What's cooking in the Indus Civilisation? Investigating Indus food through ceramic lipid residue analysis

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This dissertation is submitted for the degree of Doctor of Philosophy

August 2019

This dissertation is the result of my own work and includes nothing which is the outcome of work done in collaboration except as declared in the Preface and specified in the text.

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The length of the dissertation does not exceed the word limit set by the Degree Committee for the Department of Archaeology.

"Even the most ordinary dish follows a satisfying arc of transformation, magically becoming more than the sum of its parts. And in almost every dish, you can find, besides the culinary ingredients, the ingredients of a story: a beginning, a middle, and an end… How many of us still do the kind of work that engages us in a dialogue with the material world that concludes with such a gratifying and delicious sense of closure?"

Michael Pollan, *Cooked*

Abstract

This thesis investigates which products were used in ceramic vessels by populations of the Indus Civilisation through ceramic lipid residue analysis. It uses concepts of food choice and foodways to explore the culinary practices of Indus populations.

Specifically, the thesis examines how vessels may have been used in urban and rural Indus settlements located in northwest India during the urban period (*c*. 2600/2500-1900 BC), and identifies whether changes in vessel use occurred in the post-urban period (*c*. 1900- 1300 BC). It also analyses a small sample of Arabian and Indus-origin vessels from the Umm an-Nar period (*c*. 2400-2000 BC) in the Sultanate of Oman.

As the first large-scale investigation into Indus foodstuff and vessel-use using lipid residue analysis, the thesis first tests the viability of the method in the South Asian context. It compares lipid yields from pottery recovered from collections, washed pottery from recent excavations, and unwashed pottery from fresh excavations. It then integrates the molecular and compound-specific isotopic data with available bioarchaeological evidence from the study region to reconstruct which products were used in vessels at different sites. The results indicate that overall, lipid residues are typically poorly preserved in Indus vessels, but the acidified methanol extraction technique provides a good lipid recovery rate. No significant differences in lipid yield are observable between washed pottery samples and those collected directly from the field, which suggests that washed pottery may serve as a good source for samples for future lipid residue analysis. However, it is difficult to interpret lipid evidence from samples obtained from collections with limited contextual information, suggesting that future lipid analyses in South Asia must be carefully planned to yield optimum results.

The molecular results indicate that animal fats were primarily used in vessels, with minor indications of plant products. The compound-specific results suggest processing of different animal fats, primarily non-ruminants, however, equivocally, many vessels may also have been used to store or process mixtures of products. Inter-site differences in vessel use are observed, but there are broad similarities in vessel-use between urban and rural sites. No change over time in vessel use is observed at rural sites, suggesting stability of food choices. No correlations are observed between vessel-form and products used in vessels, indicating their multifunctionality. These results provide a new means by which to investigate Indus foodways, broadening our understanding of what ancient Indus cuisine at both urban and rural settlements may have looked like.

Acknowledgements

A big, heartfelt thank you to my supervisors, Dr Cameron Petrie and Dr Tamsin O'Connell, who helped shape a curiosity in organic residues and a fascination for the Indus Civilisation into a full-fledged PhD project. Thank you both for giving me the support I needed to see this project through. Tamsin, thank you for Thursday meetings, and making me think/write more precisely. Cameron, thank you for being one of the most approachable and available supervisors a PhD student could ask for, and for the emojis and long chats.

Thank you to my advisors who helped throughout the process: Prof. Oliver Craig, Bioarch, University of York and Prof. Carl Heron, Scientific Research, British Museum. My training in lipid residues would have never happened if not for your support, Ol; I'm grateful for the time and resources you gave to me, and glad for that chance conversation at Must Farm that started this project from a 'let's try it' to what it is now. Thanks also to you, Carl, for your kind words, support and encouragement, and for hosting me at the British Museum. At York, I would like to thank Dr Shinya Shoda, Dr Miriam Cubas, Dr Anita Radini and Dr Alex Lucquin for training me, running my samples and answering all my questions during my visits. Miriam, a special thanks to you for everything and making me feel so much at home. Anita, thank you for your support at York even during tumultuous times. Many thanks also to Matthew von Tersch for his help in the lab and with software; and a special thanks to Dr Alice Toso and Dessislava Veltcheva at Bioarch. At the British Museum, Dr Blandine Courel's guidance and methodical advice significantly improved my labwork. Thank you to Chris Mussell for solving all my technical problems, for inviting me to speak at LBMSDG, and to Adrià Breu Barcons, Edward Standall and Sana Boekenfast for being my 'lipids4lyf buddies'.

Financial support for my PhD came from Cambridge Trust and Nehru Trust for Cambridge University. A very special thanks to Dr Anil Seal for his support. Parts of this research were carried out as part of the *TwoRains* project

[\(http://www.arch.cam.ac.uk/research/projects/two-rains\)](https://www.arch.cam.ac.uk/research/projects/two-rains), which is funded by a Horizon 2020 European Research Council (ERC) Consolidator Grant (H2020, 648609), and is based in the McDonald Institute for Archaeological Research at the University of Cambridge. *TwoRains* is being carried out in collaboration with Prof. Ravindra Nath Singh and the Department of AIHC and Archaeology at Banaras Hindu University. Some

samples analysed in this thesis were also acquired from the *Land, Water and Settlement* project [\(http://www.arch.cam.ac.uk/research/projects/land-water-settlement\)](https://www.arch.cam.ac.uk/research/projects/land-water-settlement), whose work has laid the foundations for the *TwoRains* project. Thanks also to Sidney Sussex College, Anthony Wilkin Trust, Smuts Memorial Fund and McDonald Institute for Archaeological Research for research and travel funding.

This thesis would have been impossible without the tireless efforts of the *Land, Water and Settlement* and *TwoRains* team, and support of Prof. R.N. Singh. I am especially grateful for Prof. Singh's help with arranging all the necessary permissions for analysis of pottery in this thesis. I am also grateful to Prof. Vasant Shinde, Deccan College, Pune, for granting me access to pottery from Farmana and Rakhigarhi and for his support. Very special thanks to Pandeyji, Dr Dheerendra Pratap Singh, Dr Aftab Alam, Dr Amit Shah, Aarti Chowdhary, Sudarshan Chakradhari, Banaras Hindu University, and Dr Vikas Pawar, MDU, Rohtak. Thank you for your hospitality in Varanasi, your friendship, and for organising many practical details during fieldwork at Lohari Ragho I. Thanks also to Balwan Deswal for his assistance with the collection of ethnographic pots, and for Ramjatanji for his excellent cooking. At Deccan College, special thanks to Dr Nilesh Jadhav, Yogesh Yadav for their assistance with accessing materials from Farmana and Rakhigarhi.

Very special thanks to Michele Degli Esposti, Prof. Alessandra Avanzini and the Office of the Adviser to His Majesty the Sultan for Cultural Affairs for granting me permission to analyse samples from Stone Tower I, Salut, and for hosting my visit to Nizwa in 2015. A big thanks to Dr Peter Magee, Dr Steve Karacic, Dr Charlotte Cable and Dr Chris Thornton for introducing me to the archaeology of the Arabian Peninsula.

In Cambridge, there are countless people to thank. The Dorothy Garrod isotope lab group: Alice Rose, Sam Leggett, Dr Emma Lightfoot, Dr Cemre Ustunkaya, Emily Tilby, Danny Buss, Catherine Kneale and of course, Tamsin, to thank for all the fun lab meetings, for introducing me to R, making me work hard to make nice plots, think about variability and really question statistics! Our lab group has a special place in my heart. Thanks to the *TwoRains* group for all the fun and trying times during fieldwork, and for all the cake. Many thanks also to different members of the Pitt-Rivers lab and Department of Archaeology for conversations, encouragement and advice throughout the PhD process; special shout-out to Dr Penny Jones, Dr Jennifer Bates, Dr Janine Ochoa, Dr Eva Meharry,

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Dr Jess Thompson, Jeremy Bennet, Dr Hermine Xhauflair, Dr Laerke Recht, Dr. Nathan Wright, Dr Jess Beck, Laure Bonner, Jess Rippengal, Tonko Rakjovaca. A special thanks to Danika Parikh for being such a support for me in the department, for the pottery drawings and photos, and for all the sea-salt chocolate-chip cookies.

I would also like to thank Ananya Mishra and Danika Parikh for our collective effort with the Untold Histories Museum tours at MAA. They have brought a lot of meaning to me in my final year of the PhD.

Thank you to Ruth Hatfield for driving me to Huntington in a mad rush to get my passport before I left for Oman in 2015. I promised to thank you in my acknowledgements and here you are!

Thank you to Rashmi Singh and Dr Asiya Islam for their friendship, love for food, and for that (failed) trip to the beach. To Dr Wiebke Lamping and Dr Louise Purdue, thanks for making Thoday Street such a fun place to live; I will miss us commiserating together about academia and life in general.

Thank you to my friends near and far: Chhavi Kotwani, Megha Thumbunkel, Tanya Ginwala, Christine Marie Dickerson, Mine Serizawa, Meghna Singh, Neha Aurora, Rudi Vanzin, Alice Netherton, Sitara Chowfla, Nikita Ramachandran, Sebastian Selven and Varun Khanna. Many thanks also to my friends in Copenhagen who took me into the Symfuni, Plenum and DTU family.

To Meghna Nag Chowdhuri, who knows what I would have done without you! And to my other family in Cambridge: Manuel Piña Dreyer and Vibhu Tewary, thank you for all the food, fun, travels, movie-watching and gym sessions.

Thank you to my family, my dear parents and sister for their never-ending love and support. Papa, thank you for your excitement about my research and helping me organise so many aspects of my data collection in Delhi. Mummy, thank you for your constant support. Sachita, thank you for your understanding and love; I can't believe we both did it! Thank you to Tara and Kalyani didi for their endless care, and to Fuzzy.

And finally, to François: thank you for all your love, and for giving me gentleness, sustenance, and perspective when I needed it the most.

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List of Abbreviations

 Δ^{13} C...Difference in stable carbon isotope values of C_{18:0} and C_{16:0} fatty acids

- δ^{13} C...Stable carbon isotope ratio
- ALK…*n*-alkane
- ALM…Alamgirpur
- BSJ…Black-Slipped Jar
- DAGs…Diacylglycerols
- EMH…Early Mature Harappan
- FA…Fatty acid
- FRN…Farmana
- GC…Gas-Chromatography
- GC-c-IRMS…Gas Chromatography-combustion-Isotopic Ratio Mass Spectrometry
- GC-FID…Gas-Chromatography Flame-Ionisation Detection
- GC-MS…Gas Chromatography-Mass Spectrometry
- ISM…Indian Summer Monsoon
- ITCZ…Intertropical Convergence Zone
- KLB…Kalibangan
- KNK…Khanak
- LH…Late Harappan
- LHRI…Lohari Ragho I
- LMH…Late Mature Harappan
- LWS…Land, Water and Settlement Project
- MAGs…Monoacylglycerols
- MD…Mohenjo-daro

MSDI...Masudpur I

MSDVII...Masudpur VII

RGR...Rakhigarhi

STI...Stone Tower I, Salut

TAGs...Triacylglycerols

TLE...Total Lipid Extract

VPDB...Vienna Pee Dee Belemnite

Chapter One

Introduction

1.1. Food in the Indus Civilisation: research questions and approach

This thesis is focused on understanding which types of foodstuff were processed in ceramic vessels from settlements of the Indus Civilisation, South Asia's first urban civilisation, using organic residue analysis. The Indus Civilisation (*c*.3000-1300 BC) is one of the great early complex civilisations of the Old World (Marshall 1931; Kenoyer 1998; Possehl 2002; Wright 2010), but is often neglected in discussions about early urbanism due to its enigmatic nature and the lack of decipherable texts (Wright 2010; Petrie 2013). Spread across a vast geographic region, including large parts of modern Pakistan, northwest and western India and Afghanistan, our present understanding of settlement distribution suggests that the Indus Civilisation was likely the most geographically extensive of all the early Old World civilisations (cf. Wheeler 1968:4; Possehl 2002; Agrawal 2007; Petrie et al. 2017). The Indus Civilisation also occupied an environmentally diverse and climatically dynamic stretch of South Asia and experienced climatic instability in 4.2 ka BP (*c*.2100 BC) (Wright 2010; Petrie et al. 2017). Questions about environmental and regional diversity but apparent 'cultural uniformity' have pervaded archaeological literature on the Indus Civilisation (Marshall 2004[1931]; Wheeler 1953; Allchin and Allchin 1968), but for the past few decades, regional ecological and cultural variation within the Indus Civilisation has been increasingly recognised and characterised. Thus, the Indus Civilisation provides an excellent setting to investigate how quotidian human activities such as the creation of food and use of vessels are manifested in early urban societies, and how they respond to cultural or climatic change.

This study investigates lipid residues from vessels recovered from a range of settlements located in northwest India dated to the urban period (*c*. 2600/2500-1900 BC), and tests whether there was continuity or change in vessel-use practices in the post-urban period (*c*. 1900-1300 BC), a period hypothesized to be marked by dramatic societal

transformations and potential climatic instability (Staubwasser 2003, Dixit et al. 2014a/b). Three central questions are addressed:

- 1) Are there difference and/or similarities between foodstuff processed between different settlements?
- 2) Is there a relationship between vessel form and the product(s) processed in the vessels?
- 3) Did environmental and/or social changes impact what foodstuffs were processed in vessels after 4.2 ka or *c*.2100 BC?

This thesis answers these questions using organic residue analysis, specifically, lipids adsorbed within ceramic vessels. It integrates the lipid residue results with available palaeoecological, archaeobotanical, zooarchaeological and isotopic evidence from the Indus Civilisation, specifically from northwest India, to achieve a comprehensive understanding of how Indus populations grew crops; raised and managed their animals; and processed these products to create food. It also compares the results obtained with vessel products found in different regions from prehistoric contexts.

As lipid residue analysis has not been conducted at a large scale in Indus archaeology, this thesis also tests the viability of the method within the South Asian context. Methodological questions addressed by the thesis include:

- 1) Are lipids preserved in vessels from Indus sites in arid and seasonally wet environments?
- 2) What is the effect of post-excavation treatment (washing of sherds and storage) on lipid yield?

As a full range of experimental studies was outside the remit of this thesis, these preliminary methodological questions are key to address the future of organic residue analysis and biomolecular studies within South Asian archaeology.

1.2. Theoretical Relevance

The questions addressed by this thesis are important to archaeologists interested in ancient complex societies; researchers investigating the Indus Civilisation; food historians specialising in South Asia; and researchers investigating archaeological lipid residues in arid and semi-arid environments. This section will highlight why investigating ancient food
is of interest to archaeologists, and its relevance to research on ancient and contemporary South Asia and the Indus Civilisation.

1.2.1. Why study ancient food?

Humans eat every day to survive; and the regular practice of ingesting food makes it inherently 'about the body' (Ray and Srinivas 2012), as food is biosynthesized to become a part of the flesh, bone and muscle. It has been argued that in converting some part of their environment into food, humans create a peculiarly powerful semiotic device (Appadurai 1981: 494). More than any other human activity, food creates the individual as well as the community through the daily practices of eating, forming the ultimate *habitus* practice (Bordieu 1977; Atalay and Hastorf 2006). Thus, acts of acquiring, preparing and consuming foods, as regular, repeated actions, form the basis of the social (D'Anna and Jauss 2015). These acts also structure the lives of preparers and consumers, serving as a mnemonic medium through which *habitu*s is expressed and learned over generations (Bourdieu 1977; Atalay and Hastorf 2006; McCorriston 2011). As a 'highly condensed social fact', food encodes all kinds of cosmological meanings and 'collective representation' (Levi Strauss 1966; Appadurai 1981: 494). Depending on its contexts and function, food can become the site where ideology and power relations are expressed or negotiated, "signalling rank and rivalry, solidarity and community, identity and exclusion, and intimacy or distance" (Appadurai 1981: 494). Thus, commensal acts create and reinforce social relations.

Investigating food archaeologically provides a powerful lens to investigate the relationship between humans and the environment, plants, and animals, as well as gain insight into socio-political relationships. Archaeological investigations on food have ranged from traditional considerations of calories, crops, and subsistence to investigating symbolic dimensions of food used for the negotiation of power (Dietler 2003: 272). A variety of material culture has been studied to investigate food practices, including ceramics, faunal and botanical assemblages, human remains, individual domestic spaces, refuse deposits and ritual structures (Dietler 2003; Kerner et al. 2015). However, given that archaeology rarely has access to human thoughts or intentions, understanding cultural constructions of food and its edibility, delving into food avoidance, taboos or preferences (such as in anthropology, see Leach 1964; Douglas 1966; Tambiah 1969; Harris 1998; Minz and Du Bois 2002) is challenging.

1.2.2. Food practices (and politics) in South Asia

A great diversity of food traditions across South Asia is well-recognised (Appadurai 1981; Nandy 2004), but investigations into the archaeology of food, specifically from prehistoric contexts, are in a relatively nascent stage. Although there is an increase in the use of bioarchaeological techniques to study specific aspects of ancient food (for reviews, see Thomas and Joglekar 1994; Fuller 2002, 2006; Murphy and Fuller 2016), these are often characterised as subsistence strategies and their social meaning is rarely investigated. In historical periods, concepts like the relationship between food and ideas of the body, personality, health, religiosity, and identity have been primarily explored through historical, philosophical and religious texts (e.g. Khare 1976a, 1976b; Malmoud 1996; Zimmerman 1999), with important work also done in archaeology (e.g. Morrison 1994, 2001; Smith 2006). Anthropological studies of contemporary South Asian communities explore how notions of belonging and exclusion are tied into culinary choices, particularly through religious and caste identity (Appadurai 1981; Daniel 1987; Achaya 1994; Khare 1976a, 1976b, 1992; Desai 2008; Rege et al. 2009; Staples 2008, 2016), as well as how South Asian diaspora communities negotiate their identity in globalised, multicultural urban spaces through their alimentary choices and experiences (Kuper 1967; Bharati 1967; Ray and Srinivas 2012; Parveen 2016). Although food has never been apolitical in the subcontinent, present-day gastro-politics of food have transcended the private spaces of the household and entered the realms of nationalistic politics, especially in India. Meat consumption (especially cattle and buffalo meat) has become a virtual battleground where debates about identity, loyalty, morality, and nationality are waged (O'Toole 2003; Gorringe and Karthikeyan 2014; Deepak 2018; Gowen 2018). Similarly, the study of history and archaeology, particularly of the Indus Civilisation, has become highly politicised (Menon and Mishra 2018; Joseph 2018). In this current climate, investigating the connections between archaeology, food, and identity are highly relevant.

1.2.3. Food in ancient South Asia and the Indus Civilisation

Within the realm of South Asian prehistory, there is a growing focus on understanding processes of domestication of crops and animals in the Neolithic period across South Asia (Fuller 2006; Murphy and Fuller 2016), but there has been a strong preoccupation with research on the Indus Civilisation (Murphy and Fuller 2016). Despite this focus, there are long-held views about homogeneity of food practices, particularly in terms of crop choice, across the Indus Civilisation (see Chapter Two). These ideas have been challenged by

recent research indicating regional diversity of practices (Madella and Fuller 2006; Petrie and Bates 2017). Regional variations in environment, geography and crop availability within the Indus Civilisation suggest that acts of acquiring, preparing, storing, and eating food may be representative of unique culinary choices within different settlements and regions of the Indus Civilisation. Studying food choice and their change or stability across time may reveal a deeper understanding of the expression and maintenance of regional, rural/urban or relational identities in the Indus Civilisation.

Till now, discussion of food production and variability in the Indus Civilisation has been focused on crops (e.g. Petrie et al. 2016; Petrie and Bates 2017), and there has not been much effort to explore the role of plants *and* animals as food in Indus society (e.g., Fuller 2005). This thesis explores the meaningful creation of food by Indus populations, and studies it with relation to specific categories of pottery. This approach emphasizes the active use of food within ceramics against the relatively static picture of food use often presented by faunal and botanical analysis (Jones 1999: 57).

This thesis uses material from, and complements the research questions posed by the *Land, Water and Settlement* and *TwoRains* projects*,* which are two inter-disciplinary collaborative endeavours directed by Dr Cameron Petrie, University of Cambridge and Prof. R. N Singh, Banaras Hindu University. These projects have been investigating cultural and environmental landscape transformation in the plains of northwest India between *c*.3000-1500 BC, and looking into the extent of climatic, environmental, hydrological and settlement change following the weakening of the Indian Summer Monsoon (ISM) in northwest India. As key debates in Indus archaeology centre around the hypothesised relationship between climate and cultural change, food production, and the development and decline of urbanism, these projects are interested in investigating the resilience and sustainability of this region of the Indus Civilisation in the face of variable weather conditions, or climate change. This thesis examines Indus consumption and culinary choices at two scales, adopting both a synchronic approach (vessel-usage across sites during the urban period), and a diachronic approach (change in vessel-usage over time) to understand how an ancient society consciously coped with diverse and varied ecologies, and if they adapted to change in the face of shifting environmental parameters as reflected in everyday acts of cooking, consuming and storing. Thus, this research is distinct yet complementary to the greater aims of both projects.

1.3. Methodological Rationale: why ceramic lipid analysis?

One of the most ubiquitous artefacts found in the archaeological record, pottery has been widely studied and used as both a cultural and chronological marker in archaeology (Rice 1987), and especially in South Asian archaeology (Dales and Kenoyer 1986; Krishnan 2018). However, manufacturing process, shape, style or decoration are not the only defining criteria of pottery: their cultural *use* and everyday function would have served as an integral part of how they were experienced in the past. Additionally, processes of transmission of learning how to process foodstuff are closely linked to social identity (Arthur 2002, 2014; Craig et al. 2015). The study of the use of vessels provides a deeper understanding of what types of natural products were used, and allows questions related to seasonality, spatial organisation or the structuring of domestic spaces to be raised (Arthur 2002, 2014; Vieugue 2016).

Ceramic vessels are designed within limits of size and form in order to perform certain functions. For example, storage and transport vessel sizes vary according to the product form (liquid vs. solid) and the distance these items need to be transported, and serving vessels usually are smaller than storage and processing vessels to make them suitable for individual portions (Miller 1985). Researchers have used ethnographic data and morphological characteristics of archaeological vessels to correlate specific vessels forms with primary vessel function (Henrickson and McDonald 1983; Rice 1996, Abbink 1999; Skibo 2013). The difference between 'vessel function', which refers to the broad roles or activities or capabilities of ceramics, for example as containers (for storage, processing, transport), and 'vessel use', which refers to the specific way(s) in which a vessel was brought into service for a particular purpose has also been discussed (Rice 1996).

The study of adsorbed organic residues in ceramic vessels provide a unique means to study the cultural use of vessels. Depending on the number of vessels studied, it may also provide a means to study vessel function, however this is often difficult to ascertain, and vessels often are multifunctional (Heron and Evershed 1993). Details of the method are provided in Chapter Three. Investigations of lipid residues in pottery have revealed fascinating insights into the transformation of organic products for cultural uses across multiple spatial and temporal contexts in the world. There has been a heavy focus on characterising what products were processed in Neolithic pottery, a period which generally marks the transition to an agricultural lifestyle which includes the managing of various

animals, growing of crops and exploitation of secondary products (Craig et al. 2005; Evershed 2008a; Debono-Spiteri et al. 2016; Whelton et al. 2018). This focus is reflected by the attention given to degraded animal fats in the field, especially ruminant dairy and ruminant adipose products. Another major focus of interest has been in investigating products processed in early pottery produced by hunter-gatherer groups in Europe and East Asia (Cramp et al. 2014; Lucquin et al. 2016a; Shoda et al. 2017, 2018), where evidence suggests the predominance of marine and freshwater products in vessels, especially in the Mesolithic period in European, Japanese and Korean prehistory. Broadly speaking, investigating these broad temporal and spatial patterns of vessel usage demonstrates the relationship between the symbolic meaning of 'new' material culture (pottery in East Asia) or 'new' foodstuff (dairy products in Europe) and their perceived 'value' over time.

As lipid residues are one of the few methods available in archaeology to investigate the *use* of vessels (Reber et al. 2019), this method also provides a unique opportunity to examine the relationship between material culture (i.e., ceramic vessels), and natural products processed or transformed for food or other applications. Different methods of food processing associated with particular shapes of vessels have been studied, such as the presence of ketones in cooking pots,which indicate repeated heating (Evershed et al. 1995; Raven et al. 1997) and the accumulation of lipid in certain parts of a vessel which highlight differences in practices such as boiling or roasting (Charters et al. 1993). Such studies provide insight into cooking traditions that occupy an important role in how populations conceptualise their foodstuff, which is inherently linked to taste preferences and cultural choices. Examples include the preference for sticky foods in East Asia that are generally processed via boiling and steaming, as opposed to the culture of baking and roasting in western Asia (Fuller 2011; Fuller and Rowlands 2009, 2011). Being able to access this level of detail into ancient culinary choices is possible when organic residue analysis is combined with other palaeodietary information and robust ceramic analyses.

The study of the use of organic materials to seal and repair ceramic vessels, or transport other substances has been another area of interest in the field of organic residue analysis. A range of products such as waxes, resins and bituminous materials have been identified within archaeological ceramics dating from the Neolithic to the $9th$ centuries AD across different regions (Urem-Kotsou et al. 2002, Charters et al. 2003, Regert et al. 2003, Stern et al. 2003; Knappet et al. 2005, Stacey et al. 2006; Salque et al. 2013). Sometimes, the sources of these resins and bituminous materials suggest evidence of long-distance exchange of organic products (Regert et al. 2008, Connan and van de Velde 2010; van de

Velde 2015; Courel et al. 2017). The field has provided unique insight into technologies of vessel manufacture and their *chaîne opératoire*, the production of non-culinary organic substances, as well as the movement or connectivity between different regions that underlies their production.

Ceramics are one of the most ubiquitous artefacts recovered during archaeological excavation, especially in South Asia. Within the context of Indus archaeology, pottery is often embedded in typology- and form-based discussions alone and is divorced from its cultural role. The analysis of performance-based physical properties of ceramics, vessel form, and ethnoarchaeology are widely used by archaeologists to determine the intended use of vessels (Kenoyer 1997). However, the relationship of form and physical properties to actual pottery use may be ambiguous (Heron and Evershed 1993), and in some cases, vessel form variability may have little or nothing to do with performance characteristics (Miller 1985). Thus, this thesis emphasises the obvious connections between archaeological ceramics and ancient foods and organic products. The application of residue analysis on ancient pottery enables the investigation of the *use* of vessels through the determination of past vessel contents, a topic often left out of the traditional discourse in archaeological ceramic analysis in South Asia. As this method has never been applied at a large scale in Indus archaeology (a previous study analysed a single sample: see Section 2.3.5.2), this study is the first to apply it to investigate Indus foodways and vessel-use at a larger scale.

Finally, as no single dataset can reflect a people's entire diet or food technology; multiple datasets, each with their own limitations and interpretational challenges, must be combined. This thesis attempts to integrate lipid residue with archaeobotanical, zooarchaeological and isotopic data (e.g. Evershed 2008a), factoring in quantitative issues and taphonomic processes influencing the creation of different archaeological datasets. Although this exercise is challenging, this thesis demonstrates that the results can provide new, intriguing insights into ancient human-environment-food relations.

1.4. Definitions: food, vessel-use, food choice, foodways

The archaeology of food faces certain definitional issues. As an extremely multifaceted phenomenon, food has the ability to testify to many different aspects of life, but this also makes it challenging to study (Twiss 2015). As archaeologists, food is often implicitly conceptualised as 'solid consumables', and categories like beverages, inhalables, and swallowables such as 'medicines' do not usually fall under it (Twiss 2015: 93). Secondly, food is also defined as a subset of theoretically available nutrients (Twiss 2015:92), which tends to undermine the social role of food, and ignore culturally defined aspects of food such as edibility, taste, and choice. As there can be multiple appropriate definitions of food depending on the context, it is important to let research questions guide the boundaries to conceptions of food, shrinking or expanding to encompass various aspects of the environment and material culture (Twiss 2015). This thesis specifically attempts to use organic residues embedded within ceramic vessels as a proxy towards understanding of how ancient populations processed and transformed natural products to produce what was conceived as food. It is possible that not everything that was processed in vessels was consumed. However, as most vessels were recovered from domestic contexts, it is likely most of them were used for quotidian practices and were primarily involved in food processing.

Other definitions prominently used in the archaeological food literature include 'feasting', 'food choice' and 'foodways'. 'Feasting' pervades archaeological studies of food (Bray 2003; Dietler and Hayden 2010; Twiss 2015), but definitions of feasting vary in their details (Dietler and Hayden 2010) and are debated (Twiss 2015). As evidence of large-scale, "special" communal consumption has not been clearly identified in the Indus Civilisation, this term is not used in this thesis. Food choice implies the selection of "ingredients" and their consumption, and encompasses what is eaten, why, where, and how. Food choice has an important role in the social, economic, and symbolic aspects of life because it conveys information on preferences, identities, and culture (Fuller and Lucas 2017). Taking a broader approach, 'foodways', incorporates the whole interrelated system of food conceptualization, procurement, preservation, preparation, distribution, and consumption (see Figure 1.1; Camp 1982). In this thesis 'food choice' is used to highlight the active, engaged culinary practices of Indus populations. Additionally, it focuses on aspects related to procurement and preparation of food in Indus society, and emphasizes that a foodways approach in Indus archaeology may yield interesting insights into studying the diversity of Indus commensality.

Figure 1.1: Diagram illustrating foodways.

1.5. Chapter Summary and Thesis Structure

The sections above have introduced the research questions that will be addressed in this thesis and provided the theoretical aims and methodological rationale of the study. Chapter Two introduces the archaeological context and provides present knowledge about climate, urbanism, food production and cultural/climatic change in the Indus Civilisation. It also summarises what is known about food procurement, processing, and consumption in the Indus Civilisation, specifically northwest India. The scientific basis of the methods employed is elaborated upon in Chapter Three, with an introduction to lipids and organic residue analysis. Limitations and interpretational challenges faced by lipid analysts are also addressed. Chapter Four provides the background for all of the study sites, including archaeobotanical, zooarchaeological and isotopic evidence, if available. Methodological protocols and sample selection details are outlined in Chapter Five. In Chapter Six, the results of the methodological investigation into the preservation of lipid residues from various contexts are provided, and the implications of the results are discussed. Chapter Seven presents the lipid residue analysis data from every site and presents vessel-specific results of relevance. It also addresses the archaeological questions posed by this thesis. Chapter Eight draws these strands together and provides a holistic discussion of the results. Chapter Nine concludes the thesis with a discussion of the broader implications of the results for Indus archaeology and beyond.

Chapter Two

Indus Civilisation settlements, environment and subsistence: urban and post-urban periods

This chapter lays out the background for the archaeological context and details of relevant literature relating to the research questions explored in this thesis. Section 2.1 provides an introduction and brief background to the Indus Civilisation, specifically focusing on urbanism and the nature of settlements in the urban phase. The climatic and environmental contexts of the Indus Civilisation are also introduced, including discussions about the extent of cultural uniformity and variability. Section 2.2 summarises literature about the decline or transformation of the Indus Civilisation in the post-urban phase. Section 2.3 delves into a review of literature concerning the reconstruction of the subsistence practices by Indus populations in the urban and post-urban phases. This includes archaeobotanical research, zooarchaeological evidence, and biomolecular approaches including stable isotopic research and starch-grain analyses. Finally, Section 2.4 synthesises how each of these approaches have framed discourse about Indus society, economy and foodproduction in the urban period and in the transition to post-urbanism.

2.1. Contextual background to the Indus Civilisation: chronology, urbanism and environment

2.1.1. Chronology

The Indus Civilisation is best known for its urban phase which spanned from the mid-third to early second millennium BC (∼4.6– 4.5 ka BP, or *c*.2600-1900 BC), but the urban period was preceded by an extended period of village-based settlement beginning in the sixth millennium BC (Petrie 2013). Complex processes led to the development of smallscale farming societies in the pre-urban period (late fourth-early third millennium BC), which eventually gave rise to urban settlements (Chakrabarti 1995; Possehl 2002, Wright 2010). The transformation from pre-urban to urban settlements was not a linear developmental process, and there was considerable variation in lifeways throughout the region that constituted the Indus Civilisation in this formative stage (Petrie 2013: 5).

However, as this thesis focuses on the urban and post-urban period, these will not be discussed in this chapter.

Table 2.1 provides an overview of the chronology of the Indus Civilisation. As the terminology used to refer to different periods in the literature is varied (Possehl 1977; Shaffer 1992; Kenoyer 1997; Wright 2010), the table provides the different terms used as well as regional variations present in the chronology.

Chronological frameworks employed in this thesis are based on a mixture of stratigraphy, periodisation on the basis of ceramic analysis, and Bayesian radiocarbon modelling. This framework uses the generally accepted chronological divisions of 'Mature Harappan' and 'Late Harappan' based on pottery typologies found at Indus sites, which are further broken into sub-divisions that have been fine-tuned through ceramic analysis within Indus sites in northwest India. These include Early Mature Harappan (EMH) and Late Mature Harappan (LMH) (Table 2.2). This thesis favours these terms as they are used in the ceramic literature in northwest India but will also use the terms 'urban' and 'posturban' as urbanism is a fundamental aspect to discourse related to social organisation and subsistence practices in the Indus Civilisation. These cultural periods were assigned during excavation on the basis of relative stratigraphy and later clarified via the use of radiocarbon dating and Bayesian radiocarbon modelling. Samples have also been discussed according to whether they pre-date, post-date, and/or fall during the 4.2 ka climatic 'event', discussed in Section 2.1.3. This was done in order to make comparisons about the products processed in vessels, or vessel-usage before, during and after the onset of significant climatic instability in the region. Classifications have been done on the basis of radiocarbon dates obtained from strata. The approximate time window covered in this thesis are detailed in Table 2.2.

Pre-, Urban, or Post- urban	Years cal. BC	Cultural period	Regional phases	Harappa Period	Cultural trends
Pre-urban	3300- 2600	Early Harappan	Amri-Nal Kot Diji Sothi-Siswal Damb Sadaat	Period 3A	Incipient urbanism; growth and expansion of farming communities
Urban	2600- 2500	Early- Mature Transition		Period 3A	Appearance of increasingly complex craft technologies; increase in settlement size
Urban	2500- 1900	Mature Harappan	Sindhi Harappan Kulli Harappan Sorath Harappan Punjabi Harappan Eastern Harappan Quetta Late Kot Diji	Period 3B/3C	Establishment of stratified society with complex material culture; with peak of cities after $c.2100$ BC
Post- urban	1900- 1300	Late Harappan	Jhukar (1900- 1700 BC) Early Pirak $(1800-1700$ BC) Late Sorath Harappan (1900- 1600 BC) Lustrous Red Ware (1600-1300) BC) Cemetery H $(1900-1500$ BC) Late Harappan in Haryana and Western Uttar Pradesh (1900- 1300 BC)	Period 4	De-urbanisation; abandonment of many sites. Expansion of settlements towards east and proliferation of smaller settlements.

Table 2.1: Basic chronology of the Indus Civilisation

Table 2.2: Chronology used in this thesis.

2.1.2. Indus urban period: settlements and material culture

Urbanism and ancient cities have occupied a significant portion of anthropological and archaeological literature (e.g. Yoffee 2005), and they feature as important aspect of scholarship in the Indus Civilisation (see Cork 2011). As "small worlds" (Smith 2006b), cities enable the development of new social and economic roles and transform relationships between people (Chakrabarti 1995; Yoffee 2005; Smith 2006b). Urbanism has been linked to a number of features of complex societies, such as high degrees of specialisation, resource-intensive craft production, and the creation of public monuments and infrastructure (Childe 1950). Only five Indus settlements developed into sizable urban cities within a relatively short period between *c*.2600-2500 BC, and most of our knowledge about the Indus Civilisation comes from these cities, of which the two most famous were excavated in the early twentieth century: Harappa and Mohenjo-daro. The other cities, Dholavira and Rakhigarhi were excavated more recently; and although Ganweriwala remains unexcavated, it may have been much smaller than initially claimed (Kenoyer 1998; Petrie 2013; Masih 2018) (Figure 2.2). Within Harappa and Mohenjo-daro, there is evidence for multiple walled areas and three-dimensional, segregated spaces that likely reflect the existence of competing, heterarchical elite groups within cities (Kenoyer 1997; Eltsov 2008; Vidale 2010; Petrie 2013). Beyond the likelihood of the existence of polycentric cities, the nature of Indus society (its religious practices, social order, and organising principles) remain debated (Petrie 2013).

A range of Indus settlements proliferated apart from the five cities. These include smaller, medium-sized urban settlements (e.g. Kalibangan, Farmana, Banawali); small settlements with highly specialised craft production or 'factory sites' (Vidale 2000: 38) (e.g. Chanhu-Daro, Nageshwar); small settlements with substantial mud-brick or stone fortification walls, especially in Gujarat (e.g. Lothal, Shikarpur, Kuntasi, Sukotada, Kanmer); and rural settlements (e.g. Masudpur I, Masudpur VII, Burj, Dabli-vas Chugta, Alamgirpur) (Petrie 2013). While some of the 'industrial' settlements are likely to have been integrated within a complex network involved in the production and distribution of distinctive material culture (for example, shell bead production at Bagasra), the function of many smaller settlements is unclear (Parikh and Petrie 2018). There is also no simple relationship between the size of settlements and their complexity, i.e., planning or craft activities (Chakrabarti 1995; Petrie and Parikh 2018). This is exemplified through the degree or absence of specific types of Indus material culture at different settlements.

Indus material culture includes painted and non-painted pottery, terracotta bangles, figurines (likely produced locally) (Parikh and Petrie 2018), jewellery such as beads, bangles and micro-beads made from semi-precious and precious stones like agate, carnelian, lapis lazuli and gold procured from far-away sources (Law 2011), and standardised weights and stamp steatite seals (Wright 2010: 148-166) (Figure 2.1). Within cities, such as at Harappa, there was likely a diversity of production organisation including independent household, communal or kin group, and centralised production and exchange (Wright 2010: 180). Although the literature is dominated by discussions about Indus cities and exchange networks of iconic material culture (e.g. Kenoyer 1997; Wright 2010; Law 2011), many aspects of these networks and the relationship between large settlements, 'industrial settlements', and the range of smaller settlements are not well-understood (Petrie et al. 2017; Parikh and Petrie 2018). While some settlements may have been involved in the production of specific crafts, there is evidence for highly valued material culture coming from far-off distances such as lapis lazuli and gold beads present at very small settlements, for example, Alladino in Sindh (Dales and Kenoyer 1986: 9), and rural settlements in Haryana (Petrie et al. 2009, 2017). The presence of this highly valued material in small, likely rural settlements, suggests that long-distance networks of exchange were accessible by various populations (Petrie et al. 2009, 2017). It is also possible the food products moved between different settlements (Madella 2014). This suggests that relationships between settlements was complex; possibly governed by mutual economic dependence rather than overt control of cities over rural hinterlands (Wright 2010; Parikh and Petrie 2018).

As medium-sized and smaller settlements are yet to be appropriately classified (Parikh and Petrie 2018), and the dynamics between large and smaller settlements have not been systematically investigated (Petrie 2013), it is difficult to characterise the (likely varied) nature of interactions between different settlements in the Indus Civilisation. Petrie and colleagues argue that given the vast geographical spread of the Indus Civilisation, the landscape was more likely dominated by rural settlements which have received relatively little attention by scholars (Petrie 2013; Petrie et al. 2017). This thesis addresses this problem by studying vessels from range of differently-sized Indus settlements, with a focus on small rural settlements in northwest India.

Figure 2.1: Top left: Collection of jewellery from Harappa and Mohenjo-daro. Source: [www.harappa.com.](http://www.harappa.com/) Top right: Agate, gold, lapis lazuli and carnelian beads from rural Indus sites in Haryana (after Parikh and Petrie 2018: 10). Middle: Seals with Indus script from Mohenjo-daro. Source: [www.harappa.com.](http://www.harappa.com/) Bottom left: Indus perforated vessel from Lahore Museum. Source: [www.harappa.com.](http://www.harappa.com/) Bottom right: Indus pottery from rural Indus sites in Haryana (after Parikh and Petrie 2018: 7).

2.1.2.2. Rural Indus settlements

New evidence from smaller, rural settlements in northwest India are throwing fresh light on what arguably better characterises everyday life in the Indus Civilisation. Excavations at multiple small settlements (less than 10 ha) in Haryana, Rajasthan and Gujarat have revealed the complexity, diversity, and distinctiveness of the rural character of the Indus Civilisation in both the pre-urban and urban periods (Petrie 2013; Chase et al. 2014b; García-Granero 2015, 2016; Petrie et al. 2017; Lancelotti et al. 2017; Parikh and Petrie 2018). This character is particularly evinced by choices in the production of pottery and in crop selection (see Section 2.2). The diversity in different aspects of quotidian practices is suggestive of varied interactions and dynamics between small-sized settlements which may have also been regionally specific. These variations speak to larger discussions around uniformity and diversity in the Indus Civilisation and how it is characterised.

2.1.2.3. Uniformity and diversity across the Indus Civilisation

The dynamics of uniformity and diversity across the Indus Civilisation have been extensively debated due to their implications about larger questions concerning Indus socio-political structure. Although broadly speaking, similar types of material culture, including seals, weights and the script, have been found at large, medium-sized and small settlements (Chakrabarti 1999; Agrawal 2007; Kenoyer 2008; Wright 2010), variation in access to raw material, material culture and subsistence choices in particular regions and sites have long been acknowledged by several scholars (Possehl 1982, 1992; Meadow and Kenoyer 1997; Weber 1999; Wright 2010; Petrie 2013; Petrie et al. 2017, 2018). For example, although previous literature characterised the Indus Civilisation as a culturally integrated society subsisting off a suite of winter-based crops such as wheat and barley (e.g., Marshall 2004[1931]; Wheeler 1950), regional variations in crop choices have been observed which correlate with differential patterns of rainfall and crop availability (Weber 1999; Madella and Fuller 2006; Petrie and Bates 2017). Regional differences in material culture have also been noted by scholars, particularly in the pre-urban period, and then again in the post-urban period, which were used by Possehl (1982; 1992; 2002) to classify 'culture-geographic domains.' These 'domains' are an early attempt to correlate specifics of environmental diversity with cultural variation. The climatic and environmental context, and regional cultural variation of the Indus Civilisation is particularly important to consider in the context of the decline and abandonment of large Indus settlements in the post-urban period.

2.1.3. Climatic and environmental context

The Indus Civilisation stands apart from other early complex societies for several reasons, including its unique climatic and environmental context. The parts of the Indus River Basin occupied by Indus Civilisation populations incorporate areas where winter rain or summer monsoonal rain predominate independently, and where they overlap (Petrie et al. 2017; Figures 2.1 and 2.2). Furthermore, habitats and environmental contexts within which Indus settlements are occupied are diverse, and include alluvial plains, foothills, deserts, scrubland, and coastal regions (Wright 2010; Petrie et al. 2017). Thus, the Indus Civilisation provides a unique opportunity to understand how an ancient society coped with diverse and varied ecologies, as well as the impact of changes in the fundamental and underlying environmental parameters (Wright 2010; Petrie 2013, 2017; Petrie et al. 2017: 2).

Briefly, climate in the Indus region is set within two macro-regional climatic systems known as the ISM (Indian Summer Monsoon), which originates in the Indian Ocean, and the 'westerlies' or Western Disturbance storm fronts that originate in the Mediterranean (Breitenbach 2009). These two systems drive an annual climatic cycle that is characterised by cool and dry winters (December to February) with occasional rainfall from the 'westerlies', a pre-monsoon, hot and dry summers with occasional showers (March to May), hot and wet monsoons (June to September) with intense bursts of rain, and finally, a post-monsoon cool and dry period (October to December). These systems also create steep east-west and north-south rainfall gradients that guide variations in winter and monsoonal rainfall across the region (Breitenbach 2009; Jones 2017) (see Figures 2.2 and 2.3).

Variations in the intensity of these two systems have been studied at millennial, centennial and decadal scales (e.g. Berkelhammer et al. 2010; Prasad et al. 2014; Baudouin in prep.) However, detailed understanding of variations in weather and climate systems are limited by the low resolution of many of the records and an uncertainty about the interactions between the two systems (Gupta et al. 2003; Breitenbach 2009). Furthermore, correlations between global-scale climate records and local-scale cultural developments have proven to be challenging. Cultural transformations visible in the archaeological record do not typically coincide neatly with climatic variations; and as a result, inferences are often either entirely speculative or result in 'correlation equals causation' circularity (Petrie 2017; Petrie et al. 2017).

Figure 2.2: Extent of the Indus Civilisation in the urban period, with modern rainfall isohyets reflecting winter (A) and summer (B) rain gradients. Settlements are in yellow and cities in black. Courtesy Cameron Petrie.

Figure 2.3: Extent of the Indus Civilisation in the post-urban period with settlements marked in yellow, with modern rainfall isohyets reflecting winter (A) and summer (B) rain gradients. Settlements are in yellow and cities in black. Courtesy Cameron Petrie.

2.1.3.1 Climate before and after Indus urbanism

The climatic context of South Asia before the development and during the floruit of the Indus Civilisation (before 5.2 ka BP or *c*.3200 BC until 4.6/4.5 ka BP or *c*.2600-2500 BC) is characterised by an early Holocene wet phase followed by a long-term drying trend in the mid- and late-Holocene (Ponton et al. 2012; Zorzi et al. 2015; Dixit et al. 2018; Giesche et al. 2019). Although instances of the onset of monsoon weakening appears in records in the subcontinent around 8.2 ka BP and between 6.5-6.0 ka BP (Dixit et al. 2014a, 2014b; Sarkar et al. 2015) it is likely that the ISM potentially intensified between 5.3-4.2 ka BP, which includes the period that saw the development of Indus cities (Dixit et al. 2018; Giesche et al. 2019). However, this pattern is not apparent in all climate records (e.g. Thar Desert, Prasad et al. 1997), which likely reflects local hydrological responses to monsoon decline (Madella and Fuller 2006). The general trend towards a weaker monsoon no doubt affected Indus populations, but the limited number of well-dated, high-resolution, local palaeoclimatic records limits the ability to correlate these changes with archaeologically-relevant temporal scales. Furthermore, there are only limited indications of climate obtained directly from archaeological sites (Sarkar et al. 2015; Jones 2017).

Most researchers investigating the Indus Civilisation populations and their relationship to the climate have focused on a specific abrupt arid phase which began c. 4.2 ka BP or *c*. 2150 BC and lasted up to several centuries (e.g. Staubwasser 2003, Dixit et al. 2014a/b; Giesche et al. 2019). This '4.2 ka event' has been described in climatic records in the Americas, Middle East, Africa and China (Jones 2017), but its manifestation in the Indus region is contested (Staubwasser 2003; Sarkar et al. 2015). While several records provide evidence of abrupt monsoon weakening around this time (Gupta et al. 2003; Staubwasser 2003; Breitenbach 2009; Dixit et al. 2014a; Giesche et al. 2019); others indicate contrary evidence, including an arid period that begins before 4.2 ka, or no evidence of monsoon weakening at all (Ponton et al. 2012; Tiwari et al. 2015). These differences are likely reflective of the varying sensitivities of different proxies, as well as complexities driven by the interactions of winter and monsoon rainfall in some regions (Madella and Fuller 2006; Breitenback 2009; Jones 2017). Significantly, there is clear evidence for step-wise monsoon weakening at Kotla Dahar around 4.2-4.1 ka BP (Dixit et al. 2014a), which is an ephemeral lake that lies relatively close to the Indus sites investigated in this thesis. It is not clear, however, if the 4.2 ka BP weakening involved more than just a reduction in monsoon intensity, or also changes in seasonality and annual

moisture availability, as well as a reduction in winter rainfall (Staubwasser 2003; Giesche et al. 2019). Additionally, this shift must also be put within the context of an ongoing monsoon weakening, likely winter rainfall weakening after 4.3 ka (Giesche et al. 2019), and variability over decadal and centennial scales. Thus, it is still not possible to determine exactly what this 'event' meant for rainfall patterns, nor how detrimental it may have been for human populations.

