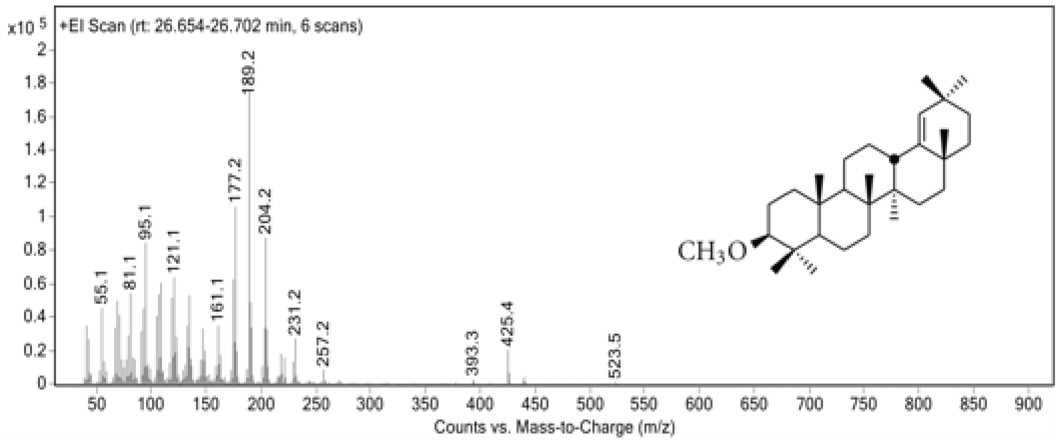

Figure 3. Mass spectra and structure of miliacin, a biomarker for millet as identified in four samples in this study. Shown here is an example mass spectra from K2165-3 from Xiaxihe.

Of the samples yielding millet biomarkers, only one (Xia-K2165-3 from Xiaxihe) dates to the Xindian period of the Early Bronze Age (1600-600 BC) and exhibits enriched $\delta^{13}C_{C16:0}$ values consistent with C_4 plant processing (Table 3). This may be due to resource mixing within the vessels and millet lipids contributing a smaller portion of the total extracted residue. Interestingly, this sample also yields large $\Delta^{13}C$ differences ranging in the isotopic area established for dairy fats (> − 3.3‰; figure 2).[36] This discrepancy may be due to enriched $\delta^{13}C_{C16:0}$ values being contributed by C_4 plants or contamination from burial environment, and/or subsequent excavation and curation. False positives for dairy have been observed previously when APAA's from aquatic resources make a significant contribution to a lipid mixture with wild ruminant fats,[37] however no APAA's were identified in this study. It is uncertain if this $\Delta^{13}C$ value denotes the presence of dairy products here, however it has been suggested that people might have moved towards pastoralism in the Early Bronze age in this area, making the presence of dairy not implausible.[38] Further residue analysis combined with isotope research and zooarchaeological work is needed to resolve the question of animal use and potential dairying practices.

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[34] Robert L. Bettinger, Loukas Barton, Peter J. Richerson, Robert Boyd, Hui Wang, and Won Choi, "The Transition to Agriculture in Northwestern China," *Developments in Quaternary Science* 9 (2007): 83–101.

[35] Ma et al., "Stable Isotope Analysis of Human and Faunal Remains in the Western Loess Plateau, Approximately 2000 Cal BC"; Liu, Li, Lisa Kealhofer, Xingcan Chen, and Ping Ji, "A Broad-Spectrum Subsistence Economy in Neolithic Inner Mongolia, China: Evidence from Grinding Stones," *Holocene* 24, no. 6 (2014): 726–42.

[36] M. S. Copley, R. Berstan, S. N. Dudd, G. Docherty, A. J. Mukherjee, V. Straker, S. Payne, and R. P. Evershed, "Direct Chemical Evidence for Widespread Dairying in Prehistoric Britain," *Proceedings of the National Academy of Sciences of the United States of America* 100, no. 4 (February 18, 2003): 1524–29.

[37] Evershed et al., "Experimental Evidence for the Processing of Marine Animal Products and Other Commodities Containing Polyunsaturated Fatty Acids in Pottery Vessels."

[38] Jaffe and Hein, "Considering Change with Archaeological Data: Reevaluating Local Variation in the Role of the 4.2 K BP Event in Northwest China."

Dehydroabietic acid and 7-oxo-dehydroabietic acid are present in four samples. These compounds are indicative of resin derived from trees in the Pinaceae family.[39] These types of resins are common in archaeological contexts. They have been identified, for example, as waterproofing agents and glues, and in funerary practices.[40] No visible resin in the form of a surface treatment is present on the samples in question, though this does not negate the possibility of its existence.[41] Nevertheless, there could also be different reason for the presence of these residues. For instance, Reber et al.[42] showed that these compounds are present in ceramic wares when wood from trees in the Pinaceae family is used as fuel for pottery firing and cooking. These compounds can derive from the thermal dehydrogenation of abietic acid naturally present in the wood, and when this occurs in an oxygenated environment, produces 7-oxo-dehydroabietic acid.[43] In the present case, it is therefore most likely these compounds derive from the production process, which likely involved wood from the Pinaceae family.