The period between *c*. 3.9-3.2 ka (during the post-urban Indus phase) is characterised by moisture recovery in parts of the Indus region, but moisture levels did not recover to early or mid-Holocene levels (Wright et al. 2008; Prasad et al. 2014; Dixit et al. 2014a; Giesche et al. 2019). In the eastern part of the Indus Civilisation (i.e. in Haryana and Rajasthan), it appears that any recovery was either absent, or insufficient to reverse the step changes from permanent to ephemeral or dry lake systems at Kotla Dahar or in the Thar Desert (Dixit et al. 2014a). Others have suggested an increase in Indus river discharge and recovery of moisture levels based off cores from the Indus delta (von Rad et al. 1999; Staubwasser 2003), and an increase in Beas River discharge based off a model derived from ITCZ (Inter-Tropical Convergence Zone) dynamics (Wright et al. 2008). At present, it is not known if there was a return to stable climatic conditions for winter and/or summer rain in the post-urban period. Further palaeoclimatic work is necessary to resolve these uncertainties and what they might suggest about climatic conditions in the post-urban period.

Thus, the spatial and temporal resolution required to make nuanced inferences about the relationship between Indus populations and the climate is insufficient at present. However, broadly speaking, the development of Indus urbanism clearly occurred in the context of a long-term decline in summer monsoon intensity. The abrupt 4.2 ka BP 'event' likely brought a further reduction in monsoon rainfall, but its expression across different zones in the Indus Civilisation is not yet clearly understood. New research also suggests the weakening summer monsoon was coupled with variations and reduction in winter rainfall (Giesche et al. 2019), which also likely had a negative impact on Indus populations. Finally, the post-urban period was characterised by recovering ISM moisture levels (Wright et al. 2008) but it is uncertain how winter rainfall systems recovered after 4.1 ka BP (Giesche et al. 2019). It is clear that far more detailed, high-resolution records of both winter and summer rainfall dynamics are necessary to better correlate the relationship between climate and human response. This thesis will address whether the processing of food products in vessels experienced change or continuity during and after the 4.2 ka BP

'event' but will frame its interpretations with the recognition of the known uncertainties and problems with linking cultural transformations with climatic change.

2.1.3.2. Fluvial dynamics and vegetation

Apart from climate and rainfall systems, access to fluvial systems and vegetation no doubt shaped the lives of Indus populations. The Indus Civilisation has been described as riverine (Marshal 1931[2004]), but settlements have been found in a range of environments, including alluvial fans, intermontane valleys, within arid regions and their margins, even islands (Possehl 2002, Wright 2010: 33–38; Petrie 2013, 2017; Petrie and Thomas 2012; Petrie et al. 2017; Petrie et al 2018: 456). Although the reconstruction of vegetation and land cover across the Indus Civilisation based on paleoenvironmental data has been limited, available data suggests the dominance of dry-thorn scrubland in the alluvial plains, as well as arid desert and steppe species (Lancelotti 2018). In Gujarat, studies suggest the existence of a C_3 rich plant environment, with an increase in arid-adapted C_4 plant (for definitions of C_3 and C_4 plants, see Section 3.5.2) in the late phase of the urban period attributed to the growing and foddering of animals on millet (Reddy 1997; Chase et al. 2018; Chakraborty et al. 2018).

Variations in water supply combined with differing vegetation, soils and hydrology would have created unique ecological niches that likely impacted on human behaviour, particularly in terms of subsistence choices (Petrie et al. 2017). For example, in lower parts of Punjab and Sindh in modern-day Pakistan, access to direct rainfall is limited, however, runoff from both winter and summer rainfall through perennial and seasonal rivers and streams would have been extremely important to Indus populations (e.g. Miller 2006, 2015). Conversely, in Haryana, the analysis of satellite imagery suggests that a profusion of relatively small-scale watercourses are preserved in the subsurface (Orengo and Petrie 2017, 2018), but the precise nature and the timing of their flow remains unclear (e.g. Durcan et al. 2017, 2019). If these watercourse(s) were indeed ephemeral, it is likely that the inhabitants also made use of a combination of wells and ponds to collect monsoon runoff (Petrie et al. 2017: 457).

Fluvial processes have also formed a key component of discourse about Indus deurbanisation. Models ranging from changes in river flow, river avulsion, flooding and drying have been put forward as major drivers of the decline of the Indus civilisation which is covered in the next section and relevant for this thesis.

2.2. Decline or transformation from urbanism

The period after *c*.2100 BC marked the peak of the Indus urban phase, and then an abandonment of settlements in Sindh and west Punjab, but an increase in settlement density in Rajasthan, Haryana and western Uttar Pradesh (Possehl 2002; Petrie 2013) (see Figure 2.3). Harappa is the only city-sized settlement for which there is evidence of continuity in the post-urban period (Kenoyer 2008; Wright 2010). By 1900 BC, many defining traits of Indus urbanism such as the use of the Indus script, seals, and weights were no longer evident.

Although there is evidence for a variety of ways of organising craft production in the urban period that emphasize communal and collective action (Wright 2010: 182-202; Green 2016, 2017), the disappearance of the circulation and use of these items in the posturban period suggests a likely shift away from specialised labour and elite economic control (Vidale and Miller 2000; Wright 2010; Law 2011). There was a clear shift to village-based settlements with less complex and less inter-connected economies during this period, with significant changes to the scale and extent of Indus exchange systems (Wright 2010; Law 2011). There was also a breakdown of civic infrastructure at settlements such as Harappa, suggesting that authority structures were weakened or being challenged (Kenoyer 2008; Wright 2010). Thus, taken together, these changes reflect a dramatic alteration to the urban character of the Indus Civilisation, resulting in reduced labour specialisation, shorter-range cultural and economic links between settlements, and changes to the prevailing socio-political systems.

'General' causes used to explain the demise of the urban phase of the Indus Civilisation range from shifts in climate to the occurrence of a natural catastrophe, warfare or cultural crisis (see Raikes 1964, 1968, 1979; Possehl 1977; 1997, 2002; Wright 2010). As mentioned above, while there is evidence for a sudden weakening of the ISM around 4.2 ka BP/*c*.2100 BC (Staubwasser et al. 2003; Dixit et al. 2014a), which broadly coincides with the peak density of occupation at Harappa, and then the onset of the decline of Indus cities, reliable evidence for a connection between the two processes is lacking. Other explanations have included the 'invasion' or influx of foreign peoples, and disruptions to maritime trade (Wheeler 1947, 1955; Kenoyer 1998). 'Local' causes potentially were specific to particular settlements or regions and must be assessed individually (Wright 2010: 309). Certain urban settlements were likely abandoned altogether, such as Mohenjodaro, Dholavira, and potentially Ganweriwala (Kenoyer 2008). For example, studies

suggest that dramatic shifts in the Indus River course placed the city of Mohenjo-daro in a perilous position (Flam 1993). Occupation at Harappa continued (Dales and Kenoyer 1986; Kenoyer 2008; Wright 2010), and the number of village-sized settlements in the eastern regions (i.e. Haryana and Gujarat) increased (Madella and Fuller 2006; Wright 2010:317- 318; Petrie et al. 2017; Green and Petrie 2018). While some have suggested that reduced seasonal (monsoonal) flooding and divergence of water flows may have caused unpredictability in water supply of the seasonal Ghaggar-Hakra River in Haryana and Gujarat (Courty 1995; Staubwasser 2003; Giosan et al. 2012), others have suggested that more reliable rainfall from a weakened monsoon was available in the region, which may have spurred increased settlement density (Petrie et al. 2017; Green and Petrie 2018). For example, there is clear evidence for an increase in sites in the post-urban period around Rakhigarhi (Singh et al 2010: 42; Green and Petrie 2018), but at present is uncertain to what extent occupation at Rakhigarhi continued. Thus, although the precise nature of the 'collapse', 'decline', or 'transformation' of urban Indus society is contested, it is increasingly recognised that Indus de-urbanisation was a gradual and uneven process that played out differently at different regions and sites (Meadow and Kenoyer 2005; Wright 2010; Petrie 2013; 2017; Petrie et al. 2017).

How did these processes affect the day-to-day lives of Indus populations, and to what extent can they be characterised and quantified? Changes in systems of pottery production are identifiable in the post-urban period, but these vary regionally and have limited chronological resolution as most regions have a small number of radiocarbon dates. For example, it is traditionally claimed that diagnostic pottery types for the post-urban/Late Harappan in Cholistan and northwest India were typically related to the Cemetery H pottery at Harappa (Wright 2010: 310). Ongoing studies suggest that ceramic production systems in northwest India were far more complex and exhibited variability and degrees of continuity in the post-urban period (Parikh and Petrie 2017, 2018; Ceccarelli in prep., Parikh in prep).

Regarding changes in subsistence strategies in the face of possible food stress in the post-urban period, there is no clear pattern. For example, Fuller and Madella (2002) have argued that the presence of low-yielding but arid-adapted millet in the post-urban period at Harappa indicates the adoption of a strategy to cope with increasing aridification. However, Weber (2003) has argued that millet was present in Harappa across time in small amounts and that fluctuations in climate were too gradual to affect agricultural strategy. There is also evidence for crop diversity in the pre-urban period from rural sites in

Haryana, as well as flexibility in crop use (Bates and Petrie 2017). Additionally, archaeobotanical remains do not reflect substantial changes of cropping patterns in northwest India (Bates 2016; Petrie et al. 2016; Petrie and Bates 2017), suggesting a broad continuation of everyday practices but slight shift towards increased summer crops. The diversity and apparent continuity in the subsistence regime across different sites (Possehl 2002: 16) exemplifies the challenges in reconciling different types of archaeological evidence and data obtained from climate proxies. Keeping these uncertainties in mind, this thesis further adds to this question by investigating whether there are measurable differences in how vessels were used over periods of fluctuations in climate, and if products processed in vessels in certain settlements varied from the urban to the post-urban period.

2.3. Indus ceramics, subsistence practices and foodways

2.3.1. Indus ceramics

Pottery from Indus settlements has been the focus of a range of studies, but the production, use and distribution of the protohistoric ceramic industries of the subcontinent are still far from completely understood (see Ceccarelli and Petrie 2018; Parikh and Petrie 2017, 2018; Petrie et al. 2018). Most reports on Indus pottery have categorised it into types and styles (e.g. Dales and Kenoyer 1986: 62), and this pattern continues today. Although broadly useful, ceramic classifications are not always clear or reproducible. Additionally, ideas about the uniformity of ceramics across the Indus region in the urban period, the use of culture-historical theoretical frameworks, and unsystematic documentation methods further hinder a wide-scale assessment of the range and variability of Indus ceramic industries (see Ceccarelli and Petrie 2018; Parikh and Petrie 2017, 2018; Krishnan 2018; Petrie et al. 2018; Ceccarelli in prep; Parikh in prep). Despite this, the possible social meaning ceramic technologies and distributions have for urban hierarchy, specialisation, organisation and transmission of knowledge have been examined extensively (e.g. Kenoyer 1989a, 1989b, 1992; Vidale 2000; Vidale and Miller 2000; Miller 1999; 2000; Ratnagar 2015), and a range of scientific approaches such as geochemical and petrographic analysis have been used to study Indus pottery and pottery from historical periods in South Asia (Hegde 1962, 1975; Krishnan and Hegde 1988; Krishnan 1992; Méry and Blackman 1996; Gogte 1997; Krishnan et al. 2005). The following section will focus on ceramics from the urban and

post-urban period, particularly in northwest India Indus sites which are relevant to this thesis.

2.3.1.1. Diversity in ceramic production and use

An understanding of a range of manufacturing methods adopted by various Indus communities is being developed, and there is an attempt to categorise the diversity of ceramic production within and between Indus settlements (Parikh and Petrie 2017; 2018; Krishnan 2018; Petrie et al. 2018; Ceccarelli in prep.; Parikh in prep.). There are several regional styles of pottery that were in use during the urban period, which have been recognised across the Indus Civilisation (see Table 2.1). Although several of these regional styles developed in the pre-urban or Early Harappan period (*c*.3000-2600 BC), many of them persisted and responded dynamically to what is considered the 'Classical Harappan' (Uesugi 2011a; 2011b, 2013, 2017) or 'Red Harappan Ware' (Dales and Kenoyer 1986) that dominated in the large settlements of Harappa and Mohenjo-daro (Figure 2.4). Far from being static archetypes as they are often described, these regional ceramic repertoires are visually distinctive in terms of their surface finish and decoration, and yet recognisably a part of the Indus material canon in terms of their forms (Parikh and Petrie 2018). They are also found in association with other types of material culture that are typical to the Indus urban phase, which suggests that pottery was a unique medium that was translated and interpreted differently across the Indus Civilisation (Parikh and Petrie 2017, 2018).

2.3.1.2. Pottery types: 'Classic Harappan'

The most comprehensive classification of Indus pottery to date is the assessment of material from Mohenjo-daro by Dales and Kenoyer (1986). This volume set out a descriptive and classificatory system that could be used across all Indus Civilisation sites, using the catalogue of pottery excavated from the University Museum of the University of Pennsylvania excavations at Mohenjo-daro (1964-5). It differentiated vessels according to forms and varieties depending on size, rim shape, surface treatment or unusual specimens, and described the ware, manufacturing, sample size, surface treatment, context and measurements of every vessel form within this pottery corpus. Typical forms include pots, jars, ledged-shouldered vessels, bowls, dishes, perforated vessels, goblets, and dish-onstands. It also compared vessels to known examples from other sites or from previous excavations at Mohenjo-daro (e.g. Marshall 1931[2004]; Mackay 1938; Wheeler 1950). Unfortunately, several examples were from surface contexts or have an undocumented stratigraphic origin, and even those with some known provenance can only be coarsely

assigned a chronological period based on relative stratigraphy or comparison with examples from other sites.

The Dales and Kenoyer (1986) volume remains the canonical publication of Indus ceramics (Petrie et al. 2018), but work in different regions of the Indus Civilization has highlighted the complexity and diversity of ceramic production and use across this area. Variations include differences in vessel manufacture, vessel forms, and surface treatments. This variability is discussed below with reference to northwest India Indus ceramics, with a focus on sites within Haryana.

Figure 2.4: Top: ledged-shouldered 'cooking pots' and small black bowl from Nausharo. Bottom: Painted and unpainted burial pottery from Harappa. Source: www.harappa.com.

2.3.1.3. Pottery types: northwest India Indus sites

Vessel assemblages from Indus sites in northwest India are visually distinctive from those described in the Mohenjo-daro canon, and are dominated by what has been referred to as Sothi-Siswal (Ghosh 1952: 37–42; Bhan 1975; Dikshit 1984: 531–537; Bala 2003; Garge 2010), 'Non-Harappan pottery' (Uesugi 2011a, 2011b) or 'Haryana Harappan' pottery (Parikh and Petrie 2017, 2018). Vessel fabrics from this region are mostly made using a red fabric of medium texture and few inclusions with enormous variety in techniques and decoration (Uesugi 2011a, 2011b; Parikh and Petrie 2018). Most of the vessels studied in this thesis belong to this fabric. Other fabrics include thick and coarse red ware with more inclusions, and a grey fabric of a fine quality that is unique to the region (Parikh and Petrie 2018).

Techniques used to manufacture vessels in northwest India are distinctive from those visible in 'Classic Harappan' ceramics, which show the predominant use of the fast wheel to form and/or finish vessels (Parikh and Petrie 2017, 2018). In contrast, 'Haryana Harappan' vessels were formed in different ways: with some evidence of coiling, handbuilding, finishing on a slow wheel, or the absence of the wheel in the manufacturing process (Parikh and Petrie 2017, 2018; Ceccarelli in prep.; Parikh in prep.). Surface treatments vary between 'Classic Harappan' and 'Haryana Harappan' as well, as the former are often slipped with a dark red glossy slip and decorated with ornate, elaborate motifs (Dales and Kenoyer 1986; Quivron 2000), while the latter have varying slips (ranging from light red to dark brown) and are painted with visually distinct geometric or naturalistic patterns (Parikh and Petrie 2017). This clear variation in approaches to surface decoration has led to the differentiation of 'Classic Harappan' and 'Haryana Harappan' or Sothi-Siswal in excavated assemblages, with the former being familiar at major centres (e.g. Harappa, Mohenjo-daro, Banawali), and the latter either predominating (e.g. comprising 80% of the assemblage at Farmana; Uesugi 2011a, 2011b) or completely dominating the assemblage at rural sites (e.g. Masudpur I, Masudpur VII) (Parikh and Petrie 2017, 2018; Petrie et al. 2018). This thesis includes both Classic and 'Haryana Harappan' ceramics from settlements where both types are found, for example, at Rakhigarhi and Farmana.

Despite the unique visual and technical language used to produce 'Haryana Harappan' pottery, there are overlaps with the 'Classical' Harappan form canon. Vessel forms like perforated jars, 'cooking vessels', and dish-on-stands are present, even at very small sites. However, the range of vessel forms is relatively limited, and storage vessels are typically smaller in size (Parikh, pers. comm.). Additionally, other forms such as basins with incised decorative motives on the interior, which were first found at Kalibangan (Thapar 1975; Bala 2003), have also been found at small sites in Haryana (Parikh and Petrie 2017, 2018; Parikh in prep.), suggesting overlapping ceramic vocabularies across northwest India (Parikh and Petrie 2018). The pottery in the region suggests a vibrant ceramic milieu that was driven by potters' choices and the consumers of pottery within different settlements (Petrie et al. 2017), hinting at the complexity of village-based craft industries that were markedly unique from, and yet not unrelated to those in larger settlements (Parikh and Petrie 2018).

2.3.1.4. Vessels and cuisine

Some reports on Indus pottery discuss certain vessels that might have been associated with specific culinary activities (e.g. Dales and Kenoyer 1986: 110; Wright 1991; Kenoyer 1998; Krishnan 2018). Wright (1991: 83) has suggested that ledge-shouldered jars and large storage jars at Harappa were likely used to store liquids such as wine and oil. Dishon-stand vessels have been linked to the display and offering of food (Dales and Kenoyer 1986). Other examples include cooking vessels or jars, which are classified so either according to their shape (based on a distinct carination on the shoulder that has also earned them the descriptor 'ledged jars')(Figure 2.4: top), or fabric (Krishnan 2018). Krishnan (2018: 266) argues that cooking vessels share a similar form across "Gujarat and the core Indus regions" and have similar non-plastic inclusions within their clay paste, suggesting there may have been specialised workshops dedicated to their production. Others have highlighted that the carinated shape of 'cooking vessels' in Indus contexts resemble *handis*, vessels made from clay and/or steel or copper that are used for cooking in contemporary contexts in Pakistan and northwest India (Dales and Kenoyer 1986), and specifically for cooking *daal* (pulses) in present-day Haryana (Singh, pers. comm.; Pawar, pers. comm.).

Another vessel form of interest to scholars and relevant to this thesis are perforated vessels. Perforated vessels come in various shapes and sizes, ranging from tall, straightsided jars to miniature globular pots (Dales and Kenoyer 1986: 110), and are documented at numerous Indus sites. The distinguishing feature of this vessel shape is that almost the entire body is pierced with holes, and the base often has a large hole through its centre. The unique form of this vessel has prompted multiple interpretations of its purported uses. While some have connected them to dairy processing based on their similarities to ethnoarchaeological dairy-processing vessels in Central Asia and Iran (Gouin 1990; Bourgeois and Gouin 1995), others have suggested they were braziers for heating (Mackay 1938: 207), colanders for draining or straining liquids, or sieves for preparing cereal pastes (Dales and Kenoyer 1986: 108-109). This thesis examines different vessel-forms, including 'cooking jars' and perforated vessels, to reconstruct their possible function in food production.

2.3.2. Indus agriculture through macro- and micro-botanical analyses

This section summarises present knowledge about Indus agricultural practices and how they relate to food production. As mentioned above (Section 2.1.3.), Indus populations occupied an area that straddled an environmental threshold where presently there is an overlap of summer and winter rainfall systems (Petrie et al. 2017). Until the 1980s it was assumed that Indus cereal exploitation was dominated by wheat and barley (Fairservis 1967, 1971), but the regional diversity of domesticated plants through the Indus region was recognised after excavations at sites in Gujarat, particularly at Surkotada, Rojdi and Rangpur, revealed a sequence of occupation dominated by summer crops and millets; particularly *Eleusine* sp., *Panicum sumatrense*, *Setaria* cf. *pumila* and *Setaria* cf. *italica* (Vishnu-Mittre and Savithri 1982; Weber and Vishnu-Mittre 1989; Weber 1991, 1999; Reddy 2003). Since then, there is increasing recognition of how diverse environmental and geographical conditions across the Indus Civilisation would have led to variable agricultural strategies (Possehl 2002; Weber 1999, 2003, Weber et al. 2010). Some examples of crops grown include winter crops (*rabi*) such as wheat, barley, oats, pulses such as peas and chickpeas, plants for fiber and oil like flax and linen, and fruits like jujube. These thrive best during cooler months, and their growing season would have been between November/December and April/May (Fuller 2006; Wright 2010). Conversely, summer cropping (*kharif*) involved cereal grasses like millets, rice (see Fuller 2006; Bates 2016; Petrie et al. 2016), mustard, grapes, dates, sesame, cotton, hemp, and jute (Wright 2010), and their planting would have taken place in May for an October harvest, with the monsoon likely providing ample rainfall for their survival (Weber 2003). Many perennial crops would have likely been grown year-long (Wright 2010; Bates 2019) (see Table 2.3).

Unravelling issues of seasonality and environmental diversity through archaeobotanical evidence has been challenging due to the unsystematic study of much archaeobotanical data. This has been regarded as a fundamental challenge for South Asian archaeology and archaeobotany for some time (e.g. Fairservis 1967; Vishnu-Mittre and Savithri 1982; Fuller and Madella 2002; Madella and Fuller 2006; Weber et al*.* 2010; Petrie and Bates 2017; Petrie et al. 2018). As reviews of the state of Indus archaeobotany exist elsewhere (Fuller and Madella 2002; Madella and Fuller 2006; Bates 2016; Petrie and Bates 2017; Petrie et al. 2017; Petrie et al. 2018), only a brief summary and a discussion about their implications for reconstructing Indus foodways will be provided here. Although several phytolith studies exist that discuss the use of plant and vegetal material within the

Indus context (e.g. Eksambedkar 1999; Fujiwara et al. 1992; Madella 1995, 1997, 2003) their primary focus is on non-culinary products. This is why they will not be considered in detail here.

2.3.2.1. Cropping processes and terminology

Indus cropping practices have often been described using terms like 'mixed-cropping' or multi-cropping' to characterise the growing of multiple crops in one or more seasons (e.g. Weber 1999, 2003; Wright 2010). However, Petrie and Bates (2017: 83) have critically addressed how much of what is said about Indus cropping is based on inference and characterised imprecisely with incorrect usage of terminology. They suggest that use of data on modern varieties of the most common crops, which have distinctive sowing times, growing periods, water requirements and harvest times can provide more nuanced assessment of how multiple crops were being grown at settlements. For example, crops like barley (*Hordeum vulgare*) and wheat (*Triticum* sp.) have different water and fertilisation requirements and generally are not cropped in close proximity; some crops have intense labour requirements and are usually mono-crops (e.g. *Macrotylma* cf. *uniflorum*); and a range of millet species (*Echinochloa* sp., *Setaria* sp. and *Panicum* sp.) share ecological and crop-processing requirements and can be grown as mixed intercropping crops (Petrie and Bates 2017: 93-94). However, the limited amount of detailed published archaeobotanical data restricts such an analysis to a handful of sites (Petrie and Bates 2017: 94); a region-based summary of which is provided below.

2.3.2.2. Regional diversity

Archaeobotanical evidence from Indus sites in Gujarat was previously used to support a model of winter/*rabi* cropping in the 'core' and summer/*kharif* cropping in the 'periphery', where the periphery was regarded as unusual and not representative of the situation across the Indus Civilisation as a whole (Meadow 1989, 1996, Fuller and Madella 2002: 353‐5). Fuller and Madella (2002: 355) also suggested that 'core' areas practised more intensive agriculture, whereas populations in the summer cropping areas utilised more extensive systems. However, newer evidence contests this claim (Petrie et al. 2017: 457). Based on an updated assessment of the data, it has been suggested that single-season winter mono-cropping was likely practised in Sindh and Baluchistan (Petrie 2017; Petrie and Bates 2017), although there is very limited archaeobotanical evidence available. In Gujarat, the lack of winter rainfall and presence of summer crops suggests populations used a single-season mono-cropping strategy focusing on a variety of summer crops, such

as millets and pulses (García-Granero et al. 2016; Petrie and Bates 2017). In Punjab, however, there is evidence that both winter and summer cropping was practised (Weber 1999; 2003). For example, it was suggested that at Harappa, the predominant cropping pattern involved the cultivation of winter plants and to a lesser extent, millets, which are present from the pre-urban period (Weber 1999; 2003). Weber (2003) suggested that the cultivation of millets and other summer crops intensified during the urban period, though wheat and barley continued to be dominant crops. Petrie and Bates (2017: 95; also Petrie et al. 2016) have noted that the evidence for this is ambiguous: although summer crops increased in ubiquity across samples; their relative abundance does not see much growth from the pre-urban to post-urban phases. Rather, it appears that there was a marked decline in the relative abundance of winter crops over time; with an increase in the 'weeds/unknown/other' category (Weber 2003; Petrie and Bates 2017: 95). In northwest India, combinations of winter and summer crops have been attested at several Indus settlements, including at Banawali, Balu, Kunal and Farmana, in contexts that appear to date before and/or during the Indus urban phase (Saraswat & Pokharia 2000, 2001, 2002; Kashyap and Weber 2010; Weber et al. 2011). In the region of central Haryana, evidence from small rural Indus settlements suggest complex agricultural strategies where wheat and barley were likely intercropped, and summer crops like rice, millets and tropical pulses were likely sequentially multi-cropped (Bates 2016; Petrie and Bates 2017). Apart from suggesting the diversity of agricultural management strategies across regions; these patterns indicate how *food choices* in the Indus may have been extremely diverse and complex (Table 2.3). Different regions likely had different ideas of what were staples versus 'special' cereals, pulses, and vegetables, accompanied with seasonal cycles to plant foods that were prepared. Similarly, certain plant foods may have had prestige associations in specific regions (Fuller 2003). Trade and exchange in staple crops between populations living in different regions may have also occurred (Fuller 2014; Madella 2014). An understanding of variations in practices will only be possible when the proportional exploitation of individual plant species in different regions is widely available (Petrie and Bates 2017).

Table 2.3: List of winter and summer crops found in the Indus Civilisation, based on approximate order of ubiquity. 'A' indicated annual, 'P' indicates perennial plant, and 'A/P' indicates a plant that can be either. (after Petrie and Bates 2017; Jones 2017, Weber and Kashyap 2010; Bates 2019).

Type	Winter (rabi)	Summer (kharif)
Cereals	Barley (Hordeum vulgare) A	Rice (Oryza cf. sativa) A
	Wheat (<i>Triticum</i> sp.) A	Signalgrass millet (Brachiaria ramosa) A
	Oats (Avena sativa)	Sawa millet (Echinochloa colona) A
		Little millet (Panicum sumatrense) A
		Proso millet (Panicum miliaceum) A
		Foxtail millet (Setaria italica) A
		Yellow foxtail millet (Setaria pumila) A
		Pearl millet (Pennisetum glaucum) A
		Kodo millet (Paspalum scrobiculatum) A
		African finger millet (Eleusine coracana) A
		Sorghum millet (Sorghum bicolor) A
Pulses	Chickpea (Cicer sp.) A/P	Horsegram (Macrotylma uniflorum) A
	Vetching (Vicia/Lathyrus sp.) A	Black/Urad bean (Vigna mungo) A
	Lentil (Lens sp.) A	Mung bean (Vigna radiata) A/P
	Pea (<i>Pisum</i> sp.) A/P	African gram bean (Vigna cf. trilobata) A/P
Fruits	Indian jujube (Ziziphus mauritiana) P	Grape (Vitis vinifera) P
	Eggplant (Macrotylma solanum) P	Cucumber/melon (Cucumis sp.) P
		Date palm (Phoenix dactylifera) P
Oilseeds and Fibre	Sesame (Sesamum sp.)	Cotton (Gossypium sp.) A/P
	Linseed/Flax (<i>Linum</i> sp.) A	Hemp (Cannabis cf. sativa) A/P
	Poppy (Papaver sp.)	Jute (Corchorus sp.) A/P
		Mustard (Brassica sp.) A/P
Roots and	Ginger (Zingiber sp.) P	Turmeric (Curcuma sp.) P
Tubers	Garlic (Allium sativum) P	

Archaeobotanical evidence from Indus sites in northwest India also suggests there may have been differences between the choices of crop cultivation between medium- and small-sized settlements *within* the region. For example, in Haryana, two rural sites located within a short distance to Rakhigarhi investigated in this thesis demonstrate varying relative abundances of crops. Present evidence from Lohari Ragho I (see Section 4.1.2.2), suggests a dominance of summer crops, especially millet, while Masudpur VII and I (Sections 4.1.1.2 and 4.1.2.1), about 10 km and 14 km away respectively, demonstrate evidence for both summer and winter crops, with generally high proportions of summer crops (Bates 2016; Petrie et al. 2016: 1498; Petrie and Bates 2017; Ustunkaya, pers. comm.). Similarly, crop proportions at Masudpur VII and I are distinct from those seen at the nearby site of Farmana (Weber et al. 2011; Petrie et al. 2016; Petrie and Bates 2017,

Section 4.1.3.1). These differences could be attributed to taphonomical factors; however, they may also hint at unique crop and food choices made by farmers in rural settlements (Petrie et al. 2016; Petrie and Bates 2017). This thesis will further explore the potential of variations in food choices and vessel usage between these rural sites.

2.3.2.3. Cropping practices, urbanism and deurbanisation

Discussions about the relationships between agricultural systems, urbanism, and changing climate are at the heart of Indus archaeology (Possehl 1982; Kenoyer 1998; Weber 1999; Fuller and Madella 2002; Madella and Fuller 2006; Wright 2010). For example, it has been argued that the transition to the urban phase of the Indus Civilisation was characterised by intensification, diversification and specialisation in agricultural, pastoralism, and foraging systems, especially at Harappa (Wright 2010: 145). Similarly, Weber (2003: 198) has argued that the continued effort at Harappa to diversify crops was interconnected with changing needs within its society to issues dealing with storage, trade, and the centralisation and control of the food supply (Wright 2010: 177). It is assumed these processes enabled the surplus to provision individuals engaging in non-agrarian, craftproducing activities (Wright 2010: 145).

Evidence for increasing crop diversity at Harappa, and evidence for the implementation of plough agriculture and draft animals have been used to suggest that large tracts of lands were farmed (Weber 2003; Miller 2003, 2004). Weber (2003:181) suggests there is an increase in the density and proportions of by-products associated with crop-processing at Harappa over its occupation. This evidence was used to suggest that crops was processed after harvesting and before storage at a location that was not an individual household, (Wright 2010: 205), indicating a communal or even centralised organisation of production (Wright 2010: 205 citing Fuller and Madella 2000; Weber 2003). Other examples of the cropping of perennial fruits, dates and grapes; and fibers such as jute and cotton harvesting and seed collecting for planting have been used to suggest the existence of complex productive systems in the urban phase (Wright 2010: 206). Unfortunately, there is not enough data to support any of these models; which have been premised on evidence from Harappa.

It is also possible that there were extensive food supply networks across the Indus Civilisation in the urban period. Kenoyer (2008) suggested that food supply to cities was derived from hinterlands, and Madella (2014) proposed that Cholistan may have exported cereal to different regions. Apart from possible luxury items like beads, everyday items

used in culinary activities like grinding-stones made from non-local stone have been found across the Indus Civilisation (Law 2011), suggesting that bulky, quotidian items moved across large distances.

The transition into the post-urban phase has also been characterised as being marked by a progressive diversification of agriculture, with the introduction of new crops such as millet and increased summer cropping, specifically at Harappa (Weber 2003; Madella and Fuller 2006). But given the evidence of the diversity of winter and summer crops in preurban contexts from northwest India (Bates 2016; Petrie et al. 2016; Petrie and Bates 2017), more nuanced approaches to characterising the shift to the post-urban phase in different parts of the Indus Civilisation are required. Petrie and colleagues suggest that in regions where only single-season cropping was possible, it is probable that some form of organised and potentially centralised storage would have been required to feed large urban populations throughout the year (Petrie et al. 2016: 1501). However, in regions where multiple crops were grown in different seasons, there may have been differential storage requirements due to the regular supply of crops and possibly reduced need of centralisation (Petrie and Bates 2017). This would have also likely affected how different populations managed and mitigated food stress in the face of changing environmental conditions in the post-urban phase (Petrie 2017; Petrie and Bates 2017). Similarly, if long- and shortdistance food supply networks existed; these would have been variably affected by changes in the environment and/or social systems in place. Keeping this in mind, this thesis investigates the use of foodstuffs in vessels across settlements in the urban period, as well as possible changes in vessel-usage patterns in the post-urban period at certain settlements. Although concepts such as intensification and diversification are difficult to assess via residues in pottery, results may be indicative of the diversity or uniformity of products processed in vessels within or across sites. The next section addresses Indus zooarchaeology, providing important background for the interpretation of results in this thesis.

2.3.3. Indus zooarchaeology

2.3.3.1. State of research

The diversity of faunal species exploited by Indus populations is well-attested through numerous zooarchaeological studies conducted in different regions. A number of reviews have highlighted the exploitation of both domestic and wild mammals, as well as nonmammalian resources (Thomas and Joglekar 1994, Meadow and Patel 2002, 2003;

Joglekar et al. 2013). Despite the increase in the number of zooarchaeological studies since the 1950's (Meadow and Patel 2002), there are still many gaps in our knowledge of human-animal interactions in different sites and regions of the Indus Civilisation. Problems include lack of systematic sampling, gaps in reporting of sample sizes, the absence of detailed contextual or stratigraphic recording or association (and as a result, poor chronological resolution), as well as the absence of standard zooarchaeological protocols for reporting faunal remains (Joglekar et al. 2013). Problems of identification are acute in South Asia because of the presence in archaeological sites of skeletal parts that are morphologically quite similar between taxa. The most common difficulties of identification relate to distinguishing the bones of different ruminants, for example, differentiating *Ovis* (sheep) from *Capra* (goat) from *Gazella* (chinkara) from *Antilope* (blackbuck) as well as large ruminants such as *Bos* (cattle) from *Bubalus* (water buffalo) from *Boselaphus* (nilgai).

Additionally, the resolution of data collected is not nuanced enough to make interpretations about the social meaning of animals. Across the Indus, Gujarat is perhaps the region with the most detailed site-based zooarchaeological studies, with work on the reconstruction of mortality profiles and systematic analyses of butchering practices (Chase 2010, 2012, 2014; Chase et al. 2014a, 2018). In Pakistani Punjab, Harappa has had the most intensive examination of animal bones (Belcher 1991; Meadow 1991; Miller 2003, 2004). Few sites in Sindh and Baluchistan have had limited systematic investigation of faunal material, except for Mehrgarh and Nausharo (Meadow 1989, 1991; Belcher 1991; Chase 2005, 2012). In Haryana, excavators have begun to conduct basic zooarchaeological analyses on faunal assemblages (e.g. Joglekar et al. 2013, 2017), but many of the reports are unpublished and interpretations that can be made are limited to a general level (Joglekar et al. 2013).

While discussions about the diversity and variability of agricultural practices across the Indus region have increased in scholarship in recent years, our understanding of cultural and regional variations in patterns of animal exploitation have been extremely limited. Crucially, as it stands, reports suggest a uniformity in the use and cultural preference for certain animals, for example, the importance of cattle across the Indus Civilisation (Possehl, 1979; Thomas 1989, 2002; Thomas and Joglekar 1994; Meadow and Patel 2003). This pattern has been highlighted by several scholars (e.g. Fairservis 1986; Thomas and Joglekar 1994; Meadow and Patel 2003); but has not been critically assessed

in terms of Indus commensality and the social context of the role of animals in Indus societies.

On average about 80% of the faunal assemblage from various Indus sites belong to domestic animal species (Thomas and Joglekar 1994; Thomas 2002; Joglekar et al. 2013), which include cattle/buffalo, sheep/goat, and pig. Out of the domestic animals, cattle (and possibly buffalo) are the most abundant type of animal discovered in faunal assemblages across the Indus Civilisation, averaging between 50 to 60% of the animal bones found across Indus sites (Thomas 2002; Miller 2004; Joglekar et al. 2013; Chase 2014) As it can be difficult to securely distinguish between cows and buffalo, differences in proportions of *Bos* and *Bubalus* at sites cannot always be determined. Meadow (1991) has claimed that the ratio of cattle to buffalo at Harappa is 34:1; indicating that buffalo made a very small contribution to Harappa's economy and diet and may have not been fully domesticated. In contrast, water buffalo appear to be present in large proportions at Dholavira (Patel 1997). Despite the arid environment of Kadir Island, it is possible their survival was assured by their immersion in the large reservoirs around the site (Bisht 1991, 2005; Patel 1997). Meanwhile, on average, sheep/goat remains account for about 10% of the total faunal assemblage across Indus sites located in alluvial plains (Thomas and Joglekar 1994; Meadow and Patel 2003). Wright (2010: 173) has noted that goats were more likely to be found in arid and semi-arid environments, such as at Nausharo and regions in Gujarat, whereas in better-watered areas, such as Pakistani Punjab, sheep probably outnumbered goats, as seen for example, at Harappa (Meadow 1991). Pigs make up about 2-3% of total faunal assemblages across Indus sites (Thomas 2002). Thomas (2002), and Chase (2014) have noted that the domestic status of the pig in Indus stock-raising is yet to be ascertained. Faunal assemblages from rural village sites that have been studied appear to be broadly similar to those from urban centres with respect to species ratios (Chase 2014; Joglekar et al. 2014, Joglekar et al. 2017).

The overwhelming proportions of cattle bones across Indus sites indicates a cultural preference/predilection for beef-consumption across Indus populations, supplemented by the consumption of mutton and lamb. It is possible that meat consumption was complemented with the widespread existence of dairy economies. Wild animal species like deer, antelope, gazelle, hares, birds, and possibly wild pigs are also found in small proportions in the faunal assemblages of both rural and urban Indus sites, suggesting a taste for game. Riverine resources such as reptiles, fish and molluscs also featured at Harappa, Alladino, Balakot and Nausharo (Belcher 1991, 1994, 2003), as did marine

resources on coastal sites such as Balakot and sites in Gujarat (Belcher 1991; Deshpande-Mukherjee 1996, 1998), suggesting that these diverse resources had a place in the Indus diet. The presence of a marine catfish species (family *Ariidae)* at Harappa (Belcher 1991) indicates that some fish may have reached this inland site in dried form through long distance exchange networks running between Harappa and sites located near coastal environments.

2.3.3.2. Approaches to Indus faunal analysis

To gain insight into the relationship between humans and animals beyond species identifications, zooarchaeologists use species ratios, animal mortality and sex profiles, and studies of butchery practices (e.g. Payne 1973; Grant 1982; Reitz and Wing 1999; O'Connor 2000; Sykes 2014). Some or a combination of these methods have been used successfully at Indus sites, but poor preservation conditions have introduced problems that limit the extent of detailed analyses. For example, many bones from Indus sites are mineralised, have salt and phosphate inclusions, or are encrusted with calcite and organic materials (e.g. Chase et al. 2014a; Joglekar et al. 2017). The taphonomic alternation of bones makes it difficult to conduct detailed studies of cutmarks or butchery practices, which would help reconstruct the *chaîne opératoire* of how cuts of meat were produced (e.g. Chase 2005, 2012). It may also obscure the pathology of animal bones that may be informative about the use of animals for traction or dairying in their lifetimes (Miller 2004). Taphonomy aside, the scarcity of ageable and sexable faunal specimens within an assemblage, or small assemblages make slaughter profiles difficult to construct, which in turn make inferences about the extent of meat-production or secondary-product exploitation challenging. These problems persist within the Indus context, (see Joglekar et al. 2013, 2017; Chase 2010, 2012, 2014).

Some studies have recorded site-specific spatial variations in animal patterning, for example, at Harappa (Meadow 1991; Miller 2004), Dholavira (Patel 1997), and Farmana (Channarayapatna 2014, 2018), as well as smaller sites such as Mehrgarh (Meadow 1989), Balakot (Meadow 1988), Shikarpur (Chase 2014), Bagasara, Jaidak, and Kotada Badli (Chase 2010, 2012; Chase et al. 2014a, 2014b, 2018; Chakraborty et al. 2018). Changes in species ratios over time have been noted at large and small sites such as Harappa, Dholavira and Nausharo, suggesting the increasing and/or decreasing importance of particular animal species, which are possibly correlated with the demands of increasing urbanism. For example, research on animal bones from Harappa indicates that cattle
became more important in the economy over time. Miller's (2004) research on the assessment of the extent of secondary products economy at Harappa suggested more intensive cattle husbandry strategies and secondary products exploitation in the urban period with a notable decrease in the post-urban period, and a higher proportion of medium or small animals being observed in later contexts (Meadow 1991: 103; Miller 2003, 2004). She suggested this pattern may be consistent with a decline in specialist pastoral producers, and a return to generalised household pastoral production systems. Alternatively, it could also be interpreted as the potential decrease in secondary products exploitation over time, perhaps as a strategy to cope with stress on resources in the post-urban period. Among the small ruminants, sheep are more numerous than goat at Harappa; suggesting further emphasis on dairying, and perhaps, on fiber products (Meadow 1991, Meadow and Kenoyer 1997). At both Nausharo and Dholavira, however, sheep and goat are found in relatively equal proportions, although there are slightly more sheep present (Meadow 1989; Patel 1997). The differences in kill-off patterns suggests that sheep may have been bred principally for wool (Wright 2010).

As faunal analyses at Indus urban sites have mostly dealt with secondary or trash deposits of bone (e.g. Meadow 1991; Miller 2004), making inferences about within-site variation in the access or consumption of different animals is challenging as there may be no correlation between the distribution, consumption and disposal patterns that eventually led to the creation of the archaeological assemblage (Meadow 1991; Miller 2004). For example, the presence of elements of cattle on all of Harappa's mounds suggests that residents in all mounds (or neighbourhoods) had access to animals that might be used for traction and as a meat source (Miller 2003). Similar patterns of large bovid bones in street deposits have been found in Dholavira (Patel 1997). In Haryana, Girawad had wild mammal remains concentrated in the central part of the site in the Early Harappan period, but at Farmana, an assessment of the different anatomical parts of different animals, including wild species, revealed that a system of distribution or sharing of high meatbearing parts may have been in place, as not all the complexes had all the parts across all time periods (Channarayapatna 2014, 2018). These patterns may be indicative of distributive systems or preferences of particular social groups within the settlement.

2.3.3.3. Slaughter and age profiles: meat and secondary-products utilisation

The construction of age and slaughter profiles is an important feature of zooarchaeological studies as it reveals patterns that help reconstruct the strategies used for the creation and

management of animal products (meat, milk, or traction). These activities also reflect ontological changes in the relationship between humans and animals (Sykes 2014). The precise origins and extent of secondary products exploitation in ancient South Asia are not well understood. Several scholars have commented on the importance of large bovids like *Bos indicus* and *Bubalus Bubalus* for dairy products and draft/traction, insisting on the overwhelming importance of cattle pastoralism for Indus society (e.g. Fairservis 1986; Possehl 1979; Meadow and Patel 2002; Miller 2004). Dairy production provides milk, butter, ghee, cheese, and yoghurt, which are all items that are high in nutritional value, but also storable and replenishable (Miller 2004: 46; Greenfield 2010). Similarly, the use of traction animals for agricultural purposes and the transport of agricultural products, craft items, and building materials (e.g. wood, clay) would have been vital for the provisioning of urban and rural populations (Miller 2004). The evidence of miniature terracotta bullock cart frames from Indus sites throughout the region, as well as yokes from Indus phase levels at Nausharo, and ploughs from Banawali, suggest that bovines were harnessed for draft (Meadow and Patel 2002).

Despite being limited in number, slaughter profiles for Indus settlements reveal a general trend of the presence of older adults (for bovine and caprine/ovine species) (Joglekar et al. 2013; Chase 2010, 2012, 2014). The present state of knowledge regarding species distinctions among the large stock and breed variation within these preclude the morphometric analyses required to distinguish cow, bulls, and castrates among cattle and buffalo (Chase et al. 2014a: 9). Miller (2004) incorporated ethnoarchaeological studies in present-day Pakistan, and artefactual and zooarchaeological analyses of cattle remains to specifically address the extent of secondary product exploitation of cattle at Harappa. She found that out of the bovine animals studied, 90% were kept alive until the age of 3-3.5 years, which indicates that females were used for dairying production, whereas males were used for traction (Miller 2004). Importantly, there were higher proportions of older adults in the urban period (42% pre-urban vs. 56% urban), which she suggested indicates the increasing importance of secondary products in the urban period and the use of animals for longer productive lifetimes (Miller 2004). Based on this, she suggested that dairying and traction activities were vital for the agro-pastoral economy at Harappa, with an intensification of secondary product exploitation and specialisation in pastoral production systems during the urban phase (Miller 2004).

Age and slaughter profiles at Bagasara, Shikarpur, Jaidak and Kotada Badli in Gujarat indicate that cattle were probably exploited for secondary products (milk and traction), but sheep/goat were likely primarily raised for meat (Chase 2014; Chase et al. 2014a, 2018; Chakraborty et al. 2018). For example, at Shikarpur during the urban period, fewer adult sheep/goat were kept in a herd (enough to maintain the long-term viability of the herd), but more than half the cattle were kept to adulthood age prior to consumption (Chase et al. 2014a). These patterns are consistent with subsistence-oriented production and secondary product exploitation (milk and traction), respectively (Chase et al. 2014a). Both Miller (2004), Chase (2014) and Chase and colleagues (2014a) have noted that most zooarchaeological studies use the presence of high numbers of young animals (nursing calves) as evidence of dairying production (e.g. Payne 1973) as most milk is available for human consumption if male lambs, kids or calves are slaughtered soon after birth. Additionally, the presence of adult males has also been interpreted as a strategy for the maximisation of meat products. This interpretative approach has also been widely criticised (Halstead 1998; Greenfield 2010), however, and may only be relevant in the case of a focus on a single-product economy. Given the absence of the slaughtered calves at Harappa and some sites in Gujarat, and a predominance of adult animals, it is likely that most Indus settlements adopted complex mixed product-economies, possibly ones that emphasised secondary-product use. Similar evidence exists at Dholavira (Patel 1997), which has a predominance of adult bovids. However, Patel (1997) has also noted the presence of higher proportions of young animals slaughtered in the 2.5 to 3.5-year ageranges, hinting at a different production economy at Dholavira. She has also observed differences in cattle size distributions, suggesting the presence of different and smaller cattle breeds at the site, as well as a high number of water buffalo bones (Patel 1997). Taken altogether, age, size, and species patterns suggest a regional diversity in subsistence strategies, particularly between Harappa and Dholavira in the urban period, but less clear differences between Harappa and smaller settlements in Gujarat such as Shikarpur, Bagasara, Jaidak and Kotada Badli (Chase 2014; Chase et al. 2014a, 2018, Chakraborty et al. 2018).

Another example of clear regional variation in cattle slaughter patterns is evident at the site of Balakot (Meadow 1979). The slaughter profiles of cattle in the urban period were recorded as 90% juvenile, with high proportion of sub-adults versus adult cattle (Meadow 1979). The high proportion of young cattle visibly contrast with patterns observed at the sites mentioned above. This pattern was interpreted as a strategy employed for optimising meat production (Meadow 1979). When compared to patterns observable from the pre-urban period, however, an increase in the slaughter of adults was observed,

possibly suggesting a gradual increase in the importance of secondary products exploitation from the pre-urban to urban period (Meadow 1979). Miller (2004) suggested that this shift may have occurred at a regional or multi-regional level, a shift towards intensive secondary products exploitation representing larger subsistence and economic trends for the civilisation as a whole. Such a claim would need much more evidence from many other Indus settlements.

In northwest India, Joglekar and colleagues (2013) reviewed evidence from fourteen sites within Haryana, Punjab, and Uttar Pradesh to make preliminary observations about the faunal diversity during the Indus period in the region. It is challenging to meaningfully compare this with faunal data obtained from other regions as the published data provides limited resolution, and slaughter profiles have not been created. Some site-specific variations, however, have been observed. They noted that Indus communities in northwest India consumed a wide range of animals, including domestic and wild mammals, birds, reptiles, riverine fish, and molluscs (Joglekar et al. 2013). Proportions of domestic animals outnumber wild species; and cattle make up the largest proportion of domestic species across sites (Joglekar et al. 2013). Out of the medium-sized domestic mammals, goats outnumber sheep across sites (Joglekar et al. 2013), except at Girawad, where more sheep appear to have been reared than goats, and at Farmana, where they have equal representation prior to and during the urban phase (Channarayapatna 2014, 2018). Pigs are found at ten out of fourteen sites (Joglekar et al. 2013). Cattle size measurements suggest that small numbers of bulls versus a dominance of castrated bulls or females were present in the assemblages studied, a herding strategy likely adopted to practice dairying (Joglekar et al. 2013). This thesis will test whether vessels were used to process dairy products in northwest India and will critically assess the evidence provided for widespread dairy production in the Indus.

2.3.3.4. Evidence for carcass processing

Butchery practices have only been systematically studied from a few sites in the Indus Civilisation. Chase (2005:51) noted that butchery practices reflect cultural preferences and technological choices, and the specific way in which an animal was prepared for consumption encapsulates important economic and social information. These practices also inform us about food preparation, which is the primary focus in this thesis. Cut-marks and charring have been observed on meat and marrow-rich bones of domestic and wild animals across the Indus Civilisation (Meadow 1979; Belcher 2003; Joglekar et al. 2013;

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Channarayapatna 2014, 2018). In Gujarat, faunal assemblages at Bagasra suggested that relatively more cut-marked pieces of bone were found within the walled precinct of the settlement, indicating that residents within the enclosure prepared meat-based dishes differently from those outside the walls, perhaps signifying a social or cultural variance, while at Shikarpur, bones from all portions of the skeleton of animals are present in all areas of the site (Chase 2012, 2014). However, at Bagasra, initial stages of the butchery process were the same for all animals that were eventually consumed as food, and the butchering process was done solely with the use of metal tools (Chase 2012). Chase (2014) suggested that this pattern would arise if both communities were raising and butchering their own animals, or if they were receiving meat from an external, common meat market. At Indus sites in northwest India that are being investigated for this thesis, cut-marks appear to have been made with a sharp metal blade, just like at Bagasra (Channaryapatna 2014; cf. Chase 2012). Additionally, completely charred and vitrified bones were dominant among the bone modifications observed at Farmana and Masudpur I and Masudpur VII (Channarayapatna 2014; Joglekar et al. 2013, 2016, 2017). At Farmana, it was also noted that nearly all anatomical elements of cattle/buffalo had charring marks, whereas only the cranial fragments, ribs, scapula, vertebrae, and phalanges of sheep/goat were charred to different degrees (Channarayapatna 2014, 2018). These differences hint at different butchery practices for different animals and may indicate preferences for the roasting of meat. Using a different approach, Goyal (2017) suggested that at Kanmer, preparation via roasting was more common in wild animals, particularly deer, than in domestic animals.

2.3.3.5. Animal-human relationships and 'continuum of lifestyles'

Sykes (2014) has argued that methods and approaches in zooarchaeology often make it difficult to conduct 'social zooarchaeology', which is an understanding of how humananimal relations transform both parties, with the two becoming mutually socialised through their exchanges (see also Mullin 1999; Mlekuz 2007; Brittain and Overton 2013). For example, the determination of kill-off patterns and age profiles focus on the productive and alimentary rather than the social significance of animals (Sykes 2014). The emphasis on the death of animals rather than their life-histories also diminishes the relationship that existed between people and the living animals around them (Sykes 2014). Other issues result from quantitative methods such as NISP, MNI, or MNE, which have come under tremendous scrutiny for what they calculate, but they have not been interrogated for what they might mean or represent socially (Sykes 2014: 9). It is also well-recognised that

different parts of an animal carcass will be deemed variously as 'good' or 'poor' depending on cultural attitudes (Crabtree 1991). Additionally, these distinctions may not just be in terms of high or low value foodstuffs; they likely represent ontologies that are representative of power relations and extend into ritual and/or political lives (Davis 2008; Choyke 2010).

Direct correlations between the number of bones found in a faunal assemblage and the importance of animal are also found in Indus archaeology (e.g. Meadow and Patel 2003; Joglekar et al. 2013). The frequency of cattle bones is high in Indus faunal assemblages, leading to the assumption that cattle/buffalo were the most important animal(s). However, high levels of archaeological representation need not equate to an animal's social importance, and there may also be taphonomic variables contributing to biases in the archaeological record. The presence of adult cattle in a majority of faunal assemblages at Indus sites (see above) does suggest that bonds would have developed between people and their cattle. Ethnographical parallels exist with modern pastoral communities; the Suri herders of Northeast Africa see cattle as extensions of human society (Abbink 2003: 342), while the Skaha of northeastern Sibera see cows as central to society (Crate 2008), even though they do eventually consume them.

Some scholars have discussed how the relationship between people and their animals framed the lives of various Indus societies. For example, Wright (2010: 173-174) has discussed how a 'continuum of lifestyles' marked by degrees of mobility would have accommodated the keeping of animals in the Indus context, including nomadic pastoralists, semisedentary pastoralists, and sedentary pastoralists. A full range of animal specialisation types was likely present in the Indus Civilisation (Wright 2010). Wright (2010) has suggested that sedentary pastoralism is reflected by the large numbers of animal bones and cart tracks found in street deposits at Harappa, and water storage tanks in Dholavira. She claims that the current evidence indicates that animals were kept by urban inhabitants to supply dairy products, such a milk or yoghurt (Wright 2010), but more evidence is needed to support this. Settled pastoralists on the alluvial plains most likely took advantage of its varied landscape, seeking out well-watered areas for water buffalo. Sheep and goats could graze on marginal lands on the outskirts of agricultural lands and on stubble from harvested fields. The evidence from Bagasra shows that all the animals were pastured in the region with no seasonal migration; however, unlike sheep/goat, which were raised from birth locally at Bagasra, many of the cattle/buffalo were acquired at a young age from different locations, but probably from within the same region (Chase et al. 2014a, 2018).

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Semisedentary pastoralism was possibly practised at settlements such as Nausharo where pastoralists moved seasonally between upland and lowland settlements to avoid harsh winters and hot summers when grazing areas were reduced (Wright 2010), and at Oriyo Timbo in Gujarat, which was seasonally occupied (Rissman 1985; Reddy 1994; Wright 2010). Examples of nomadic pastoralism in the pre-urban (Hakra) period are suggested by the presence of surface scatters of small camp sites discovered by Mughal (1997) in Cholistan. It is possible that nomadic pastoralists lived beyond the limit of agricultural zones returned to the same location on a seasonal basis (Wright 2010). Elsewhere, it has been suggested that hunting and gathering populations possibly engaged in symbiotic relationships with village and town dwellers (Possehl 2002; Wright 2010), but there is limited evidence to support any of these ideas. Overall, evidence from Indus faunal assemblages has led scholars to focus on questions of the extent of urban pastoralism and/or mobile pastoralism practised across settlements, and the relationship between urban dwellers and hunter-gatherer groups across time. As the history of the development of pastoral systems in South Asia remains sketchy (Meadow and Patel 2003: 84), archaeologists often rely on ethnographic and historical record to make interpretations about the past (Guha 1994). It is likely that the mix of environments and specialisation of husbandry could have provided rich resource viability and exchange of products that was mutually beneficial. No pastoral system exists, or is likely to have existed for very long, without agricultural products being available through one means or another (Meadow and Patel 2003: 75). Nomadic pastoralists may have facilitated the movement of trade goods and their own products through their mobility. However, it is possible that these relationships were complex and required negotiation, especially during periods of food stress or crisis.

The different species of wild plants, animals and forest products in archaeological assemblages across the Indus Civilisation suggests that these resources were exchanged between hunters and gatherer groups, nomadic populations, and/or permanently-settled people (Wright 2010: 175). Although, it is also possible that rural and urban inhabitants hunted and consumed wild animals. The consistent presence of the bones of wild species of animals and fish at both large and small Indus sites, attests to the continued utilisation of wild animal resources by inhabitants. The possible complex systems of animal production and distribution are not easy to untangle with present evidence as they may have involved networks of multiple 'animal specialists': urban, rural, and nomadic, or may not have involved 'specialists' at all. As these has implications for how food sources were produced,

accessed, and valued across different Indus societies, these considerations are vital to how Indus foodways are imagined.

2.3.4. Indus isotopic studies

The use of isotopes of C, N, O and Sr from human and/or animal bone and teeth give an indication of diet and mobility (see Richards and Hedges 1999; Balasse et al. 2002; Schulting and Richards 2002; Evans et al. 2006; Hedges and Raynard 2007; Pollard et al. 2007). Carbon isotope values (δ^{13} C) are used to estimate dietary proportions of C₃ versus C⁴ terrestrial plants (see Section 3.5.2) eaten and to assess marine versus terrestrial inputs (DeNiro and Epstein 1978; Heaton 1999; Balasse et al. 2002; Kohn 2010). Nitrogen isotope values ($\delta^{15}N$) are commonly used to determine trophic level (DeNiro and Epstein 1981; Hedges and Raynard 2007). Oxygen isotope values (δ^{18} O) reflect the isotope values of the water ingested (as water or from food) at the time of tissue formation, providing a palaeoclimatic indicator (Bryant et al. 1996; Evans et al. 2006; Lightfoot and O'Connell 2016). Finally, the strontium isotope signature (${}^{87}Sr/{}^{86}Sr$) of an individual or animal can be compared to a biologically available signature determined by the surrounding biosphere to determine mobility of humans and animals (Lee-Thorp and Sponheimer 2003; Price et al. 2008).

In the South Asian context, and especially in the Indus Civilisation, only a small number of studies using isotopes have been conducted. Poor preservation of bone collagen has meant that researchers can typically only analyse carbonate from bioapatite available from human or animal teeth, which limits our understanding of diet.

2.3.4.1. Human isotopic studies

C, O and Sr isotopic values of tooth enamel have been studied from individuals buried at Harappa, Farmana and Sanauli (Valentine 2013; Valentine et al. 2015). The results suggested that individuals at Farmana consumed more C_4 plants than at Harappa and Sanauli (Valentine 2013: 193-194), and that millet, or other arid-adapted C_4 plants, were an important proportion of the diet at Farmana. The results pertaining to diet of individuals suggest that there are inter-individual differences in diet at all three sites, but there was no relationship between age and sex and the relative contribution of C_3 and C_4 foods in diet (Valentine 2013: 124, 147, 169). $\delta^{15}N$ values were not available, so it is not possible to know the contribution of animal protein to the individuals studied. Unfortunately, the aggressive sample preparation methods used by Valentine (2013) suggest that these results may be unreliable (Lightfoot et al. in prep). Re-analysis of these results is currently

underway (Lightfoot et al. in prep; Nayak in prep.). As this thesis investigates vessels from Farmana, the results obtained will be able to provide further insight into the foodways and dietary habits of populations at the site.

2.3.4.2. Faunal enamel carbonate isotopic studies

Carbon isotopic results from domestic and wild ungulate tooth enamel from Indus sites in northwest India provide further information about animal management and serve as a proxy for reconstructing the various inputs into human diet. Bioapatite carbonate values from animal teeth from small, rural Indus settlements demonstrate differential animal management practices and give insight into the diet of different species of domestic animals and wild ruminants (Sarkar et al. 2016; Jones 2017; Lightfoot et al. in prep). The data demonstrates that in northwest India, most domestic and wild bovine species had primarily C_4 diets, with evidence for the mixed consumption of C_4 and C_3 plants for the wild ungulates and sheep/goat at most sites. There is little evidence for clear seasonal variation in diet in any of the species. Crucially, there is clear difference between bovine and ovine/caprine feeding; both cows and buffaloes ate C_4 plants throughout the year, whereas sheep/goat followed patterns consistent with wild ungulates, indicating mixing of C³ and C⁴ plants, and a less controlled diet, except at the site of Alamgirpur in Uttar Pradesh (see Chapter 4). Although the information available for different time periods is limited, there appears to be no change in δ^{13} C values for most species across time.

Results from enamel carbonate δ^{13} C values from Gujarat (Chase et al. 2014a, 2018) reveal patterns very similar to data from Haryana, except at the site of Kotada Badli (Chakraborty et al. 2018). Evidence from most sites in Gujarat suggests that cattle were consistently feeding on C_4 plants, whereas sheep/goat demonstrate some mixing of C_3 and C⁴ plants (but data for wild ruminants is not provided) (Chase et al. 2014a, 2018; Chakraborty et al. 2018). The authors concluded that the high proportion of C_4 in cattle diets is an indication of cattle being fed millet throughout the year (Chase et al. 2014a). However, at Kotada Badli, individual cattle/buffalo demonstrate considerable variation and appear to have consumed a mixed diet of C_4 and C_3 vegetation, whereas sheep possibly consumed more C4 plants than goats (Chakraborty et al. 2018).

The input of C_4 plants into the diet of wild ruminants and sheep/goat suggests the presence of C_4 plants in the surrounding landscape. Although the consistent C_4 diets for cattle/buffalo are suggestive of specific feeding practices for cattle/buffalo in many sites in Gujarat and Haryana, the possibility that cattle were feeding on wild C_4 vegetation cannot

be excluded. According to Reddy (1994) and Pokharia and colleagues (2017), the soil organics from sites in Gujarat indicate a C3-dominated environment during the Mature Harappan period, with increased input of C_4 plants towards the Late Mature Harappan period due to the increased cultivation of millets (Chakraborty et al. 2018). Thus, in Gujarat, it is possible that the primary source of C_4 for cattle/buffalo was from agricultural millets through specialized foddering. However, other studies around the world have demonstrated the fallacy of the assumption that any C4-based dietary input in animals or plants arises from millet (or maize in the Americas) (e.g. Cadwallader 2013; Warinner 2013). This is especially true if cattle/buffalo were valued for dairy production. Since millets are generally sown to take advantage of the monsoon and harvested in October and November, cattle would have to be fed dry millet in spring and summer. Ethnographic research conducted by Miller (2004) demonstrated that dairy animals are dependent on fresh green fodder supplies because the lactation process depletes animals of minerals, proteins, and sugars, components vital for their nutritional health. Although animals that consume dry fodders (chaff, grass) will continue to produce milk, the quantity and quality (fat content) of the milk is severely reduced. Miller (2004) noted that in Pakistani Punjab, when green fodder is scarce during the summer, cattle and buffalo are milked only once a day, and the volume of milk produced is much lower than in the winter and spring, when they are milked twice a day. During the driest summer months, many animals cease producing milk entirely and dry up (Miller 2004) but recover later in the year. Thus, dairy products would likely be subject to seasonal availability. These seasonal patterns are important to consider when reconstructing the likely contribution of different sources into animal and human diet. Considerations of animal management practices and animal diets thus serve as important proxies for reconstructing direct or indirect inputs into human foodstuff. As this thesis will demonstrate, evidence from dietary isotope studies can provide insight into interpreting the potential source(s) of lipid residues into vessels.

2.3.5. Indus biomolecular archaeology: starch-grains, ceramic vesselfunction, and lipid residues

Methods to directly investigate food remains associated with archaeological artefacts, such as the study of starch, lipids and proteins have been limited in the South Asian context. Studies of starch-grains in association with artefacts such as grinding stones, pottery and human/animal teeth have been conducted in Gujarat and Haryana, from time periods spanning from the Chalcolithic and Mature Harappan periods (García-Granero et al. 2015,

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2016, 2017), and Mature Harappan period, respectively (Kashyap and Weber 2010; Weber et al. 2011). There is only one example of a lipid residue study from Indus ceramics (Bourgeois and Gouin 1995), and another that has assessed Indus vessel-function (Gouin 1990). This thesis aims to fill this gap.

2.3.5.1. Starch-grain analyses

Kashyap and Weber (2010; Weber et al. 2011) reported the preliminary results of the first starch grain-analysis on several Indus artefacts from Farmana. They examined the surfaces of stone tools, pottery, and human and cattle teeth for charred residues and dental calculus respectively, extracting starches from encrustations (Weber et al. 2011). The authors found starches of barley, mango, and small millet adhering to the surfaces of pounders and grinding stones; lentils, curcuma, *Macrotylma solanum* (cf. aubergine), *Zingiber* (cf. ginger) in storage jars; and starches of lentils, small- and large-grained cereals, fruits, vegetables, roots and tubers in human tooth calculus (Kashyap and Weber 2010; Weber et al. 2011: 820) . They also mention correlations between certain artefact types and starches, suggesting that certain vessels were used to process specific plant matter (Kashyap and Weber 2010). These results are extremely exciting, and suggest it is possible to study specific *ingredients* that were a part of food production, and the relationship between foodstuff and material culture like grinding stones and pottery. However, starch-grains associated with archaeological objects are particularly susceptible to contamination from the surrounding sediment, or airborne starch in the storage or laboratory environment (e.g. Haslam 2004; Crowther et al. 2014; Barton and Torrence 2015) Thus, as details of the sample collection, laboratory or storage environment, reference collection, and full details of the results are yet to be published, it difficult to assess the robustness of the study.

2.3.5.2. Ceramic form and function and residue analysis

Few researchers have attempted to test the relationship between Indus artefacts and their purported function in antiquity using organic residue analysis or use-wear analysis. Bourgeois and Gouin (1995) conducted the chemical analysis of a single fragment of a perforated vessel from Nausharo. They used the chemical methods available at the time to extract fatty acids from the potsherd and compared it with proportions of fatty acids present in modern-day milk collected from sheep, goat, and cattle (Bourgeois and Gouin 1995). They suggested that the results confirmed the ethnoarchaeological hypothesis that perforated vessels were used as 'cheese-drainers' and went as far as to suggest that the lipids suggest the use of goat milk for cheese or ghee production. This was used to support Gouin's (1990; 1992) hypothesis of a large-scale dairying industry in the Indus Civilisation at the time. Although such an approach was novel for its time; the study was limited by: 1) lack of acknowledgement of the degradation of lipids within the ceramic matrix; 2) accounting for potential post-excavation contamination; 3) and by the number of samples analysed. It is now also well-established that it is not possible to achieve species-specificity of animal products based on degraded fatty-acid profiles (Evershed 2002). This thesis analyses a large sample of vessels with different shapes and sizes from different sites across northwest India and Pakistan, as well as a single site in south-eastern Arabia, and uses lipid analysis in conjunction with compound-specific isotopic analysis to determine the possible use of different vessels and products being processed in Indus vessels to address the limitations of the Bourgeois and Gouin's study.

2.4. Urban and post-urban food production in Indus agropastoral societies

The sections above highlight how questions about Indus subsistence and food production in different regions are deeply tied into debates about the nature of urbanism and the processes affecting or contributing to post-urban transformation. In most cases, the lack of systematic, detailed, and *testable* data severely limits nuanced understanding of the changing climate, agricultural strategies, animal management, plant and animal consumption, as well as ceramic production and use across the Indus Civilisation. The degree to which the diversity and variability of practices within and between regions can be measured and connected to something that was socially meaningful in both the urban and post-urban periods remains challenging. At present, there are many unanswered questions about how urban populations obtained their food, as well as how land may have been owned and labour organised (Miller 2015). It is assumed that most urban centres likely relied on external food production due to the number of non-agricultural specialists residing within cities (Yoffee 2005; Fuller and Stevens 2009; Wright 2010; Miller 2015). For example, at Harappa, there is clear evidence of labour specialisation (Kenoyer 1998; Wright 2010), participation in long-distance exchange networks (Law 2011), and urbanrural exchange (Wright 2010). At the same time, there is also evidence of animal husbandry and crop processing within the settlement, suggesting that urban centres produced at least some of their own food (Meadow 1991; Miller 2003, 2004; Weber 2003; Wright 2010; Miller 2015). However, to what extent were urban centres dependent on rural agricultural production? Was this dependence influential in determining their resilience and response to climatic changes and food stress in the post-urban period? How were rural and urban relational identities defined (cf. Chase et al. 2014b; Parikh and Petrie 2018)? Many models attribute the decline of Indus cities to decreasing agricultural yields caused by climatic shifts, which increased stress on their rural hinterlands to produce and supply food and other resources, resulting in the 'collapse' of civic authority (Kenoyer 1998; Staubwasser and Weiss 2006). Given how little is known about social/cultural diversity and political organisation in the Indus Civilisation, and the minimal evidence for centralised control within urban centres, Miller (2015) has proposed that goods-distribution and exchange may have been tied with social norms and conventions of group responsibility. Indeed, if urban centres obtained food and other resources via some form of reciprocal economic exchange (Parikh and Petrie 2018), rural populations may have become less likely to exchange food for urban products if agricultural yields declined (Jones 2017). Evidence from rural settlements in northwest India, however, suggest diversity of agricultural practices from the pre-urban period, their continuity into the posturban period (Bates 2016; Petrie et al. 2016; Petrie and Bates 2017), and minimal measurable impacts of climate change on crop water availability in the region (Jones 2017). Thus, it is clear that rural settlements had more complex food production practices than we might have expected, and there is no simple linear relationship between climate, subsistence practices, and society in the Indus Civilisation.

This strongly suggests that:

1) rural, small settlements across the Indus Civilisation deserve closer examination;

2) the relationship between large, medium-sized and small rural settlements, particularly in terms of food-production, must be tested;

3) regional patterns must be examined independently; and,

4) generalised relationships between climate, subsistence and society in the region must be interrogated.

This thesis addresses gaps in previous research by studying food production and vessel usage within large, medium-sized, *and* small, rural settlements in northwest India. Vessels from the urban and post-urban periods have been examined via organic residue analysis to examine how quotidian commensal practices were constructed within settlements in the

region, and if they changed over time. Importantly, this thesis also focuses on addressing the interaction between Indus material culture and foodstuff, on a regional, and local, sitespecific scale.

2.5. Chapter Summary

This chapter has provided an introduction to the Indus Civilisation with an overview of chronology, urbanism, climate, ceramics, agricultural and pastoral practices and possible human and animal dietary practices. This chapter sets the necessary groundwork to contextualise and interpret the results from lipid residue analyses conducted in this thesis. The next chapter provides a background for the scientific analyses conducted in this thesis.

Chapter 3

Background to ceramic organic residue analysis

This chapter defines and characterises the different concepts integral to the study of organic residues within archaeology, particularly to those related to organic residues adhering to ceramics, which is the focus of this thesis. Section 3.1 introduces different organic compounds and key terminology that will be used throughout the thesis. Sections 3.2 and 3.3 describe various aspects that influence our understanding of lipids within archaeological contexts including preservation, degradation and contamination. Section 3.4 details aspects of methodology by describing extraction processes, Section 3.5 describes analytical techniques with a focus on the ones used in this thesis, and Section 3.6 discusses interpretive methods. This chapter aims to summarise key concepts related to organic residues within archaeological contexts, particularly related to ceramics, as well as contribute critical insights concerning the assumptions and interpretations made within the field.

3.1. Organic compounds in archaeological contexts

Different types of organic compounds are found and investigated within archaeological contexts (Evershed 1993). These encompass a wide range of molecules belonging to different families of compounds with varying potential for preservation. These include nucleotides such as RNA and DNA, proteins, carbohydrates, and lipids (Evershed 1993). Of these, lipids have demonstrated a higher rate of survival, which is generally attributed to their easy entrapment in organic or mineral matrices and their inherent hydrophobicity which aids their long-term survival (Evershed 1993). Investigations of organic remains in ceramics have studied charred, visible residues ('foodcrusts') adhering to pottery vessel surfaces (Heron et al. 2016) as well as amorphous compounds adsorbed within the ceramic matrix (Evershed 1993; 2008). The sections below will focus on different types of lipids adsorbed into pottery vessels.

3.1.1. Lipids

Lipids are defined by their solubility in organic solvents such as chloroform, ethers and alcohols (Christie 1989; Wade 2013). Lipids are composed mainly of carbon, hydrogen

and oxygen, and to a lesser degree, phosphorus, nitrogen and sulphur (Christie 1989; Evershed 1993; Wade 2013).They appear in a wide range of carbon skeleton structures (linear, branched and cyclic) which are commonly fully substituted with hydrogen atoms (Evershed 1993). The high proportions of hydrocarbon moieties in lipids confers nonpolar properties on them which reduce their solubility in water. The nonpolar, hydrophic nature of lipids makes them extremely valuable within archaeological contexts as it makes them resistant to leaching and reduces their availability as a substrate for microorganisms (Evershed 2008a). However, lipids have the reputation of being some of the most complex biomolecules to study as they are abundant in plants, animals, and micro-organisms and are subject to different types of chemical or microbiological alterations that can complicate interpretations of their origin (Evershed 1993).

Lipids encompass a variety of compounds with different functional groups (Figure 3.1). Lipids such as fatty acids (long-chain carboxylic acids), alcohols, and alkanes are less susceptible to alteration and are more commonly found in archaeological contexts. Lipids such as triglycerides and waxes are susceptible to alteration and degradation processes and often break up into their simpler constituents.

Figure 3.1: Structural formulae of different lipids. In a) carboxylic acids, a carbon atom is bonded to an oxygen atom by a double bond and to a hydroxyl group (–OH) by a single bond. Alkanes (b) are hydrocarbon chains with single bonds between each atom. An alcohol (c) is any organic compound in which a hydroxyl group (–OH) is bound to a carbon. Triaglycerides are composed of a glycerol base and three of the hydroxy (—OH) groups are attached to a fatty acid via an ester linkage (after Evershed 2008a).

3.1.1.1. Fatty Acids

Fatty acids are usually straight-chain, monocarboxylic acids (Scrimgeour and Hardwood 2007). The broadest definition includes all chain lengths, but most natural fatty acids have even chain-lengths between C4:0 and C28:0 (Malainey 2011: 56).They are composed of a hydrocarbon chain $(CH_3(CH_2)_n)$ and a carboxylic group (COOH), resulting in a non-polar region attached to a more polar end (Figures 3.1 and 3.2). The carboxylic group's polar structure interacts with water, making fatty acids with shorter non-polar structures more soluble in water. While solubility of fatty acids decreases with chain length, the melting point of fatty acids is also affected by chain length and the presence of double bonds. Saturated (single bond) long-chain fatty acids have high melting points but unsaturated (one double-bond or more) fatty acids have low melting points; the kink(s) in the chain preventing packing into a solid lattice (Wade 2013: 1204). These factors also affect definitions of fats and oils: most plant oils contain high concentrations of unsaturated fatty acids, lowering their melting point, and rendering them liquid at room temperature. Conversely, animal fats contain high concentrations of saturated fatty acids, making them solid at room temperature. Unsaturated fatty acids degrade rapidly and are rarely found in archaeological contexts (Eerkens 2007; Evershed et al. 1999; Steele et al. 2010), however, C16:1 and C18:1 are usually detectable in small quantities. Fatty acids are often found in relation to other compounds such as triglycerides or wax esters.

Fatty acids are classified as short-, medium-, and long-chain based on their carbon chain length. Certain chain-lengths are more ubiquitous in certain natural products (see Table 3.1). For example, mid-chain fatty acids such as $C_{16:0}$, $C_{18:0}$, and unsaturated fatty acids such as $C_{18:1}$ and $C_{18:2}$ form the basic building blocks of adipose (storage) tissue in animals and plants, while unsaturated fats are more common in plants and aquatic animals (Christie 1989). Even-numbered long-chain fatty acids $(C_{20:0}$ - $C_{28:0})$ are found mostly in plants (Kolattukudy 1970; Correa-Ascencio et al. 2014; Dunne et al. 2018). Although the occurrence of certain fatty acids in specific products may aid their identification in archaeological contexts, their differential degradation hinders their use for altered materials (Section 3.2.1).

cis-9-octadecenoic (oleic) acid $(C_{18:1})$

Figure 3.2: Structures of commonly occurring fatty carboxylic acids including saturated straightchain carboxylic acids such as a) palmitic acid and b) stearic acid; saturated branched-chain carboxylic acids in b) iso- and c) anteiso-forms; e) monounsaturated carboxylic acids $(C_{18:1})$.

*with a methyl group on the n-2 or n-3 carbon

3.1.1.2. Alcohols

Linear alcohols, sometimes referred to as fatty alcohols, have a hydroxy group at the end of their hydrocarbon chain. Like fatty acids, they exist rarely in free form, but rather in combination with other molecules, for example in the form of wax esters (Christie 1989: 149, Killops and Killops 2009: 44). *n*-alcohols found in association with *n*-alkanes in archaeological contexts are likely indicative of plant products (Harwood 1996, Dunne et al. 2017), but sterols of both animal (cholesterol) and plant (campesterol, sitosterol and stigmastanol) origin are also reported (Dudd et al.1999). However, sterols are rarely preserved in archaeological organic residues (Evershed 1993) as they oxidise rapidly and dissolve into groundwater under oxic burial conditions. Often, the presence of cholesterol in vessel extracts may indicate post-excavation contamination arising from human skin.

3.1.1.3. Alkanes

Alkanes are characterised by a hydrocarbon chain and the absence of functional group (Killops and Killops 2009: 30). Linear alkanes with an odd number of carbon atoms are among the main compounds of waxes, be they of vegetable or apicultural origin (Eglinton and Hamilton 1967; Kolattukudy 1970; Heron et al. 1994; Charters et al. 1995; Regert et al. 2001, 2005; Kimpe et al. 2002; Roffet-Salque et al. 2016). As waxes may be absent in archaeological contexts, the presence of *n-*alkanes can help distinguish between products derived from the epicuticular waxes of leaves, beeswax, or aquatic or terrestrial plants (Evershed et al. 1997, Dunne et al. 2017; Whelton et al. 2018).

3.1.1.4. Triacylglycerides

Triacylglycerides (TAGs) are the major component of fats and oils of natural origin (Christie 1989). They are composed of a glycerol base and all three of the hydroxy (—OH) groups are attached to a fatty acid via an ester linkage (Christie 1989:10; Wade 2013: 1202, Figure 3.1). The position of each fatty acid composing the TAG, as well as its chain length, number and position of unsaturation are highly variable and dependent on the natural origin of the fat (Christie 1989:10). It is thus necessary to know the composition of fatty acids and their distribution on the glycerol backbone to determine the origin of TAGs. TAGs are affected by various degradative processes such as chemical or enzymatic hydrolysis. Hydrolysis of TAGs results in the breakage of the bonds between the glycerol backbone and the fatty acids, leading to diacylglycerides (DAGs), monoglycerides (MAGs) and free fatty acids. While TAGs, DAGs and MAGs may be preserved in some

archaeological contexts (e.g. Regert et al. 1998; Dudd 1999), they rarely survive and usually hydrolyse completely to their fatty acid constituents.

3.1.1.5. Waxes

Waxes are esters of long-chain fatty acids linked to long-chain alcohols via an ester bond (Christie 1989: 12; Wade 2013: 1202). They occur widely in nature and serve several purposes in plants, animals and microbes (Wade 2013). A variety of alcohols and constituent fatty acids are linked to various natural waxes origins: animal, plant or microbial (Christie 1989). For example, ketones are restricted to higher plants and bacteria, whilst terpenoids are found widely in plants and animals and aldehydes are mainly found in higher plants. Wax esters are susceptible to hydrolysis, releasing fatty acids and alcohols which compose them. In archaeological contexts, waxes are often distinguished on the basis of the presence of long-chain *n*-alkanes, which may be indicative of leafy plants or apicultural origin. For example, beeswax has a characteristic odd- carbon numbered *n*alkanes (C_{21} -C₃₃), even-numbered free fatty acids (C_{22} -C₃₀) and long-chain palmitate esters in the carbon range $(C_{40}-C_{52})$ (Frith et al. 2004). The brief overview of different types of lipids provides necessary context for the organic compounds discussed later in this thesis.

3.2. Preservation and degradation of lipids

Variations in temperature and precipitation affect the survival of all organic matter (Pollard et al. 2007), and specific conditions of the depositional environment may facilitate or reduce the preservation potential of organic compounds (Eglington et al. 1991; Evershed 2008a). Rodents, insects, fungi, and bacteria can all affect the survival of all buried organic matter, but microbial degradation is accelerated in most depositional environments through fluctuations in temperature, moisture, and redox conditions (Eglington et al. 1991; Evershed 2008a; Gregg 2009). Thus, both burial conditions coupled with the physical and structural characteristics of different lipids influence their preservation potential. For example, the most common compounds to survive in archaeological pottery are the most non-polar and hydrophobic lipid classes in animal fats and vegetable oils which are $C_{16:0}$ and C18:0 saturated fatty acids (Evershed 2008a; Gregg 2009). Certain classes of lipids are more susceptible to degradation than others; thus, survival of TAGs, DAGs and MAGs, short-chain fatty acids and alcohols is strongly dependent on depositional environment and have been recovered from archaeological pottery only in certain contexts (Regert et al. 1997; Dudd 1999; Stern 2000; Copley et al. 2001; Copley 2002). This section will provide

a short summary of the transformative processes affecting certain classes of lipids during diagenesis generated by a combination of biological and chemical factors.

3.2.1. Lipid diagenesis

Our understanding of lipid diagenesis in ceramics comes primarily from foundational laboratory decay experiments and studies on ethnographic pottery samples (Charters et al.1993, 1997; Evershed et al. 1995; Dudd 1999; Aillaud 2002). For example, experiments involving a range of fatty acids and other aliphatic lipids dosed into replica ceramics have allowed degradation in oxic versus anoxic environments to be investigated (Evershed et al. 1995; Charters et al. 1997; Dudd 1999; Aillaud 2002; Evershed 2008b). The results showed unequivocally that there is little fatty acid diagenesis under anoxic conditions, while little or no lipid remained after only a few weeks of microbial degradation under oxic conditions (Evershed et al. 1992; Dudd et al. 1998; Evershed 2008b). While our knowledge about degradation processes is incomplete, it is well-established that the two main chemical processes that result in the diagenesis of lipids are oxidation (reactions in the presence of oxygen) and hydrolysis (breakage of bonds in the presence of water).

Of the pathways of oxidation (α -, β -, and ω-), β -oxidation is the most prevalent (Pollard and Heron 2008: 394) This is a biological mechanism of fatty acids metabolism by enzymatic action which occurs in aerobic conditions (Evershed et al. 1992). This mechanism affects all fatty acids, whether linear or branched, particularly leading to a decrease in chain length (Aillaud 2002: 11), although saturated fatty acids are less sensitive than unsaturated fatty acids. Polyunsaturated fatty acids oxidise much faster than their unsaturated or monosaturated equivalents, and longer-chain molecules oxidise slightly faster than shorter compounds (Eglington and Logan 1991; Eerkens 2005). The oxidation of unsaturated fatty acids, either by atmospheric oxygen or enzymatic processes, can lead to the formation of many products and their isomers, such as α , ω-dicarboxylic acids (diacids) and dihydroxy fatty acids (Passi et al. 1993; Regert et al. 1998; Aillaud 2001; Hansel et al. 2004;Frankel 2005; Hansel and Evershed 2009). As distinctive oxidation products, the chain length of a diacid and the position of the diol on the dihydroxy acid can reflect the position of the double bond on its original unsaturated fatty acid (Regert et al 1998; Dudd 1999). For example, 9- and 10-hydroxyoctadecanoic acids and C⁹ diacid are usually derived from the oxidation of $C_{18:1}$ (Figure 3.3). These components are thought to be formed by extensive oxidation of lipids in commodities during processing in vessels. Only those components which became bound within the matrix of the pottery (e.g. through

chemical bonding to the clay matrix) are found preserved since oxidised moieties are unlikely to survive as free lipids (Regert et al. 1998; Dudd 1999). Thus, certain extraction methods such as the acidified methanol protocol, have demonstrated an increased release of diacids and dihydroxyacids from the 'bound' lipid fractions (Correa-Ascencio and Evershed 2014).

Mechanisms of hydrolysis affect ester bonds of molecules which can occur either via enzymatic process or in the presence of water (Evershed et al. 1991; Dudd et al. 1998). Variations in water solubility of different lipid species also means that some compounds are more likely to experience hydrolysis than others. For example, diagnostic TAGs or wax esters rarely survive in archaeological pottery as they are often hydrolysed to molecules that they are composed of (fatty acids and linear alcohols/glycerol) (Dudd et al. 1998:1481). Hydrolysis products do not survive very often (Dudd et al. 1998), but they have been identified in ethnographic pottery (Evershed et al. 1997; Dudd 1999). Hydrolysis may also be catalysed by heating; thus, the hydrolysis of TAGs and wax esters may take place during the repeated heating of ceramic containers (Evershed 2008a).

Figure 3.3: Structures of various fatty acid oxidation products. A) C_9 α, ω-dicarboxylic acid or azelaic acid, which when present in archaeological contexts suggests the prior existence of moieties with unsaturation mainly at position 9 (most probably a $C_{18:1}$ fatty acid), however unsaturated fatty acids with double bonds at other positions may have also existed, b) 9-hydroxyoctadecenoic acid, c) 9, 10-dihydroxyoctadecanoic acid and (d) α -hydroxydodecanoic acid, which are formed by various oxidation processed (after Regert et al. 1998).

3.2.2. Burial conditions

While hydrolysis and oxidation result in the degradation of lipids, various biological and chemical conditions including pH levels, level of microbial activity, fluctuating water conditions and temperature changes strongly influence these processes (Eglington et al. 1991). It has been suggested that 99% of extractable lipid is lost through microbial action; and most degradation takes place in the first year, or first weeks of burial (Dudd et al. 1998; Aillaud 2002: 61-62); the surviving lipids protected due to their adsorption into the clay fabric microstructure of vessels (Evershed et al. 1995; Evershed 2008a; Budja 2014). Soil pH is known to affect lipid preservation. Several authors suggest that acidic soils are considered the most favourable environments for the survival of lipids after burial (Evershed 1993; Evershed 2008b; Gregg and Slater 2010; Debono Spiteri 2012; Debono Spiter et al. 2016). Degradation experiments suggest that pH levels between 8 and 5 are optimum for the preservation of organic matter (DeLaune et al. 1981; Debono Spiteri 2012). However, microorganisms responsible for hydrolysis are active in neutral soils (Aillaud 2001: 145), and acidic soils can lead to the rapid hydrolysis of ester linkages in triacylglycerides (Evershed 1990; Eglinton and Logan 1991). Meanwhile, calcareous soils with high pH levels can lead to the formation of water-soluble salts of free fatty acids, which would be susceptible to leaching from potsherds via percolating groundwater (Copley et al. 2005a; Cramp 2008). These reactions can further be catalysed by the presence of metal ions such as iron, manganese or magnesium (Aillaud 2001: 145). Thus, although the relationship between soil pH and lipid preservation in pottery is not straightforward, acidic soils appear to have better preservation potential than basic soils.

Apart from pH conditions, alternating wetting and drying conditions affect microbial activity, and may also result in the leaching of more soluble lipids by percolating groundwater moving through the stratigraphic profile (Dudd et al. 1998; Evershed 2008a). Thus, it is suggested that areas where seasons of high rainfall are followed by hot, dry periods might be detrimental to the survival of organic residues (Cramp 2008; Evershed 2008a). As most of the study sites in this thesis are located in regions with alternating wetdry conditions, experiencing both heavy monsoon rains and winter rains interspersed with hot, dry periods, it was important to test the extent of the preservation of lipids in pottery (see Chapter Six).

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Figure 3.4: Map marking pH of soils and pre- and protohistoric sites in Europe from where pottery has been analysed for residue analysis. Preservation of lipids in ceramics is indicated with percentage of ceramics with lipid yields below and above 5 μg/g out of analysed assemblage. A: Northern Europe, B: Central Europe, C: Southern Europe. pH map made from data available from scientific service of the European Commission [\(http://esdac.jrc.ec.europa.eu/content/soil-ph](http://esdac.jrc.ec.europa.eu/content/soil-ph-europe)[europe\)](http://esdac.jrc.ec.europa.eu/content/soil-ph-europe) (reproduced from Drieu 2017: 126).

Certain physical and chemical environments unquestionably enhance the survival of lipids in pottery. Pottery found in environments that are frozen, or very arid, have extreme waterlogging, or anaerobic conditions demonstrate remarkable preservation of a range of lipid species (e.g. Regert et al. 1998; Malainey et al. 1999; Colombini et al. 2005; Copley et al. 2005a; Spangenberg et al. 2006; Stern et al. 2008). However, these are exceptional cases. Although some assert that adsorbed organic residues survive in >80% of domestic cooking pottery assemblages worldwide (Evershed 2008a: 904; Brown and Brown 2011:194), other investigations demonstrate that lipids appear to be less well preserved in the Mediterranean and Middle East than in northern Europe (Gregg 2009; Drieu 2017), and that lipid residue recovery is variable and unpredictable. The mapping of the preservation of lipids within pottery across Europe based on soil pH (Drieu 2017: 126; Figure 3.4) reveals that a comparison of lipid preservation in archaeological pottery across different

geographies is challenging as there are several factors influencing the preservation of adsorbed residues, and micro-environments of exceptional lipid preservation may exist within regions with poor lipid preservation.

3.2.3. Vessel characteristics and use

Manufacturing characteristics of the vessel and its usage in antiquity also affect preservation of organic remains. For example, vessel fabrication such as the size and shape of ceramic pores, firing conditions and surface treatments will impact vessel porosity and the degree of absorption of lipids (Evershed et al. 1995, Raven et al. 1997; Debono Spiteri 2012; Correa-Ascencio and Evershed 2014; Drieu 2017). The use of the vessel in antiquity will also influence the preservation and composition of organic matter.

3.2.3.1. Vessel porosity and fabric

Several researchers have highlighted the complexity of interactions between lipids and the clayey matrix of ceramics (Evershed 2008a, Debono Spiteri 2012; Drieu 2017; Drieu et al. 2019). Clays are an active matrix due to their structure that comprises of sheets of aluminosilicates layered between water and cations. Firing the clay creates a collapsed, ridged porous unit (Raven et al. 1997), with pores of molecular dimensions that trap organic compounds and possible reaction products. Thus, vessels with high porosity (with large pore-sizes) are likely to adsorb a large amount of organic matter. However, experimental research suggests that ceramics with pore sizes between 0.5 to 3 μm diameter appears to be more favourable compared to larger pores (greater than $4 \mu m$), as the latter may be easily accessible to microorganisms (Drieu 2017). The porosity of terracotta is reported to be between 20-25 μm (Rice 1987: 106), which facilitates adsorption of organic products, but may not be conducive to their preservation against microbial action.

It is also likely that functional groups of lipid molecules influence adsorption properties (Matlova et al. 2017), as non-functional molecules like alkanes are only weakly bound to the ceramic matrix (Craig et al. 2004), while stronger interactions are likely to exist between carboxylic acids and metal cations within the ceramic matrix (Craig et al. 2004; Correa-Ascencio and Evershed 2014; Matlova et al. 2017) Additionally, when metal oxides and salts such as calcium carbonate, iron oxide or magnesium oxide within the ceramic matrix are heated, they catalyse fatty acid ketonic decarboxylation, producing long mid-chain ketones derived from precursor fatty acids (Evershed et al. 1995; Raven et al. 1997). These examples highlight how the relationship between pore size and lipid

preservation is complex and the interaction of lipids and the vessel matrix is dependent on different factors.

3.2.3.2. Vessel manufacture

Organic matter may be incorporated into the original clay as temper before firing (Orton and Hughes 2013), but it is well-established that lipids do not survive exposure to temperatures ≥600 °C, and thus, contamination from lipid signatures of organic temper within pots is unlikely to be a concern during lipid analysis of well-fired pottery (Heron et al. 1991; Drieu 2017). However, the porosity of the fabric of vessels does influence the preservation of lipids, and high levels of small pores are considered generally favourable for lipid preservation (Drieu et al. 2019). Questions of surface treatment and the addition of organic sealants at the final stage of vessel manufacture have also been raised by many researchers (Charters et al. 1993; Heron and Evershed 1993; Diallo et al. 1995; Copley et al. 2005a; Craig et al. 2005). Plant adhesive applied to the surfaces of ceramics, such as resins of birch pitch and pine have been identified by chemical analysis (Urem-Kotsou et al. 2002; Regert et al. 2003; Colombini et al. 2005; Reber and Hart 2008; Stern et al. 2008), which were possibly used to seal the vessels. Similarly, there are ethnographic examples that describe potters dipping vessels in substances like milk or lining with plants after production and before cooking to reduce the porosity of the vessel and make it usable for greater retention of liquids (Arnold 1985: 140). As archaeological parallels for this might exist, it is important to analyse and compare lipids signatures in both the exterior and interior surfaces of vessels in order to confirm the use of organic sealants.

3.2.3.3. Vessel-use

Lipids, proteins and carbohydrates are liberated from foodstuffs during the boiling, roasting or cooking of food and adsorbed by the ceramic fabric. Researchers hypothesise that adsorbed residues contain compounds from the entire life-use of a vessel, representing an integrated signature as opposed to its first or last use (Evershed 2008a; Budja 2014). Thus, the use-history of vessel will affect not only the type of lipids obtained, but also lipid recovery. The frequent use of a vessel for either processing or storing fatty or oily products will lead to a higher chance of lipids being found. Additionally, vessels in which commodities are processed, such as cooking pots, often contain higher concentrations of lipid rather than those used for serving or dry storage (Correa-Ascencio and Evershed 2014) as the processing of foodstuff usually involves the addition of water, prolonged heating and/or mechanical action, which helps transfer the fats in liquid form into the

ceramic walls (Correa-Ascencio and Evershed 2014). Many chemical reactions may be catalysed by inorganic elements, especially by ions of salts and metals either present in the ceramic matrix or introduced as components of commodities processed in vessels (Evershed et al. 1995; Raven et al. 1997; Evershed 2008a). This is also influenced by cooking temperatures. For example, the creation of ketones (especially K_{33}) by interaction with metal salts or oxides in the ceramic matrix occurs when animal fats are heated at temperatures above 300 °C and are used as evidence of repeated heating of a vessel (Raven et al. 1997). Similarly, the presence of long-chain ω-(o-alkylphenyl) alkanoic acids in vessels suggests the protracted heating of products with significant amounts of polyunsaturated fatty acids (i.e. marine or freshwater fats and oils) at temperatures around 270°C (Evershed 2008a; Cramp et al. 2014; Lucquin et al. 2016a, 2016b). Together with dihydroxy acids and isoprenoid acids, the presence of these compounds provides a sensitive means of characterising the processing of marine products in ceramic vessels (Cramp et al. 2014).