Conclusions

This pilot study revealed that the preservation of organic residue was very low (less than 5 µg/g) in the 14 sherds analysed. However, given that this is a pilot study the sample size examined here is limited, and future examinations with larger sample sizes, a selection of samples from different contexts, and/or different methods may yield different results. In spite of these limitations, the study has also yielded a few interesting results. For instances, it has been shown that the lipid yields are determined more by the burial environment than by sherd composition (i.e., coarse vs fine wares). Finds from other sites held in the Andersson collection may thus have higher lipid yields that provide more robust insights into food preparation and subsistence practices. The lipids extracted in this study show that the vessels in question were used to process non-ruminant and freshwater aquatic resources which is consistent with faunal assemblages at these sites that include pig and freshwater fish remains. Millet may also have been included in the meals prepared and/or served in these vessels which is consistent with

[39] Erika Ribechini, Francesca Modugno, Maria Perla Colombini, and Richard P. Evershed, "Gas Chromatographic and Mass Spectrometric Investigations of Organic Residues from Roman Glass Unguentaria," *Journal of Chromatography. A* 1183, no. 1–2 (2008): 158–69.

[40] Ilaria Bonaduce, and Maria Perla Colombini, "Characterisation of Beeswax in Works of Art by Gas Chromatography-Mass Spectrometry and Pyrolysis-Gas Chromatography-Mass Spectrometry Procedures," *Journal of Chromatography. A* 1028, no. 2 (2004): 297–306; M. P Colombini, G. Giachi, F. Modugno, P. Pallecchi, and E. Ribechini, "The Characterization of Paints and Waterproofing Materials from the Shipwrecks Found at the Archaeological Site of the Etruscan and Roman Harbour of Pisa (Italy)," *Archaeometry* 45, no. 4 (2003): 659–74; Thibaut Devièse, Camille Vanhove, Rémy Chapoulie, Philippe Blanchard, Maria Perla Colombini, Martine Regert, and Dominique Castex, "Détermination et Fonction Des Substances Organiques et Des Matières Minérales Exploitées Dans Les Rites Funéraires de La Catacombe Des Saints Pierre-et-Marcellin à Rome (Ier-IIIe Siècle)," *De Corps En Corps: Traitement et Devenir Du Cadaver*, 2010. https://www.academia.edu/download/31537691/MSHA_2010.pdf.

[41] Jelmer Eerkens, "The Preservation and Identification of Piñon Resins by GC-MS in Pottery from the Western Great Basin." *Archaeometry* 44, no. 1 (2002): 95–105.

[42] E. A. Reber, M. T. Kerr, H. L. Whelton, and R. P. Evershed, "Lipid Residues from Low-Fired Pottery," *Archaeometry* 61, 1 (2018): 131-144.

[43] Maria Perla Colombini, and Francesca Modugno, *Organic Mass Spectrometry in Art and Archaeology* (Chichester: John Wiley & Sons, 2009).

both palaeobotanical and isotope data from the region. There is even an indicator for the potential presence of dairy, though this will have to be tested with larger-scale research on ceramic residues combined with isotope and faunal data. The study has furthermore provided some insights into the nature of the fuel used in pottery production and/or cooking which seems to have involved wood from the Pinaceae family.

Future work on this collection would benefit from employing more advanced extraction methods such as supercritical fluid extraction, as this method yielded the higher lipid quantities in this study and presents the advantage of being non-destructive.[44] Additional purification using $AgNO_3$-impregnated silica gel packed in Pasteur pipette columns for FAME isolation would be beneficial to mitigate contamination hereafter.[45] Future work may also benefit from examining ceramics from other sites which may have a better lipid preservation. In particular, it might be worth-while to investigate complete ceramic vessels retrieved from graves as some of them show evidence for having contained food. The issue remains, of course, that at least part of the collection - and especially the complete vessels shown in the museum display - underwent cleaning with harsh chemicals that likely destroyed much of the remaining residue. Future work thus has to be preceded by an investigation into museum records to learn about previous cleaning and restoration methods that may prevent successful residue analysis. This study has thus shown the potential and challenges for the study of such a unique collection and suggested avenues for research on this collection in particular as well as legacy collections at large.

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[45] Papakosta, Oras, and Isaksson, "Early Pottery Use across the Baltic--a Comparative Lipid Residue Study on Ertebølle and Narva Ceramics from Coastal Hunter-Gatherer Sites in Southern Scandinavia, Northern Germany and Estonia."