The processing of two or more materials in a vessel simultaneously may also affect levels of lipid recovery by archaeologists. For example, recent cooking experiments suggest that the cooking of cereals in vessels liberates only small quantities of lipid into the ceramic matrix, however, the processing of both meat and cereals at the same time enables the transfer of cereal lipids such as alkylresorcinols into the vessel matrix (Hamman and Cramp 2018). Similarly, cooking experiments involving the cooking of lamb meat and brassica leaves led to considerably higher levels of adsorbed lipids leading to high lipid concentrations in vessels (Evershed 2008a). Thus, the absence of lipid in a vessel may be indicative of its use for non-fatty products, or less frequent use (Regert 2007; Šoberl et al 2014). For example, Drieu (2017) demonstrates that vessels with very low concentrations of lipid (0-7 μg/g) probably contained very little fatty products, as analysis of carbonised surface residues from the same vessels also revealed low quantities of lipid whereas other surface residues contained high $(100+\mu g/g)$ lipid concentrations.

Finally, vessel form may be indicative of vessel use, which would influence the mechanism of accumulation of organic residues during use (Charters et al. 1993). As the transfer of lipids is dependent upon the methods of food processing, i.e., boiling, roasting, or frying, certain locations of the sherd on the vessel (rim, body or base) may contain more concentrations of lipid than others (Charters et al.1993). Experimental evidence suggests that the boiling of products in vessels would lead to lipids accumulating at the rims of vessels (Charter et al. 1993), which has influenced sampling strategies in this thesis.

3.3. Contamination

Lipid residues present in archaeological vessels do not only reflect the use-history of vessels but also their abandonment, recovery, and post-excavation treatment. As these factors strongly affect archaeological interpretation, it is important to control for all possible contamination of extraneous lipids into archaeological vessels that are not related to its use-history. These include the influence of the burial environment, recovery conditions, processing and handling techniques, and storage environments. Full knowledge of these conditions enables more careful assignment of organic products to their potential role in the life-history of the vessel. Most archaeological literature does not highlight this explicitly enough. In this thesis, a small selection of soil samples adhering to vessel fragments or directly from contexts were collected for comparative purposes, and possible sources of contamination have been discussed in lipid extracts (Chapter Six).

3.3.1. Migration of soil lipids

The effects of post-depositional intrusion of soil organic matter on organic residues retained in the walls of pottery vessels was a key concern in early studies of organic residue analysis. As lipids are ubiquitous in soils, bacteria, algae, fungi, plants and animals, this concern was well-founded. A few key studies initially ruled out the effects of intrusion of soil organic matter within pottery residues. For example, Heron and others (1991) assessed the possible migration of lipids from the soil on the organic residues preserved in buried potsherds from West Cotton, Northamptonshire, U.K. It was observed that the composition of the soil and sherd lipid exhibited marked variation, with soil lipid content falling within a more closely defined range (30-510 μ g/g) and the lipid content of the sherds exhibiting much greater variation (60-4800 μ g/g) (Heron et al. 1991: 648). Additionally, soil lipids were found to be of relatively consistent composition, made up of a complex mixture of lipids originating from plant, animal and microbial detritus; whereas sherd extracts were generally of simpler and more variable composition (Heron et al. 1991; Evershed 1993). The major compounds present in every soil sample investigated were long-chain free fatty acids, particularly pentacosanoic acid $(C_{25:0})$ which is absent in the vast majority of foodstuffs and only present in the cuticular leaf waxes of some plants as a minor trace component (Heron et al. 1991). These results suggested that negligible migration of soil lipids occurs during burial. Several others (Craig et al. 2003, 2005; Evershed 2008a; Correa-Ascencio and Evershed 2014; Drieu 2017) have supported this finding.

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However, it is important to be cautious and account for potential sources of contamination, and samples must be assessed on a case-by-case basis. In order to account for potential contamination, the exterior surface of the sherd is removed via drilling (e.g. Heron et al. 1991), and samples with lipid concentrations below 5 μ g/g are usually excluded from analysis (Heron et al. 1991; Evershed et al. 1999; 2008a). The interaction between soil organic matter, soil microorganisms and soil structure and properties is extraordinarily complex, and many aspects of the diagenesis of organic residues in archaeological contexts are not fully understood (Haslam 2004). Differences in decomposition factors and rates between artefact surfaces and in sediments have also not yet been thoroughly investigated (Haslam 2004). This makes it important to test for the possible effects of migration and microbial activity by comparing the organic matter content of the buried sherd and its burial context.

3.3.2. Other sources of contamination

Apart from exposure to lipids in sediment, potsherds may adsorb additional compounds, or lose organic compounds after their recovery. Most potsherds are washed and scrubbed after excavation. The washing and scrubbing of pottery may result in the loss of less hydrophobic compounds. Excavators may also introduce lipids from their fingertips onto the surface of the vessel, write on potsherds, use adhesives to stick potsherds together, and store them in plastic bags, exposing the potsherds to a range of synthetic compounds and modern sources of contamination. Phthalates are often found within lipid extracts of ancient pottery. Phthalates are a group of colourless, odourless liquids which are most often used as plasticisers (or softeners). A plasticiser is a substance added to plastic that makes it more flexible, resilient, and easier to handle. They are produced by the simple reaction of alcohols with an acid (e.g. phthalic anhydride) (Meeks et al. 2009). These compounds are components of plastics, safety glasses, rubber coating agents, moulding powders, insect repellents and pesticides. Although phthalates are easily recognisable via their mass spectra and can be excluded from analysis, a high degree of plastic contamination may interfere with the recognition and interpretation of other organic molecules within lipids extracts.

Finally, contaminants may also be introduced into at any time during the sample preparation process in the laboratory (Stern et al. 2000). Although extraction protocols are carefully designed to minimise laboratory contamination and cross-contamination (see Chapter Five), with the use of nitrile gloves during the handling of the sample and

extraction of lipids, the repeated cleaning of drill bits, syringes and needles with dichloromethane or hexane between transfer of samples, etc.), it is always possible to introduce organic compounds from the laboratory environment into the sample. For this reason, method blanks are also processed during extraction of samples, making it possible to identify potential introduced organic compounds during analysis, if any, and exclude them from analysis.

3.4. Extraction methods

Extraction involves the separation of relatively non-volatile analytes in analysis of the organic fraction from solid samples (food, animal and plant tissues, ceramics, sediments). The extraction of lipids from archaeological pottery operates on a basic principle about the solubility of lipids in non-polar organic solvents. Ad-hoc experimentation and the use of a variety of extraction methods, especially soxhlet extraction, was common in the early days of organic residue analysis in archaeology (e.g. Condamin et al. 1976; Shackley 1982; Bourgeois and Gouin 1990; Oudemans and Boon 1991). Later, researchers suggested using a microwave-assisted extraction (Gregg et al. 2009; Gregg and Slater 2010) and automatic shaker extraction (Zagorevski and Loughmiller-Newman 2012). Microwave radiation can heat the whole sample in a short time and accelerate extraction, cutting solvent consumption and extraction time (Gregg et al. 2009). However, recently ultrasonication and other extraction protocols have found more favour among researchers (e.g. Evershed et al. 1990; Craig et al. 2013; Correa Ascenscio and Evershed 2014) The two commonly used extraction methods are detailed below. Details of the extraction of lipids from archaeological pottery and instrumental analyses used in this thesis are provided in Chapter Five.

3.4.1. Solvent extraction

The solvent extraction method was established in 1990s, using chloroform/methanol and trimethylsilylation derivatisation (Evershed et al. 1990). Recently, this method has been adapted to use dichloromethane/methanol instead of chloroform (e.g. Lucquin et al 2016; Oras et al 2018). This method involves the repeated washing of the powdered sherd sample with the prepared solvent mixture through ultrasonication. The solvent extract is then evaporated to obtain a total lipid extract (TLE). Subsequently, TMS ether and ester derivatives, or methyl ester derivatives are prepared by silylation or methylation to

improve the separation, resolution and detection of peaks via analysis with Gas Chromatography-Mass Spectrometry.

This method enables the detection of a range of lipids including fatty acids, longchain ketones, wax ester, *n*-alkanols, *n*-alkanes, acylglycerols (MAGs, DAGs and TAGs), terpenes and sterols, however, it is unable to extract polar lipids that have stronger interaction with the ceramic matrix, such as diacids and hydroxyacids (Correa Ascencio and Evershed 2014). Thus, often this method does not extract appreciable lipid yields from pottery recovered from burial contexts that are not amenable to good preservation of organic matter. For example, Gregg (2009; Gregg and Slater 2010) was unable to extract appreciable quantities (above 5 μ g/g) of lipid from Near Eastern vessels using this method. The method is also time-consuming as derivatisation is necessary after extraction.

3.4.2. Acidified methanol extraction

The second method was developed in order to improve the efficiency and effectiveness of the solvent method. The use of acidified methanol demonstrated enhanced recovery of organic residues from vessels that did not yield lipid concentrations above 5 μg /g using the solvent method.

The use of a reagant such as concentrated sulphuric acid (95%) enables the cleavage of chemical bonds that may exist between lipid species and the clay fabric of the vessels. Correa-Ascencio and Evershed (2014: 1334) suggest that aluminosilicates within the ceramic fabric can promote lipid polymerisation reactions (the joining of small molecules to form long molecules) and strong intermolecular interactions, especially with molecules with one or more polar functional groups (e.g. hydroxyl and carboxyl). The number and strength of intermolecular interactions will affect their extraction with organic solvents. Thus, the use of an acid solution removes both polar and non-polar lipids from the surface and deeper layers of the ceramic matrix (Correa-Ascencio and Evershed 2014). The extraction also includes the derivatisation step with the simultaneous production of methyl esters of fatty acids, thereby making the extraction process more efficient (Correa-Ascencio and Evershed 2014).

The acidified methanol extraction process enables the recovery of long-chain fatty acids and usually reveals a wide range of oxidation products, such as series of dicarboxylic acids, dihydroxy $C_{16:0}$ and $C_{18:0}$ fatty acids and ketoacids (Aillaud 2002; Correa-Ascencio and Evershed 2014). Compounds such as terpenes and sterols are also revealed (Correa-Ascencio and Evershed 2014). The position of the double bond within the original

molecule may still be revealed by oxidation products, although caution must be exercised in interpretation. Long-chain fatty acids have also been observed in pottery vessels containing degraded animal fats when extracted with acidified methanol (Correa-Ascencio and Evershed 2014), suggesting that they may arise through routing from the plant diet into the carcass and dairy fats (Whelton et al. 2018).

Overall, the acidified methanol extraction process has improved the efficiency and feasibility of lipid extraction. This is especially when analysing vessels from regions where lipid preservation may be low, as traditional solvent extraction may fail to yield any lipids, resulting in the waste of the sample and money. However, this extraction process also affects the structure of larger and more complex molecules such as triacylglyerols and wax esters (Correa-Ascencio and Evershed 2014). All these compounds are hydrolysed to their constituent acids and alcohols, resulting in loss of compositional information (Correa-Ascencio and Evershed 2014). However, other lipid classes such as *n*-alkanes, *n*-alkanols and ketones remain unaltered (Correa-Ascencio and Evershed 2014). This method has thus enabled the investigation of lipids in pottery from areas reporting poor lipid preservation; such as the Mediterranean (Barcons 2015); southern France (Drieu 2017); parts of Northern Africa (Dunne et al. 2017, 2018), western India (García-Granero et al. in prep.) and in this thesis, northwest India.

3.5. Instrumental analyses

The analyses of amorphous archaeological organic residues have involved the use of multiple techniques including Liquid Chromatography-Mass Spectrometry (LC-MS), High-Performance Liquid Chromatography (HPLC), and Thin-layer Chromatography (TLC) (e.g. Passi et al.1981; Hurst et al. 1987; Charters et al. 1993; Heron and Evershed 1993). More recently, the use of Gas Chromatography (GC) and Gas Chromatography-Mass Spectrometry (GC-MS) have become the common instrumental analyses for characterising organic residues in pottery. Lately these have been coupled with the use of Gas-Chromatography-combustion-Isotope Ratio Mass Spectrometry (GC-c-IRMS), allowing for increased scope of distinction between the potential source(s) of the archaeological residue.

3.5.1. GC and GC-MS

GC enables the separation of compounds within heterogenous complex mixtures based on their boiling point and polarity. GC is particularly well-suited for volatile compounds and

for the analysis of complex, degraded molecular mixtures. Detection is often carried out via flame ionization detection (FID) where molecules are identified by comparing their retention times (i.e. time elapsed between the injection of the sample and detection of the molecule after its separation) with those of standard molecules and according to the elution order of homologous series (Evershed et al. 1991). The response from the detector is amplified by a data system and plotted against time, giving rise to a 'chromatogram' (see Fig 3.5). The data system can also be used in association with various softwares to perform different quantitative and qualitative operations on the chromatogram, which assist with sample identification and quantification. Compounds (analytes and sample components) that are retained elute as approximately 'Gaussian' (following the shape of a 'normal' distribution) shaped peaks in the chromatogram. The retention time of a compound will always be the same under identical chromatographic conditions. The chromatographic peak height or peak area is related to the quantity of analyte. For determination of the actual amount of the compound, the area or height is compared against standards of known concentration.

Samples analysed by GC must be volatile under conditions in the GC, i.e., they must have significant vapour pressure below 250° C. Within organic residue analysis, derivatisation of molecules is conducted during sample preparation to increase volatility, such as methylation (e.g. Condamin et al. 1976) or trimethylsilylation (Evershed et al. 1990) to derivatise -OH groups with CH³ and SiCH³ respectively, reducing the polarity of molecules, and improving the resolution of the peaks during chromatographic analysis. Thus, often the molecules identified via GC-MS are methylated or silylated compounds. For example, FAMES (Fatty Acid Methyl Esters) are identified and described in the literature instead of unmethylated fatty acids. Silylation may be used after methylation for the identification of *n*-alcohols (Correa-Ascencio and Evershed 2014). The fast, efficient and highly sensitive nature of GC, along with its ability to couple to MS makes it a good choice for the analysis of volatile samples. MS is an instrument that measures the masses of individual molecules that have converted to ions, i.e., molecules that are electrically charged.

Figure 3.5: Representation of a chromatogram (after Christie 2018).

Figure 3.6: Mass spectrum of methyl palmitate. The height of the bar is related to the intensity of the signal of the ions against mass-to-charge (*m/z*).The molecular ion at the end of the high mass range at m/z is 270, while the ion at m/z 74 is diagnostic for a methyl ester and especially abundant with saturated fatty acids (after Christie 2018).

GC-MS enables the separation, identification, and quantification of lipid species in complex mixtures. MS alone is a highly sensitive technique for both separation and detection, but the presence of complex mixtures in archaeological pottery residues may contain several compounds with similar molar mass and fragmentation pattern, thus requiring an additional separation process. Once separated components leave the GC

column, they pass through a transfer line into the ion source of a mass spectrometer (MS), where the sample is vaporised and ionised (Rouessac and Rouessac 2002: 290). The ions are filtered according to their atomic mass to charge ratio (m/z) (Pollard et al. 2007), and the detector measures electrical charge (Rouessac and Rouessac 2002: 290; Khoury et al. 2018). Thus, for each sample, GC-MS analyses provide two sets of data: (i) a chromatogram on which each peak corresponds to a molecular constituent (Figure 3.5), and (ii) a series of mass spectra for each molecular constituent (Figure 3.6). It is thus possible to quantify all the different constituents present in the sample and to characterize the structure of each by MS (Regert et al. 2003: 1623). The abundance of a particular compound within a total lipid extract is calculated by comparing the peak area of the compound with that of the internal standard, whose amount is known. Identification is aided by comparison with database libraries available via NIST (National Institute of Standards and Technology), or through selective-ion-monitoring (SIM) mode (Khoury et al. 2018). The GC-MS conditions for samples used in this thesis are described in Chapter Five.

3.5.2 GC-c-IRMS

This technique determines the carbon isotope ratio (δ^{13} C) of single fatty acids and other lipid molecules within an archaeological lipid extract that are isolated after their separation and identification via GC-MS (Evershed et al. 1994). The introduction of GC-c-IRMS in the early 1990s transformed the field of organic residue analysis as it increased the scope of differentiating between terrestrial animal products (adipose vs. dairy), marine products, and the input of plant lipids $(C_3$ vs C_4 plants). The combined approach of molecular and isotopic analysis enhances interpretive power.

3.5.2.1. Background to isotopic analysis

All atoms of an element have the same number of positively charged particles (protons) in their nucleus, but the number of particles with no charge (neutrons) may vary. Atoms of the same element with different numbers of neutrons are classified as isotopes. Different isotopes have different atomic masses, which means that while their chemical properties are almost identical, their physical properties vary (Pollard et al. 2007). Differences in physical properties of heavier and lighter isotopes lead to isotopic fractionation during various physical, chemical and biological processes, which result in different isotopic ratios to the original reactants (Van de Merwe 1982; DeNiro 1987).

Isotopic measurements are reported as a ratio of the heavier to the lighter isotope. This ratio is measured with reference to an element-specific international standard, with the isotopic deviation from the standard expressed using the δ notation as illustrated in the equation 3.1. Here, X denotes the heavy isotope and R is the ratio of the heavier to lighter isotope. The measurements are generally expressed in parts per mil (‰).

$$
\delta X = \frac{R_{sample} - R_{standard}}{R_{standard}} \, X \, 1000 \tag{3.1}
$$

This thesis specifically focuses on stable isotopes of carbon of specific compounds within lipid extracts, particularly C_{16:0} and C_{18:0} fatty acids, however the δ^{13} C values of other compounds are also used (e.g. Copley et al. 2001; Dunne et al. 2017). Carbon stable isotope ratios are expressed as delta values (e.g. δ^{13} C) on the VPDB (Vienna Pee Dee Belemnite) international scale (Coplen et al. 2006; Coplen 2011). The precision of δ^{13} C values is typically $\pm 0.3\%$ (Regert 2011).

3.5.2.2. Compound-specific isotopic analysis

The use of compound-specific isotopic analysis in organic lipid residue analysis uses basic principles in animal digestive metabolism and plant photosynthesis to distinguish between potential food sources in vessels. There are also differences in how lipids are biosynthesised and routed to tissues of different animals (Christie 1981; Dudd et al. 1998; Dudd 1999)

In plants, the stable isotopic values of carbon in tissues are primarily related to the mechanisms of photosynthesis that enable metabolism of $CO₂$ (Smith and Epstein 1971). These differ according to the type of plant and the ecosystem to which they are adapted: C3, C⁴ and Crassulacean Acid Metabolism (CAM) (O'Leary 1981). About 85% of plant species in the world are C_3 plants, including all trees, and crops like wheat, barley, and rice (O'Leary 1981). C_4 plants are better adapted to arid climates, such as crops like millets and maize. Each photosynthetic pathway involves very different amounts of fractionation and responds differently to environmental and physiological controls (O'Leary 1981; DeNiro 1987; Farquhar et al. 1989), allowing for distinctions to be made based on their isotopic values. For example, δ^{13} C values for C₃ plants are around -27.1‰ $\pm 2\%$; C₄ plants have values of -13.1‰ $\pm 1.2\%$; and marine values fall around -20‰ (Regert 2011).
Isotopic fractionation also occurs in consumers. As carbon isotopic fractionation between the tissues of the consumer and its diet is very small, from 1% to 2% (DeNiro and Epstein 1978), δ^{13} C values of animals are often directly linked to those of the plants/herbivores consumed (Regert 2011). Fundamental differences between the digestive physiology of ruminants (cattle, sheep/ goat) and non-ruminant animals such as pigs also exist, which enable distinction between the δ^{13} C values of their fats. For example, while the primary source of $C_{16:0}$ and $C_{18:0}$ is all animals is through dietary carbohydrates, in ruminant dairy products, C16:0 is synthesised in the mammary gland *de novo* from acetate (derived mainly from dietary carbohydrate) while $C_{18:0}$ is derived from dietary fatty acids and other sources in the rumen (Dudd et al. 1998). In ruminant adipose fats, the $C_{16:0}$ and $C_{18:0}$ fatty acids are derived directly from the rumen (Copley et al. 2003). As the mammary gland cannot biosynthesize $C_{18:0}$ directly, the routing of dietary fatty acids of milk during lactation gives rise to more negative $\delta^{13}C$ values for the C_{18:0} fatty acid of milk compared to the adipose of animals feeding on the same diet (Dudd et al. 1998; Copley et al. 2003). Meanwhile, non-ruminants such as pigs use carbon from both acetate and glucose to produce C_{16:0} and C_{18:0}, creating more positive δ^{13} C values than those obtained by ruminant adipose fats. These differences in δ^{13} C values of fatty acids and carbohydrate components of forage and fodder in animals' diets make it possible to distinguish between ruminant dairy fats, ruminant adipose fats and non-ruminant adipose fats (Dudd et al. 1998; Copley et al. 2003).

However, isotopic values of animal tissues are influenced both by biosynthetic processes, specific to each species (and each organ or tissue) and are also related to the environment and the type of diet (DeNiro and Epstein 1978; Van Klinken et al. 2000; Boecklen et al. 2011). The δ^{13} C values obtained from archaeological lipids are compared with modern reference values of the δ^{13} C values of C_{16:0} and C_{18:0} fatty acids of known animal or plant origin (Figure 3.7), and corrected for the modification of atmospheric $CO₂$ due to the burning of fossil fuels (Friedli et al.1986; DeNiro 1987; Evershed et al. 1994; Dudd and Evershed 1998; Spangenberg et al. 2006). All these factors must be accounted for during interpretation.

As stable carbon isotopic values are known to be stable even after degradation and transformation of the source fatty acids (Dudd and Evershed 1998; Dudd et al. 1999, Evershed et al. 2002; Steele et al. 2010) they provide means to establish the potential source/s of the archaeological lipid, albeit with the potential of providing a 'mixed' isotopic signal when multiple products have been processed in the same vessel (Copley

2002; Mukherjee et al. 2008; Craig et al. 2011; Regert 2011; Cramp et al. 2014). However, regionally-specific reference animal fats are required for the precise interpretation of archaeological sources in different regions due to environmental differences (Coupley et al. 2003; Mukherjee et al. 2008; Gregg et al. 2009). An effort has been made in the past ten years to add to previously existing references from the United Kingdom (Figure 3.7) (Dudd and Evershed 1998, Dudd 1999; Evershed et al. 1997a; 2002). Isotopic values of modern animal fats now exist from the Middle East (Gregg et al. 2009), Eastern and Northern Africa (Dunne et al. 2012), the Mediterranean (Debono Spiteri 2012) and Central Europe and Asia (Spangenberg 2006, 2008; Outram et al. 2009, 2011). A systematic collection of reference fats from South Asia has not yet occurred, although values for an ethnographic milk pot from Gujarat and milk fat values from cow fed on a mixed C_3-C_4 diet have been reported by Craig and colleagues (Craig et al. 2005).

The difference in the δ^{13} C values of fatty acids is commonly expressed in Δ^{13} C values, where $\Delta^{13}C = (\delta^{13}C_{18:0} - \delta^{13}C_{16:0})$ is plotted against $\delta^{13}C_{16:0}$ (Craig et al. 2003; Copley et al. 2005a, 2005b, 2005c; Evershed et al. 2008; Mukherjee et al. 2008; Regert 2011; Cramp et al. 2014; Whelton et al. 2018) (Figure 3.7). The arithmetic transformation to Δ^{13} C values removes the influence of varying proportions of C₃ and C₄ plants in ruminant forages; seasonal changes in the δ^{13} C values of the plant biochemical components in the animals' diet; as well as other influencing factors such as altitude, latitude, and atmospheric CO² concentrations (Copley et al. 2003; Dunne et al. 2012; Roffet-Salque et al. 2017b). Plotting against $\delta^{13}C_{160}$ reveals any isotopic variation in dietary carbon, indicating whether the animal had more of a C_3 , C_4 or marine diet (Copley et al. 2003; Dunne et al 2012). As the Δ^{13} C proxy has been argued as globally applicable (Dunne et al. 2012), it has been applied in this thesis.

Figure 3.7: Examples of scatterplots of $\delta^{13}C$ values of $C_{16:0}$ and $C_{18:0}$ fatty acids measured from sites from prehistoric pottery from the United Kingdom (a, b, and c). The ellipses represent 1SD confidence ellipses, while curves from the porcine adipose ellipse to the ruminant dairy and adipose ellipses and between the ruminant dairy and adipose ellipses represent theoretical δ^{13} C values for mixture of these fats. Scatterplots d, e and f are examples of Δ^{13} C values against δ^{13} C values of $C_{16:0}$ fatty acid, often used in the absence of modern reference fats (reproduced from Mukherjee et al. 2008).

3.6. Interpretation

After the completion of analyses, the interpretation of the constituents of the organic residue is undertaken. The assignment of a specific source or constituent of a residue based on the presence of a particular biomarker component or mixture of components demands a high degree of rigour (Evershed 2008a: 898). The direct comparison of ancient and contemporary organic products is not straightforward, as archaeological residues are extremely complex mixtures, complicated by effects of decay over archaeological time (possibly in different burial environments) and effects of human intervention (the mixing or alteration of natural products during processing or cooking) (Evershed 1993). Furthermore, certain compounds in natural products may not survive, or may transform

over time, giving rise to altered rather than original chemical structures (Evershed 2008a). A good knowledge of the chemical and biochemical pathways involved in the degradation and transformation of organic compounds is required to make nuanced interpretations of the potential source of the archaeological residue. A variety of different methods enable the identification of specific or general lipids. These are highlighted below.

3.6.1. 'Biomarkers', molecular signatures and proxies

Certain chemical compounds, or a combination of compounds might be unique to a specific product in nature. A compound or mixtures of compounds in an archaeological organic residue must be compared to those present in contemporary plants and animals (known as modern references) (Evershed 1993). A unique chemical 'fingerprint' or signature may enable the determination of the presence or use of a specific organic product in the past. Several types of molecular markers have been defined by bioarchaeologists: (i) 'biomarkers' that unambiguously correspond to native molecules whose association can be linked with natural sources (e.g. theobromine for cacao) (e.g. Zarillo et al. 2018); (ii) anthropogenic transformation markers that are the result of chemical transformations induced by different human activities; (iii) natural degradation markers that are formed by natural decay of the initial biomarkers or transformation markers in the archaeological deposits by chemical or biochemical processes (Evershed et al. 1992; Regert 2007, 2011; Evershed 2008a).

Archaeologists have used the molecular signature concept to determine the origin of a constituent of an organic residue in multiple geographic and archaeological contexts (see Evershed 2008a for detailed summary). Certain compounds of organic products like beeswax, plant resins, fish oils, and other plant organisms have been widely studied and robustly associated as components in ancient archaeological residues. These are summarised in Table 3.2.

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Table 3.2: Examples of molecular signatures, proxies and ratios used to identify different natural products commonly used as foodstuff.

Archaeologists also use the presence of specific compounds to make inferences about the way the product has been processed in antiquity. Within the environmental and plant sciences, the term 'molecular proxy' has come into use to include individual compounds characterising specific biogenic sources and individual compounds acting as a proxy for another compound. For example, the presence of long-chain ketones within pottery lipid extracts is used as a molecular proxy for the continuous heating of plant waxes to temperatures above 300°C (Regert et al. 1998; Evershed 2008a). Other examples of molecular proxies for heat alteration include ω -(o-alkylphenyl) alkanoic acids with 16-22 carbon atoms, which are produced when polyunsaturated fatty acids (PUFAs) are heated to over 270°C, and dihydroxy fatty acids and dicarboxylic acids that are produced by the oxidation of unsaturated fatty acids. The production of short-chain α, ω-dicarboxylic acids (diacids) occurs by oxidative degradation of a carbon-carbon double bond in an unsaturated fatty acid (Regert et al. 1998). Various diacids are known to be formed, and assumptions can be made from the distribution pattern of the homologues (compounds belonging to a series, but differing from each other by a repeating unit), for example, the prominence of the C⁹ diacid implies the presence of compounds with unsaturation at position 9 (most probably $C_{18:1}$ fatty acid) (Regert et al 1998: 2030). Similarly, with dihydroxy acids, the position of the hydroxyl groups added to the carbon atoms bearing the original double-bond record the original position of unsaturation, which in combination of the carbon number of the fatty acids can be related back to the original compound in the vessel (Copley et al. 2003; Colombini et al. 2005; Evershed 2008a). Thus, the presence of diacids in a vessel may be indicative of the presence of plant-based residues or fish, which contain high proportions of unsaturated fatty acids. Examples of such 'molecular proxies' are also included in Table 3.2.

3.6.2. Molecular ratios

Archaeologists and plant scientists also use molecular ratios of sets of compounds to distinguish between sources of products (Jansen and Wiesenberg 2017). Examples include the carbon preference index (CPI), palmitic and stearic acids ratio (P/S ratio), average chain length (ACL) and P_{aa} proxy ratio (Dunne et al 2017). CPI measures the relative abundance of odd over even carbon chain lengths, P/S ratio measures the ratio of the relative abundance of palmitic $(C_{16:0})$ and stearic $(C_{18:0})$ fatty acids, ACL measures the weight-averaged number of carbon atoms of the higher plant C25:0 to C33:0 n-alkanes, and P_{aq} proxy ratio measures the emergent and non-emergent aquatic macrophyte input (Dunne et al. 2017: 4) respectively, in lipid extracts (Table 3.2). In general, these ratios may be indicative of the presence of plant species (for example, CPI values for plant species have strong odd-chain preferences, P/S ratios greater than 4 indicate a plant origin, and ACL values and the Paq proxy ratio are indicative of different plant taxa and aqueous plant species, respectively (Ficken et al 2000; Diefendorf et al. 2011; Dunne et al. 2017). The use of molecular ratios in plant sciences and archaeology is common as the direct comparison of lipid compositions from contemporary biogenic sources is not possible due to degradation, whereas broadly speaking, ratios of compounds may be more stable indicators of biological origin of residues (Eerkens 2005). Archaeologists are known to use not only P/S ratios, but also $C_{18:1}/C_{16:0}$, $C_{16:1}/C_{18:1}$ or $C_{16:0}/C_{14:0}$. (e.g. Malainey et al. 1999), and ratios of odd-chain vs. even-chain fatty acids $((C_{15:0} + C_{17:0})/C_{18:0})$ $C_{16:0}+C_{18:0}/(C_{12:0}+C_{14:0}$ (e.g. Eerkens 2005). Caution must be exercised, however, as while ratios are effective in discriminating against fresh animal fats or plant oils, the differential degradation of fatty acids prevents their use for heavily altered or degraded fats. For example, the ratio $C_{16:0}/C_{18:0}$ cannot be regarded as constant over archaeological time as $C_{16:0}$ is twice as soluble in water at 20 \degree C than $C_{18:0}$ and may be preferentially leached from the residues at all but the driest of burial sites (Steele et al. 2010). Fatty acid ratios are also affected by the solvents used and method employed to extract and analyse organic residues (Steele et al. 2010). As a result, they cannot be considered diagnostic for the degraded fats found in archaeological samples but may be broadly indicative.

3.6.3. Interpretational challenges

The sections above have described a variety of methods by which archaeologists interpret the likely origins of the compounds present in lipid extracts from archaeological vessels. But interpretation of the likely source(s) can be challenging due to several factors. These include complex formation processes, lack of available modern reference fats, and challenges of resolving mixtures of products in vessels.

3.6.3.1. Complex formation processes

Understanding the various formation processes of organic residues can be very complex. Potsherds may adsorb a variety of organic compounds during their life-, depositional- or excavation histories. The porosity and fabric of the vessel may influence lipid adsorption (Drieu 2017). The use-history of the vessel may be varied, and compounds within the pot may represent an aggregate of all or most of the vessel's uses over its lifetime. Upon deposition of the vessel, a variety of factors conducive to diagenesis could affect the

adsorbed residues, including soil pH levels, microbial activity, fluctuating water conditions and temperature changes. Compounds may leach out of the pot, hydrolyse and oxidise, or transform beyond recognition. Some chemical signals may also be more robust than others, for example, the richness of lipid of the source foodstuff and the hydrophobicity of certain molecules will favour their survival. Finally, vessels may adsorb a variety of synthetic or organic compounds once they are excavated and processed by archaeologists, including skin lipids from human contact, phthalates from plastic storage, or other organic compounds from conservation treatment (see Figure 3.8).

Figure 3.8: Inputs, losses and transformation processes affecting the survival and composition of organic residues in archaeological ceramics (reproduced from Roffet-Salque et al. 2017a: 628)

3.6.3.2. Modern reference fats and resolving mixtures

The development of any biomolecular or stable isotope proxy for reconstructing past environments or processes requires measurements of reference collections of organisms from contemporary or past environments of known provenance (Roffet-Salque et al. 2017b). Lipid researchers generally use modern references that are specific to the study region, enabling greater specificity with identifying the potential source(s) of ancient lipids (e.g. Spangenberg et al. 2006; Gregg et al. 2009). However, recent studies have demonstrated the potential pitfalls in creating reference fats from meat or dairy products sourced from animals fed on unknown diets or those exposed to modern practices (Roffet-Salque et al. 2017a). This is because the diet of animals can alter the $\delta^{13}C$ values of fatty acids by affecting pathways of fatty acid synthesis during metabolism (Roffet-Salque et al. 2017b). For example, cattle fed on high starch diets like modern silage produce milk with altered $\delta^{13}C_{18:0}$ values, creating an unreliable proxy for archaeological samples (Roffet-Salque et al. 2017b). Thus, extreme care must be exercised in selecting animals raised on diets isotopically similar to those that would have existed in prehistory (Roffet-Salque et al. 2017b). However, access to animals with known diets, and the export and analysis of modern animal products can be challenging.

Reference fats from different animals and regions have demonstrated that isotopic values can overlap (Evershed et al. 2002, Steele et al. 2010; Craig et al. 2012). Additionally, some demonstrate high variability, for example, the subcutaneous fats of deer (Dudd 1999; Evershed et al. 2002a; Spangenberg et al. 2006; Craig et al. 2012) and freshwater fish (Craig et al. 2007, 2011, 2013; Outram et al. 2009, 2011; Cramp and Evershed 2014; Taché and Craig 2015), creating interpretational challenges. Similarly, although the Δ^{13} C proxy has been used by researchers with limited access to regionallyspecific modern animal fats as it is applicable in regions with both C_3 and C_4 plants (Evershed et al. 2002; Copley et al. 2005a; Evershed et al. 2008; Dunne et al. 2012; Whelton et al. 2018), it is limited in scenarios where animal-based products were mixed with plant-based products in vessels (Hendy et al. 2018). As this was ostensibly was common throughout different regions in prehistory, it is important to develop more strategies to resolve potential mixtures.

Resolving mixtures of products in vessels is one of the major challenges faced by lipid residue studies (Craig et al. 2011; Regert 2011). Researchers have attempted to resolve these issues by creating models that consider the concentration of each fatty acid

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determined from reference samples (Mukherjee et al. 2008; Craig et al. 2011). This makes it possible to isolate mixtures of two products, for example, mixtures of non-ruminant products with ruminant products (e.g. Mukherjee 2008, see Figure 3.7) but resolving mixtures of multiple products remains challenging. Others have used Bayesian mixing models such as FRUITS to determine the proportional contribution of different food sources to a series of different mixed food compositions, using data generated both by simulation and by experiment (Fernandes et al. 2018). However, plant products are often rendered 'invisible' in mixtures where both products were processed in the same vessel due to their low-fat content (Hendy et al. 2018). Plants also have a much higher $C_{16:0}$ to $C_{18:0}$ ratio than animal fats and produce significant deviations in Δ^{13} C values depending on the absolute δ^{13} C values of the end-members (Steele et al. 2010; Hendy et al. 2018: 6).

Figure 3.9: Density distributions of Δ^{13} C values obtained by theoretical mixing of a) dairy and b) ruminant adipose fats with an increasing amount of C_3 plant lipids (Reproduced from Hendy et al. 2018: 6).

In a mixing model that created hypothetical Δ^{13} C values of mixtures of C₃ plants and dairy products from animals grazing on both C_3 and C_4 plants, Hendy and others (Hendy et al. 2018) demonstrated that the Δ^{13} C values generated are the same as those created by ruminant adipose fats. This was so even when relatively small amounts of plant lipid (750g of barley 2.5 wt% lipid) was mixed with dairy products (1 L of raw sheep's milk; 7 wt\%) lipid). Figure 3.9 demonstrates how the increasing contribution of a) C_3 plants versus dairy products in a vessel would generate Δ^{13} C values above -3.3‰, creating a 'false' ruminant adipose fat signal. When plants were b) mixed with ruminant adipose fats, the likelihood of creating a non-ruminant adipose fat signal was increased. This suggests that resolving mixtures of products in vessels, particularly those of plants and animals, is notoriously tricky. Environments with high abundance of C_4 plants and/or marine and freshwater resources add further complexities as they increase the isotopic variability of products available for both humans and animals to consume. In contexts with both C_3 and C_4 plants in the landscape, such as in the Sahara and in Anatolia, and in this thesis, northwest India, it is possible the applicability of the Δ^{13} C proxy, particularly in contexts where animal fats and plant oils were processed in vessels together, is limited. These interpretational complexities are discussed in detail in Section 8.2.4.

In summary, although lipid researchers use a variety of interpretive strategies to understand the possible source(s) processed within a vessel's use-history, there are degrees of uncertainty associated with different interpretive processes. The combination of molecular and isotopic techniques enable the narrowing of potential ancient source(s) into vessels, but a consideration of the available palaeoecological and bioarchaeological evidence; possible formation processes and burial environment affecting preservation or diagenesis of the lipid residues; pyrolytic chemistry and experimental literature; excavation conditions and storage environment influencing contamination are vital in order to make any interpretation of their past use (e.g. Mazow et al. 2014). It is also essential that the full extent of the possibilities and limitations of lipid residue analysis are understood by archaeologists. This means that archaeologists and lipid specialists must work closely and readily share information with one another for an accurate characterisation of organic residues within ceramics.

3.7. Chapter Summary

This chapter has provided the necessary background to lipid residue analysis, including an introduction to lipids, issues surrounding preservation, degradation and contamination, a brief description of common extraction methods and instrumental analyses. This chapter also outlines techniques or proxies used to interpret lipid residue data and highlights some of the challenges faced during the interpretive stage. The information provided in this chapter is necessary to understand the results and discussions presented in Chapters Six and Seven, and for the synthesis in Chapter Eight. The next chapter provides a detailed background to the archaeological sites investigated in this thesis, also providing archaeobotanical and zooarchaeological data that aid in the interpretation of results from the analyses.

Chapter Four

Background to study sites

This chapter outlines the relevant background information for sites investigated in the thesis. The information is presented as 1) Indus sites in northwest India from where recently-excavated and freshly-excavated pottery samples were studied (Section 4.1); 2) sites from where pottery samples lying in collections have been analysed (Section 4.2), and 3) samples from outside the Indus Civilisation, namely, Sultanate of Oman (Section 4.3) (see Table 4.1). Details about each site's environment, excavation and periodisation, archaeobotanical, faunal, and stable carbon isotopic evidence from animal (and human) tooth enamel, and summaries for the types of ceramics found are provided, when available. This information sets the relevant context for the interpretation of ceramic lipid residue analyses detailed in Chapters Six and Seven and in Appendix A. Figure 4.1 is a map with all the site locations.

Haryana Farmana 18^{**} town Group II Haryana Rakhigarhi 80** city Group II Rajasthan Kalibangan 11.5 town Group I

Salut Group III

Pakistan Sindh Mohenjo-daro 100 city Group I

Stone Tower I,

Table 4.1: Names and sizes of sites analysed in this thesis. Group I: samples from collections; Group II: samples from sites that were excavated between 2008-2014; Group III: samples from fresh excavations

* likely larger as modern village lies on top of site

** criteria for calculation uncertain

Sultanate of Oman

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Figure 4.1: Map of northwest India and Arabian Peninsula with urban Indus settlements and cities (in yellow and white, respectively) and sites investigated in this thesis (in red). Courtesy Cameron Petrie.

4.1. Indus sites in northwest India

The sites selected were targeted to include a range of Indus settlement types: small, rural settlement ("small villages" and "villages"), medium-sized settlements ("towns") and large settlements ("cities"). Sites were classified into these categories on the basis of estimated settlement size (see Table 4.1). Very small settlements range from between 1-3 hectares in size; small settlements range from 3-10 hectares in size; medium-sized settlements range from 10-20 hectares in size; and large-sized settlements have estimated sizes greater than 20 hectares. Although the size of Indus sites is not directly related to their function or role in the past (Chakrabarti 1995; Petrie 2013; see Section 2.1.2), and terminology may reflect inherent cultural biases about life-ways of 'village', 'town' and 'city'-populations, this categorisation is based upon evidence obtained from excavations which suggests these terms likely accurately describe their function or urban status.

4.1.1 Very small settlements ('small villages': 1-3 ha)

4.1.1.1 Alamgirpur (ALM)

Alamgirpur (29 °00.206'N, 77 °29.057'E) is one of the eastern-most settlements of the Indus Civilisation. Locally known as Parasuram-ka-Khera, it is located in the Yamuna-Ganges doab in Uttar Pradesh. A 1-hectare mound, it was discovered in the 1950s, and interpreted as an outpost of Indus settlement by Sharma (1989). Later, the discovery of other prominent sites such as Hulas and Sanauli (Dikshit et al. 1993; Joshi 1993; IAR 2007), in the region indicated that groups using Indus-affiliated cultural material were settled here. Detailed surveys and excavations in the future are likely to reveal more about the timing and intensity of settlement in the Yamuna-Ganges doab (Singh et al. 2013), however, presently our knowledge about the details of the occupation of the region remains incomplete.

Site environment

Alamgirpur lies at the eastern edge of the floodplain of the Hindon River (Singh et al. 2013). The site sits on a consolidated sand dune that rises 1.5m above the surrounding modern floodplain and is likely to have been at the margin of the Hindon's flood zone during the Indus period (Neogi 2013; Neogi et al. in press). The Hindon is almost entirely monsoon-fed, indicating seasonal access to fluvial sources (Mondal et al. 2012). However, Alamgirpur's climate is sub-humid with mean annual rainfall averaging 850 mm per year (Fick and Hijmans 2017). Thus, despite the seasonality of the rainfall, Alamgirpur has a

moderate climate, with cooler summers, warmer winters, and higher moisture availability compared to the sites in Haryana (Jones 2017). It has been suggested that the humid climate at Alamgirpur might have rendered it relatively insensitive to changes in floodwater supply during the decline of the ISM (Jones 2017).

Geoarchaeological analyses have revealed that Alamgirpur is located within a region with deep alluvial soils, with a mosaic of silts and silty clay loams that are calcareous, alkaline and relatively low in organic matter (Neogi 2013; Neogi et al. in press). It is highly likely that the soil alkalinity and salinity (rated moderate; Fischer et al. 2008) have affected the preservation of amorphous lipids within the pottery (see Chapter Six).

Periodisation and radiocarbon dates

Based on early excavations at the site, Sharma (1989) suggested that there were four distinct cultural phases at Alamgirpur, beginning with Harappan occupation (Mature and Late Harappan) followed by Painted Grey Ware, Early Historic and Medieval phases. Ceramic typologies were used to assign periods to the different phases, and thus, ambiguity remained about the precise periods of occupation of the site; particularly the relationship between the Late Harappan and Painted Grey Ware phases.

Fresh excavations carried out by a Banaras Hindu University team in 2008 aimed to establish the periodisation of the cultural sequence through a systematic program of radiocarbon dating and gathering of environmental data from the site, which had not been a focus of previous excavations. Five trenches and a Section Cutting (SC) were opened, however, only samples from the SC were analysed from this thesis as it has the highest number of radiocarbon dates. A figure of the SC trench with labelled context numbers and table with obtained radiocarbon dates are provided below (Figure 4.2 and Table 4.2).

Archaeobotanical and faunal evidence

Limited archaeobotanical analysis was originally conducted at Alamgirpur, which was presented as a list of taxa (Singh et al. 2013). The list suggested dominance of C_3 cereals within the assemblage, with the presence of winter crops such as *Hordeum/Triticum* (barley/wheat), *Vicia* sp. (vetch/wild pea), *Vigna* sp. (mung bean) and winter weeds such as *Chenopodium* sp. (Singh et al. 2013). A small number of summer crops were reported, such as rice, millet and *Zizyphus* fruit (Singh et al. 2013). The presence of both *rabi* (winter) and *kharif* (summer) crops and 'weeds' led to the interpretation that a mixed season cropping system was being employed by inhabitants at Alamgirpur (Singh et al., 2013:49; Bates et al. in prep.). A detailed analysis of the charcoal remains revealed

evidence of wood species from dry thorn scrubland, while the phytolith evidence suggested that dung fuel and grass inflorescences were used for fuel exploitation due to scarcity of tree resources (Lancelotti 2010, 2018; Lancelotti and Madella 2012). Notably, the site today is used for the storage of dung fuel.

An updated assessment of the archaebotanical remains suggest that a diverse range of cereals were used, with the ubiquitous presence of pulses across time, as well as a wide range of wild plant taxa (Bates et al. in prep.). It appears that pulses and cereals were the most frequently used crops at Alamgirpur over time (Bates et al. in prep.). Although a mix of summer and winter crops is seen across phases, evidence suggests that *kharif* cereals and tropical *kharif* pulses (summer crops) were present in higher density and proportion than *rabi* crops, suggesting they were more important and used in greater numbers at the site across time at the site (Bates et al. in prep.).

The faunal evidence from Alamgirpur indicates the dominance of *Bos indicus*/*Bubalus Bubalus* (cattle/buffalo) (<75%), and a smaller proportion of (*Ovis aries*/*Capra hircus*) sheep/goat across all periods (Singh et al. 2013). The role of *Sus* sp. (domestic and wild pig) is considered marginal, contributing to less than 4% of the NISP (Singh et al. 2013) (Table 4.3). Unfortunately, details about the data available is very limited and grouped by chronological phases categorised as 'Harappan', 'Harappan-Painted Grey Ware Overlap Phase' and 'Painted Grey-Ware phase'. Thus, as fine-grained assessment of the different Indus occupation phases is not possible, data from the 'Harappan Phase' have been provided here (Table 4.3).

Trench	Context	<i>Material</i>	Lab code	Uncal BP	cal BC (2σ)
_{SC}	128	Barley/Wheat	OxA-21881	$3737+31$	2274-2034
	126	Barley	OxA-21859	3652 ± 28	2135-1942
	118	Seed	OxA-21858	$3610+27$	2031-1896
	115	Barley	OxA-21857	$3508 + 26$	1904-1750
	107	Barley	OxA-21586	3630 ± 26	2122-1916

Table 4.2: AMS radiocarbon results from SC trench from Alamgirpur. Calibration was performed by C.A. Petrie in OxCal version 4.2 using the IntCal13 calibration curve.

Species name/category	NISP	NISP	% NISP	
	(SC Trench)	(from other trenches & excavations)		
Bos/Bubalus	92	294	80.5	
Capra/Ovis	25	34	9.3	
Sus	4	14	3.8	
Wild ruminants	$\overline{4}$	19	5.2	
Canis familiaris (dog)		2	0.5	
<i>Vulpes vulpes</i> (red fox)			0.3	
<i>Pavos cristatus</i> (peafowl)			0.3	
Total	128	365	100	

Table 4.3: Faunal remains from 'Harappan levels' at Alamgirpur (after Singh et al. 2013)

Figure 4.2: Incremental tooth enamel carbonate data from domestic and wild animals from Alamgirpur. Distance REJ indicates distance of the point from the apex of the tooth. Wild ruminants are in grey; domestic animals in colour. Higher values indicate higher input of C_4 plant into the diet of the animal. Data analysed by Smith (2016) and Jones (2017) see Lightfoot et al. (in prep). Courtesy Emma Lightfoot.

Animal enamel carbonate data

Enamel carbonate data from domestic animals has revealed insight into the diet of animals (Lee-Thorp et al. 1989; Codron et al. 2018). At Alamgirpur, δ^{13} C values of enamel carbonate from the teeth of domestic animals suggests that cattle/buffalo and sheep/goat were largely consuming high proportions of C_4 plant in their diet throughout the year, with no clear differences between animal feeding practices between *Bos* and *Capra/Ovis* at the site (Lightfoot et al. in prep., Figure 4.3) Wild ruminants such as *Bos gaurus* and *Antilope cervicapra* appear to have consumed increasing amounts of C₄ plants over time, while

Bosephalus tragocamelus ('*nilgai*'), the bovine wild ruminant species, consistently consumed C_4 plants. The high proportion of C_4 plants in the diet of wild ruminants is suggestive of the presence of wild C_4 plants in the surrounding environment.

Ceramic evidence

The ceramic assemblage at Alamgirpur is distinct to those from other Indus sites, demonstrating the presence of both Indus, or Indus-inspired ('Harappan') and a regional variant ('Bara') types of pottery (Singh et al. 2013; Ceccarelli in prep.). While the Indus or Indus-inspired pottery comprise red-slipped and dark-slipped ("chocolate") wares, the Bara types are red wares and have shapes such as long-necked jars with flaring mouths, dish-onstand vessels with sloping or drooping rims, and lids with out-turned and painted rims (Singh et al. 2013). At Alamgirpur, a variety of differently shaped vessels were found, including goblets, bowls, perforated jars, cylindrical vases, shallow dishes and basins, long-necked jars and miniature vessels.

4.1.1.2 Masudpur VII (MSDVII)

Masudpur VII (29°12.445"E, 75°57.090"N) is a 1 hectare mound site lying 4 km to the SSW of Masudpur I. Surveys indicated Early, Mature and Late Harappan material on the surface (Petrie et al. 2009), providing an opportunity to examine a rural settlement's cultural developments over time, especially in relation to Rakhigarhi, an Indus city located 16 km away.

Site environment

Locally known as Bhimwada Jodha, Masudpur VII lies within a belt of alluvial plains about 50km away from the modern Ghaggar river (Saini and Mujtaba 2012). It lies close to one of the major incursions of aeolian sediments that extends from the Pleistocene palaeoextension of the Thar desert (Saini and Mujtaba 2012). Neogi (2013; Neogi et al. in prep.) mentions the presence of sandy loams interspersed with patches of clay loams which were seasonally watered by ephemeral channels fed by summer monsoon rain. It is possible that the weakly alkaline, relatively lower levels of salinity, soil conditions, and the site's location near aeolian deposits enabled the relatively better preservation of organic remains and lipids within the pottery. The location receives an average of 432 mm of rainfall per year (Fick and Hijmans 2017), mostly during the *kharif*, or summer monsoon months (July-September), and an average of 42 mm of rainfall in the *rabi* season (November-April).

Excavation, periodisation and radiocarbon dates

The site was excavated by the *Land, Water and Settlement* project in 2009 (Petrie et al. 2009). Two trenches were excavated: YA2 and YB1 (Petrie et al. 2009, 2016). YA2 was a 2x2m sounding excavated at the highest point in the mound. Thirty-one stratified deposits comprising thirteen phases of occupation were identified. YB1 was opened south-west of YA2 to confirm the consistency of the stratigraphic contexts and expose more structural and occupational deposits. Twenty-eight stratigraphic levels with twelve phases of occupation were excavated. Section drawings are provided in Chapter 5.

AMS radiocarbon dates combined with ceramic analysis confirmed the presence of Early Harappan (*c*. 2800-2500 BC), Early Mature Harappan (EMH) (*c*. 2500-2300 BC), and Late Harappan (date is intrusive: cal. 994-831 BC) deposits in trench YA2. AMS radiocarbon dates from YB1 revealed Early Harappan (*c*. 2800-2600 BC, Late Mature Harappan (LMH) (*c*. 2100-1900 BC) and Late Harappan (*c*. 1900-1700 BC) contexts (see Figure 4.3).

Figure 4.3: Radiocarbon dates from Masudpur VII demonstrating occupation during the Early Harappan, Early Mature and Late Mature Harappan (EMH and LMH) and Late Harappan periods (after Petrie et al. 2016).

Archaeobotanical and faunal evidence

The ubiquity of cereals, pulses and fruits was high across all periods from the archaeobotanical assemblage at Masudpur VII, with lower ubiquities of oilseeds across time (Bates 2016). In the Early Harappan period there was a dominance of millet species (*Echinochloa* sp*.*, *Setaria* sp. and *Panicum* sp.) in ubiquity, density and proportion (Bates 2016). Barley was the dominant winter (*rabi*) cereal crop, but was found in 40% of the contexts, and wheat and rice contributed a minor component of the assemblage (Bates 2016). *Rabi* and *kharif* pulses were also exploited, but in low levels (Bates 2016).

There appeared to be an increase in proportions of wheat and barley in Mature Harappan contexts. Although wheat was low in ubiquity (8% of contexts), it appeared in high densities and formed nearly 20% of the crop assemblage. The proportion of millet species decreased from the Early to the Mature Harappan period, and the assemblage mostly comprised of the 'SEB' (*Setaria*/*Echincochloa*/*Bracharia*) category of small millets that are difficult to distinguish between (Fuller 2000), with no *Panicum* sp. and *Setaria* sp. present. The phytolith evidence supports the macrobotanical data from the Mature Harappan period, with increased wheat/barley phytoliths, and relatively less millet-derived phytoliths (Bates 2016). A range of summer (*kharif*) pulse taxa were also cultivated. The evidence from the Late Harappan period, which is the period the samples analysed date to, was very limited. Only three macrobotanical samples were available from Late Harappan contexts (Bates 2016). A large deposit of *Coccinia* cf. *grandis* deposited together as a vitreous clump skewed the analysis, however, rice and barley dominated the assemblage along with *Echinochloa* sp., and two *rabi* pulses – *Pisum* (pea) and *Cicer* (chickpea) (Bates 2016). The presence of *Coccinia* cf. *grandis* ('ivy gourd') is interesting as it is commonly cooked and consumed as a vegetable in northern India today. The phytolith evidence showed a slight decrease in the wheat/barley phytoliths during this period (Bates 2016).

Table 4.4: Faunal remains from Mature and Late Harappan contexts from Masudpur VII (after Joglekar et al. 2016).

Figure 4.4: Incremental tooth enamel carbonate data from domestic animals from Masudpur VII. Distance REJ indicates distance of the point from the apex of the tooth. Higher values indicate higher input of C_4 plant into the diet of the animal. Data analysed by Smith (2016) and Jones (2017); see Lightfoot et al. (in prep). Courtesy Emma Lightfoot.

Figure 4.5: Examples of Mature Harappan pottery from Masudpur VII (Petrie et al. 2009; Parikh and Petrie 2018).

Zooarchaeological analyses conducted by Joglekar and colleagues (2016) revealed that the faunal assemblage at Masudpur VII shared similar patterns to other sites in the region, with the dominance of *Bos/Bubalus* (cattle/buffalo) consumption (74% NISP), supplemented by *Ovis/Capra* (sheep/goat) (19% NISP) across time periods. The authors suggest that there was no clear evidence of sheep and did not report evidence of *Sus* sp. (wild or domestic pig) in any time period (Joglekar et al. 2016). Domestic animals dominated the Mature and Late Harappan faunal assemblages. Five fragments of *Bos/Bubalus* were the only identifiable remains from the Mature Harappan period. Domestic ruminants and a small number of wild ruminants were present from Late Harappan contexts, such as *Tetracerus quadricornis* (four-horned antelope) and *Antilope cervicapra* (blackbuck). Fragments of *Canis familiaris* (dog) and a single bone of *Labeo rohita* (rohu), a carp freshwater fish, were also found in Late Harappan contexts (Joglekar et al. 2016) (Table 4.4).

Partially charred (22%NISP) and completely charred bone fragments (14%NISP) were relatively high in proportion from Late Harappan contexts, consisting mostly of cattle/buffalo vertebrae and ribs (Joglekar et al. 2016). Cut-marks appeared on seventeen (10% NISP) skeletal elements; these were observed mostly on vertebrae, ribs, and scapulae of cattle/buffalo and sheep/goat, but also on wild ruminants such as *nilgai* and blackbuck.

Animal tooth enamel data

Isotopic evidence from enamel carbonate from domestic animals (Lee-Thorp et al. 1989) from Masudpur VII indicate diverse animal management practices (Lightfoot et al. in prep). Evidence suggests that while cattle/buffalo were consuming a high C_4 plant diet throughout the year, goat ate a more mixed C_3 and C_4 plant diet throughout the year (Figure 4.4) (Lightfoot et al. in prep.). It is hypothesised that these differences may indicate specific animal feeding practices for cattle/buffalo, as the proportion of C_4 plant input into their diet appears to be very high. Evidence from wild ruminants and more sheep/goat would create a clearer picture of wild animal diets and would serve as a proxy for reconstructing the vegetation available in the region.

Ceramic evidence and cultural material

Pottery recovered from Masudpur VII revealed the presence of locally-produced pottery classified as 'Haryana Harappan' pottery (Parikh and Petrie 2017, 2019) (Figure 4.5). Other local material culture included terracotta cakes, lumps, beads and bangles (Petrie et al. 2009, 2016). Non-local stone (steatite, carnelian) beads, faience bangles, and one gold

bead were recovered, indicating that even this small one-hectare settlement had access to broader exchange networks across the Indus Civilisation (Petrie et al. 2009).

4.1.2. Small settlements ('villages': 3-10 ha)

4.1.2.1 Masudpur I (MSDI)

Locally known as Sampolia Khera, Masudpur I (29°14.636" E, 75°59.611" N) is a 6 hectare mound site located in the Hissar district in present-day Haryana. It lies approximately 12 km to the west of Rakhigarhi and 4 km to the NNE of Masudpur VII. It has been suggested that the relative proximity of these sites to Rakhigarhi indicates that they were within the hinterland of the city during the phases of Indus occupation and may have had established networks of exchange (Singh et al. 2010).

Site environment and background

Masudpur I lies in an environment very similar to Masudpur VII. Today it is located on an alluvial plain about 50 km south of the modern-day channel of the Ghaggar river (Saini and Mujtaba 2012), but sedimentary evidence suggests that ephemeral channels seasonally aggraded the area during Indus occupation (Neogi 2013; Neogi et al. in press). Like Masudpur VII, it receives an average of 432 mm of rainfall per year (Fick and Hijmans 2017), mostly during the summer monsoon months (*kharif*) (July-September), an average of only 42 mm of rainfall in the winter (*rabi*) season (November-April). Thus, it is possible that monsoon-driven run-off would have been the primary water source for the settlement during the Indus period.

Geoarchaeological evidence suggest that much like today, Indus-period soils around Masudpur I were weakly alkaline, low salinity sandy-loams with patches of clay-loams (Singh 2007; Neogi 2013; Neogi et al. in press). High levels of calcium carbonate and leaching of nutrients down soil profiles were observed by Neogi (2013; Neogi et al. in press), however, the Indus period soils were characterised as reasonably fertile (Neogi 2013; Neogi et al. in press). As at Masudpur VII, it is possible that the weakly alkaline and relatively lower levels of salinity enabled relatively better preservation of organic remains and lipids within the pottery. Today the site has been heavily truncated by agricultural activity (Petrie et al. 2009).

Excavation, periodisation and radiocarbon dates

Excavations at Masudpur I were conducted in 2008 by the *Land, Water and Settlement* team, a joint University of Cambridge and Banaras Hindu University collaboration (Petrie et al. 2009). Three trenches were excavated, namely XA1, YA3 and XM2. XAI was

opened at the top of the mound to expose the sequence of occupation. Trench YA3 was opened next to XAI to test the consistency of the stratigraphic sequence. Trench XM2 was opened on the west-side of the mound where an exposed section revealed mudbrick structures. Pottery samples for organic residue analysis were not selected from Trench YA3 due to questions about the stratigraphic integrity and lack of radiocarbon dates from the trench (see Chapter 5).

The ceramics and small finds from Masudpur I indicate that it was occupied in the Early, Mature and Late Harappan periods (Petrie et al. 2009). However, the Late Harappan levels were severely truncated and radiocarbon dates point to the site's occupation predominantly dating to the Mature Harappan period (Figure 4.6). Thus, sherds to be sampled for this thesis were only selected for analysis from contexts with radiocarbon dates between *c*. 2200-1900 BC, or the Late Mature Harappan (LMH) period (see Chapter 5).

Figure 4.6: Radiocarbon dates from Masudpur I indicating occupation during Early Harappan and Late Mature Harappan (LMH) periods (after Petrie et al. 2016).

Archaeobotanical and faunal evidence

Masudpur I experiences both summer and winter rainfall systems, and archaeobotanical analysis indicates the use of both summer- and winter-based cropping, with macrobotanical remains of barley-type, rice $(C_3$ crops) and millets $(C_4$ crop). However, Bates and colleagues (Bates 2016; Petrie and Bates 2017; Petrie et al. 2017) have demonstrated that millets were more regularly used and in greater proportions than wheat/barley, and to a lesser extent, rice. Inhabitant at Masudpur I grew a high percentage of small-grained millets, which appear to have been either used to fodder animals and/or were directly consumed. Although the phytolith data was dominated by wheat/barley types, appearing nine times more than millet-type phytoliths, it has been suggested that their profusion likely relates more to crop-processing activities (Bates 2016).

Apart from cereals, the archaeobotanical assemblage from Masudpur I is extremely diverse, characterised by a range of pulses, oilseeds and fruits. Pulses and oilseeds are ubiquitous, with summer (*kharif*) pulses and oilseeds found in more contexts and greater quantities than the winter (*rabi*) pulses. It is likely that both *rabi* and *kharif* crops were grown at Masudpur I, but *kharif* cropping was more intensive (Bates 2016). Thus, evidence suggests that the site's inhabitants were consuming a range of summer- and winter-grown cereals, pulses and oilseeds. However, crops may have also been traded.

The faunal evidence from Masudpur I is similar to that of the rest of the region, with high proportions of domestic versus wild mammal remains, and the dominance of cattle/buffalo bones compared to the remains of smaller ruminants (but evidence for buffalo and sheep appears limited across contexts) (Joglekar et al. 2017). As Table 4.6 indicates, *Bos/Bubalus* comprise 83% NISP, *Capra/Ovis* make up 10% NISP, and *Sus* comprise 2% NISP at the site during the Mature Harappan period. The consumption of wild ruminants such as *T. quadricornis* and *A. cervicapra* (antelopes and black buck) and *Lepus nigricollis* (hares) is also attested, as well as freshwater species (carp, mussel and gastropod) (Joglekar et al. 2017).

Charring marks and cutmarks were observed on several bones from the Mature Harappan contexts at Masudpur I (Joglekar et al. 2017). Crucially, 31% NISP of the bones showed evidence for charring, and about 12% NISP showed evidence for cut-marks in the Mature Harappan period (Joglekar et al. 2017). 19% NISP of the bones were completely charred (Joglekar et al. 2017). The high degree of charring of bovine bones might reflect the end-products of cooking activities (the roasting of meat cuts over fire and discarding of bones in the fire), or of using bone as a fuel source. Patterns of chopping and butchering

Species name/category	Trench XAI (Phases III-VII)		Trench YA3 $(Phases II-IV)$		Trench XM2 (Phases III-IX)		Total NISP	%NISP
	NISP	%NISP	NISP	%NISP	NISP	%NISP		
Bos/Bubalus	376	76.7	595	90.4	163	73.1	1134	83.0
Capra/Ovis	84	17.1	18	2.7	40	18	142	10.4
Sus	3	0.6	22	3.3	2	0.9	27	2.0
Wild ruminants	15	3.5	15	2.3	11	5	41	3.0
Lepus nigricollis								
(hare)	4	0.8	$\boldsymbol{0}$		6	2.6	10	0.7
Freshwater fish and molluscs	3	0.6	$\boldsymbol{0}$				3	0.2
Canis/Vulpes								
(dogs/foxes)	1	0.2	$\overline{2}$	0.3			3	0.2
Pavo cristatus								
(peafowl)	$\mathbf{0}$		$\overline{2}$	0.3			$\overline{2}$	0.1
Hystrix indica								
(Indian-crested)								
porcupine)	$\overline{0}$		$\overline{4}$	0.6	1	0.4	5	0.4
Total	486	100	658	100	223	100	1367	100

Table 4.5: Faunal remains from Mature Harappan contexts from Trenches XAI, YA3 and XM2 at Masudpur I (after Joglekar et al. 2017).

Figure 4.7: Incremental tooth enamel carbonate data from domestic and wild animals from Masudpur VII. Distance REJ indicates distance of the point from the apex of the tooth. Wild ruminants are in grey; domestic animals in colour. Higher values indicate higher input of C₄ plant into the diet of the animal. Data analysed by Smith (2016) and Jones (2017); see Lightfoot et al. (in prep). Courtesy Emma Lightfoot.

cattle, buffalo, and goats appeared to be similar across the site, with chop-marks visible on the humerus, femur, tibia, radius-ulna, vertebrae, ribs, phalanges and mandibles (Joglekar et al. 2017).

Animal enamel carbonate data

Enamel carbonate $\delta^{13}C$ values of fauna (Lee Thorp et al. 1989) from Masudpur I were similar to those from Masudpur VII (Lightfoot et al. in prep). Domestic and wild bovine species demonstrate very high C_4 plant consumption throughout the year, whereas domestic and wild small ruminants appeared to be eating a more varied C_3 and C_4 diet (Figure 4.7), suggesting variable animal management and feeding practices (Lightfoot et al. in prep.).

Ceramic evidence and cultural material

Excavations conducted in 2009 by the *Land, Water and Settlement* team recovered a large number of black on red local ceramic vessel fragments, which are described as 'Haryana Harappan' (Parikh and Petrie 2017, 2018). Other material such as terracotta cakes, lumps, beads and bangles that were likely to have been produced locally were also found (Petrie et al. 2009). Non-local material such as fragments of faience and shell bangles, agate, steatite, carnelian, lapis and gold beads and fragments of grinding stones made from Delhi quartzite were also recovered (Petrie et al. 2009). The presence of these objects demonstrates that the settlement was embedded in broader resource acquisition and trade networks within the Indus Civilisation, ranging from northern Afghanistan and Pakistan to Gujarat (Petrie et al. 2009).

4.1.2.2 Lohari Ragho I (LHRI)

Lohari Ragho I (76°03.473" E, 29°24.66" N) was one of the sites discovered during the surveys undertaken by the *Land, Water and Settlement* project in 2009 and 2014 within the hinterland of Rakhigarhi (Singh et al. 2018). Surface mapping, survey and preliminary excavations undertaken in 2015 demonstrated that this was one of the most significant settlement sites in close proximity to Rakhigarhi, appearing to be around 8-9 hectares in extent (Singh et al. 2018).

Site environment and background

The settlement was possibly situated at the distal end of a raised area in a braided floodplain, similar to Masudpur I (Petrie et al. 2009; Singh et al. 2018; Neogi et al. in press). Ongoing geoarchaeological investigations suggest that Lohari Ragho I lay close to a riparian environment (Walker, pers. comm.; in prep.).

Excavation, periodisation and radiocarbon dates

Preliminary excavations at the site were carried out in 2015 to assess the degree of preservation at the site (Singh et al. 2018). A small 2x2m sounding (Trench A) was opened at the highest point in the NW quadrant of the mound and a 5x2m sounding (Trench B) was opened next to a standing section in the SW (Singh et al. 2018). In 2017, larger-scale excavations were conducted. A $10x10m$ trench (Trench EA) was opened in the NE quadrant of the mound, revealing 97 stratigraphic contexts related to at least 3 distinct phases of occupation (Singh et al. 2018). Deposits included evidence of structural remains and collapse and distinct activity areas (Singh et al. 2018). Ceramics and radiocarbon dates obtained from Trench EA suggest that Lohari Ragho I was occupied in the Early Harappan period (3000-2500 BC) and Early Mature Harappan (EMH) (2500-2200 BC) period into the Late Mature Harappan period (LMH) (2200-1900 BC) (Figure 4.8). Other dates obtained were from Medieval and modern periods.

Archaeobotanical and faunal evidence

The archaebotanical assemblage from Lohari Ragho I is currently under investigation (Ustunkaya, pers. comm.). Preliminary analyses suggest poor preservation of charred seeds. Out of the seeds preserved, a high percentage of millet species and legumes and a small percentage of rice (*Oryza* sp.) have been recovered, suggesting that summer cropping may have been dominant. Faunal analyses from Lohari Ragho I are ongoing; however, flotation revealed the presence of a high proportion of fishbone, suggesting inhabitants at the settlement had access to freshwater resources.

Animal enamel carbonate data

Enamel carbonate ẟ ¹³C values (Lee-Thorp et al. 1989) are only available from *Bos/Bubalus* (cattle/buffalo) teeth from Lohari Ragho I. The results obtained are similar to those for other cattle/buffalo in the region, indicating very high C_4 plant consumption throughout the year (Figure 4.9) (Lightfoot et al. in prep.) It is hypothesised this suggests specific cattle/buffalo management and feeding practices (Lightfoot et al. in prep.).

Figure 4.8: Radiocarbon dates from Trench EA at Lohari Ragho I. Courtesy Cameron Petrie.

Figure 4.9: Incremental tooth enamel carbonate data from *Bos*/*Bubalus* from Lohari Ragho I. Distance REJ indicates distance of the point from the apex of the tooth. Higher values indicate higher input of C₄ plant into the diet of the animal. Data analysed by Smith (2016) and Jones (2017); see Lightfoot et al. (in prep). Courtesy Emma Lightfoot.

Ceramic evidence and cultural material

The pottery excavated from Lohari Ragho I is currently under study (Ceccarelli in prep.), but the ceramics recovered closely match black on red 'Haryana Harappan' pottery found at sites in Masudpur VII, Masudpur I, and Farmana (Singh et al. 2018; see Parikh and Petrie 2017, 2018). In contrast to Masudpur VII and Masudpur I, a small number of examples of pottery that resembled 'Classic Harappan' shapes and decoration were also found. Further analysis is ongoing (Cecccarelli in prep.) Nearly 240 fragments of grinding stones were recovered from the surface of the site; matching types similar to those recovered at Rakhigarhi (Nath 2014; see Singh et al. 2018). Although no formal identifications have taken place, most of the stones appear to be made from red/pink Delhi quartzite from the Kaliana Hills, Haryana, with others are visually similar to granite from Tosham (near Khanak), Pab sandstone from the Sulaiman Range in Pakistan and rounded cobbles of possible Himalayan origin (Law 2011; Singh et al. 2018). The ceramic and cultural material suggest that inhabitants of Lohari Ragho I were integrated into the same raw material acquisition networks that were accessed by those living at Rakhigarhi, and producing and obtaining local and 'non-local' pottery, respectively (Singh et al. 2018; Ceccarelli in prep.). The movement of quotidian, bulky items involved in food-processing is particularly significant.

4.1.2.3. Khanak (KNK)

Khanak's (28°54'26.5" N, 75°52'08.9" E) location within southern Haryana is unique in contrast to the other sites discussed in this chapter. It lies adjacent to an inselberg, which is an isolated outcrop of the Aravalli hills. The geology of the region comprises a range of igneous, granite and quartzite rocks, including a type of the infamous Delhi quartzite. The stone from these outcrops is red-pink to pinkish-grey in colour and is criss-crossed with thin haematite and quartz-filled fractures (Law 2011). This specific type of stone has been found in Harappa in the form of grinding stones (Law 2011) and other sites in Haryana, including Lohari Ragho I (Singh et al. 2018). Tin has also been reported in the form of cassiterite from the region (Singh et al. 2015).

The proximity of the site to rocks and minerals that were important within Indus trade networks suggests it played a role in stone mining and tin or copper smelting activities (Singh et al. 2015). Although the estimated size of the site was calculated to be 1.5 hectares, there is evidence that the modern village of Khanak is now lying on top of

more of the ancient settlement. It is thus likely that this settlement was larger in antiquity, but it has been damaged due to modern occupation.

Site environment and background

In contrast to other sites discussed in the thesis, Khanak lies within the margins of an arid climate zone, with mean annual rainfall of 400mm, receiving most of it between June-September (data.gov.in). This suggests that the residents of the settlement possibly had a higher reliance on summer crops. Agricultural and pastoral strategies were probably suited to adapt to the paucity and unpredictability of water availability in the region.

Excavation, periodisation and radiocarbon dates

Excavations were conducted at Khanak in 2014 by a team from Banaras Hindu University, Uttar Pradesh. The excavations were conducted on the grounds of a government school. The goal of the excavations was to conduct small-scale rescue excavations to examine the potential of early exploitation of stone and minerals. Other goals included obtaining secure radiocarbon dates and bioarchaeological information for the site, obtaining geological samples for provenance studies; and to explore potential locations of ancient mining activity near the settlement. Five trenches were excavated within three weeks. Excavations revealed over 20 contexts with evidence of structural material and occupation surfaces. The preservation of contexts was better than other sites in the region, probably due to the depth of contexts, arid environment and the location of the trenches in a schoolyard, which meant it had been undisturbed by modern-day agricultural activities.

Based on the cultural material, the site was dated to the Early and Mature Harappan periods (Singh et al. 2015). Radiocarbon dates from Trench A05 confirmed that the site was occupied in the Early Harappan (3000-2500 BC), Early Mature Harappan (EMH) (2500-2200 BC) and Late Mature Harappan (LMH) (2200-1900 BC) periods (Figure 4.12). Pottery samples were chosen from this trench for organic residue analysis.

Figure 4.10: Radiocarbon dates from deposits at Khanak (Courtesy Cameron Petrie).

Archaeobotanical and faunal evidence

The archaeobotanical and faunal analyses from excavations at Khanak are ongoing. Preliminary results suggest that the archaeobotanical assemblage at the site is distinctive as it contains very limited evidence of charred seeds and demonstrates high ubiquity of charred wood species (Ustunkaya, pers. comm.). Out of the studied assemblage, economic crops at Khanak involved *Hordeum/Triticum* (wheat/barley) as dominant species (24%) followed by millets (12.5%) and a small percentage of legumes (2.5%). Dominant economic non-crop species included *Ziziphus* sp. (20%) (fruit), along with *Plantago* sp.; plants that are generally found in riparian environments (Ustunkaya, pers. comm.). Unexpectedly, a seed of *Allium* sp. (garlic) was preserved in the assemblage (Ustunkaya, pers. comm.). Thus, it appears that summer and winter crops were grown at Khanak but details about agricultural practices are presently being collated. The faunal assemblage from Khanak is also under study, however a significant number of fishbones were recovered via flotation (Ustunkaya, pers. comm.), which suggests that inhabitants of Khanak had access to freshwater resources.

Ceramic evidence and cultural material

The ceramic assemblage at Khanak included typical 'Haryana Harappan' pottery, with black on red painted jars, chocolate slipped jars, vases, bowls, basins, perforated jars, and examples of dish-on-stand vessels. Some vessel shapes typical of 'Classic Harappan' pottery were also found, such as goblets and miniature vessels (Singh et al. 2015). Examples of nearly complete vessels were recovered from several trenches. Other objects included terracotta cakes, steatite beads, semiprecious stones including lapis lazuli, large quantities of slag and a copper celt.

4.1.3. Medium-sized settlements ('towns': 10-20 ha)

4.1.3.1. Farmana (FRN)

The site of Farmana (29 ° 02' 22" N, 76 ° 18' 21" E) is located in Rohtak district, Haryana and is locally known as Daksh Kheda (Shinde 2011). It has been suggested that nearly 18 hectares of the site were densely occupied in the past (Shinde et al. 2008), although it is unclear how this estimation was calculated, as the preserved architecture has been revealed across a much smaller area.

Site environment and background

Farmana lies about 30km away from the modern Chautang River, a tributary of the Ghaggar River, within a large alluvial plain. The site is located on an elevated sand deposit that the excavators suggested was formed by fluvial action (Shinde 2011). However, aeolian deposits are located close to the site and are interspersed with alluvial sediments (Shinde 2011). Detailed information about the sedimentological conditions and soil quality at Farmana are not available to make inferences about how they might have influenced the preservation of organic remains.

Excavation, periodisation and radiocarbon dates

Excavations at Farmana were led by Prof. Vasant Shinde from Deccan College, Pune, Dr. Manmohan Kumar, Maharishi Dayanand University, Rohtak, and Prof. Toshiki Osada, Research Institute for Humanity and Nature (RIHN), Kyoto, between 2006-2009. Excavations revealed that only the central portion of the mound (80x60m) was preserved, with the upper levels being heavily truncated by modern agricultural activity (Shinde 2011).

Excavations at Farmana revealed evidence of an extensive settlement area, and a cemetery located less than a kilometre away. Burials in the cemetery date to the Mature Harappan period, but as pottery from the cemetery has not been investigated in this thesis, only details of the settlement area are provided here. Large-scale excavations revealed at least three architectural complexes with multiple rooms, bathing platforms, drains, storage units and courtyards, with a main street and smaller streets dividing them. Although multiple phases of occupation (at least five) were exposed in soundings, the precise extent of occupation at different points has been interpreted in contradictory ways. The radiocarbon dates suggest that the settlement was occupied in the Early Harappan and Early Mature Harappan period (EMH) (*c*.2500-2250 BC) (Figure 4.11) (Shinde et al. 2011: 831). The excavators, however, suggest that the central area of the site is composed of both Early Harappan ('Period I': *c*. 3500-2600 BC) deposits and Mature Harappan deposits, with the latter being represented by three sub-phases: 'Period IIA' (early Mature: *c*.2600- 2400 BC), 'Period IIB' (middle Mature: *c.* 2400-2200 BC) and 'Period IIC' (late Mature: *c*.2200-2000 BC) (Shinde 2011: 6).These classifications were based on stratigraphy, pottery, and burials from the cemetery.

Pottery samples selected for analysis from Farmana in this thesis are from three architectural complexes in the 'Central Area' where the excavators and the radiocarbon dates are broadly in agreement; and occupation is dated to the Early Mature Harappan period (EMH) (*c*.2500-2200 BC), or 'Period IIB' for the excavators. However, it is not always possible to securely associate pottery samples with their precise find-location within a trench (see Chapter 5).

Figure 4.11: Radiocarbon dates obtained from Trench 1D5 in the 'Central Area' from Farmana (after Shinde et al. 2011: 835).

Archaeobotanical and faunal evidence

The plant remains at Farmana were subjected to diverse types of analyses. Macrobotanical analyses were complemented by phytolith and starch grain analysis from ceramics, stone blades, pounders and grinders, human and animal dental calculus, and soil (Weber and Kashyap 2010; Weber et al. 2011). The macrobotanical remains primarily provided evidence for wheat, barley and several small millets such as *Panicum* sp. and *Setaria* sp., although poor preservation (high ubiquity but low density) of seeds was observed (Weber et al. 2011). Seeds from a variety of pulses such as *Mung* sp*.* (green gram), *Macrotylma* sp*.* (horse gram) and *Sesamum* sp*.* (sesame) were also recovered: after cereals, these were the most frequently recovered seeds (Weber et al. 2011). Fruits and oilseeds such as *Cucurbita* sp., *Linum* sp., (linseed) and mango were present (Weber et al. 2011). A carbonised fragment of *Allium* sp. (garlic) (Weber et al. 2011), like at Khanak, was also found. Weber and colleagues (2011) argued that wheat, barley, and millets were the primary cereal crops for Farmana. They also suggested that winter cereals declined in importance over the

occupation of the site (from 61% to 20% ubiquity), that millets remained important, but that rice never played an important role (Weber et al. 2011).

Starch grains and phytoliths were collected from 240 surfaces from both the cemetery and settlement area, including human and animal teeth, ceramic vessels, stone tools and sediment (for control; Weber et al. 2011). Out of the 50 specimens that were subsequently analysed, starch remains of millet, barley and gram were found on human dental calculus (n=9; 3 individuals). *Solanum* sp. (aubergine) and mango were found on long, narrow stone blades (n=2). In contrast, spices such as *Curcuma* sp*. and Zingiber* sp*.* (turmeric and ginger respectively), were only found on the surface of ceramics. Starches of barley, millet and mango were found on grinders and pounding stones (n=8). Additionally, the phytolith remains revealed evidence of *Oryza sativa* (rice), which was found on three artefacts, wheat/barley, *Panicese*-type (possibly derived from millets), and *Poacese* (grasses and reeds) phytoliths.

The authors (Weber et al. 2011) used this evidence to suggest that individuals at Farmana had a broad plant diet and consumed wheat, barley, a variety of small-grained cereals, pulses, fruits, vegetables and roots and tubers. Although the starch-grain analyses provide direct evidence for the processing and consumption of plant-remains, there is inadequate data and information presented about the measures taken to prevent and check for contamination that may have occurred from starches in the sediment and surrounding environment (Chapter Two, Section 2.3.5.1). This is particularly important as many of the starch remains are ingredients used in Indian cooking today. Since these details, as well as detailed information and analyses remain unpublished, the results must be treated with caution.

The faunal remains at Farmana were studied by Sharda C.V. The results from her thesis and other publications (Channarayapatna 2014, 2018; Joglekar and Channarayapatna 2018) suggest that just as at other sites in the region, the proportions of domestic animals outnumber wild species at Farmana. In the Mature Harappan period, *Bos/Bubalus* (cattle/buffalo) made up the largest proportion of domestic species (78% NISP) (Table 4.7). Out of the medium-sized domestic mammals, which make up about 11% NISP, *Ovis* and *Capra* (sheep and goat) appear to have equal representation (Channarayapatna 2014, 2018). Although distinguishing between sheep and goat is very difficult and depends on the presence of specific anatomical elements, if this interpretation is correct, it suggests that residents of Farmana may have practised different animal management systems from other settlements, as not many sites have clear evidence for sheep (Joglekar et al. 2016;
2017). *Sus* (wild and/or domestic pig) remains comprise 0.6% NISP from the Mature Harappan period. Remains of several wild deer, including *Antilope cervicapra, Tetracerus quadricornus, Gazella benneti*, hare, small-, medium-, and large-sized birds and freshwater fish and molluscs were found in all parts of the settlement. However, except for a single trench (1D5), which yielded high proportions of freshwater fish (58.2% NISP), the remains of wild ruminants, hare, birds and freshwater fish and molluscs account for a small proportion of the fauna. Despite the low ubiquity of wild faunal remains, Farmana exhibits the highest level of wild mammal utilisation diversity compared to other sites in the region (61% compared to 20-40%) (see Joglekar et al. 2013). It is also notable that the presence of ruminants such as *Tetracerus quadricornus* (four-horned antelope) suggests that residents possibly had direct or indirect access to humid evergreen open jungles, where these animals are found (Joglekar et al. 2013). Overall, the presence of over fifteen species of wild ruminants and freshwater fish indicate the diversity of animals brought to the site (Channarayapatna 2014, 2018).

The study of butchery practices at Farmana revealed cut marks and charring on meat and marrow-rich bones of domestic and wild animals (Channarayapatna 2014, 2018). Completely charred and vitrified bones were dominant among the bone modifications observed (Channarayapatna 2014, 2018). It was also noted that nearly all anatomical elements of cattle/buffalo had charring marks, whereas only the cranial fragments, ribs, scapula, vertebrae, and phalanges of sheep/goat were charred to different degrees (Channarayapatna 2014, 2018). These differences might hint at different butchery practices for different animals at the settlement, and preferences for certain cuts of meat amongst residents. More than half of the fish bones recovered from Trench 1D5 were charred (Channarayapatna 2014, 2018). The wide range of animal types and high percentage of fish remains is unique to Farmana. No clear patterns of discard and deposition of animal bones within the settlement could be discerned, with a variety of anatomical elements of different animals present across the settlement. This suggests that hunting was an activity practised by urban residents, or that urban residents procured whole carcasses of wild mammals from other communities and butchered them on site before consumption.

Table 4.6: Faunal remains from Farmana from Mature Harappan and 'General Mature Harappan' (which includes both periods IIA and IIB) contexts. Channarayapatna (2014) provided additional faunal remains from the 'Early Harappan', 'Early-Mature Transition' periods, 'Surface Layer' and 'Kiln' area, but these were excluded. (after Channarayapatna 2014)

Figure 4.12: Incremental tooth enamel carbonate data from domestic and wild animals from Farmana. Distance REJ indicates distance of the point from the apex of the tooth. Higher values indicate higher input of C_4 plant into the diet of the animal. Data analysed by Tames-Demauras (2018), see Lightfoot et al. (in prep). Courtesy Emma Lightfoot.

Animal and human isotopic data

The carbon isotope values of the tooth enamel (Lee-Thorp et al. 1989) from domestic and wild animals at Farmana revealed similar patterns to other sites in the region (Lightfoot et al. in prep.) (Figure 4.12). An assemblage of multiple cattle and two sheep/goat teeth, and a single wild ruminant tooth were investigated as part of a MPhil dissertation (Tames-Demauras 2018). Cattle, like at other sites, demonstrated very little change in diet throughout the year, foddering consistently on C_4 plants. A single sheep/goat demonstrated a change in diet from C_3 plant consumption to higher inputs of C_4 plant consumption throughout the formation of the tooth. The wild ruminant (*Antilope cervicapra*) tooth reflected δ^{13} C values that were extremely similar to cattle, suggesting a C₄ plant-based diet, which is unlike the δ^{13} C values of other wild ruminants from the region that demonstrate a mixed C_3 and C_4 plant-based diet across the period of tooth formation (Tames-Demauras) 2018). The lack of diet variation exhibited in the δ^{13} C values of this wild ruminant might be suggestive of its origin from a different habitat, but extrapolating meaning from a single outlier in the assemblage is difficult (Tames-Demauras 2018).

Oxygen isotope values (Bryant et al. 1996) demonstrated another aspect of the animal management at Farmana. Sinusoidal curves were observed in the $\delta^{18}O$ values of six cattle teeth, reflecting a change in oxygen isotope values throughout the year based on seasons (Tames-Demauras 2018). However, no clear pattern was observed, suggesting that the cattle were born in different seasons. It was hypothesised that the variable time of birth of the cattle may reflect an active controlling of the breeding season of cattle by residents at Farmana to maintain a constant source of meat and secondary products (Tames-Demauras 2018). A correlation between the sinusoidal curves of $\delta^{18}O$ and $\delta^{13}C$ values for the single sheep/goat analysed was observed, suggesting C_3 plant consumption in winter, and higher C⁴ plant consumption in summer, reflecting an environmental shift in access to plants (Tames-Demauras 2018). When considered together, the isotopic values from cattle teeth suggest the active monitoring of foddering practices and possible breeding control at Farmana (Tames-Demauras 2018).

Results from Valentine's (2013) analysis of enamel carbonate from humans buried in the cemetery at Farmana suggested that populations at Farmana were eating both C_3 and C_4 plants. There was a limited variation in the relative contribution of C_3 and C_4 consumption at the population level (δ^{13} C range = 4.7‰), but this variation did not correlate with tooth type, sex or age (Valentine 2013). Unfortunately, as bone collagen was not available for

analysis, $\delta^{15}N$ values were not obtained, limiting an assessment of protein input into individuals' diet.

Ceramic evidence and cultural material

The ceramic assemblage at Farmana was studied in detail by Uesugi (2011). He reported the presence of 'Harappan' and 'Non-Harappan' pottery, which has been termed here as 'Classic Harappan' and locally-produced 'Haryana Harappan' pottery, respectively (Figure 4.13). Uesegi (2011) and Shinde (2011) noted the persistence of 'Haryana Harappan' pottery throughout the sequence; suggesting that no particular trend in the relative frequencies of both types of pottery could be observed across time or in different areas (Uesugi 2011). Although there are concerns about the degree to which taphonomic processes were accounted for while conducting the analysis, the overwhelming presence of 'Haryana Harappan' pottery at the site suggests that locally-produced pottery predominated in medium-sized Indus settlements like Farmana.

Other cultural material recovered from the excavations link it to larger urban centres and long-distance exchange networks (Konasukawa et al. 2011). For example, four steatite seals, and two seal impressions were found, out of which two seals and one seal impression were found in Structural Complex No. 3 (Konasukawa et al. 2011), from where pottery samples for organic residue analysis have been selected. Other finds included a variety of stone beads made from steatite, carnelian, chert, agate, jasper, chalcedony, lapis lazuli, and precious metals like gold (Konasukawa et al. 2011). Faience and shell bangles and beads were also reported from the excavations (Konasukawa et al. 2011).

Figure 4.13: Top: 1-18: Examples of 'Classic Harappan' ("Harappan") pottery from the Settlement area at Farmana. Bottom: 1-13: Examples of 'Haryana Harappan' ("Non-Harappan") pottery from the Settlement area at Farmana (reproduced from Uesugi 2011: 180-181).

4.1.4 Large-sized settlements (>20 ha)

4.1.4.1 Rakhigarhi (RGR)

Today, Rakhigarhi (29°17'19" N, 76°06'47" E) is a large village in Hissar district in the state of Haryana, but in 1963, Suraj Bhan (1975) identified one of the largest Indus sites in the region located underneath it. At least seven mounds exist in the area; five in close proximity, and two others a distance away, numbered RGR-1 to RGR-7 (see Figure 4.14). A cemetery area (RGR-7) with multiple burials lies between 300-750m away from RGR-1 and RGR-2, respectively (Nath et al. 2015; Shinde et al. 2018a, 2018b). It has been argued that archaeological remains at Rakhigarhi extend to 300 hectares (Nath 2014: 128), but this estimation includes the area of all seven mounds. Thus, it is unclear if the mounds effectively made up a single settlement in antiquity, and there has been a tendency to overestimate its size. Despite this, there is no doubt that the archaeological remains at Rakhigarhi are substantial, possibly comparable to Harappa. The cemetery area (Shinde et al. 2018a, 2018b) is not described in this thesis as pottery samples were only taken from the settlement, specifically, RGR-4.

Site environment and background

Like other sites discussed in this thesis, Rakhigarhi lies on the Satluj-Yamuna alluvial plain. The landscape comprises of mixed alluvial and aeolian deposits. Aeolian deposits of varying thicknesses overly the Pleistocene alluvium, which is exposed at various instances in topographic depressions or beneath a veneer of sands (Nath 2014: 59-62). The region immediately around Rakhigarhi (1km) has a series of sandy loam to moderately developed loam soils that are well-drained. In the larger vicinity, soils range from sandy and heavily eroded to loamy and calcareous, with variable levels of fertility (Nath 2014: 60). Geomorphological evidence of pluvial and fluvial processes (multiple rivers and braided drainage and deposition as well as major stream action) are indicated with the presence of riverine sediments comprising of clay, sand, silt and gravel (Ahuja and Singh 1983; Nath 2014: 60-61). Seasonal flooding continues to take place between July through to September during the monsoon season.

It has been suggested that Rakhigarhi was located on a now-dried river bed (Bhan 1975: 95), and idealised plans of the site show a channel encompassing most of the mounds (e.g., Nath 1998: 40; Fig 4.14). It is notable that the channel is not clearly visible today, or on the British maps from the early decades of the twentieth century (Singh et al. 2010). Nath (2014: 105) also mentions evidence of 60cm of flood deposits at RGR-1. Such

flooding events were likely seasonal as they are even today, which has implications for how inhabitants at Rakhigarhi secured a consistent water supply, and how they managed the settlement in circumstances of flooding. Evidence for palaeo-ponds and palaeo-lakes near the site have also been mentioned (Nath 2014), and the modern villages of Rakhi Khas and Rakhi Shahpur are surrounded by very large ponds, some of which as permanently filled, and others that are seasonally flooded (Singh et al. 2010). It is possible these ponds served as more reliable water sources for residents.

Figure 4.14: Plan of archaeological mounds at Rakhigarhi. RGR-7 lies to north of RGR-1 and is not on the map (reproduced from Nath 1998: 40).

Figure 4.15: Trenches excavated on RGR-4 by Deccan College, Pune. Courtesy Yogesh Yadav.

Excavation, periodisation and radiocarbon dates

Two large-scale excavation projects have taken place at Rakhigarhi. Three seasons of excavations were first organised between 1997-2000 by Dr. Amarendra Nath, Archaeological Survey of India (Nath 2014). More recently, the site was excavated between 2013-2015 by a team from Deccan College, Pune, led by Prof. Vasant Shinde. The pottery samples from this thesis were obtained from the 2014 excavations, specifically from excavations conducted on RGR-4.

The excavation between 1997-2000 uncovered what was termed as 'pre-formative' occupation (*c*. 4500-3900 BC) on RGR-6, Early Harappan occupation (*c.* 3900-3000 BC) on RGR-6, and Mature Harappan occupation (*c*. 2500-2300 BC) on RGR-1 and RGR-2 (Nath 2014, 2018; Nath et al. 2015). The 'pre-formative' and Early Harappan occupation phases are characterised by large circular mudbrick structures and rectangular mudbrick buildings with rooms, courtyards and hearths, next to E-W and N-S running streets (Nath 2001). A large (25x12m) plastered enclosure wall encircling RGR-2, with evidence of platforms and brick wells at the entrance, was dated to the Mature Harappan period (Nath 2001). Nath (2001) reported the presence of a lapidary workshop from the Mature Harappan period at RGR-1, with evidence of thousands of unfinished and finished beads made from carnelian, chalcedony, agate, and jasper as well as bead polishers and hearths for firing the stones. A crafting area with debitage of *Turbinella pyrum*, a type of marine shell, were also reported in the vicinity of the settlement (Nath 2014).

At RGR-4, early excavations reported evidence of platforms and streets running N-S (Nath 1997, 2000). More recently, the Deccan College team laid out 6 stepped trenches across the northern slope of RGR-4, from Trenches 1.A to 1.F (Figure 4.15). The stepped trenches uncovered structural remains and a series of fills and pits that possibly represent episodes of ephemeral occupation and filling events. While Trench 1.A mostly contained fill material, Trench 1.B revealed evidence of a structure with individual rooms and a large hearth structure with an *in-situ* storage jar (Yadav, pers. comm.). Remains of a mudbrick platform were excavated at Trench 1.E, and a large storage area with multiple 'bins', termed 'granary' by the excavators was excavated in Trench 1.F (Yadav, pers. comm.). A report on the recent excavations is currently under preparation.

Stylistic assessment of the pottery vessels from these trenches suggests that date to the Mature Harappan period. The radiocarbon dates obtained from RGR-4 range between *c*. 2500-2300 BC, placing it within the Early Mature Harappan period (EMH) (Vahia et al. 2016). These dates were obtained from depths ranging from 9 to 20 metres into the mound (Vahia et al. 2016), suggesting a large amount of deposition within a relatively short period of time. Another set of radiocarbon dates have recently been published from RGR-1, RGR-2 and RGR-6 (Nath 2018). This paper reports dates from 5500 BC, pushing the origins of occupation at Rakhigarhi to the "Neolithic," upto 1800 BC (Nath 2018: 116-125). Unfortunately, the published dates are not accompanied by stratigraphic profiles, or details, drawings or analyses of cultural material excavated. This makes it difficult to confidently assess the nature of establishment and abandonment of the settlement.

Archaeobotanical and faunal evidence

The archaeobotanical and faunal analyses from the most recent excavations at Rakhigarhi are ongoing. Detailed information about previous analyses are extremely limited. The presence of rice, wheat and barley in the Early and Mature Harappan periods (Nath 2014, 2018) is the only information we have regarding the archaeobotanical remains from the entire site. As far as the zooarchaeological remains are concerned, the excavators report the remains of domestic cattle, buffalo, sheep/goat and pig, with cattle dominating the assemblage. A large percentage of buffalo bones are said to have been found from all occupation periods at Rakhigarhi (Nath 2014), though this claim is not supported by detailed publication. Although we currently have a poor understanding of the agropastoral activities and subsistence strategies at Rakhigarhi, it is very possible that a range of both *rabi* and *kharif* crops were grown around the settlement as the area receives both summer and winter rainfall.