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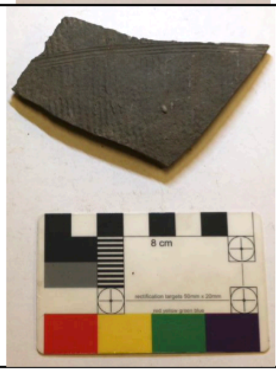
Appendix I Sample Overview

Table A1. Ceramic samples analysed in this study

Sherd number	Box no./label	Site	Period	Photo
K2165:3	8-28B	Xiaxihe	Xindian	
K2165:5	8-28B	Xiaxihe	Xindian	
CJP_2019_0029	Caojiaping (T.C.P) GANSU sherds	Caojiaping	Qijia	

CJP_2019_0039	17-88/16-83	Caojiaping	Qijia	
TEMP-2698	16-79	Siwashan	Majiayao	
TEMP-2715	16-79	Siwashan	Majiayao	
ZJZ_2019_0001	8-24	Zhujiazhai	Qijia	

K2055:226	8-20	Zhujiazhai	Qijia	
QY_2019_0001	8-13	Qiayao	Kayue	
QY_2019_0002	8-12	Qiayao	Kayue	
K3248:2	9-21A	Dashiquan	Majiayao	

K3241:38	9-21A	Dashiquan	Majiayao	
MCY_2019_0001	10-25A	Machangyan	Xindian	
K-2360:143	10-23	Machangyan	Xindian	

Table A2. Molecules identified in 3 selected samples in this study

	Glycerol	Caproic acid	Enanthic acid	Caprylic acid	Pelargonic acid	Capric acid	Undecylic acid	Lauric acid	Myristic acid	Pentadecanoic acid	Palmitic acid	Margaric acid	Stearic acid	Oleic acid	α-Linolenic acid	Phthalates	Benzoic Acid	Dehydroabietic acid (DHA)	7-oxo-DHA	Cholesterol	Abietic Acid	Monostearin
DASHI-K3248-02 (SFE-GCMS)	x			x	x	x	x	x	x	x	x	x	x		x	x	x	x	x			
SIWA-2715 (SFE-GCMS)	x			x	x	x		x	x	x	x	x				x	x	x	x	x		
XIAXIHE-K2165-3 (SFE-GCMS)	x			x	x	x	x	x	x	x	x	x	x	x	x	x	x				x	x
DASHI-K3248-02 (CH3-GCMS)																x						
SIWA-2715 (CH3-GCMS)		x	x	x	x						x					x						
XIAXIHE-K2165-3 (CH3-GCMS)		x	x	x	x	x			x		x		x			x						
DASHI-K3248-02 (H2SO4-MeOH)											x	x	x	x		x						
SIWA-2715 (H2SO4-MeOH)											x		x			x						
XIAXIHE-K2165-3 (H2SO4-MeOH)										x	x	x	x	x								

Table A3. Summary of lipid extracts from each sample in this study

	Lipid extract summary
XIA-K2165-3	High concentrations of palmitic and stearic acids for IRMS, abietic acids, miliacin, saturated and unsaturated free fatty acids, monostearin
XIA-K2165-5	High concentrations of palmitic and stearic acids for IRMS, saturated and unsaturated free fatty acids
CJP-2019-0029	Phthalates, saturated free fatty acids, concentrations not high enough for IRMS, Miliacin
CJP-2019-0039	Phthalates, saturated free fatty acids, concentrations not high enough for IRMS
SIWA-TEMP-2698	Phthalates, saturated free fatty acids, concentrations not high enough for IRMS, Squalene
SIWA-TEMP-2715	Phthalates, saturated free fatty acids, concentrations not high enough for IRMS, cholesterol, DHA
ZJZ-2019-0001	High concentrations of palmitic and stearic acids for IRMS, saturated free fatty acids, phthalates
ZJZ-K2055-226	High concentrations of palmitic and stearic acids for IRMS, saturated and unsaturated free fatty acids, phthalates
QY-2019-0001	High concentrations of palmitic and stearic acids for IRMS, saturatedfree fatty acids, phthalates
QY-2019-0002	High concentrations of palmitic and stearic acids for IRMS, saturatedfree fatty acids, phthalates
DASHI-K3248:2	High concentrations of palmitic and stearic acids for IRMS, Abietic acids, miliacin, saturated and unsaturated free fatty acids
DASHI-K3241:38	Phthalates, unsaturated and saturated free fatty acids concentrations not high enough for IRMS, DHA
MCY-K2360-143	High concentrations of palmitic and stearic acids for IRMS, saturated free fatty acids, phthalates and contaminants
MCY-2019-001	High concentrations of palmitic and stearic acids for IRMS, saturated and unsaturated free fatty acids, phthalataes and contaminants

Table A4. Reference of extraction and characterisation procedures samples have been subjected to in this study.

	Chlorform & Methanol Extraction	Acidified Methanol Extraction	Supercritical Fluid Extraction	GC-MS	GC-C-IRMS
XIA-K2165-3	X	X	X	X	X
XIA-K2165-5	X	X	X	X	X
CJP-2019-0029	X		X	X	
CJP-2019-0039	X		X	X	
SIWA-TEMP-2698	X		X	X	
SIWA-TEMP-2715	X		X	X	
ZJZ-2019-0001	X	X	X	X	X
ZJZ-K2055-226	X	X	X	X	X
QY-2019-0001	X	X	X	X	X
QY-2019-0002	X	X	X	X	X
DASHI-K3248:2	X	X	X	X	X
DASHI-K3241:38	X		X	X	
MCY-K2360-143	X	X	X	X	X
MCY-2019-001	X	X	X	X	X