Ceramic evidence and cultural material

Detailed knowledge about the types of ceramic industries that operated at Rakhigarhi is lacking. Observations suggest that both 'Classic Harappan' and regionally- or locallyproduced 'Haryana Harappan' pottery are present; but their relative abundances over time are not known. Both types of pottery are found in high ubiquity in contexts from Mound-4 in Rakhigarhi, but observations made in the field suggest the regional 'Haryana Harappan' pottery outnumbers the 'Classic Harappan' pottery (Petrie, pers. comm.) No kilns or largescale production workshops have been reported from the site.

A range of non-local cultural material such as beads made of semi-precious stone have been reported from different mounds at Rakhigarhi (Nath 2007, 2014, 2018). The absence of raw material sources for stone in the region and the presence of marine shell so far inland (Law 2011) suggests that the settlement was embedded in regional and longdistance networks for the sourcing of raw and finished stone and mineral products.

Provenance studies for a variety of stones and minerals at the site have revealed that materials such as steatite, carnelian, agate, lapis lazuli, sandstone and quartzite were possibly being sourced from present-day Khyber Pakhtunkwa, northern Gujarat, northern Afghanistan, Sulaiman ranges and Kaliana Hills in southern Haryana (near Khanak), respectively (Nath 2014). Although the studied stone and mineral assemblage was small, it demonstrates that resource acquisition networks of residents at Rakhigarhi were similar to those at Harappa, Mohenjo-daro, and Dholavira (Law 2011; Nath 2014).

4.2. Samples from collections

4.2.1. Mohenjo-daro (MD)

One of the most famous sites of the Indus Civilisation, Mohenjo-daro is situated on the Indus River floodplain in Sindh (Pakistan), a few kilometres west of the Indus River. Pottery samples from Mohenjo-daro analysed for this thesis, but they were acquired from legacy collections, and the find-spots of the pottery are unknown, so only a brief background of the site is provided here.

Site environment and background

Lying in an area of high aridity and high temperatures, Mohenjo-daro experiences a mean average rainfall of 116 cm annually (Goudie 1977). The site encompasses over 100 ha (Jansen 1994), with an estimated population of 40,000 inhabitants (Wright 2010:107-110). Excavations have demonstrated that there was a build-up of at least seven metres of alluvial silt immediately around the site (Jansen 1994). Today, the preservation of the archaeological site is severely threatened by rising ground water and salinization, problems that possibly led to the settlement's abandonment in the Indus period (Jansen 1994).

The site is made up of two main mounds. Recent discoveries suggest that the overall occupied area may have covered c.250 hectares (Jansen 1994), making it the largest protohistoric urban settlement in the subcontinent. The higher mound is comprised of high brick platforms topped by fired-brick structures, some of which are unusual buildings such as the 'Great Bath', the Granary/Great Hall/Warehouse, the 'College of Priests', the 'Pillared Hall', and what was originally termed a later-period Buddhist Stupa, but is likely an Indus structure that contained votive objects and offerings (Verardi 1984; Verardi and Barba 2010; Petrie 2013). A mudbrick fortification wall with at least one gateway surrounded at least part of this mound, and although many of these buildings may have been elite structures, several contained evidence for craft activities, including lapidary,

shell, and leather-working (Verardi 1984; Verardi and Barba 2010; Petrie 2013; Green 2018). The lower mound consisted of habitation and workshop areas that were also raised on brick platforms. At least one building has been identified by the excavator as a palace, and it is likely there are other elite structures present, but this is debated (Verardi 1984; Verardi and Barba 2010; Vidale 2010; Petrie 2013; Green 2018).

Excavation, periodisation and radiocarbon dates

Mohenjo-daro was the focus of several major phases of archaeological research during the twentieth century (1920s, 1930s, 1940s and 1950s) and has seen the most extensive investigation of any Indus settlement (Jansen 1994; Petrie 2013; Green 2018). The site and the excavations have been discussed in detail in several publications (e.g. Marshall 2004[1931]; Mackay 1938; Wheeler 1953, 1966; Dales 1965; Jansen and Urban 1984, 1987; Dales and Kenoyer 1986; Jansen 1994, 1999; Wright 2010).

Mohenjo-daro has been dated to the Mature Harappan period, with suggestions of both 'Early' and 'Late' Mature occupation (Jansen 1994). Previous excavators mention at least six phases of occupation (Mackay 1938). Due to the nature of excavations in the early twentieth century, however, radiocarbon dates are sparse. It has been proposed that the settlement was not occupied before the Mature Harappan period, with foundations built on platforms for flood defence, but it is possible that there were pre-urban phases of occupation that were not reached during excavations because of the high modern water table (Dales and Kenoyer 1986; Jansen 1994; Petrie 2013). Later remains are characterised by roughly built houses (Mackay 1938), but their dating is uncertain (Jansen 1994). However, the large-scale exposures at Mohenjo-daro set the model for understanding Indus urban layout by revealing houses arranged in coherent blocks separated by wide main streets, narrow side streets, and alleyways (Jansen 1994; Petrie 2013; Green 2018).

Beyond architecture, the material culture excavated from Mohenjo-daro has also set the 'standard' for Indus material culture studies. It is comprised of some of the most wellknown examples of Indus statuary, seal-making, lapidary, and pottery. These include the infamous 'Priest King' statue, the bronze 'Dancing Girl', and examples of ceramic 'stoneware' and other iconic forms of Indus pottery including dish-on-stands, perforated and miniature vessels, and large storage vessels including Black-Slipped Jars (Wright 2010: 249-257). One of the few comprehensive corpuses of Indus pottery published so far includes the pottery from Mohenjo-daro (see Dales and Kenoyer 1986).

Archaeobotanical and faunal evidence

There is minimal information about the archaeobotanical and faunal assemblages from the site. Early excavations at the site did not prioritise the systematic collection of bioarchaeological remains; however, a small assemblage of remains was analysed. Wheat and barley (Mackay 1931[2004]: 586–587; Luthra 1936), and field pea (Wheeler 1968: 84–85) were found at Mohenjo-daro, suggesting that agriculture was carried out using late monsoonal rain and sustained via winter rain and runoff (Petrie and Bates 2017; Petrie et al. 2018). This pattern observed in Sindh was subsequently extrapolated as the norm for other regions (Fairservis 1967, 1971; Petrie 2013). Sewell and Guha (1931) conducted a small assessment of the zooarchaeological remains, citing evidence for the remains of cattle, sheep/goat, pigs, and domestic chicken. Beyond this, nothing is yet known about the agricultural and pastoral practices of the inhabitants of Mohenjo-daro.

4.2.2. Kalibangan (KLB)

Site environment and background

Kalibangan (KLB) (29°25' N; 74°5' E) is located on the southern edge of a now-dried but seasonal river channel within the Ghaggar-Hakra alluvial plain in Hanumangarh district, Rajasthan. Kalibangan receives a mean annual rainfall of 100 cm, with more rainfall in winter (Lal et al. 2003, 2015).

The settlement is c.11.5 hectares in size, and it falls within the category of Indus 'towns' that probably had an important role within the political and economic landscape. The site is comprised of three mounds named KLB-1 to KLB-3. While KLB-1 only has evidence for Early Harappan occupation, KLB-2 and KLB-3 were occupied during the Mature Harappan period. Both KLB-2 and KLB-3 were walled mounds, each with possibly different functions. The excavators claim KLB-2 was made up an elite residential area and a separate area with several brick platforms, which possibly had a ritual function. KLB-3 is a lower eastern mound that comprised of several 'fire-altars' against a wall. This has been interpreted as a 'ritual mound' (Joshi 2015).

The architecture exposed at KLB-2 reveals a grid plan of numerous streets and houses consisting of a courtyard, a well, six or seven rooms, as well as what the excavators call 'fire altars' within at least a single room within a housing complex (Joshi 2015). The material excavated from the mounds at Kalibangan presents much of the well-known Indus cultural material, but the ceramic types used by the pre-urban population continued in use

for at least part of the later period of occupation, suggesting both continuity of the local population and a progressive emulation of non-local material (Petrie 2013: 9).

Excavation, periodisation and radiocarbon dates

Large-scale excavations at Kalibangan were conducted between 1960 until 1969 by Prof. B.B. Lal, Jagat Pati Joshi and the Archaeological Survey of India. A report on the Early Harappan remains was published in 2003 (Lal et al. 2003), and a volume which includes the stratigraphy, radiocarbon dates and descriptions of the objects from the Mature Harappan occupation levels at KLB-2 and KLB-3 was published in 2015 (Lal et al. 2015). Radiocarbon dates from the Mature Harappan occupation levels suggest the site was occupied between 2600-2100 BC (Lal et al. 2015). The dates demonstrate some mixing in contexts; however, they largely point at occupation of the settlement in the Early Harappan and Mature Harappan period, with minimal evidence for occupation in the Late Harappan period (Lal et al. 2015).

Archaeobotanical and faunal evidence

Knowledge about plant use at Kalibangan is very limited and there is a lack of published material. Evidence of a ploughed field dating to the Early Harappan period is often cited as early evidence for plough agriculture in this region (Lal et al. 2003; Joshi 2015: 714). The plough marks cross each other at right angles, which is argued as evidence for 'mixed crop cultivation' (Joshi 2015: 714). Reports mention the growing of winter crops, such as barley, wheat, peas, and horse gram (Joshi 2015: 714), but there is not published quantified evidence. Similarly, the zooarchaeological assemblage at Kalibangan has not been adequately studied or published. The site report mentions a large proportion of cattle bones compared to goat, pig and fowl, and the presence of small and large hooks that were used for fishing (Joshi 2015).

Other evidence for subsistence practices include stone saddle querns, mortars and pestles that were probably used during food preparation. The excavators noted the presence of grains in large jars at Kalibangan, as well as an underground lime-plastered pit that was interpreted as a granary (Joshi 2015). The multiple hearths and ovens found inside the residential structures are said to be reminiscent of modern-day *tandoors* that are used for baking flat bread (Joshi 2015). The excavators also mention evidence for the presence of ceramics and other tools that are used in modern-day Indian cooking to roll and roast *chapattis* made from cereals (Joshi 2015: 824).

4.3. Sites outside the Indus Civilisation (Sultanate of Oman)

4.3.1. Stone Tower I, Salut (STI)

Site environment, excavations and periodisation

Stone Tower I, Salut (STI) is a 22 m circular stone tower near the modern town of Bisyah, central Oman. An ancient agricultural oasis, Salut is situated in a large valley north of Bisyah, close to w*adi* Bahla and *wadi* Sayfam. The stone tower dates to the second half of the third millennium BC (*c*. 2400-2000 BC), also known as the Umm an-Nar period in southeastern Arabia (Frenez et al. 2016). Near the tower lie the remains of a large Iron Age fortress that are on a rocky outcrop (Avanzini et al. 2005). Although excavations of the Iron Age remains at Salut began in 2004, the Bronze Age tower was excavated by the Italian Mission to Oman at the University of Pisa in collaboration with the Office of the Adviser to His Majesty the Sultan for Cultural Affairs in 2010.

The tower had a central stone-lined well and was surrounded by a large ditch (11- 13m wide and upto 3m deep) with two connecting channels (see Figure 4.16). These features were interpreted as related to water management and storage (Frenez et al. 2016). In a late phase of the tower's occupation (*c*. 2460–2145 BC), waterborne sediments gradually filled the main ditch, which eventually became used as a dumping area (Frenez et al. 2016). Pottery samples for analysis in this thesis were collected from this ditch. For more details about the site and the Umm an-Nar period in southeastern Arabia, see Appendix A.

Archaeobotanical and faunal evidence

Analyses of archaebotanical and faunal material from the site are ongoing.

Indus cultural material at Stone Tower I, Salut

A wide range of Indus and Indus-related pottery types, including utilitarian pottery and specific forms used for food production, presentation, and storage were recovered from the stratigraphic levels associated with the ditch at Stone Tower I, Salut. Indus seals and carnelian beads possibly manufactured with non-Indus raw materials were also recovered from another part of the ditch.

The presence of Indus material culture is attested for at several sites in southeastern Arabia dated to the second half of the third millennium BC (2500-2100 BC), out of which Indus Black-Slipped Jars (BSJs) are one of the most common vessel-type found. BSJs were also found at Stone Tower I, some of which are analysed in this thesis (Figure 4.17). Geochemical analyses of the pottery have indicated that a bulk of these vessels were

produced in areas near or at Mohenjo-daro (Méry and Blackman 1996, 1999, 2004), but they are found more widely in coastal and interior settlements in the Omani peninsula than they are within the Indian subcontinent (Méry and Blackman 2004). Indus BSJs are unmistakably transport or storage vessels; shaped not unlike amphoras from classical antiquity (Méry & Blackman 2004). Although there are variations in size and capacity (they range between 19-22cm in external rim diameter, and estimated volumes vary from 30-80 litres), most BSJs appear to have been made to specific orders to meet transport needs; their bases are tapered which makes them easy to stack and ship by river or sea, and they are slipped on the both interior and exterior surfaces (Kenoyer 1998; Méry and Blackman 1996, 1999, 2004). It has been suggested that Indus BSJs were used to transport a specific foodstuff from the Indus Valley to south eastern Arabia (Kenoyer 1998; Méry & Blackman 2004); however, it is possible that upon their arrival in the Omani peninsula they were emptied and refilled with different foodstuffs or for the transport of other commodities. It is also possible that BSJs had secondary or multiple uses and were used to store a variety of foodstuffs.

Emerging evidence from the site and other sites in the area suggests that merchants and craftsmen from the Indus region may have been living and working in interior Oman during the second half of the third millennium BC. It is thus possible that that the interaction between Indus communities and eastern Arabia was much more extensive than previously thought (Frenez et al. 2016).

Figure 4.16: Plan of the tower and location of Indus objects at Stone Tower I, Salut (STI) (reproduced from Frenez et al. 2016: 110).

Figure 4.17: Examples of Indus Black-Slipped Jars found at Stone Tower I, Salut (STI) (after Frenez et al. 2016: 111).

4.3. Chapter Summary

This chapter has reviewed and summarised relevant contextual information for every site investigated in this thesis. The information provided makes it possible to integrate the available bioarchaeological and cultural evidence with the results from lipid residue analysis from pottery fragments. The integration of results provides a more nuanced interpretation of food-production/acquisition, processing and consumption at different sites, as will be demonstrated in Chapters Seven and Eight.

Chapter Five

Materials and methods

This chapter describes the rationale behind the sampling strategies and analytical methods used in this thesis. Section 5.1 covers the rationale behind selection of pottery samples and specific details of the collection of pottery from sites. Section 5.3, Section 5.4 and Section 5.5 provide the lipid extraction protocol, study design, and details of the instrumental analyses conducted, namely Gas Chromatography, Gas Chromatography-Mass Spectrometry and Gas Chromatography-Combustion-Mass Spectrometry, respectively. Details of the statistical tests performed are provided in Section 5.6 and details of data reproducibility in Section 5.7.

5.1. Pottery Selection

Pottery from Indus sites in northwest India and Sindh, and Umm an-Nar and Indus pottery from the Sultanate of Oman were selected for analysis. Samples of pottery were collected from selected sites in 2015 and 2017. Three groups of samples were collected, representing the 'worst-' and the 'best-' case scenarios for pottery sampling for organic residue analysis. The first group of sherds (n=10), 'Group I', came from legacy collections and had no contextual information associated with them except for which site they were collected from. The sherds had been washed, and were likely exposed to a variety of synthetic contaminants, such as plasticisers from being stored in plastic bags for several years, skin lipids from individuals handling them, and other products like adhesives, nail varnish, marker pens, etc. that are used to process pottery after collection. It is also likely they were susceptible to the vicissitudes of being exposed on the surface of a site for an indeterminate period.

The second group of sherds (n=135), 'Group II', were excavated between 2008-2014 by different excavation projects. Although mostly recovered from well-stratified contexts, these sherds had been washed and processed, and touched and handled by multiple individuals. Most sherds were written on with marker pens and stored in plastic bags; and some had traces of adhesive on them. Care was taken to avoid drilling portions of the sherd that had writing on them and areas with adhesive were avoided.

The third group of sherds (n=48), 'Group III' were collected from well-stratified contexts and only handled by individuals wearing nitrile gloves. They were wrapped in aluminium foil in the field and not washed, only cleaned with sterile equipment prior to drilling. This protocol minimised the potential unknown synthetic contaminants that the pottery was exposed to. Additionally, sediment samples from the surrounding context and dirt adhering to some of the sherds were also collected for control $(n=7)$, so that comparisons of lipid profile from sediment and potsherds could be conducted. The details of the different sample types are provided in Table 5.1.

Table 5.1: Number of potsherds analysed from Groups I, II and III with site names and site codes.

5.1.1. Group I: Pottery from collections

The precise history of the samples analysed from Group I is uncertain. It is presumed the samples were collected from these sites between the 1950s and 1970s and brought to Cambridge where they were stored and used as part of a private teaching collection. The potsherds were stored in bags labelled 'Kalibangan' and 'Mohenjo-daro'. Although it was not possible to confirm the provenance of the samples, they closely matched known examples of pottery from both sites (Cameron Petrie & Danika Parikh, pers.comm; also Dales and Kenoyer 1986; Lal et al. 2003). The uncertain context, limited information about collection and treatment of the pottery, and lack of supporting faunal or botanical data available relating to this group of samples represented the 'worst-case'-scenario of

sampling options for lipid analysis. It was not possible to collect specific data about the preservational environment immediately surrounding the sherds, but broadly speaking, both sites are located in areas of high aridity and experience seasonal rainfall. At the same time, all the potsherds had been stored in poor-quality plastic bags for several years, and 6 out of 10 potsherds samples from Group I had been labelled with varnish and marker pen. It was thus expected that lipid extracts from these samples would also contain high concentrations of synthetic compounds. Five samples were selected from a plastic bag labelled 'Kalibangan' (KLB), and 5 samples were selected from a bag labelled 'Mohenjodaro' (MD) Details are provided in Tables 5.2 and 5.3.

5.1.2. Group II: recently-excavated samples

Group II samples were collected from: Alamgirpur (ALM), Masudpur VII (MSDVII), Masupdur I (MSDI), Farmana (FRN) and Rakhigarhi (RGR). The potsherds from these sites were excavated between 2008-2014. All these sites lie within an alluvial plain and experience seasonal variation in temperature and rainfall (see Chapter Four). Assessing the potential for these samples to undergo routine lipid residue analysis was essential since most potsherds available for organic residue analysis in archaeology come from similar collection and storage conditions.

Pottery fragments were collected from Group II based on four key parameters: chronological period, shape of vessel, context, and location of sherd on vessel. Following these parameters, sherds were generally selected from contexts that had radiocarbon dates associated with them (when available), and/or from contexts that were indicative of occupational surfaces, such as fills of floor surfaces, deliberate fills or hearth contexts. Pottery from pits or dumps were avoided, however, vessel fragments of interest were chosen even if they originated from these contexts (e.g. perforated vessels). Potsherds likely to have been used in the cooking of foodstuffs, for example, pots/jars versus bowls or dishes, were selected. Rims or body sherds from upper parts of vessels were selected where possible, as these have previously been linked to high abundances of absorbed lipid (Charters et al. 1993). All except a single potsherd showed minimal signs of post-firing exposure to heat with no extensive exterior soot marks.

5.1.2.1. Alamgirpur (ALM)

Rims of 15 vessels (9 jars of different sizes, 3 necked jars 2 dishes), and 1 body sherd were selected from floor and fill contexts from Trench SC (Section Cutting) (Figure 5.1). Detailed descriptions of individual fragments are provided in Appendix B. Radiocarbon

dates from these deposits range from *c*. 2130-1942 BC to *c*.1904-1750 BC, suggesting that they span the transition between the Late Mature Harappan (LMH) and into the Late Harappan (LH) periods, and were obtained from charred seeds found within these contexts. The pottery selected for analysis primarily dated to the LH period. Details and descriptions of the contexts are provided in Table 5.4. Ten out of fourteen samples were selected for further analysis via GC-c-IRMS.

Figure 5.1: Section drawing of the ALM SC trench. Samples from contexts 114, 117, 119, 121, 122, 124 and 125 were analysed (reproduced from Singh et al. 2013: 37).

5.1.2.2. Masudpur VII (MSDVII)

Rims of 29 vessels (14 jars of different sizes, 5 ledged jars, a perforated bowl) and body sherds of 6 perforated vessels were selected from fill contexts from trenches YA2 and YB1 at Masudpur VII (Figure 5.2). Detailed descriptions of individual fragments are provided in Appendix B. Vessel fragments from Masudpur VII were selected primarily from Late Harappan (LH) contexts (*c*. 1900-1700 BC), except four vessels which were from Early Mature Harappan (EMH) (*c.*2500-2200 BC) contexts. Descriptions of the contexts and chronological information for each sample is provided in Table 5.5. Twenty-three of the samples were selected for analysis via GC-c -IRMS.

Figure 5.2: Stratigraphy of trench YA2 (top) and YB1 (bottom) at Masudpur VII, with red dots marking the contexts from where pottery was selected for analysis. (Courtesy Cameron Petrie).

Table 5.2: Details of samples selected from Kalibangan

Table 5.5: Details of samples selected for analysis from Masudpur VII

Table 5.6: Details of samples selected for analysis from Masudpur I.

S. no.	Sample ID	Vessel form/size	Trench and context no.	Context description	Chronological period
$\mathbf{1}$	FRN02	body	1C8-9021	Complex 3? Depth 65-77cm	EMH
$\overline{2}$	FRN04	small jar	3Y17-9023	NW Area. Depth 2.57m	EMH
3	FRN08	small jar	1G7-9022	Inside Complex 3. Depth 58-60cm	EMH
4	FRN09	medium jar	1G7-9022	as above	EMH
5	FRN10	large jar	1G7-9022	as above	EMH
6	FRN11	perforated vessel	1G7-9022	as above	EMH
7	FRN12	large jar	1D5-8008	Complex 3. Depth 40-56cm	EMH
8	FRN13	small jar	1B3-8004	Main Street. Depth 33-40cm	EMH
9	FRN14	large jar	1G3-8007	Outside Complex 4. Depth 29-46cm	EMH
10	FRN15	small jar	1D5-8007	Complex 3. Depth 39-56cm	EMH
11	FRN16	perforated vessel	1D5-8007	as above	EMH
12	FRN17	perforated vessel	1D5-8007	as above	EMH
13	FRN18	small jar	1B3-8004	Main Street. Depth 33-40cm	EMH
14	FRN19	medium jar	1G3-8007	Outside Complex 4. Depth 29-46cm	EMH
15	FRN20	small jar	1G3-8007	as above	EMH
16	FRN21	small jar	1E3-8005	Complex 3. Depth 35-45cm	EMH
17	FRN22	large jar	1E3-8005	as above	EMH
18	FRN23	medium jar	1E3-8005	as above	EMH
19	FRN24	small jar	1E3-8005	as above	EMH
20	FRN25	medium bowl	1B5-8002	Complex 3. Depth 41-44cm	EMH
21	FRN26	jar	1C6-8003	Complex 3. Depth 41-43cm	EMH
22	FRN27	body	1E5-8006	Complex 3. Depth 39-56cm	EMH
23	FRN28	perforated	1D3-8008	Complex 3. Depth 35-47cm	EMH
24	FRN29	large bowl	1D3-8008	as above	EMH
25	FRN30	body	1B5-8004	Pit in Complex 3. Depth 41-43cm.	EMH
26	FRN31	large bowl	1B5-8002	Complex 3. Depth 41-44cm	EMH
27	FRN32	perforated	1D3-8008	Complex 3. Depth 35-47cm	EMH
28	FRN33	medium jar	1G4-8005	Lane No. 2. Depth 36-48cm	EMH
29	FRN34	small jar	1G3-8007	Outside Complex 4. Depth 29-46cm	EMH
30	FRN35	small jar	1E3-8005	Complex 3. Depth 35-45cm	EMH

Table 5.7: Details of samples selected for analysis from Farmana.

Table 5.9: Details of samples selected for analysis from Rakhigarhi.

5.1.2.3. Masudpur I (MSDI)

Thirty-one vessel fragments from Masudpur I were selected from trenches XA1 and XM2 (Figure 5.3). Samples included rims of jars of different sizes ($n = 22$), necked jars ($n = 7$) and body sherds of perforated vessels $(n = 2)$. Detailed descriptions of individual fragments are provided in Appendix B.

Samples were selected primarily from contexts dated to the Late Mature Harappan period (LMH) (c. 2287-2040 cal. BC and c. 2025-1888 cal. BC) and two vessels from an Early Mature Harappan (EMH) (c.2431-2141 cal. BC) context. A description of the contexts and chronological periods for each sample is provided in Table 5.6. Thirteen of the thirty samples were selected for GC-c-IRMS analysis.

Figure 5.3: Stratigraphy of trench XM2 (top) and XA1 (bottom) at Masudpur I, with red dots marking the contexts from where pottery was selected for analysis. (Courtesy Cameron Petrie).

5.1.2.4. Farmana (FRN)

Thirty vessels from Farmana were selected for analysis. Vessel forms included differentsized jars (n=15), necked jars (n=2), perforated vessels (n=4) and bowls (n=3) (Table 5.7). Detailed descriptions of individual fragments are provided in Appendix B. Most vessels were black-on-red vessels, however, other surface treatments included 'red wash', 'mud applique' and 'chocolate slip' varieties (cf. Uesugi 2011). 26 vessels were classified as 'Haryana Harappan' and 4 vessels were classified as 'Classic Harappan' (Uesugi, pers. comm.).

Vessel fragments from Farmana were selected in March 2017 from Deccan College, Pune after obtaining permission from Prof. Vasant Shinde, and with the assistance of research scholar Yogesh Yadav. Samples from Farmana were selected from a large structural complex (Structural Complex No. 3), outside Structural Complex No. 4, the Main Street outside Structural Complex No. 3, and from area 3Y17. Within Structural Complex No. 3, sample find locations ranged from the 'central courtyard' to small rooms that were possibly used for storage. Detailed descriptions of the contexts that sherds were selected from were unavailable, but the approximate location within a trench or structure and depth are shown in Table 5.7. As the pottery comes from different depths, it is likely that they are from different chronological phases during the occupation of the structural complex. Despite this chronological uncertainty, it is likely that the vessels analysed were used and discarded during the Early Mature Harappan period (EMH) as all radiocarbon dates from Structural Complex No. 3 fall within this period (Shinde et al. 2011). The location of vessel fragments analysed are depicted in Figure 5.4. Seven of the thirty samples were selected for GC-c-IRMS analysis.

Figure 5.4: Locations of selected sherd samples from Farmana. Samples analysed via GC-MS analysis are represented by filled black circles, and those further analysed by GC-c-IRMS analysis are represented by black circles outlined by red. The samples represented by blue circles had low lipid concentrations and were not analysed in detail (After Shinde et al. 2011: 97).

Figure 5.5: Photographs from the hearth structure with *in situ* storage vessel from Trench 1.B (top) and mud-brick platform (Trench 1.E) (bottom left) and storage bin feature (Trench 1.F) (bottom right) at Rakhigarhi. (Courtesy Yogesh Yadav).

Figure 5.6: Examples of 'Classic Harappan' vessels studied in this thesis. A: FRN14, B: FRN26, C: RGR17, D: RGR30.

5.1.2.5. Rakhigarhi (RGR)

Thirty rim sherds from Rakhigarhi were selected for lipid analysis, including sherds of jars of different sizes (n= 16), ledged jars (n= 5), dishes (n=2), necked jars (n=2), one globular jar and body sherds of 4 perforated vessels (Table 5.8). Detailed descriptions of individual fragments are provided in Appendix B. Most vessels were slipped with red, or dark red slips and black-painted. Other surface treatments included 'mud applique' or rusticated and 'chocolate-slipped', or dark-slipped varieties. Twenty-five vessels were classified as 'Haryana Harappan', while 5 vessels were 'Classic Harappan' (Jadhav, pers.com; Figure 5.6).

Samples from Rakhigarhi were selected from areas on Mound RGR-4 excavated by Deccan College in 2013-14 and 2014-15, specifically from Trenches 1.B, 1.E and 1.F. The stepped trenches uncovered structural remains and a series of fills and pits that possibly represent episodes of ephemeral occupation and dumping. While the dating of the contexts from which the pottery samples are collected is uncertain, a stylistic assessment of the pottery suggests that they were manufactured, used and discarded during the Mature Harappan period.

Pottery samples analysed from Trench 1.B were possibly associated with a hearth structure and an *in-situ* storage jar, along with a large collection of terracotta cakes and lumps. Samples collected from Trench 1.E come from fills above a mudbrick platform and from within storage bins located next to the platform. Finally, samples from Trench 1.F come from within filling events inside a storage bin, what was called a 'granary' by the excavators. Unfortunately, precise information about several contexts is unavailable (see

Table 5.8). Seven vessel fragments were analysed via GC-c-IRMS to provide differentiation between degraded animal fats.

5.1.3. Group III: freshly-excavated samples

Group III samples were considered the 'best-case scenario' for sampling potsherds for organic residue analysis as all the steps from excavation to extraction were controlled. Freshly-excavated samples were recovered from three sites: Khanak (KNK) and Lohari Ragho I (LHRI) in Haryana, northwest India and Stone Tower I, Salut (STI) in the Sultanate of Oman. The details of each site are provided in Chapter Four.

Potsherds were chosen directly from the site during excavation and selected from contexts of interest. Sediment samples up to 2-4g from the locations from where sherds were retrieved, or from soil adhering to the sherd were sampled. Care was taken to minimise the potsherds' exposure to contaminants: the pottery was not washed but only cleaned using nitrile gloves before being photographed and wrapped in aluminium foil. Samples were handled with nitrile gloves and wrapped in aluminium foil to avoid any contact with human fingers or plastics.

5.1.3.1. Lohari Ragho I (LHRI)

The samples from Lohari Ragho I were selected from contexts associated with floors and hearths; the shape of vessel fragments was also a determining factor for selection. The vessels were selected from contexts that likely date to the Early Mature Harappan (EMH) and Late Mature Harappan (LMH) on the basis of pottery typologies. Radiocarbon dates from the site confirmed that most of the vessels selected come from contexts dated to the Early Mature Harappan (EMH) (2500-2200 BC) period and three vessels come from a context dated to the Late Mature Harappan (LMH) (2200-1900 BC) period. Description of the contexts are provided in Table 5.9.

Rims of twenty-five vessels were selected for lipid analysis, as well as sediment samples adhering to the surface of potsherds $(n=2)$, and terracotta cakes $(n=2)$. Vessel forms included rims of globular jars ($n = 3$; 2 were from complete vessels), necked jars $(n=3)$, jars of different sizes $(n=14)$, perforated jars $(n = 2)$, a ledged jar, a dish and a base fragment (Table 5.9). Detailed descriptions of individual fragments are provided in Appendix B. Twelve of the twenty-six ceramic samples were selected for analysis via GCc-IRMS.

5.1.3.2. Khanak (KNK)

Pottery and sediment samples were selected from Trench A05 from the 2016 excavations at Khanak. Nine pottery samples and two sediment samples were selected from fill contexts, a pit with evidence for pyrotechnical waste (burnt clay lumps) which contained a smashed vessel at the bottom of the pit, and a floor surface. Description of the contexts that sherds were selected from are provided in Table 5.10.

From the ceramic assemblage, jars of different sizes (n=8) and a bowl were analysed (Table 5.10). Detailed descriptions of individual fragments are provided in Appendix B. Samples in this thesis mostly date to the Early Mature Harappan (EMH) period, with one sample possibly dating to the Early Harappan period and four samples dating to the Late Mature Harappan (LMH) period.

Four of the potsherds were from semi-complete vessels and appeared to contain visible residues inside. Upon closer examination, it was apparent that these residues were encrustations of sediments and intrusive organic material, and likely unrelated to the actual contents of the vessels. Two sediment samples from soil adhering to two sherds were analysed via GC-MS in order to compare their lipid profiles with those obtained from the vessels they were found in. Three ceramic samples with relatively high lipid concentrations were selected for GC-c-IRMS analysis.

5.1.3.3. Stone Tower I, Salut (STI)

Pottery and sediment samples from the excavations at the Stone Tower I (STI) at Salut, near Nizwa, Sulatate of Oman were collected in December 2015. The excavation was headed by the Italian Archaelogical Mission to Oman in collaboration with the Office of the Adviser to His Majesty the Sultan for Cultural Affairs.

Eleven potsherds were analysed from Stone Tower I, Salut. These included the rims of 5 Arabian Umm an-Nar vessels and rims, bases and body sherds of 6 Indus Black-Slipped Jars (BSJs) (see Chapter 4 and Appendix A, Table 5.11). Potsherds were chosen from a context that contained evidence of ephemeral occupation, demonstrated by hearths in sandy fills and concentrations of pottery. Several sediment samples were collected, out of which three were analysed. Seven of the eleven ceramic samples were selected for analysis via GC-c-IRMS. Descriptions of the pottery and lipid yields are provided in Section 6.1.3.1 and GC-c-IRMS results are provided in Appendix A.

5.1.4. Vessel forms

Of the entire analysed assemblage, vessel profiles could be determined from most preserved rim or body fragments. Based on these, vessels were characterised as very small or small jars, medium jars, large jars, jars (of unknown diameter), perforated vessels, necked jars, ledged jars, globular jars, bowls, dishes or goblets. Some base and body fragments (n=4) could not be reliably characterised. The size categorisations were done following Dales and Kenoyer (1986), but these measurements reflect rim sizes and may not accurately represent the size of the overall vessel, or its volumetric capacity; they merely provide a sense of the shape of the mouth of the vessel. The rim sizes and vessel categorisation of every potsherd are provided in Appendix B.

5.1.5. Documentation

The exterior and interior surfaces, and profiles of all potsherds were photographed prior to, and after drilling. Photographs of selected sherds are provided in Appendix B.

S. no.	Sample ID	Vessel form	Trench and context no.	Context description	Chronological period
$\mathbf{1}$	LHR03	globular jar	EA-511	Fill on surface with complete vessels and hearth	LMH
$\overline{2}$	LHR06	globular jar	EA-511	as above	LMH
3	LHR07	globular jar	EA-511	as above	LMH
4	LHR08	terracotta cake	EA-520	Occupation deposit on top of mudbrick floor/platform	EMH
5	LHR09	jar	EA-520	as above	EMH
6	LHR10	large jar	EA-520	as above	EMH
7	LHR11	large jar	EA-520	as above	EMH
8	LHR12	perforated jar	EA-520	as above	EMH
9	LHR13	jar	EA-520	as above	EMH
10	LHR14	perforated jar	EA-520	as above	EMH
11	LHR15	jar	EA-520	as above	EMH
12	LHR16	terracotta cake	EA-520	as above	EMH
13	LHR17	perforated jar	EA-520	as above	EMH
14	LHR ₂₀	necked jar	EA-520	as above	EMH
15	LHR21	medium jar	EA-520	as above	EMH
16	LHR22	perforated jar	EA-520	as above	EMH
17	LHR23	jar	EA-520	as above	EMH
18	LHR24	ledged jar	EA-520	as above	EMH
19	LHR25	jar	EA-525	Second deposition within hearth fill	EMH
20	LHR26	jar	EA-520	Occupation deposit on top of mudbrick floor/platform	EMH
21	LHR27	small necked jar	EA-520	as above	EMH
22	LHR29	jar	EA-522	Fill abutting various structures	EMH
23	LHR32	jar	EA-541	Occupation deposit w/ bioturbation brick fragments	EMH
24	LHR33	dish	EA-541	as above	EMH
25	LHR36	body	EA-553	Fill/occupation?	EMH
26	LHR37	sediment	EA-565	Ashy fill	EMH
27	LHR38	perforated jar	EA-553	Fill/occupation?	EMH
28	LHR40	large jar	EA-553	as above	EMH
29	LHR41	sediment	EA-524	Hearth fill	EMH

Table 5.7: Details of samples selected for analysis from Lohari Ragho I.

Table 5.8: Details of samples selected for analysis from Khanak

Table 5.9: Details of samples selected for analysis from Stone Tower I, Salut

S. No.	Sample ID	Vessel form	Context description	Manufacturing characteristics and surface treatment	Calibrated radiocarbon date range
$\mathbf{1}$	ST101	Indus Black- Slipped Jar	207: Sandy fill. Ephemeral occupation deposit	Distnctive curvilinear profile on rim. Thick layers of blackish to brown-purplish slip completely coating the internal and external surfaces. Mica inclusions.	$c.2460-$ 2145 BC
$\overline{2}$	ST102	as above	as above	as above	
3 $\overline{4}$	ST109 ST1018	as above Umm an-Nar jar	as above as above	Base, as above. Arabian Fine Sandy Ware. Rim appears Indus-inspired and could have been produced by an Indus potter	
5	ST1021	Suspension vessel	as above	Fine Red Omani Ware.	
6	ST022	Umm an-Nar jar	as above	Umm an-Nar style Orange Sandy Ware. Shape and rim are very local.	
τ	ST1039	unknown	as above	Very fine Sandy Orange Ware. Made on fast wheel	
8	ST1040	Umm an-Nar jar	as above	Orange light sandy ware. Ledge-shaped rim with potter's mark that appears Indus-inspired.	
9	ST1074	Indus Black- Slipped Jar	as above	Thick layers of blackish to brown- purplish slip completely coating the internal and external surfaces of all sherds. Mica inclusions.	
10	ST1078	as above	as above	as above	
11	ST1079	as above	as above	as above	
5.3. Drilling of pottery and extraction method

5.3.1. Drilling of pottery

Group I and samples from Stone Tower I, Salut were drilled in the McDonald Institute for Archaeological Research at the University of Cambridge. Out of the samples from Group II, the samples from Farmana and Rakhigarhi were drilled at Deccan College, Pune, (in Maharastra, India) and the remaining were drilled at my parents' residence in Noida (Uttar Pradesh, India). Out of the samples from Group III, some potsherds from Lohari Ragho I were drilled during fieldwork at the excavation house, while the rest were drilled in my parents' residence in Noida (Uttar Pradesh, India).

In India, care was taken to procure HPLC grade dichloromethane from local vendors in New Delhi and Pune. All other laboratory and safety equipment was transported from University of Cambridge. Working surfaces were covered in aluminium foil and cleaned with dichloromethane. Sterile nitrile gloves were worn at all times and changed regularly while handling potsherds and drilling. Vessel fragments were never handled with bare hands. All pottery grinding took place under controlled conditions with PPE (Protective Personal Equipment) including eye and face masks.

When sampling archaeological ceramics, the relevant area of the outer surface of the ceramic was cleaned with a modelling drill to remove the influence of exogenous lipids that may have been absorbed the vessel during deposition and post-excavation (cf. Heron et al. 1991; Correa-Ascencio and Evershed 2014). The removal of potentially contaminated external surfaces of ceramic is now an accepted approach that is applied to all samples (Roffet-Salque et al. 2017a). The sherd fragments are then usually ground to a powder in a mortar and pestle that has been washed and cleaned with solvents (e.g. Dunne et al. 2012; Correa-Ascencio and Evershed 2014; Roffet-Salque et al. 2017a). Alternatively, a modelling drill can also be used to remove the internal surface or sub-surface of the sherd which powders the ceramic very finely (Craig et al. 2005, 2011; 2015). The latter approach was used in this thesis as most ceramic fragments were extremely hard and breaking off the potsherd or drilling the exterior surface would have caused irreversible damage to the pottery. Crushing or drilling of the ceramic increases the surface area of each individual particle, increasing the rate of reaction of the ceramic particles with solvents used for lipid extraction. About 2-3g of pottery powder was collected which provided enough material for repeat analysis if necessary. Excess powder is stored in the freezers in the Pitt-Rivers Laboratory.

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For samples from Group III, 2-3g of soil adhering to the potsherds was collected using tweezers cleaned with dichloromethane. This is was done in order to compare lipid yields of the sediments versus the sub-surface powder of the sherds. 2-3g of soil was also collected from specific contexts of interest (such as hearths).

Extensive precautions were taken to prevent contamination from environment and cross-contamination during drilling of pottery. Abrasive tungsten drill bits were cleaned prior to use with dichloromethane via sonication. Drill bits were changed between the drilling of the interior surface and sub-surface of the potsherd samples and between samples.

5.3.2. Lipid Extraction

5.3.2.1. Acidified methanol extraction

The direct acidified methanol extraction protocol (Correa-Ascencio and Evershed 2014) was used to extract lipids from all potsherds in the thesis. This method was chosen as it has demonstrated the recovery of lipids from contexts that are not conducive to the preservation of organic remains.

The extraction protocol is provided in Appendix C. For Group I and Stone Tower I samples, 10 μg of an internal standard, C_{34} alkane (n-tetratriacontane) was added at the start of the extraction to compare with obtained peak heights. For Group II and III samples, two internal standards were used: C³⁴ alkane (*n*-tetratriacontane) was added at the start of the extraction to assess lipid recovery, and C³⁶ alkane (*n*-hexatriacontane) was added at the end of the extraction process before analysis to quantify lipids.

The acidified methanol protocol enables the direct hydrolysis and methylation of lipids and facilitates high recovery of lipids (Correa-Ascencio and Evershed 2014). Since methyl esters are produced simultaneously during the extraction process, this method takes less time than the conventional solvent extraction method, however, it does result in the loss of compositional information, as triacylglycerols or wax esters (if preserved) may be hydrolysed (Correa-Ascencio and Evershed 2014, See Section 3.4.2).

It is not possible to predict lipid preservation within potsherds, but the burial conditions of the potsherds analysed in this thesis are not optimal for organic preservation. Thus, it was hypothesised that lipid preservation would be low, and the most aggressive lipid extraction protocol would be ideal to gauge the degree of lipid preservation within the pottery. One gram of pottery powder for each sample was lipid-extracted via acidified

methanol extraction, and the remaining pottery powder was reserved for re-examination via solvent extraction if required.

5.3.2.2. Solvent extraction

Six samples yielding high lipid concentrations (FRN11, KNK02, LHR10, MSD1326, MSD3795, RGR24) via the acidified methanol extraction protocol were lipid-extracted using the solvent extraction method (see Appendix D for protocol) to check for the preservation of more complex compounds that may have been hydrolysed via the acidified methanol extraction method.

Importantly, none of potsherds extracted via solvent extraction yielded appreciable quantities of lipid; the only visible peaks in chromatograms were some those of contaminants such as plasticisers. These were thus excluded from further analysis.

Precautions were taken during both types of extraction to avoid any potential contamination from the laboratory environment. Glassware was washed, combusted, and rinsed sequentially with methanol, dichloromethane and hexane before use. Sample blanks were prepared and extracted at the same time as every batch of sample, following the same protocol(s) as outlined in Appendices D and E. Blanks were also used to isolate any contaminants that may have been introduced during the extraction or analytical procedures.

5.4. Instrumental Analyses

5.4.1. Gas Chromatography Flame-Ionisation Detection (GC-FID)

The obtained total lipid extracts (TLEs) from Group I samples and samples from Stone Tower I, Salut, were screened using gas chromatography‐flame ionization detection (GC‐ FID) in order to determine the lipid concentrations. This data was used to identify the internal standard, and screen for possible contaminants, specifically plasticisers, which might otherwise have been included in quantitative analyses. GC-FID analysis was done using an Agilent 7890 A Series chromatograph (Agilent technologies, Cheadle, Cheshire, UK) at the University of York (Table 5.10).

5.4.2 Gas-Chromatography-Mass Spectrometry (GC-MS)

5.4.2.1. Group I and STI samples

GC-MS analysis was carried out on Group I samples and samples from Stone Tower I, Salut (n=21) using an Agilent 7890 A Series chromatograph attached to an Agilent 5975 C Inert XL mass-selective detector with a quadrupole mass analyser (Agilent technologies,

Cheadle, Cheshire, UK) at the University of York (Table 5.10). All samples were initially screened using a splitless injector maintained at 300 °C. The carrier gas used was helium, and inlet/column head-pressure was constant. The column (DB-5 ms) was coated with 5% phenyl-methylpolysiloxane (30 m × 0.25 mm x 0.25 μm; J&W Scientific, Folsom, CA, USA). The oven temperature was set at 50 °C for 2 min, then raised by 10 °C min⁻¹ until 325 °C was reached, where it was held for 15 minutes until the end of the run. The GC column was inserted directly into the ion source of the mass spectrometer. The ionization energy of the mass spectrometer was 70 eV and spectra were obtained in scanning mode between *m*/*z* 50 and 800 (Craig et al. 2007, 2012).

Samples from Group I were also tested for the presence of miliacin using selected ion monitoring in the MS. The oven temperature was set at 50 °C for 1 min, then raised by 20 °C min⁻¹ until 280 °C, then raised at 10 °C min⁻¹until reaching 325 °C, where it was held for 10 minutes until the end of the run. In SIM mode, a first group of ions $(m/z, 189, m/z, 204, m/z, 231, m/z, 425, m/z, 440)$ corresponding to miliacin fragmentation were monitored. After 16 minutes, a second group of ions (*m/z* 57, *m/z* 71, *m*/*z* 85, *m/z* 478, *m/z* 506) were monitored to record the internal standard.

An authentic standard of miliacin was injected in each sample run to monitor the retention time and confirm the presence of this compound. Hexane 'blanks' were injected regularly throughout the sequence to monitor for any carry-over between analytical runs.

After GC-MS analysis, seven samples from Group I and Stone Tower I, Salut with high lipid concentrations were divided into two. An aliquot was reserved for GC-c-IRMS analysis; another aliquot was derivatized with *N*,*O-bis*(trimethylsilyl)trifluoroacetamide (BSTFA to derivatize neutral lipids in the extract to trimethylsilyl (TMS) esters (See Appendix E for protocol) (Craig et al. 2007, 2012).

5.4.2.2. Group II and Group III samples

GC-MS was carried out using an Agilent 7890 B Series Gas Chromatograph attached to an Agilent 5977 B Mass Spectromer with a quadrupole mass analyser (Agilent technologies, Cheadle, Cheshire, UK) at Scientific Research, British Museum (Table 5.10). All samples were initially screened using a split/splitless injector in splitless mode which was maintained at 300 °C. The GC carrier gas was helium, configured at a constant flow rate of 1ml min⁻¹. The column (HP-5MS) was coated with 5% phenyl-methylpolysiloxane (30 m × 0.25 mm x 0.25 μm; Agilent technologies, Cheadle, Cheshire, UK). The oven temperature was set at 50 °C for 2 min, then raised by 10 °C min−1 until 325 °C was

reached, where it was held for 15 min until the end of the run. The ionization energy of the mass spectrometer was 70 eV and spectra were obtained in scanning mode between m/z 50 and 800.

After GC-MS analysis, three samples (LHR10, KNK02, MSD329) with high lipid concentrations were divided into two. An aliquot was reserved for GC-c-IRMS analysis; another aliquot was derivatized with *N*,*O-bis*(trimethylsilyl)trifluoroacetamide (BSTFA to derivatize neutral lipids in the extract to trimethylsilyl (TMS) esters (See Appendix E for protocol) (Craig et al. 2007, 2012).

Compounds revealed in GC-MS profiles were present as methyl esters, TMS esters and ethers (not detected), or underivatised in the case of *n*-alkanes.

5.4.2.3. Interpretation

Data were acquired using HP Chemstation and Masshunter software (Agilent Technologies). Phthalate concentrations were calculated by using EIC mode and monitoring for an abundant *m/z* 149 ion. Quantification of lipid was based on the known amount of internal standard $(C_{36}$ alkane; n-hexatriacontane) introduced during sample preparation. Compounds were identified by comparison with the National Institute of Standards and Technology (NIST) mass spectra library (v. 2.0) or with reference to external sources such as The Lipid Library [\(www.lipidlibrary.aocs.org\)](http://www.lipidlibrary.aocs.org/). This method enabled the identification of compounds however, the results were not trusted at face-value as the library is not designed to identify heavily fragmented or degraded compounds. The mass spectra of compounds were often manually checked to assign their origin.

Group or site	GC-FID	$G C$ -MS	GC - c -IRMS
Group I	University of York	University of York	University of York
Stone Tower I, Salut	University of York	University of York	University of York
Group II	Scientific Research,	Scientific Research,	University of York
	British Museum	British Museum	
Group III	Scientific Research,	Scientific Research,	University of York
	British Museum	British Museum	

Table 5.10: Details of where different analyses in this thesis were conducted. All analyses were performed by the author.

5.4.3. Gas Chromatography-combustion-Isotopic Ratio Mass Spectrometry (GC-c-IRMS)

Ninety-one samples were analysed with a GC-c-IRMS system comprising of an Isoprime 100 (Isoprime, Cheadle, UK) linked to a Hewlett Packard 7890B series Gas Chromatograph (Agilent Technologies, Santa Clara, CA, USA) with an Isoprime GC5 interface (Isoprime, Cheadle, UK), according to previously described protocols (Lucquin et al. 2016a) at the University of York (Table 5.10). The results from the analyses are reported relative to an international scale (VPDB). Replicate measurements of the sample and a mixture of fatty acid methyl esters (FAMEs) with δ^{13} C values comparable to international standards were used to determine instrument precision $(\pm 0.3\% \text{ sE})$ and accuracy $(\pm 0.5\%$ SE). Values were further corrected to account for the methylation of the carboxyl group (Lucquin et al. 2016a). Calibration and corrections are provided in the repository (see Section 5.6).

Reference fats from South Asia were not available except for two dairy references obtained from previous publications (Craig et al. 2005). Following previous publications (e.g. Evershed et al. 2008; Dunne et al. 2012), $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values obtained from fatty acids were compared to modern reference animal fats from Africa (Dunne et al. 2012, United Kingdom (animals raised on a pure C3 diet) (Dudd and Evershed 1998), Kazakhstan (Outram et al. 2009), Switzerland (Spangenberg et al. 2006) and the Near East (Gregg et al. 2009).

5.5. Statistical Analysis

All statistical tests were performed in R (version 3.4.1). The comparison of lipid concentrations from vessels from different sites was performed via various statistical tests. Comparisons were conducted between vessel forms from sites within Groups I, II and III and between vessel forms from sites across Groups I, II and III.

Lipid concentrations for most groups were not normally distributed. Thus, nonparametric tests such as the Kruskal-Wallis and Mann-Whitney-U tests were used for comparing multiple groups or two groups, respectively. One-way ANOVA tests were used for comparing lipid yields from multiple groups of vessels when data was normally distributed.

Differences in the distribution of Δ^{13} C values and 1) vessel-types, 2) chronological periods, and 3) climatic periods between sites were also conducted using non-parametric (Kruskal-Wallis) tests.

5.6. Reproducibility

To enable re-use of the data and improve reproducibility and transparency (cf. Marwick 2017; Marwick et al. 2018), the GC-MS data, analysis files, GC-c-IRMS calibration and correction files, and R code for statistical analysis and visualisations in Chapters Six and Seven are available at the University of Cambridge data repository at [https://doi.org/10.17863/CAM.42339.](https://doi.org/10.17863/CAM.42339) All the figures and statistical tests presented in this thesis can be reproduced with the data and code in the repository. The next chapters provide the results of the lipid and compound-speciific isotopic analysis.

Chapter Six

Lipid yields in archaeological pottery from South Asia and Sultanate of Oman

This thesis presents the first large-scale investigations into lipid residues in South Asian archaeology. This study has necessitated an examination of the probability of preservation of lipid residues recovered from different contexts. As mentioned in Chapter Three, several factors influence the preservation of lipids within ceramics. The following questions have been explored based on a comparative study of lipid concentrations of archaeological pottery from private collections (Group I), recently excavated (Group II), and freshly excavated pottery samples (Group III):

- Are sufficient quantities of lipids preserved within potsherds from sites in northwest India, Sindh (Pakistan) and the Sultanate of Oman?
- What is the effect of post-excavation treatment (washing of sherds) on lipid yield?
- To what degree does potential contamination from storage environment and/or adhering sediment affect the interpretation of the lipid profiles from vessels?

These questions address the viability of conducting future studies in lipid residue analysis in the subcontinent.

6.1. Results: Lipid Yields

This section presents the lipid concentrations obtained from vessels from Groups I, II and III. Lipid yields obtained from each site within groups are provided, and the extent of contaminants within samples are also characterised. Lipid concentrations and presence of contaminants within samples across sites are compared, and their possible links with burial environment are explored.

6.1.1. Group I: Pottery from collections

6.1.1.1. Lipid yields

Kalibangan

Five vessel fragments (4 rims, 1 base) from Kalibangan were selected for analysis (Chapter Five). Table 6.1 provides details about the vessel form/type, manufacturing characteristics

and surface treatment, and the lipid yield, from every analysed sherd from Kalibangan. Lipid concentrations of samples ranged from 64 to 3514 µg/g, with a mean lipid concentration of 823 μ g/g and median of 178 μ g/g. Rim fragments of small jars and a perforated jar (KLB04, KLB02, KLB01 and KLB03, respectively) contained high quantities of lipids, while the base fragment (KLB05) contained relatively lower lipid quantities. The lipid profiles obtained from vessels (e.g. Figure 6.1) are discussed in detail in Appendix F.

Vessel form	Sample ID	Location of vessel on sherd	Lipid concentration $(\mu g/g)$	Quantity of phthalates $(\mu g/g)$
Jar	KLB01	Rim	68.4	5.8
	KLB03	Rim	178	6
	KLB04	Rim	3514.2	20.5
Perforated jar	KLB02	Rim	290.4	7.4
Dish	KLB ₀₅	Base	64.1	2

Table 6.1: Sample descriptions and lipid concentrations for individual sherds from Kalibangan.

Figure 6.1: Partial ion chromatograms of vessel fragments from Kalibangan. KLB04 contains the highest lipid concentrations from the entire analysed assemblage. Cn:x indicates fatty acid with n carbon atoms and x double bonds. IS: Internal Standard, P: phthalate, diacid: dicarboxylic acid. For detailed interpretation see Appendix F.

Mohenjo-daro

Five sherds comprising a fragment of a single rim, three body fragments and a base fragment of a goblet were analysed from Mohenjo-daro. Lipid concentrations of samples ranged from 15.6 μ g/g to 82.1 μ g/g, with a mean of 41 μ g/g and median of 36.5 μ g/g. Table 6.2 provides details about the vessel form/type, manufacturing characteristics and surface treatment, and the lipid yield from every analysed sherd from Mohenjo-daro. Unusually, the body fragment of a perforated vessel contained the highest lipid concentration. The body and rim fragments contained comparable amounts of lipid, whereas the base fragment contained the lowest quantity of lipid relative to other sherds. Interpretations of the lipid profiles from Mohenjo-daro are provided in Appendix F.

Vessel form	Sample ID	Location of vessel on sherd	Lipid concentration $(\mu g/g)$	Quantity of phthalates $(\mu g/g)$
Jar	MD02	Rim	41.4	2.8
Goblet	MD03	Base	15.6	409.2
Perforated jar	MD01	Body	82.1	7.2
Unknown	MD ₀₄	Body	28.8	2.5
Unknown	MD ₀₅	Body	36.5	5.7

Table 6.2: Sample descriptions and lipid concentrations for individual sherds from Mohenjo-daro

6.1.1.2. Lipid yields across sites

The samples from Kalibangan exhibit significantly higher lipid concentrations than those from Mohenjo-daro (Mann-Whitney test, $W = 23$, $n_1 = 5$, $n_2 = 5$, $p = 0.03$) (see Figure 6.2). However, the lack of specific geoarchaeological and bioarchaeological information from Kalibangan makes it difficult to make a detailed assessment of preservation conditions at the site.

Figure 6.2: Boxplot demonstrating lipid concentrations (summarised in Table 6.2) obtained from pottery from Kalibangan and Mohenjo-daro. Lipid concentrations are represented on a log-scale for better visualisation. Red diamonds represent mean values.

Figure 6.3: Boxplot demonstrating phthalate concentrations (Summarised in Table 6.2) obtained from pottery from Kalibangan and Mohenjo-daro. Lipid concentrations are represented on a logscale for better visualisation. Red diamonds represent mean values.

Lipids were preserved in low quantities in pottery from Mohenjo-daro. The lack of specific contextual information for the potsherds makes it difficult to connect this with preservation conditions at the site. It is possible that the type of vessels selected for analysis from Kalibangan and Mohenjo-daro have influenced the preservation of lipids. As the samples analysed belong to different vessel forms (or are from unknown forms) across both sites, it is not possible to compare lipid concentrations across vessel forms. Additionally, as the specific context from where the pottery comes from is unknown, it is also not possible to discuss their use in antiquity.

6.1.1.3. Presence of contaminants

Samples from both Kalibangan and Mohenjo-daro contained varying levels of phthalates (Tables 6.1-6.2 and Figure 6.3). The origins of these compounds may be from the exposure to contaminants on the surface of a site, the storage of sherds in plastic bags, or their exposure to varnish, marker fluid, and/or glues. Phthalates like dimethyl phthalate and phthalic acids were found in Group I samples. Although the presence of phthalates in archaeological ceramic lipid extracts is widely acknowledged (e.g. Evershed et al. 1990; Cramp et al. 2011), they are not usually discussed in detail. However, the presence of phthalates is problematic as they can interfere with the identification of other archaeologically meaningful compounds. Phthalate concentrations in samples from Kalibangan and Mohenjo-daro range from 2-410 μ g/g. Most samples contain less than 10 µg/g of phthalates, but sample MD03 had high concentrations of di-n-octyl phthalate (DnOP). The high quantity of plasticiser in this sample makes it unreliable for further study. The lack of contextual information, absence of palaeoenvironmental data from these sites, the fact that the samples were subjected to long-term storage and handling, and the high degree of phthalates in several samples reduce the degree of confidence with one can attribute the lipid concentrations to archaeological vessel-usage and make interpretations challenging. For this reason, lipid profiles of vessels from Group I are excluded from further discussion (results are provided in Appendix F).

Figure 6.4: Partial ion chromatograms of vessel fragments from Mohenjo-daro. MD02 contained short-chain fatty acids that rarely survive in archaeological contexts and may originate from storage environment; MD03 contained very high concentrations of phthalates. Cn:x indicates fatty acid with n carbon atoms and x double bonds. P: Plasticiser; IS: Internal Standard. For detailed interpretation see Appendix F.

6.1.2. Group II: 'recently excavated samples'

Samples from recently excavated sites were located in environments that are not particularly conducive to the organic survival. Since the samples have been washed, handled by various people, and stored in poor-quality plastic bags (see Chapter Five), it was hypothesised that lipid survival in pottery would not be very high. However, the input of synthetic contaminates would be minimal as the samples have only been exposed to them for a few years. The sites from which the recently excavated samples were obtained are listed along with the vessel forms, range, mean and median of lipid yields in Table 6.3.

6.1.2.1. Lipid yields

Alamgirpur (ALM)

Fourteen out of fifteen sherds from Alamgirpur yielded lipid concentrations above 5 μ g/g, with a body sherd yielding less than 5 μ g/g. Potsherds with such low lipid yields are normally excluded from analysis as they may not adequately represent ancient vessel use. The mean lipid concentration of these 14 vessels was 14.3 μ g/g and median was 10.5 μ g/g. A painted medium-mouthed jar had the highest lipid concentration from the assemblage (42.8 μg/g) (Table 6.3).

Table 6.3: Range, mean and median lipid concentrations of vessel-type from recently excavated sites.

Masudpur VII (MSDVII)

Lipid yields of samples from Masudpur VII were relatively higher compared to other sites in the region. A total of 28 out of the 31 vessel fragments analysed had appreciable quantities of lipid (over 5 μ g/g). The two samples with very low lipid concentrations (under 5 μ g/g) are not included in the discussion about the lipid composition of the sherds (see Appendix H). The mean lipid concentration of vessels from Masudpur VII that contained interpretable quantities of lipids was $30.2 \mu g/g$ and median was $17.3 \mu g/g$. (Table 6.3) A necked jar had the highest lipid concentrations out of the different vessels (219.3 μg/g). Overall, ledged jars demonstrated relatively higher mean lipid concentrations than other vessel forms but the difference was not significantly higher (one-way ANOVA test, $F(7, 20) = 0.96$, $p = 0.48$). Perforated body sherds had lipid concentrations similar to rim sherds of jars, which may be indicative of their use for processing similar products.

Masudpur I (MSDI)

Thirty vessel fragments from Masudpur I were analysed. Twenty-four out of 30 sherds had appreciable quantities of lipids, while six sherds contained less than $5 \mu g/g$. These latter sherds were excluded in the discussion about lipid composition (see Appendix H). The mean lipid concentration of vessels from Masudpur I that contained appreciable quantities of lipids was 23.3 μg/g and median was 12.3 μg/g (Table 6.3). A small red-slipped jar (MSD343) had the highest lipid concentration out of the entire assemblage (122 μ g/g).

Farmana (FRN)

The lipid yields from vessels analysed at Farmana were typically low. Out of 30 vessels, only 18 vessels contained interpretable quantities of lipid (above 5 μg/g) (see Appendix H), and the mean lipid concentration of these vessels was 14.5 μ g/g and median was 9.8 μ g/g (Table 6.3) There were no observable differences between the lipid concentrations of different vessel forms. It is notable that the lipid concentrations of the body sherds of perforated vessels are comparable to the rims of other vessels, but it is not possible to make further interpretations about the frequency of use or function of vessels due to low organic preservation across the analysed assemblage.

Rakhigarhi (RGR)

Samples from Rakhigarhi also had poor lipid yields. Only 18 out of 30 vessels had interpretable concentrations of lipid (above 5 μ g/g) (see Appendix H). The mean lipid concentration of these vessels was 15.7 μ g/g, with a median value of 11.8 μ g/g (Table 6.3). There were no differences between the lipid concentrations of different vessel forms. A

very small necked jar (RGR20) had the highest lipid concentration out of the assemblage (48.8 μg/g) It is not possible to make further interpretations about the frequency of use of vessels, or ways in which the vessels might have been used due to the low organic preservation across the assemblage.

6.1.2.2. Lipid yields across sites

As hypothesised, yields of total lipids residues in vessels across recently-excavated sites located in northwest India's alluvial plains (n=102) was not high, ranging from trace amounts up to 219 μg/g, and, after excluding the vessels with lipid concentrations below 5 μg/g (n=33), averaged 21 μg/g. Samples from Masudpur VII demonstrated relatively higher lipid yields overall, but the difference was not statistically significant (Kruskal-Wallis test $\chi^2(4) = 101$, $p = 0.48$) see Tables 6.3-6.4, Figure 6.4).

Figure 6.5: Lipid concentrations of samples from Group II sites. Values are represented on a logscale for better visualisation; red diamonds represent mean values.

The overall low yields of lipids from samples from these recently excavated sites may be explained by their location within a semi-arid alluvial plain with alkaline soil conditions that has witnessed major landscape transformations in the past centuries, mostly due to recent heavy agricultural activity. Additionally, all sites experience seasonal variations in temperature and rainfall which includes high-intensity monsoonal rainfall and winter rain; these fluctuating conditions are not conducive to the preservation of organic matter (Section 3.2.2). Furthermore, all the sherds analysed had been washed and scrubbed after excavation, which may have led to some loss of lipid within the ceramic fabric. Since lipids are sensitive to various alteration processes, it is likely that a variety of chemical or biological processes have led to the significant loss of the initial compounds (Regert et al. 2003).

The relatively higher yields of lipid from samples from Masudpur VII is interesting and suggests that certain sites may have unique micro-environments that facilitate the preservation of lipids. This possibility is further attested by the better preservation of both archaeobotanical and faunal assemblages at Masudpur VII (Bates 2016). Other vessels across the analysed assemblages have relatively higher lipid concentrations (over 100 μ g/g). (e.g. MSD3412 and MSD343 (see Figure 6.6), and some vessels have lipid concentrations above 40 μg/g (see Figure 6.6). The factors influencing high lipid

concentrations in these sherds relative to other samples may be their more frequent use in antiquity, or due to the sherd's unique depositional environment that enabled higher preservation. However, as several samples were selected from the same locus/context (e.g., from Masudpur I, Masudpur VII and Rakhigarhi), and therefore would have similar depositional conditions, it is entirely possible that the higher lipid concentrations of these sherds are linked to differences in their use in antiquity. The possible relationship between lipid concentration and vessel use-history is detailed in Chapter Seven.

6.1.2.3. Presence of contaminants

Every sample analysed from Group II contained synthetic compounds such as phthalates or plasticisers, but mean concentrations were below 2.5 μg/g and concentrations did not exceed 10 μg/g (Figure 6.7). These compounds likely originate from the sherds' storage in plastic bags, or their processing on plastic work surfaces, as well as their exposure to compounds present in sun creams or lotions during handling by excavators and subsequent archaeologists. It is also possible these contaminants were introduced during sample drilling or lipid extraction, either from the sampling or laboratory environment.

Additionally, an unsaturated long-chain fatty acid $(C_{22:1})$ was noticed in many samples but was missing from sample blanks and standards (see Figure 6.5). As this compound is unlikely to preserve in archaeological samples and was noticed in samples analysed within the same run, it may have been introduced during laboratory processing. Another example of possible contamination was seen in ALM125-479, where even- and odd-chain *n*-alkanes were seen in the lipid profile in equal proportions (Figure 6.6). Thus, even though some sherds had been excavated as recently in 2013; their exposure to synthetic compounds after excavation resulted in their adsorption into the ceramic fabric. Although the quantities of synthetic components were relatively low for several samples; it is possible that their presence interfered with the detection of other organic compounds. Other contaminants were also likely introduced during the sample preparation period.

Figure 6.6: Partial total ion chromatograms of vessel fragments from Masudpur VII, Masudpur VI, Farmana and Alamgirpur. Cn:x indicates fatty acid with n carbon atoms and x double bonds. IS: Internal Standard; P: Phthalate, diacid: dicarboxylic acid.

Figure 6.7: Phthalate concentrations of samples from recently excavated sites. Values are represented on a log-scale for better visualisation; red diamonds represent mean values.

6.1.3. Group III: freshly-excavated samples

Pottery from all sites within Group III experienced arid conditions with seasonal precipitation and fluctuating temperatures (see Chapter Four). Although it would be impossible to control for other variables within the burial environment, the effect of postexcavation handling of pottery on the survival, composition, and detection of lipid residues could be assessed. Additionally, comparisons between the lipid quantity and composition of the sediments adhering to the potsherds and the lipid yield of potsherds could also be investigated.

6.1.3.1. Lipid yields

Khanak (KNK)

The lipid concentrations of vessels from Khanak was variable. Four out of seven vessels (42.8%) had lipid concentrations above 5 μ g/g (see Appendix H for details of vessels with lipid yields below 5 μg/g). The mean lipid concentration of these vessels was 49.1 μg/g and median was 28.6 μg/g (see Table 6.5).

Two sediment samples from Khanak were analysed. These were KNK14 and KNK15, which were collected from sediment adhering to ceramic samples KNK02 and KNK18, respectively. The sediment samples had lipid concentrations above 5 μ g/g, but they were still relatively low in lipid yield, containing 8.6 μ g/g and 13 μ g/g of lipid respectively (Table 6.12). The molecular profiles of KNK14 and KNK15 contained relatively higher concentrations of very long chain fatty acids (up to $C_{30:0}$) generally not observed in the ceramic samples from this thesis, as well as odd-chain fatty acids, branched-chain (e.g. $C15_{\text{Br}}$, $C17_{\text{Br}}$) fatty acids and unsaturated fatty acids. Figures 6.8 and 6.9 provide total ion chromatograms of the lipid extracts from pottery and sediment adhering to the fragments. The clear distinguishing characteristics between lipid profiles from sediment and pottery is the presence of very long-chain fatty acids in both sediment samples and odd-long-chain *n*-alkanes in KNK15. Oddly, KNK15 contains relatively high proportions of $C_{16:0}$ and $C_{18:0}$, and both pottery fragments contain odd-chain and branchedchain fatty acids.

Site Name	Vessel form	Number of vessel fragments	Lipid concentration range $(\mu g/g)$	Mean lipid concentration $(\mu g/g)$	Median lipid concentration $(\mu g/g)$
Khanak	Small jar	1	131		
	Jar (rim unknown)	2	18-39	28.7	
	Base	-1	8		
	Total	4	8-131	49.1	28.6
Lohari Ragho I	Small jar		19.2		
	Medium jar	2	$9-21$	15	
	Large jar	3	22-214	76.4	24.3
	Necked jar	3	17-30	24.7	27.3
	Globular jar	3	$6 - 45$	28.3	34.5
	Ledged jar		10.7		
	Perforated jar	2	$12 - 16$	14.7	
	Base		5.6		
	Total	16	$6 - 214$	32.2	20.2

Table 6.5: Range and mean lipid concentrations by vessel form from Group II sites in northwest India

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Figure 6.8: Partial total ion chromatograms of KNK02, fragment of a red-slipped jar, and KNK14, sediment adhering to the sherd. KNK14 contains relatively higher quantities of long-chain fatty acids up to C30:0 rarely found in archaeological pottery, branched-chain and odd-chain fatty acids and unsaturated fatty acids. Cn:x indicates fatty acid with n carbon atoms and x double bonds. IS: Internal Standard, P: phthalate, Br: branched-chain fatty acid.

Figure 6.9: Partial total ion chromatograms of KNK18, base fragment, and KNK15, sediment adhering to the sherd. KNK15 contains relatively higher quantities of long-chain fatty acids up to C30:0 rarely found in archaeological pottery, and odd-chain *n*-alkanes. Cn:x indicates fatty acid with n carbon atoms and x double bonds. IS: Internal Standard, P: phthalate, Br: branched-chain fatty acid; ALK: *n*-alkane.

Lohari Ragho I

Twenty-six pottery fragments were analysed from Lohari Ragho I, and out of these, sixteen contained appreciable concentrations of lipid (above 5 μ g/g). Two terracotta cakes revealed trace quantities of lipids and were thus not included in further analyses (see Appendix H for details of vessels with lipid yields below 5 μ g/g). The mean lipid concentration of these vessels was $32.2 \mu g/g$ and median was $20.2 \mu g/g$ (Table 6.5).

Two sediment samples from Lohari Ragho I were analysed to compare lipid yield and lipid composition of sediments versus ceramic sherds. Unusually, a sediment sample (LHR41) obtained from context number 524, a hearth, contained 38.6 μ g/g of lipids (Figure 6.10), which is comparable to yields obtained from pottery vessels in this thesis. This concentration may be suggestive of mixtures of lipids from sediment and remains of food-processing activities associated with the hearth. In contrast, LHR37, a sediment sample from locus 520, from which several sherds were analysed, contained only 7.9 μ g/g of lipid. The low quantities of lipids in LHR37 suggest it is unlikely to have influenced lipid composition in the sherds. The lipid composition of both sediment samples included relatively higher proportions of very long-chain fatty acids, odd-chain fatty acids, branched-chain fatty acids and unsaturated fatty acids, which were not present (e.g. $C_{20:1}$), or present in very low quantities (e.g. $C_{18:1}$), in the ceramic samples.

Small peaks of plasticisers were detected in the lipid extracts, which may have been introduced during sample preparation. A compound $(C_{22:1})$ was noticed in all samples, especially in sediment sample LHR41, but was missing from sample blanks. As this compound is unlikely to preserve in archaeological samples and was noticed in virtually all the samples analysed within the same run, it may have been introduced during laboratory processing.

Figure 6.10: Partial total ion chromatograms of two sediment samples from archaeological loci at Lohari Ragho I. Both contain long-chain fatty acids upto C28:0, branched-chain and odd-chain fatty acids, and high quantities of unsaturated fatty acids that are rarely found in archaeological pottery. LHR41 contained relatively high quantities of lipid and high proportion of $C_{22:1}$, which may have been introduced during sample preparation. Cn:x indicates fatty acid with n carbon atoms and x double bonds IS: Internal Standard, P: phthalate, Br: branched-chain fatty acid.

Stone Tower I, Salut (STI)

Eleven sherds were analysed from Stone Tower I at Salut, including 5 local Arabian Umm an-Nar vessels and 6 Indus Black-Slipped Jars (BSJs). The mean lipid concentration of all pottery samples was 40.7 μg/g and median was 22.9 μg/g, but the mean lipid concentration of Arabian Umm an-Nar pottery was 36.8 μg/g and median was 12.4 μg/g, and mean lipid concentrations for BSJs was 44 μg/g and median was 36.45 μg/g (Table 6.6).

Given the arid conditions at the site and the virtual absence of rainfall in the region, it was hypothesised that lipid preservation at Stone Tower I, Salut would be very high. However, it is likely that overall lipid preservation and the preservation of a variety of lipid species have been negatively affected by sandy, alkaline soils (Bellini et al. 2011) and wetting and drying cycles experienced via seasonal rainfall and flash floods in this region (Horowitz 1992; Méry and Tengberg 2009). Thus, even though the concentrations of lipid preserved within samples from Stone Tower I, Salut are relatively higher than the rest of the study sites, the range of lipid concentrations is narrow, and overall lipid yields of vessel fragments is low.

Vessel form/type	Location of vessel on sherd	Lipid concentration $(\mu g/g)$
Umm an-Nar jar	Rim (ST1018)	96.1
	Rim (ST1022)	10.5
Unknown	Body (ST1021)	57.8
	Body (ST1039) 12.4	
	Mean	36.8
	Median	12.4
Indus Black-Slipped Jar (BSJ)	Rim (ST101)	85.2
	Rim (ST1074)	21.9
	Body (ST102)	22.9
	Body (ST109)	18.4
	Base (ST1078)	65.4
	Base (ST1079) Fits with ST1078	50
	Mean	44
	Median	36.5

Table 6.6: Lipid concentrations for samples analysed from Stone Tower I, Salut with details of vessels

Due to the nature of vessel-fragment preservation at Stone Tower I, Salut. it is possible that the available lipid concentrations may be informative of vessel-use. A comparison of the lipid concentrations of different fragments of Indus BSJs (see Table 6.4, Figure 6.3) may reveal vessel-usage characteristics as six fragments from different locations on a vessel of the same vessel-type have been analysed. Additionally, the special characteristics of this vessel-type (see Chapter Four) make it an interesting case-study when compared with local Arabian vessels.

Lipid concentrations of the BSJs from Stone Tower I, Salut indicate that lipid content from a single rim sherd, ST101 and two base fragments, ST1078 and ST1079 are relatively higher than other fragments (85.2 μ g/g, 65.4 μ g/g and 50 μ g/g respectively). Body sherds yielded relatively lower quantities of lipids (22.9 µg/g and 18.4 µg/g respectively). Low yield of lipids from body sherds have been previously demonstrated (Charters et al. 1993). However, the overall low lipid yield from the vessel fragments may be because of their large size. As storage vessels, it is possible that BSJs were not exposed to high temperatures in their use-history, hindering lipids from foodstuff being easily mobilised

and incorporated into the ceramic fabric. It is also possible that the interior slip acted as a barrier for the adsorption of lipids into the vessel.

The lipid yields from two rim sherds (i.e., at least two vessels) are 85.2 μ g/g and 21.9 μ g/g, respectively, indicating that these vessels were not used in a similar fashion, and may have use-histories of storing different foodstuffs. This finding adds support to the suggestion that BSJs had secondary or multiple uses (Méry and Blackman 2004). The high concentration of lipids in the base fragments (ST1078 and ST1079) is unique and may also indicate another use of the vessel-type. Additionally, the two base fragments analysed fit together to form a large fragment and have lipid concentrations of 65.4 μ g/g and 50 μ g/g respectively. Although a difference of 15 µg/g between two different locations on the same sherd is considerable, it indicates the relative consistency in measurement of lipid yields from specific parts of vessels.

When compared with the BSJs, two out of five of the local Arabian Umm an-Nar vessels have relatively high concentrations of lipid. ST1018, a rim sherd from a fine Sandy Ware pot has a lipid concentration of 96.1 µg/g, and ST1021, a body sherd from a suspension vessel has a lipid concentration of 57.8 µg/g. ST1018 is made of local clay, but

it appears to be 'Indus-related' as it has a very distinctive rim shape and may have been produced by an Indus potter (Méry, pers. comm.). Another rim-type that appears Indusinspired is ST1040, but its lipid concentration is very low $(7.4 \mu g/g)$. The other two fragments from locally-produced vessels (rim sherd ST1022 and body sherd ST039) also have relatively low lipid content (10.5 μ g/g and 12.4 μ g/g respectively. The differences in lipid concentration suggests that ST1018 and ST1021 may have been used more frequently or used for the processing of fatty/oily products compared to other vessels.

Even though potsherds were collected freshly from the ground, only cleaned (minus water) and wrapped in aluminium foil prior to lipid extraction, peaks attributed to di-isooctyl phthalate, a plasticiser commonly used in plastic and rubber products, was observed in very small concentrations in each sample (see Figure 6.12). Additionally, some samples (e.g. ST102) contained high quantities of column bleed or siloxanes (m/z 207), which is the normal background signal generated by the column stationary phase within the GC or GC-MS (see Figure 6.12). If bleed is minimised, there is a better chance of mass spectral matching against a database of reference spectra, however, it is easily identifiable and does not usually interfere with elution of peaks. High column bleed is caused by degradation of the stationary phase of the column and can occur if the column is in use for long periods of time.

Figure 6.12: Partial total ion chromatograms of two vessel fragments from Stone Tower I, Salut. ST101 has phthalates and column bleed is visible. Cn:x indicates fatty acid with n carbon atoms and x double bonds IS: Internal Standard, P: phthalate

Table 6.7: Lipid concentrations from interior and exterior surfaces of pottery and sediment samples from Stone Tower I, Salut

Figure 6.13: Partial total ion chromatograms of the interior and exterior surface of ST102 from Stone Tower I, Salut. Cn:x indicates fatty acid with n carbon atoms and x double bonds IS: Internal Standard, P: phthalate.

Figure 6.14: Partial total ion chromatograms of ST1074 and soil adhering to the surface of the sherd (ST1074SOIL) from Stone Tower I, Salut. Cn:x indicates fatty acid with n carbon atoms and x double bonds IS: Internal Standard, P: phthalate.

Three sediment samples (ST1SOIL74, ST1SOIL79 and ST1SOIL082) and samples of the exterior surfaces of two vessels (ST102E and ST1018E) were analysed to compare the quantity and composition of lipids in sediments adhering to sherds during collection, or from the context from which the sherd was collected. It is also possible to assess the difference between the exterior and interior surfaces of sherds.

In these instances, lipid yields obtained from the interior surface of vessel fragments were higher than those obtained from the exterior surface of the vessel (Table 6.7). However, there was no major variation in the types of compounds observed in their lipid profiles (Figure 6.12). This potentially has implications for understanding the use of these specific vessels (see Appendix A). The lipid concentrations obtained from ST1SOIL74 and ST1SOIL082 were very low (trace quantities 9 µg/g, respectively) (Figure 6.13), suggesting that the lipid content of analysed sediments at Stone Tower I, Salut, was minimal. Similarly, lipids obtained from the sediment adhering to ST1079 (ST1SOIL79) was 4 μ g/g, suggesting that it was unlikely for lipids from the sediment to have an influencing factor on the quantity or composition of lipids within the sherd.

6.1.3.2. Lipid yields across sites

Despite being situated in different environmental contexts (and in the case of Stone Tower I in different countries; see Chapter Four), samples from sites within Group III demonstrated comparable lipid concentrations to each other. Samples from Khanak demonstrated the smallest range of lipid yields (between 131 μ g/g and 8 μ g/g), but their mean value was 49.2 µg/g and median was 28.6 µg/g, which is higher than samples from Lohari Ragho I and Stone Tower I, Salut (mean lipid yield of 31.6 μ g/g and 40.7 μ g/g, and median of 20.7 and 22.9 respectively) (Table 6.8 and Figure 6.14). There were no significant differences in lipid concentrations across sites (Kruskal-Wallis test, $\chi^2(2) = 30$, $p = 0.47$).

It is possible that Khanak's location in a consistently arid environment ensured better lipid preservation, thus enabling relatively higher lipid yields from samples. The site's location within a school playground also has protected it from being disturbed by agricultural activity, and deposits were considerably deeper than those found at Lohari Ragho I. Conversely, Lohari Ragho I is in a landscape where there have been significant fluctuations in the water table, the land is heavily cultivated, and the excavated remains are very close to the surface (see Figure 6.15). Fluctuations in fluvial processes are evinced by the build-up of large calcretic nodules in the environment. These conditions have likely created a sub-optimal environment for lipid preservation (Cramp 2008). Similarly, it is likely that at Stone Tower I, Salut, the presence of silty sediment with a high percentage of gravel and exposure to seasonal saturation due to the fluctuating water table and minor flooding events resulting from its location in a *wadi* (valley) have not aided lipid preservation. However, it is possible that the relatively quick burial of the sherds (Cremaschi, pers. comm.) and the arid environment contributed to the survival of lipids.

Table 6.8: Total vessel fragments analysed, samples with appreciable lipid concentration (above 5.0 μg/g), % of samples with appreciable lipid yield, and mean lipid concentrations for vessels per site.

Site code	KNK	LHRI	STI
Total ceramic samples analysed		28	11
Samples with lipid concentrations above 5.0 μ g/g	$\overline{4}$	16	11
Success rate $(\%)$	43%	60%	100\%
Mean lipid concentration $(\mu g/g)$	49.2	31.6	40.7
Median lipid concentration $(\mu g/g)$	28.6	20.2	22.9

Figure 6.15: Boxplot of lipid concentrations of pottery from Khanak, Lohari Ragho I and Stone Tower I, Salut (Tables 6.5 and 6.6). Values are represented on a log-scale for better visalisation; red diamonds represent mean values.

Figure 6.16: Left: Site of Lohari Ragho I. Middle: School yard at Khanak where excavations were conducted with view of inselberg (Courtesy Cameron Petrie). Right: Remains of Umm an-Nar tower at Stone Tower I, Salut showing calcretic deposits in the profile, demonstrating fluctuating water movement.

6.1.3.3. Presence of contaminants

The presence of phthalates and plasticisers was minimal in the samples excavated from Khanak and Lohari Ragho I (below 5 μg/g). In contrast, however, the samples from Stone Tower I contained relatively high concentrations of phthalates. These contaminants may have been introduced during sample preparation, and there was also column bleed originating from the analyser (Figure 6.16). Both Lohari Ragho I and Khanak also contained organic compounds that had been introduced during sample preparation or extraction (e.g. $C_{22:1}$).

Figure 6.17: Boxplot of phthalate concentrations in lipid extracts from pottery samples from Khanak, Lohari Ragho I and Stone Tower I, Salut. Values are represented on a log-scale for better visualisation; red diamonds represent mean values.

6.2. Discussion: lipid yields and preservation

The analysis presented in Section 6.1 was designed to address questions about the relationship between lipid yield and preservational environment, synthetic contamination, and influence of lipids from the burial sediment. In the following section, the comparison of lipid content between Group I, II and III will be discussed, and the cumulative results between comparisons of lipid content from the interior versus exterior surface of sherds, and sherd versus sediment samples will be summarised in order to make inferences about lipid preservation.

6.2.1. Comparison of lipid yield with other regions of the world

Lipid concentrations from vessels across study sites in this thesis were overall low. Table 6.9 provides a comparison of lipid yields from the study sites with other arid regions of the world. The data is not directly comparable as several studies have used alternative extraction protocols and analysed vessels of greater antiquity than those in the present study, but it provides an indication of the poor preservation of lipids within archaeological vessels in South Asia and south-eastern Arabia relative to other arid sites. This is despite the use of acidified methanol extraction method which increases ceramic lipid yield compared to solvent extraction (Correa-Ascencio and Evershed 2014; Chapter Three).

The results obtained from the study likely best compare with those obtained from the Near East. Gregg (2009; Gregg and Slater 2010) suggests that poor preservation of organic matter in the Neolithic ceramics of the Near East can be attributed to the calcareous nature of the soil, with pH levels above 6.5. Microbial activity is optimal in these pH conditions (DeLaune et al. 1981) and the fatty acids present in the form of soluble salts are more easily removed by leaching (Evershed et al. 1997), which reduce the chances of lipid preservation. All the study sites in this thesis have calcareous, weakly alkaline soils (Neogi 2013; Neogi et al. in press; Chapter Four). The sites also experience seasonal, heavy rainfall and hot temperatures. The combination of these conditions likely creates an unfavourable environment for organic preservation.

Table 6.9: Details of recovery rates, range and mean of lipid concentrations obtained from different sites in the Near East, Anatolia and North Africa (when available) compared with sites studied in this thesis. nr: not-reported.

6.2.2. Lipid yields across vessel fragments

Details of the types of vessel fragments analysed are provided in Tavle 6.10. Only the 143 sherds containing more than 5 μ g/g of lipid were investigated further. These included 6 base fragments, 23 body fragments, 2 neck fragments, and 112 rim fragments, suggesting 74% of the assemblage contained interpretable concentrations of lipid.

Table 6.10: Details of ceramic fragments analysed, lipid concentrations, % of samples with lipid concentrations above 5 μg/g, range, mean and median of lipid concentration per sample type.

Of the different vessel fragments analysed, the success rate of extracting appreciable quantities of lipid (i.e. above 5 μ g/g) from different types of sherds ranged between 66 and 75% (Table 6.10). Mean and median lipid concentrations from rim sherds were lower than those obtained from bases when excluding samples from Group 1, but higher than lipid concentrations obtained from body or neck sherds. This is attributable to the relatively higher concentrations of lipid obtained from potsherds from Stone Tower I, Salut. The
range of lipid quantities obtained from rim sherds was wide (upto $216 \mu g/g$), but much narrower for base, body, and neck sherds (upto 65 μ g/g, upto 58 μ g/g, and 12-14 μ g/g respectively). Only trace amounts of lipids were obtained from the two terracotta cakes analysed, which may have implications about their use. Although only two terracotta cakes were analysed, it is possible that terracotta cakes had a range of functions. While some have clearly been associated with heat management and food processing (some terracotta cakes have been discovered with charred seed remains adhering to them (Vishnu-Mittre and Savithri 1975; Manuel 2010; Ceccarelli in prep.), it is possible the samples analysed were not directly involved in cooking, and did not absorb lipids.

6.2.3. Comparison of lipid yields across Groups I, II and III

As demonstrated by Table 6.11, the overall lipid yields of Group I are higher than Groups II and III. Within Group I, four out of five samples from Kalibangan demonstrate relatively high yields (see Fig 6.1). It is possible that unique site-specific conditions enabled better preservation of lipids within pottery from Kalibangan, but no detailed study on the organic or bioarchaeological material from the site has been undertaken that would make it possible to crosscheck this finding. Furthermore, the high presence of plasticisers and the unknown context of the sherds suggest that results from Kalibangan and Mohenjo-daro may not reliably represent archaeological vessel-use. Thus, the results from collections are not included in the future discussions about vessel contents and vessel usage.

Samples from the recent excavations (Group II) did not exhibit high lipid yields. Within Group II, Masudpur VII demonstrated the highest lipid concentrations relative to other recently excavated samples. This does not seem to be attributable to how recent the excavations were, as samples from both Masudpur I and Masudpur VII were excavated in 2009, and samples from Alamgirpur, Farmana and Rakhigarhi were excavated in 2008, 2009 and 2014, respectively. Observations from site surveys (Walker, pers. comm.) and results from archaeobotanical analysis at Masudpur VII (Bates 2015) reveal better overall organic preservation at Masudpur VII. This factor is likely attributable to either the site's location at the edge of a sand dune, thus creating an arid environment. Consistent burial conditions in some parts of the site may also have enabled better organic preservation.

Table 6.11: Range, mean and median lipid concentrations from all three groups. Samples with lipid concentrations below 5 µg/g are excluded

Figure 6.18: Boxplots of lipid concentrations of Group II and Group III (Table 6.11). Values are represented on a log-scale for better visualisation; red diamonds represent mean values.

The differences between the Group II and III samples suggests there may be exceptional micro-conditions conducive to the survival of organic remains within environments that are generally low in organic preservation. The possibility of the presence of microenvironments supporting better organic preservation is vital to consider when designing future excavations in regions with poor organic preservation. Small 'proof-of-concept' studies testing preservation should be planned before budgeting a dedicated programme of organic residue analyses. Additionally, as the samples were chosen from well-documented and specific archaeological contexts of interest (e.g. occupational surfaces and hearth contexts) and could be complemented with other site-specific faunal, archaeobotanical, starch-grain, or isotopic data, they are more reliable and provide the necessary palaeoecological context to deduce the likely source(s) of the lipid residues detected within vessels. This deductive aspect is essential to interpreting the remains found within lipid extracts of vessels, as only the presence of a constituent of a residue that is consistent with the archaeology and palaeoecology of the settlement, region, and/or period from which the vessels are obtained are considered (Evershed 2008a).

Samples from freshly excavated pottery (Group III) demonstrated higher lipid yields than those from recent excavations (Group II), though the difference was not statistically significant (Kruskal-Wallis test, $\chi^2(7) = 132$, $p = 0.48$). This finding is interesting as Group III includes samples from Stone Tower I, Salut, in the Sultanate of Oman, which experiences a more arid climate than Lohari Ragho I and Khanak. However, Salut is prone to flooding events as it is in a flat valley and lies within a region with alkaline soils. This suggests that overall, environmental conditions in the alluvial plains of northwest India and *wadis* (valleys) in Oman may not be conducive to the survival of organic remains, particularly lipids in pottery (see Section 3.2.2). Nonetheless, the Group III samples represented the 'best-case' scenario for collection of pottery samples for organic residue analysis as all the steps from excavation to extraction and analysis were controlled. This opportunity reduced the likelihood of incorporation of extraneous compounds unrelated to the archaeological use of the vessel into the fabric and increased the degree of confidence with which one could attribute organic remains within the vessel to its use-history. Additionally, ceramic samples were not washed or scrubbed, which can lead to the removal of lipids. Thus, despite the low levels of preservations, these samples are the most reliable as their removal was controlled and the interpretations could be contextualised within site-specific bioarchaeological studies when available.

6.2.4. Presence of contaminants across groups

Phthalates and synthetic contaminants were present across Groups I, II and III. Samples from Group I exhibited the highest quantities of phthalates (Figure 6.19), with a single sample (MD03) containing the highest quantities from all analysed samples (Figure 6.4). These samples were excluded from discussions in later chapters. Samples from Group II also contained phthalates, but they occurred in relatively lower proportions than those observed in samples from Group I. Within Group II, samples from Masudpur VII and Farmana included examples with relatively high concentrations of phthalates, but none exceeded 10 µg/g (Figure 6.7). Plasticisers were also present in samples from Group III, with concentrations comparable to those from Group II (Figure 6.18). The presence of phthalates in freshly excavated material is unexpected as the samples were wrapped in aluminium foil after excavation and they did not come in direct contact with plastic bags at any stage. This finding suggests that phthalates and other compounds can easily be introduced during the sample preparation and lipid extraction process. Figure 6.20 suggests that there is no relationship between lipid and phthalate concentrations between Groups II and III. This result is positive, however, it is not always possible to distinguish between what may be introduced from the laboratory and/or post-excavation/storage environment, making interpretations challenging and increasing the need for caution.

6.2.5. Lipids in sediment versus interior surfaces of sherds

Seven sediment samples (1g each) from three sites were analysed via GC-MS. Lipid profiles of sediment samples varied from those obtained from the interior surfaces of potsherds. The samples either had negligible quantities of lipid (e.g. ST1074SOIL) (Table 6.12) or very long-chain fatty acids (up to $C_{30:0}$), odd- and branched-chain fatty acids and/or long-chained *n-*alkanes, which were not present, or present in relatively lower quantities in the ceramic samples. This finding correlates with the distinctive nature of lipid moieties within soils noted by others, generally attributed to their origins in higher plants or microbial organisms (Heron et al. 1991; Eckmeier and Wiesenberg 2009). For example, the presence of long-chain fatty acids and odd-, long-chained *n*-alkanes is indicative of higher plants, whereas odd-chain fatty acids and short-chain odd *n*-alkanes are derived from microbial organisms (Heron et al. 1991; Eckmeier and Wiesenberg 2009).

Figure 6.19: Phthalate concentrations from sherds from Groups I, II and III. Values are represented on a log-scale for better visualisation; red diamonds represent mean values.

Figure 6.20: Scatterplot of lipid and phthalate concentrations from Groups II and III. Values are presented on a log scale for better visualisation.

The lipid content of the analysed archaeological sediments ranged from trace amounts to 38.6 µg/g. Overall, sediment samples from Stone Tower I, Salut, contained the lowest lipid concentrations, with both Khanak and Lohari Ragho I demonstrating relatively higher lipid preservation from sediments and soil adhering to vessel fragments. Overall, however, lipid concentrations in the sediments were relatively low. It has been previously established that there is a relationship between pH and overall lipid content of soils, with the highest amounts occurring in highly acidic soils and lower values occurring in alkaline soils (pH 7.1 to 7.5) ((DeLaune et al. 1981; Debono Spiteri 2012). Sites located on the alluvial plain in northwest India have weakly alkaline soils (Neogi 2013; Neogi et al. in press), and Stone Tower I, Salut lies in an area with extremely alkaline soils (Bellini et al. 2011; Cremaschi, pers. comm.). Thus, it is possible that the low lipid concentrations of the analysed samples are linked to their relatively high pH levels (Cramp 2008, see Section 3.2.2).

A single sediment sample from a hearth context from Lohari Ragho I (LHR41) had the highest lipid content of the analysed soil samples, with a lipid content of 38.6 µg/g. A closer look at its lipid profile reveals relatively high concentrations of unsaturated fatty acids such as $C_{16:1}$, $C_{18:1}$, $C_{22:1}$ (which is likely a laboratory contaminant) and relatively high concentrations of odd-chain fatty acids (see Figure 6.10). The sample is lacking high concentrations of long-chain fatty acids and long-chain odd *n*-alkanes. It is possible that this lipid profile reflects vestiges of ancient food-processing activities mixed with soil organic matter.

Table 6.12: List and details of archaeological sediments and soils analysed via GC-MS. FAs: fatty acids.

Overall, the low lipid content and lipid profiles of the sediment samples suggest that it is unlikely that lipids within soils influenced the lipids absorbed within pottery after deposition. Previously, researchers have suggested that quantitative and qualitative differences in soil lipids compared to pottery rule out the possibility of migration of soil lipids into buried potsherds (Heron et al. 1991; Craig et al. 2004, 2005; Correa-Ascencio and Evershed 2014). Additionally, the hydrophobic nature of lipids limits their solubility in groundwater, hence reducing the likelihood of them migrating into (or out of) potsherds by dissolution and diffusion. On this basis lipids show potential for the study of vessel use,

diet and other cultural activities. Despite the affirmative nature of the results, it is important to be cautious and account for potential sources of contamination, and samples must be assessed on a case-by-case basis. The interaction between soil organic matter, soil microorganisms and soil structure and properties is extraordinarily complex, and many aspects of the diagenesis of organic residues in archaeological contexts are not fully understood (Haslam 2004). Differences in decomposition factors and rates between artefact surfaces and in sediments have also not yet been thoroughly investigated (Haslam 2004). Thus, it is important to test for the possible effects of migration and microbial activity by comparing the organic matter content of the buried sherd and its burial context. Additionally, different pottery may have different rates of porosity and unique burial conditions that may influence the leaching of lipids. Further experimental studies with various types of pottery buried in different sediments with controlled degradation would help assess and model the scenarios under which the migration of soil lipids into pottery may occur, but these were not attempted for the research presented in this thesis.

6.2.6. Effect of time since excavation on lipid yield

One of the objectives of the first stage of the study was to assess the effect of time between excavation and analysis on the survival of lipid residues in pottery. Samples from Group I, II and III originated from sites that were broadly similar in terms of their environmental characteristics. The differences lie in the fact that Group I sherds were stored in plastic bags after collection (for over twenty years), Group II sherds were washed, processed and stored in plastic bags from between three to nine years prior to organic residue extraction and analysis, and Group III sherds were unwashed, did not come into direct contact with plastic, and analysed soon after removal (between one month to one year) from their original depositional context.

The analytical results suggest that lipid yield of potsherds is not visibly affected by the time interval between excavation and analysis; instead, lipid preservation in pottery appears to be most influenced by its original depositional environment. This has also been suggested by other studies that report that most degradation of lipid takes place shortly after abandonment or burial of the vessel (Aillaud 2002; Stacey 2009). Although lipid concentrations from Kalibangan are higher than those obtained from samples from Group II and Group III, which may be attributed to site-specific characteristics or post-excavation organic input, samples from Mohenjo-daro have lipid yields comparable to samples from both other groups. This suggests that time since excavation does not have a measurable

impact on lipid yields or on the degree of lipids preserved in pottery. In the future, comparison between stored versus pottery freshly-excavated from the same site would be necessary to make meaningful interpretations about the effect of time since excavation on the preservation of lipid yields in pottery.

6.3. Chapter Summary and Future Directions

This chapter has discussed issues related to the the overall preservation of lipids in South Asian and Arabian ceramics. Specifically, it: i) compared lipid preservation in ceramic samples from collections, recently excavated, and freshly excavated samples; ii) addressed issues concerning the input of synthetic contamination into samples; iii) discussed the likelihood of the migration of lipids in burial sediments into pottery; and iv) hypothesised the likely use of different vessel forms on the basis of their lipid preservation.

In summary, it was possible to extract lipids from pottery using the acidified methanol extraction protocol from sites located in northwest India, Sindh and the Sultanate of Oman, with over half of the samples yielding enough concentrations of lipid to be connected to archaeological use (over 5 μg/g of lipid) (Heron et al. 1991). Unfortunately, the overall preservation of lipids is extremely low. This difference is despite the use of the acidified methanol extraction method, which is much more aggressive than conventional solvent extraction method. The low lipid preservation in ceramics is likely connected to the original depositional environment of the vessels, as arid environments with seasonal variations in temperature and rainfall and alkaline sediments are not conducive to organic preservation (Evershed 2008a; Cramp 2008). However, certain vessel fragments within the analysed assemblage contain relatively high concentrations of lipid. This finding suggests that lipid degradation does not operate on a simple linear scale and predicting the decay pattern in individual samples involves the detailed consideration of multiple complex processes resulting from use of the vessel, the primary depositional environments, as well as conservation and subsequent storage (Barker et al. 2018). Thus, the analysis of lipids from pottery from sub-optimal burial conditions should not be undervalued.

Regarding synthetic contamination and burial environment, the concentration of plasticisers and synthetic compounds found within pottery from Group I was relatively high. Although their identification is easy as they have distinctive mass spectra, they may co-elute with archaeological lipids, making comparisons with extant mass spectral databases difficult. However, other organic compounds within samples from collections may not reflect archaeological use. Thus, all the samples from Group I have been excluded from further analysis in this thesis. Samples from Group III demonstrated that contaminants might be also incorporated into freshly excavated samples and may be introduced at any point during the extraction or analysis period. This discovery suggests it is advisable to know as much about the post-excavation burial environment of selected sherds so as to be more confident about the likely origins of the lipid species obtained from ceramic samples.

Comparisons with lipids from soil (adhering to pottery and collected from the original depositional contexts) with those found in the interior surfaces of pottery suggest the likelihood of migration of soil lipids into ceramics is low. However, certain soils may contain anthropological input and results may vary depending on individual scenarios. Thus, a systematic analysis of sediments and pottery from exterior and interior surfaces, and more experimental research is recommended for future studies.

The results of this chapter suggest that researchers should not devalue the possibility of gaining valuable results from lipid residue analysis studies in regions with sub-optimal preservational conditions. As Barker and others (2018) suggest, more effort should be given towards analysing ceramics from poor preservational contexts as negative results provide a framework against which more positive results can be better understood; and spur further methodological developments (Barker et al. 2018). The development of the acidified methanol extraction protocol is one such example. It is essential, however, that the examination of lipid residues in non-ideal conditions for organic preservation must be meaningfully supplemented with archaeobotanical and zooarchaeological analyses so as to develop trustworthy claims regarding past vessel use and subsistence patterns. This can only occur if a comprehensive bioarchaeological programme has been implemented at the site(s) of interest and may not always be possible. Thus, going forward it is important to acknowledge the challenges associated with lipid preservation in ceramics and the strengths and limitations of existing methodologies.

Now that the nature of preservation of lipids in the samples has been assessed, Chapter Seven will discuss the site-specific lipid and compound-specific isotopic results and address key archaeological questions posed by this thesis.

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Chapter Seven

Lipid and isotopic analyses of pottery from Indus sites in northwest India

This chapter presents the results of lipid analyses of vessels from seven Indus sites in northwest India. Section 7.1 presents overall results. Section 7.2 presents the site-specific results, which are organised according to settlement size, from smallest to largest. (see Chapter Four). Here, the lipid composition and possible contents of the vessels from each site based on GC-MS and GC-c-IRMS analyses are discussed, taking account of the bioarchaeological evidence available. Section 7.3 summarises the vessel-specific results and considers the purported use of specific vessel forms in the sites investigated in this thesis. Finally, Section 7.4 provides a discussion of the results, addressing the two main archaeological questions posed by this thesis:

1) Are there differences in vessel-usage across settlements?

2) Are there changes in the use of vessels over time?

7.1 Summary of analyses

A total of 168 ceramic vessel fragments from seven Indus sites in northwest India were analysed via GC-MS. Out of these, 121 samples contained lipid concentrations above 5.0 μg/g. Of those, 73 samples were selected for GC-c-IRMS analysis. Table 7.1 summarises the number of samples for each type of analysis from the study sites in northwest India.

Site name	Site code	Ceramic samples analysed via GC- ΜS	Ceramic samples with appreciable quantities of lipid $($ >5 µg/g)	Ceramic samples analysed via $GC-c-IRMS$
Alamgirpur	ALM	15	14	9
Masudpur VII	MSDVII	31	28	21
Masudpur I	MSD1	29	23	14
Khanak	KNK	7	4	3
Lohari Ragho I	LHRI	26	16	12
Farmana	FRN	30	18	7
Rakhigarhi	RGR	30	18	7
Total		168	121	73

Table 7.1: Details of samples analysed via GC-MS and GC-c-IRMS analyses

The lipid profiles of vessel fragments from all sites were almost entirely composed of fatty acids of varying chain lengths, but dominated by medium-, odd- and branched-chain fatty acids, which suggest the presence of degraded animal fats such as dairy or carcass fats. There is minimal evidence for direct plant-processing, however, some vessels have evidence for dicarboxylic acids, long-chain fatty acids and *n*-alkanes, which could suggest plant input. There is no direct evidence for the processing of aquatic products. Compoundspecific isotopic analyses were used to further characterise the differences between the sources of animal fats processed in the vessels. The evidence demonstrated that a range of animal products and mixtures of animal (or plant) products were being processed in different vessels across different sites. Descriptions of sherd samples from each site, lipid concentrations, brief description of lipid composition, and isotopic values of the two main fatty acids $(C_{16:0}$ and $C_{18:0}$) where available, are provided in Appendix B.

The next section presents the site-by-site results of the GC-MS and GC-c-IRMS analyses and provides short discussions on vessel usage and food processing.

7. 2. Site-based results

7.2.1. Very small settlements (small villages)

This section presents and discusses the GC-MS and GC-c-IRMS results from samples from Alamgirpur and Masudpur VII. Given the very small estimated size of these settlements (1- 3 ha), it is likely the vessels are indicative of household food production, possibly reflecting food choices of a small number of families. However, as Alamgirpur and Masudpur VII are located in unique environmental contexts; respectively, by the edge of a summer-fed seasonal river, receiving little winter rainfall, and in an area receiving both summer and winter rain, it was expected that lipid residues within vessels would reflect varying food-processing strategies that reflect these differing environments.

7.2.1.1. Alamgirpur (ALM)

Rims of fourteen vessels were analysed from Alamgirpur. The vessel form and periods from which they originate have been introduced in Chapter Five, Section 5.1.2.1. Some of these contexts likely dated to a period after the 4.2 ka BP 'event' as indicated by Bayesian modelling (Jones 2017), whereas most contexts were deposited during 4.2 ka BP. The chronological span of these phases of occupation suggests the vessels investigated represent at least 300 years of vessel use-history at the site and may reflect changes in food processing strategies in response to increasing aridification experienced in the region by a small group of individuals at the village.

Lipid composition

The lipid profiles of the sherds from Alamgirpur contained saturated medium-chain fatty acids $(C_{12:0}, C_{14:0}, C_{16:0}, C_{18:0})$ typical of degraded animal fats, and in several instances, minor peaks of long-chain fatty acids $(C_{20:0}, C_{22:0}, C_{24:0})$ (see Figures 7.1 and 7.2), which could indicate plant input. All samples contained odd-chain fatty acids (such as $C_{15:0}$ and $C_{17:0}$, but only eight contained odd-branched-chain fatty acids (C_{15Br} and C_{17Br}), which are typical of ruminant fats. Unsaturated fatty acids such as $C_{16:1}$ and $C_{18:1}$ were present in nearly all samples, except three samples did not contain $C_{16:1}$. None of the samples contained dicarboxylic acids or *n*-alkanes except for ALM125-479, although this was likely a contaminant introduced during storage or processing (See Chapter 6, Figure 6.5). All vessels had P/S ratios between 1 and 3. All except two samples contained $C_{22:1}$ which likely has a laboratory or post-excavation origin as it rarely survives in archaeological contexts.

Compound-specific isotopic data

Nine vessels from Alamgirpur that were analysed were sampled for GC-c-IRMS. All isotopic data is tightly clustered, revealing no obvious differences between vessel forms (see Table 7.2, Figure 7.3).

Figure 7.1: Heatmap showing percentage contribution of different fatty acids to the total lipid content of individual samples from Alamgirpur, and presence/absence of branched-chain fatty acids, dicarboxylic acids and n -alkanes. FA: Fatty acid, where C_{nx} represents the carbon chain length and the degree of unsaturation. When unspecified, unsaturation is 0, Br: branched-chain fatty acid.

Figure 7.2: Partial total ion chromatogram of sample ALM117-276. Cn:x indicates fatty acid with n carbon atoms and x double bonds. IS: Internal Standard; P: Phthalate, Br: branched-chain fatty acid.

Sample ID	Rim/ Base/ Body	Vessel shape	Chronol ogical period	Lipid concentrati on $(\mu g/g)$	P/S ratio $(C_{16:0}$ $C_{18:0}$	$\delta^{13}C$ $C_{16:0}$	$\delta^{13}C$ $C_{18:0}$	$\varDelta^{13}C$ $(C_{18:0}$ - $C_{16:0}$
ALM114- 252	rim	large jar	LH	8.5	2.4	-29.0	-28.7	0.3
ALM117- 275	rim	$jar - rim$ diameter unknown	LH	11.1	1.8	-29.0	-29.0	0.0
ALM117- 279	rim	small jar	LH	9.9	1.5		-28.9 -29.4 -0.5	
ALM119- 370	rim	small jar	LH	25.1	2.1	$-27.8 - 28.6$		-0.7
ALM122- 397	rim	small jar	LH	15.2	2.6	$-28.5 -28.6$		-0.1
ALM124- 460	rim	small jar	LH	8.4	1.8	-28.0	-28.0	0.0
ALM125- 475	rim	small necked jar	LH	26.3	2.1	-27.8	-28.0	-0.1
ALM125- 481	rim	large necked jar	LH	11.9	1.6	-29.5	-29.8	-0.3
ALM125- 494	rim	small jar	LH	9.5	1.7	-28.7	-28.3	0.4
ALM125- 491	rim	large dish	LH	13.2	1.5	$-28.9 -29.2$		-0.3

Table 7.2: Details of samples analysed for GC-c-IRMS analysis from Alamgirpur.

Figure 7.3: $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values plotted against $\delta^{13}C_{16:0}$ values of vessel forms from Alamgirpur, colour-coded by trench and context number. The shaded areas in-between dotted lines indicate the regions where dairy fats and ruminant adipose fats, and ruminant adipose and nonruminant fats overlap respectively, indicating mixing of products.

Discussion

The molecular results from Alamgirpur suggest a remarkable degree of consistency in the products being processed in vessels. The lipid profiles of all the vessels are characteristic of degraded animal fats. For example, the presence of branched-chain fatty acids $(C_{15Br}$ and C_{17Br}) in most extracts may be indicative of the presence of ruminant fats (Dudd 1999).

When only considering the molecular results and contextualising these results within the available bioarchaeological evidence from the site, one could hypothesise that either cattle/buffalo or sheep/goat adipose fats or milk were processed in vessels, as zooarchaeological data from Alamgirpur reveal high percentages of cattle/buffalo remains with a minor presence of goats in the faunal assemblage (no clear evidence for sheep), and very minor presence of domestic/wild pig (Singh et al. 2013). Additionally, tooth enamel carbonate δ^{13} C values from domestic and wild animals suggest that cattle/buffalo and sheep/goat were consuming a high proportion of C_4 plants throughout the year (Jones 2017; Lightfoot et al. in prep.). Archaeobotanical evidence from the Late Harappan period at Alamgirpur indicates a dominance of C³ *rabi* (winter) cereals, especially barley and pulses, with minor evidence for *kharif* (summer) cereals such as small millets and tropical pulses (Singh et al. 2013; Bates in prep.). However, direct evidence of plant-processing is not visible in the lipid extracts.

But the fatty acid-specific isotopic results within lipid extracts from Alamgirpur vessels do not suggest the sole processing of ruminant adipose or dairy fats. Instead, comparisons with global reference fats suggest that these vessels were used to process nonruminant fats or, for a single vessel (ALM117-279, a painted jar with unknown rim diameter), mixtures of ruminant and non-ruminant fats (Figure 7.3). Given that there is no molecular evidence for direct processing of plant products within the vessels analysed, the δ^{13} C values from C_{16:0} fatty acids are likely indicative of plant input via animal diet, which is primarily C_3 with some evidence of mixing with C_4 plants. As both cattle/buffalo and sheep/goat at the site had high proportions of C_4 plants in their diet (Lightfoot et al. in prep.), this data suggests the meat of animals dominantly consuming C_3 plants were more often processed in vessels at Alamgirpur.

Importantly, there appears to be no evidence for dairy processing or use within the vessels. This absence of evidence does not unequivocally mean that dairy was not a component of the diet at Alamgirpur, as it is possible that vessels used for storing or processing dairy products were not sampled. It is also possible that dairy products were

mixed with other products during processing, or that the proportion of dairy products being consumed by populations was minimal.

It is also not possible to rule out the likelihood of other mixtures of products in vessels. Recent mixing models (Hendy et al. 2018, see Section 3.6.3.2) suggest vessels with mixtures of C_3 plants and dairy products from ruminants grazing on C_3 and C_4 plants yield Δ^{13} C values that plot within the ranges of ruminant adipose fats. Thus, vessels from Alamgirpur may also represent the use of vessels for cooking C_3 plants (wheat, barley, winter legumes, vegetables) and ruminant adipose products. Chapter Eight (Section 8.2.4) will demonstrate these values are also consistent with mixtures of C_3 plants and dairy products from C⁴ fed animals. Thus, at present the data is ambiguous and multiple interpretations are possible.

Both the molecular and isotopic data from the Alamgirpur vessels are remarkably consistent. There appear to be no differences in how specific vessels forms were being used; jars of different sizes and dishes seem to have been used to process, store, and/or serve similar foodstuff. However, lipid concentrations may be indicative of the frequency of vessel usage. Small- and medium-sized jars have relatively higher lipid concentrations compared to other vessels, but the difference is not statistically significant. Higher lipid concentrations in some vessels might suggest the more frequent usage of these vessels. There are also no observable differences in vessel-use over time or across different contexts, which suggests that there is no visible shift in vessel usage from during 4.2 ka BP to the period after 4.2 ka BP. It is possible that the populations of Alamgirpur were already well-adapted to seasonal fluctuations in rainfall and the relatively humid environment in the region enabled the maintenance of cropping or pastoral strategies at the settlement over long time periods. Although the number of samples investigated is small, the consistency of results reflects a uniformity in vessel usage over hundreds of years.

7.2.1.2. Masudpur VII (MSDVII)

Twenty-eight vessel fragments were selected for analysis from Masudpur VII. Details of the samples and chronological periods are provided in Section 5.1.2.2. The selected vessel fragments represent diversity in form and two unique chronological periods in Masudpur VII's occupation history. Several vessel fragments were selected from the same depositional context, enabling comparison of vessel usage within a relatively short time interval, and what likely represents the subsistence practices of a small number of individuals at the settlement.

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Lipid composition

Lipids preserved in the sherds from Masudpur VII contained medium-chain fatty acids $(C_{14:0}, C_{16:0}, C_{18:0}; 11)$ samples also contained $C_{12:0}$, and 22 out of 28 samples contained long-chain fatty acids like C_{20:0}, C_{22:0}, C_{:24:0}, C_{26:0}. All samples contained C_{15:0}, and most contained C17:0 fatty acids. MSD3586 also contained long-chain odd-chain fatty acids (Figure 7.5). Thirteen samples contained small peaks of series of long-chain *n*-alkanes from C_{26} -C₃₃ (odd-over-even carbon number predominating), and 21 of 28 (75%) of the samples contained dicarboxylic acids (diacids) ranging from C_6 to C_{12} carbon-chain-length, with C₉ (azelaic acid) the most abundant homologue. All vessels had P/S ratios between 1 and 3.

Compound-specific isotopic data

The 23 vessels sampled for compound-specific isotopic analyses included vessels from both the Early Mature Harappan (EMH) and Late Harappan (LH) periods, out of which three vessels from the EMH period were analysed. Most of the Δ^{13} C values plot between 1‰ and -1‰, except for two vessels (Table 7.3)

Figure 7.4: Heatmap showing percentage contribution of different fatty acids to the total lipid content of individual samples from Masudpur VII, and presence/absence of branched-chain fatty acids, dicarboxylic acids and *n*-alkanes. FA: Fatty acid, where C_{n:x} represents the carbon chain length and the degree of unsaturation. When unspecified, unsaturation is 0, Br: branched-chain fatty acid, Diacids: dicarboxylic acids.

Figure 7.5: Partial total ion chromatogram of samples MSD3603 and MSD3586. Cn:x indicates fatty acid with n carbon atoms and x double bonds. IS: Internal Standard; P: Phthalate, Br: branched-chain fatty acid; diacid: dicarboxylic acid; ALK: n-alkane

Sample ID	Rim/ Base/ Body	Vessel shape	Chronol- ogical period	Lipid concent- ration $(\mu g/g)$	P/S ratio $(C_{16:0}$ $C_{18:0}$	$\delta^{13}C$ $C_{16:0}$	$\delta^{13}C$ $C_{18:0}$	$\varDelta^{13}C$ $(C_{18:0}$ - $C_{16:0}$
MSD1788	body	perforated vessel	LH	23.8	0.7	-26.7	-27.3	-0.6
MSD1799	body	perforated vessel	LH	16.0	1.2	-28.2	-28.6	-0.4
MSD1873	body	perforated vessel	LH	17.4	1.7	-29.0	-28.5	0.5
MSD2115	rim	medium jar	EMH	23.5	1.8	-26.7	-25.6	1.1
MSD2116	rim	small jar	EMH	39.6	1.3	-27.9	-28.3	-0.4
MSD2209	rim	large bowl	EMH	17.2	2.4	-29.0	-28.7	0.3
MSD3392	rim	bowl	LH	11.6	2.2	-26.7	-27.7	-0.9
MSD3402	rim	jar	LH	9.2	1.2	-23.3	-24.7	-1.5
MSD3412A	rim	necked jar	LH	219.3	1.0	-24.8	-24.5	0.4
MSD3576	rim	jar	LH	14.1	1.0	-30.0	-30.3	-0.3
MSD3585	rim	ledged jar	LH	63.3	2.6	-27.8	-28.5	-0.7
MSD3586	rim	ledged jar	LH	66.7	1.4	-15.4	-19.6	-4.2
MSD3587	rim	small jar	LH	17.1	2.0	-29.0	-29.0	0.0
MSD3602	rim	jar	LH	23.9	2.0	-29.0	-30.0	-1.0
MSD3603	rim	jar	LH	31.0	1.6	-25.6	-24.9	0.7
MSD3788	rim	jar	LH	13.5	2.0	-28.7	-30.3	-1.5
MSD3794	rim	jar	LH	20.1	2.2	-28.7	-29.5	-0.8
MSD3795	rim	jar	LH	46.4	1.4	-26.7	-25.6	1.2
MSD3809	rim	ledged jar	LH	59.8	1.9	-23.1	-21.3	1.9
MSD3810	rim	ledged jar	LH	21.0	1.1	-28.3	-28.6	-0.3
MSD3813	rim	small jar	LH	14.0	1.8	-29.4	-29.7	-0.3

Table 7.3: Details of samples analysed for GC-c-IRMS analysis from Masudpur VII

Figure 7.6: $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values plotted against $\delta^{13}C_{16:0}$ values of vessel forms from Masudpur VII and colour-coded according to time period (EMH: Early Mature Harappan; LH: Late Harappan). The shaded areas in-between dotted lines indicate the regions where dairy fats and ruminant adipose fats, and ruminant adipose and non-ruminant fats overlap respectively, indicating mixing of products.

Figure 7.7: $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values plotted against $\delta^{13}C_{16:0}$ values of vessel forms from Masudpur VII and colour-coded by trench and context number. The shaded areas in-between dotted lines indicate the regions where dairy fats and ruminant adipose fats, and ruminant adipose and non-ruminant fats overlap respectively, indicating mixing of products.

Discussion

The molecular evidence from vessels from Masudpur VII suggest the predominance of animal fats in vessels. The ubiquity of odd-chain $(C_{15:0}$ and $C_{17:0}$) and branched-chain fatty acids (C_{15Br} and C_{17Br}) suggests that ruminant fats may have been processed in many vessels (after Dudd 1999). Minor plant input into the vessels is suggested by the presence of fatty acids such as $C_{12:0}$ and $C_{14:0}$, which are present in seed oils (Copley et al. 2001; Dunne et al. 2017), dicarboxylic acids, which are degradation products of unsaturated fatty acids generally present in high proportions in plant products (Regert et al. 1998), and *n*alkanes, which are compounds that originate from epicuticular waxes of vascular plants (Eglinton and Logan 1991; Dunne et al. 2017).

The molecular data appears to correlate with the zooarchaeological evidence from Masudpur VII, which demonstrates a preference for cattle/buffalo meat, with a minor presence of goats (there is no clear evidence for sheep) (Joglekar et al. 2016). The compound-specific isotopic data, however, demonstrates a variety of sources for the animal fats found in the vessels, including the processing of non-ruminant fats, mixtures of non-ruminant and ruminant fats, ruminant adipose fats and dairy fats.

It is notable that the $\delta^{13}C$ values from C_{16:0} fatty acids suggest variable input of C₃ and C⁴ plant products, with a single small ledged jar, MSD3586, indicating relatively higher C₄ plant input compared to other vessels. The Δ^{13} C value of this vessel suggests it lies within the global range for dairy products, which is unusual within this assemblage. Since tooth enamel carbonate values from domestic animals from Masudpur VII indicate that cattle/buffalo were predominantly feeding on C_4 plants throughout the year versus sheep/goat, which were feeding of mixtures of C_3 and C_4 plants (Lightfoot et al. in prep.), it is likely this vessel was used to process cow/buffalo milk or dairy products.

The archaeobotanical evidence suggests that there was a shift from a focus on a millet-based diet to wheat/barley cereal crops from the Early Harappan period to Mature Harappan period at Masudpur VII, with little further change in the Late Harappan period (but the evidence from this time period is limited) (Bates 2016; Petrie et al. 2016; Petrie and Bates 2017). The predominance of macrobotanical and phytolith evidence for wheat, barley, rice and pulses in the Mature and Late Harappan periods suggests that inhabitants of the settlement were predominantly consuming C_3 plants. Although there is very limited molecular evidence of the direct processing of plant products within the vessels analysed, the compound-specific isotopic data indicates that nearly all vessels have evidence for the input of both C_3 and C_4 plant products. Since seasonal shifts or differences in habitat

composition affect isotopic variations in plants that have been incorporated into the tissues of animals consuming them (Roffet-Salque et al. 2017b), the isotopic values of lipid extracts from vessels possibly demonstrate variable inputs of plants into different animals' diets.

Combining all the palaeo-ecological and bioarchaeological evidence together, it appears likely that the isotopic values from the vessels are either representative of the processing of the carcass of small ruminants that have mixed C_3-C_4 diets, mixed with nonruminant animal fats, or alternatively, of the cooking of C_3 plants mixed with dairy products from ruminants grazing on C_3 and C_4 plants, as suggested by recent mixing models (after Hendy et al. 2018). Chapter Eight (Section 8.2.4) will demonstrate these values are also consistent with mixtures of C_3 plants and dairy products from C_4 fed animals. Thus, at present the data is ambiguous and multiple interpretations are possible. Extracts with positive Δ^{13} C values are better explained by mixing models (Hendy et al. 2018; see Section 8.2.4) than by the assumption that non-ruminant fats were processed in vessels due to the paucity of these animals in the Masudpur VII faunal record.

Lipid concentrations of vessels from Masudpur VII suggest that ledged jars have higher mean lipid yields compared to other vessel forms (44.7 μg/g versus 23.6 μg/g [small jars], 19.2 μg/g [jars] and 16.4 μg/g [perforated jars]), suggesting their more frequent or prolonged use for processing oily or fatty products over other vessels. However, the compound-specific isotopic results demonstrate that the two ledged jars analysed were used to process different products: one for dairy products, and the other for non-ruminant fats or mixtures of plant and dairy products. Additionally, a single necked jar (MSD3412A) (Chapter Six, Figure 6.5) with the highest lipid concentration out of the entire assemblage (219.3 μ g/g) falls within the range for non-ruminant fats, which suggests it was either consistently used for storing lard or cooking pork, or for processing plant and dairy products intensively over a long period of time. As the data is complex and local references are unavailable, at present it is not possible to distinguish between these two diverse interpretations.

Overall, the results from Masudpur VII demonstrate the multifunctionality of vessels, as foodstuff derived from range of animal products, and possibly, mixtures of plants and dairy fats, were processed in vessels. Several vessels were obtained from the same context (Fig. 7.5), indicating they were deposited broadly within the same time period, and may also have been used contemporaneously. The spread of fatty acid-specific values from vessels within individual contexts reflects a diversity of animal products being processed in

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vessels, and importantly, a diversity of animal diets. This pattern may have broader implications for the conception of the organisation of animal management strategies in small Indus settlements. Additionally, although evidence from the EMH period is limited to three vessels from Masudpur VII, these vessels provide a picture of vessel usage across two distinct time periods from what likely represents quotidian practices of small households within this 1 ha village.

7.2.2. Small settlements ('villages')

This section presents and discusses the GC-MS and GC-c-IRMS results from samples from Masudpur I, Lohari Ragho I and Khanak. The estimated size of these settlements is between 3-10 hectares, which suggests that they were larger than Masudpur VII and Alamgirpur, but still represent the vessel use of populations living in villages. It is likely the vessels are indicative of household food production, possibly reflecting food choices of a small number of families. A comparison of the vessel-use practices of Masudpur I and Lohari Ragho I is useful, as although they are located about 5 km from each other, ongoing geoarchaeological research indicates that each had unique environmental contexts, with evidence of a riparian or pond environment surrounding Lohari Ragho I (Walker in prep), but not at Masudpur I. Khanak is located in a very arid environment and is considerably far away from both these sites.

7.2.2.1. Masudpur I (MSD I)

Twenty-four vessel fragments were analysed from Masudpur I. The vessel forms and chronological details of every sample are provided in Section 5.1.2.3. Several samples were selected from the same depositional context, enabling comparison of vessel usage within a relatively short time interval, and what likely represents the subsistence practices of a small number of individuals at the settlement.

Lipid composition

Of the 24 samples analysed, all contained medium-chain fatty acids such as $C_{12:0}$, $C_{14:0}$, $C_{16:0}$, $C_{18:0}$ (with all except 2 samples containing $C_{12:0}$ and/or $C_{14:0}$). Most samples contained odd-chain fatty acids such as $C_{15:0}$ and $C_{17:0}$ as well as branched-chain fatty acids like C_{15Br} and C_{17Br} . Several samples contained long-chain fatty acids such as $C_{20:0}$, $C_{22:0}$, $C_{24:0}$ and fifteen contained dicarboxylic acids (diacids) ranging from C_7 to C_{12} carbonchain-length, with C_9 (azelaic acid) the most abundant homologue (see Figure 7.8). All vessels had P/S ratios between 1 and 3. Nearly all samples contained $C_{22:1}$, which is an

unsaturated fatty acid that rarely survives in archaeological contexts and was likely introduced via the burial, post-excavation or laboratory environment (see Chapter Six).

Figure 7.8: Heatmap showing percentage contribution of different fatty acids to the total lipid content of individual samples from Masudpur I, and presence/absence of branched-chain fatty acids, dicarboxylic acids and n-alkanes. FA: Fatty acid, where Cn:x represents the carbon chain length and the degree of unsaturation. When unspecified, unsaturation is 0, Br: branched-chain fatty acid, Diacids: dicarboxylic acids.

Figure 7.9: Partial total ion chromatogram of sample MSD329. Cn:x indicates fatty acid with n carbon atoms and x double bonds. IS: Internal Standard; P: Phthalate, Br: branched-chain fatty acid; diacid: dicarboxylic acid.

Sample ID	Rim/ Base/ Body	Vessel shape	Chronol ogical period	Lipid concentrati on $(\mu g/g)$	P/S ratio $(C_{16:0}$ $C_{18:0}$	$\delta^{13}C$ $C_{16:0}$	$\delta^{13}C$ $C_{18:0}$	$\varDelta^{13}C$ $(C_{18:0}$ - $C_{16:0}$
MSD191	\lim	medium jar	LMH	23.3	2.1	-27.3	-27.4	-0.2
MSD192	rim	medium necked jar	LMH	6.6	2.0	-28.1	-28.0	0.1
MSD199	rim	medium jar	LMH	79.4	1.5	-20.4	-19.9	0.5
MSD200	rim	medium jar	LMH	11.6	2.0	-28.4	-28.7	-0.3
MSD214	rim	medium jar	LMH	30.1	2.5	-28.0	-27.7	0.4
MSD218	rim	large jar	LMH	57.7	2.5	-27.7	-27.0	0.7
MSD264	rim	medium necked jar	LMH	24.6	1.5	-30.3	-30.3	0.0
MSD273	rim	small jar	LMH	15.8	1.5	-29.8	-30.3	-0.5
MSD329	rim	medium necked jar	LMH	38.3	1.2	-14.7	-18.7	-4.1
MSD343	rim	small jar	LMH	122.6	1.2	-22.4	-22.6	-0.2
MSD1326	rim	small jar	LMH	23.5	1.7	-26.7	-27.3	-0.6
MSD1557	body	perforated vessel	LMH	23.3	0.8	-29.2	-29.3	-0.1
MSD1597	body	jar - rim diameter unknown	LMH	13.0	1.4	-29.5	-29.6	-0.1
MSD1712	rim	small necked jar	EMH	10.2	1.2	-28.2	-27.0	1.2

Table 7.4: Details of samples analysed for GC-c-IRMS analysis from Masudpur I.

Figure 7.10: $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values plotted against $\delta^{13}C_{16:0}$ values values of different vessels from Masudpur I, colour-coded according to context. The shaded areas in-between dotted lines indicate the regions where dairy fats and ruminant adipose fats, and ruminant adipose and non-ruminant fats overlap respectively, indicating mixing of products.

Compound-specific isotopic data

Fourteen of these vessel fragments were sampled for GC-c-IRMS analysis (Table 7.4). While most values cluster together, three vessels are distinctive: these include a mediummouthed necked jar $(\Delta^{13}C = -4.1\%)$, and two vessels (small- and medium-mouthed jars) with more positive $\delta^{13}C_{16:0}$ values (-22.4 ‰ and -20.4 ‰) relative to the other samples. The rest of the Δ^{13} C values range from -0.5‰ to 1.2‰ (Figure 7.10).

Discussion

The lipid profiles of the vessels from Masudpur I mostly appear to be characteristic of degraded animal fats. The ubiquity of odd-chain $(C_{15:0}$ and $C_{17:0}$) and branched-chain fatty acids (C_{15Br} and C_{17Br}) suggests a dominance of ruminant fats as these bacterial lipids originate within the rumen (Dudd et al.1999) (Figure 7.8). More than half of the samples contain small proportions of dicarboxylic acids, which are degradation products of unsaturated fatty acids (Regert et al. 1998), also contain $C_{12:0}$ and $C_{14:0}$ fatty acids (present in seed oils) (Dunne et al. 2017) and have even-numbered long-chain fatty acids, all of which may be indicative of their being derived from plants. However, as these compounds occur in low proportions, they are not sufficient to conclusively suggest plant origin.

When contextualised with the available zooarchaeological evidence, the presence of degraded animal fats in vessels is supported by the presence of domestic animals at the site, especially cattle/buffalo and a smaller proportion of sheep/goat (Joglekar et al. 2017). However, the fatty acid-specific isotopic values from Masudpur I are similar to those from the very small settlements, with the Δ^{13} C values of lipids from all vessels falling within the reference range for non-ruminant products, except one that demonstrates evidence for dairy processing (MSD329, a medium-mouthed necked jar). Like at other settlements, this pattern is unusual, as non-ruminant animals such as pigs only comprise about 2% NISP of faunal remains found at the site (Joglekar et al. 2017). Just as at other sites, these values are also explained by mixtures of C_3 plants and mixed with dairy products from ruminants grazing on C_3 and C_4 plants (Hendy et al. 2018), and other hypothetical mixtures (see Section 8.2.4) in these vessels. Thus, the results are ambiguous, and a single interpretation is not possible at present.

While most vessels have $\delta^{13}C_{16:0}$ values that indicate the input of both C₃ and C₄ plants, three vessels (MSD199, a medium-mouthed jar; MSD343, a small jar; and MSD329; see Chapter Six, Figure 6.5, and Figure 7.9) have relatively positive $\delta^{13}C_{16:0}$ values, suggesting a higher input of C_4 plants in them relative to other vessels. Although this pattern may indicate the direct processing of C_4 plants (millets or wild plants) in these vessels, it is most likely that they reflect plants incorporated into the tissues of animals as there is no clear molecular evidence for direct plant-processing in the vessels. This could suggest that the fat of non-ruminant animals with high C_4 diets were processed in vessels MSD343 and MSD199. Both vessels also have relatively high lipid concentrations (79.4 μ g/g and 122.6 μ g/g, respectively) which may indicate these vessels were used more frequently. Similarly, it is likely that vessel MSD329 was predominantly used for the processing of dairy products from cow/buffalo milk, as enamel carbonate isotopic analyses of domestic and wild animals from the site indicate that cattle/buffalo consumed higher proportions of C_4 plant than sheep/goat (Lightfoot et al. in prep). Archaeobotanical analysis suggests that both summer and winter crops were grown at Masudpur I, with millets being grown more frequently and in greater proportions than wheat/barley (Bates 2016; Petrie and Bates 2017). Thus, it is possible that these crops or their by-products were also used to feed domestic animals.

No clear relationship between vessel form and vessel content emerges, suggesting the multi-functionality of vessels. The δ^{13} C values of a fragment of a perforated vessel (MSD1710) do not indicate that it was used for the processing or straining of dairy

products, but rather for the processing of animal products with a mixed C_3-C_4 plant dietary input. The vessel with clear evidence for dairy is a necked jar with a narrow mouth. Such a vessel would be ideal for the storage and pouring of dairy products such as ghee, yoghurt, or milk. However, as Figure 7.10 suggests, small- and medium-mouthed vessels were also used for the processing of other products. Additionally, a large proportion of the analysed vessels from Masudpur I originated from the same context (Trench XA1, context 110, fill), suggesting their deposition occurred within narrow spatial and chronological loci within a domestic setting. The spread of fatty acid-specific values from the vessels from this single context, however, suggest that a range of products were being processed or transformed in vessels by inhabitants. These results provide a snapshot into quotidian practices of populations at small Indus villages.

7.2.2.2. Lohari Ragho I (LHRI)

Twenty-four vessel fragments from Lohari Ragho I were selected for analysis. Vessel forms and chronological details for every sample are provided in Section 5.1.3.1. Overall, the samples are indicative of vessel-usage by a small group of individuals or families in the EMH period living within the settlement.

Lipid composition

Lipid extracts from sixteen out of twenty-four vessels from Lohari Ragho I contained medium-chain fatty acids such as $C_{12:0}$, $C_{14:0}$, $C_{16:0}$, $C_{18:0}$ and unsaturated fatty acids such as $C_{16:1}$ and $C_{18:1}$. Eleven out of 16 samples contained odd-chain fatty acids (C_{15:0} and C_{17:0}, and in some cases $C_{19:0}$, and 14 out of 16 contained long-chain fatty acids between $C_{20:0}$ -C26:0, although generally in low proportions (see Figures 7.11 and 7.12).

Less than half of the samples contained branched-chain fatty acids (e.g. C_{15Br} and C_{17Br} , with one sample containing C_{13Br} and C_{19Br}). Four out of 17 samples contained dicarboxylic acids (diacids) ranging from C_7 to C_9 carbon-chain-length, with C_9 (azelaic acid) as the most abundant homologue. All vessels had P/S ratios between 1 and 3. Nearly all samples contained unsaturated fatty acids such $C_{20:1}$ and $C_{22:1}$, and some contained *n*alkanes, which were likely introduced via the burial or laboratory environment as they rarely survive in archaeological contexts (see Chapter Six).

Figure 7.11: Heatmap showing percentage contribution of different fatty acids to the total lipid content of individual samples from Lohari Ragho I, and presence/absence of branched-chain fatty acids, dicarboxylic acids and n -alkanes. FA: Fatty acid, where C_{nx} represents the carbon chain length and the degree of unsaturation. When unspecified, unsaturation is 0, Br: branched-chain fatty acid, Diacids: dicarboxylic acids.

Figure 7.12: Partial total ion chromatogram of sample LHR10. Cn:x indicates fatty acid with n carbon atoms and x double bonds. IS: Internal Standard; P: Phthalate, Br: branched-chain fatty acid; diacid: dicarboxylic acid.

Sample ID	Rim/ Base/ Body	Vessel shape	Chronolo gical period	Lipid concentrati on $(\mu g/g)$	P/S ratio $(C_{16:0}/$ $C_{18:0}$	$\delta^{13}C$ $C_{16:0}$	$\delta^{13}C$ $C_{18:0}$	$\varDelta^{13}C$ $(C_{18:0}$ - $C_{16:0}$
LHR03	rim	globular jar globular	LMH	44.6	0.8	-30.7	-30.7	0.1
LHR07	rim	jar	LMH	34.6	0.9	-28.3	-28.8	-0.5
LHR09	rim	necked jar	EMH	29.9	0.8	-29.3	-29.9	-0.6
LHR10	rim	large jar	EMH	214.7	1.0	-14.2	-15.1	-0.9
LHR11	rim	large jar perforated	EMH	24.3	1.2	-23.0	-24.3	-1.3
LHR12	body	vessel	EMH	16.4	0.8	-30.2	-30.7	-0.5
LHR13	rim	necked jar	EMH	27.3	1.0	-24.9	-24.7	0.2
LHR25	rim	small jar medium	EMH	19.2	0.7	-31.0	-31.2	-0.2
LHR ₂₆	rim	jar	EMH	21.1	0.7	-18.8	-19.2	-0.4
LHR27	rim	necked jar	EMH	17.2	0.7	-31.2	-31.3	0.0
		perforated						
LHR38	body	vessel	EMH	12.9	1.0	-27.8	-27.7	0.0
LHR40	rim	large jar	EMH	22.0	1.0	-15.5	-17.2	-1.7

Table 7.5: Details of samples analysed for GC-c-IRMS analysis from Lohari Ragho I

Figure 7.13: $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values plotted against $\delta^{13}C_{16:0}$ values of different vessel forms from Lohari Ragho I, colour-coded according to time period. The shaded areas in-between dotted lines indicate the regions where dairy fats and ruminant adipose fats, and ruminant adipose and non-ruminant fats overlap respectively, indicating mixing of products.

Figure 7.14: $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values plotted against $\delta^{13}C_{16:0}$ values of different vessel forms from Lohari Ragho I, colour-coded according to context. The shaded areas in-between dotted lines indicate the regions where dairy fats and ruminant adipose fats, and ruminant adipose and nonruminant fats overlap respectively, indicating mixing of products.

Compound-specific isotopic results

The 12 vessels sampled for compound-specific isotopic analyses included jars with small, medium-sized and large-mouths (n=8), two perforated jars and two globular jars. The results are presented in Figures 7.13 and 7.14. The plots indicate that the range of $\delta^{13}C$ values of the $C_{16:0}$ and $C_{18:0}$ fatty acids is very large (Table 7.5). Jars of different sizes have a wide range of isotopic values, but the two perforated vessels plot closely together (Figure 7.14). The isotopic values of the two globular jars are also similar. The $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values of the vessels, however, do not show as wide a range, with nine out of the twelve vessels (75%) with values higher than -0.8‰.

Discussion

The lipid profiles and isotopic data from vessels from Lohari Ragho I are intriguing. As at the other sites, the molecular profiles of the vessels are characteristic of degraded animal fats, with additional evidence required to ascertain any substantial contribution of plant products to the amorphous residues. Similarly, the compound-specific isotopic results demonstrate little variation in terms of the type of animal product being processed in

vessels, but the key difference is that they reflect tremendous diversity of C_3 and C_4 plantinput into vessels.

The δ^{13} C values from fatty acids from nine out of twelve vessels extracts fall within the threshold of non-ruminant fats. Values from two vessels (LHR11, LHR40) fall within the ruminant adipose range, and one jar (LHR10) suggests mixing of non-ruminant and ruminant adipose fats. None of the vessels demonstrate evidence for dairy processing. The range of $\delta^{13}C_{16:0}$ values within the lipid extracts is very wide (-31‰ to -14.2‰), possibly suggesting a wide range of C_3 and C_4 plants incorporated into the tissues of animals consuming them (Roffet-Salque et al. 2017b). Since isotopic values in plants are influenced by seasonal shifts or differences in habitat composition, these values may also indicate that animals were moving across different landscapes or foddering on different types of plants in their lifetimes (Roffet-Salque et al. 2017b). Although most vessels have $\delta^{13}C_{16:0}$ values indicating predominantly C₃ plant input in the vessels, five vessels demonstrate increasingly high $\delta^{13}C_{16:0}$ values, suggesting higher input of C₄ plants. Out of these, three have Δ^{13} C values that fall within the global ruminant adipose threshold, and one vessel (LHR26: medium-mouthed, dark-slipped jar) suggests it was used to process a mix of ruminant and non-ruminant fats. As enamel isotopic data demonstrates that cattle/buffalo fed off C_4 plants throughout the year at Lohari Ragho I (Lightfoot et al. in prep), it is likely that jars LHR10, LHR11 and LHR40 were predominantly used to process ruminant carcass fats, particularly those of cattle/buffalo. Thus, vessels from Lohari Ragho I provide relatively unambiguous evidence of beef processing. Sample LHR10 also contained the highest concentration of lipid out of the assemblage (214.7 μg/g) and had relatively high proportions of dicarboxylic acids in its lipid profile, which may suggest the vessel was also used to process plant products (Regert et al. 1998).

It is not possible to contextualise the results with bioarchaeological information as the archaeobotanical and faunal analyses from Lohari Ragho I are currently under analysis. Preliminary results from archaeobotanical data indicate the presence of millet species and legumes. As the enamel isotopic evidence demonstrates that cattle/buffalo fed off C_4 plants throughout the year, it is possible that millets were being used to fodder animals at the site. Faunal analyses are yet to be undertaken, but proportions of riverine fishbone recovered have been relatively high, which supports ongoing geoarchaeological analysis that suggests it was located near a riparian area (Walker in prep). The presence of fishbone adds another possible strand of interpretation, as some of the $\delta^{13}C_{16:0}$ values obtained may also be indicative of the processing of freshwater aquatic products in vessels. However, as aquatic

biomarkers from lipid extracts are absent, and the carbon isotope compositions of fatty acids from freshwater fish are highly variable (Craig et al. 2007), this is purely speculative. Further analysis of lipids from a range of reference freshwater, estuarine and marine organisms in South Asia would be needed to confirm this interpretation.

Due to small sample sizes, determining a relationship between vessel forms and what vessels were used for is not straightforward. The $\delta^{13}C_{16:0}$ values of lipid extracts from large- and medium-mouthed jars are more variable (n=5), whereas small-mouthed and perforated jars cluster more closely together (n=5), possibly indicating their use for similar products. This may be suggestive of specific vessel-use conventions or practices unique to Lohari Ragho I. It is also notable that most samples date to the EMH period and many originate from a single context (EA-520, fill above a mudbrick floor), indicating that inhabitants at the site processed products derived from animals that were fed on variable diets within a relatively short span of time. However, as all vessels seem to have been used to process non-ruminant and/or ruminant carcass fats, the differences in fatty acid-specific δ^{13} C values may not actually translate to significant variation in foodstuff.

Zooarchaeological analyses from Lohari Ragho I are not yet available, but it is likely the results will be similar to those from other Indus sites in the region, which makes the high percentage of non-ruminant fats in vessels difficult to interpret. Just like at other sites, while it is possible that porcine or avian products were preferentially processed in these vessels, it is also possible that the δ^{13} C values from lipids are a result of the mixing of different foodstuffs such as C_3 plants and dairy products from ruminants grazing on C_3 and C⁴ plants (Hendy et al. 2018), and other hypothetical mixtures (see Section 8.2.4). These may either reflect the multi-use of vessels, or that foodstuff was prepared with multiple, mixed ingredients. As the results are ambiguous, presently, a single, conclusive interpretation is not possible.

7.2.2.3. Khanak (KNK)

Located within an arid climate zone, Khanak occupies an environmental context that is different from all other Indus sites investigated in this thesis. The vessel fragments analysed originate from deposits that demonstrated evidence of pyrotechnical waste, possibly suggesting that inhabitants at the site were engaging in craft-production or metallurgical activities. Only a small number of vessels were studied and their details are provided in Section 5.1.3.2.

Lipid composition

Only four vessel fragments contained lipid concentrations above 5 μg/g. Of these vessels, lipid extracts usually comprised of fatty acids between $C_{14:0}$ to $C_{22:0}$, with high concentrations of C16:0 and C18:0. One vessel, KNK11 (small jar) had an unusual lipid profile (Figure 7.16), with the presence of fatty acids between $C_{14:0}$ to $C_{28:0}$, including oddchain long-chain fatty acids and relatively high concentrations of $C_{22:0}$. All extracts contained unsaturated fatty acids $C_{16:1}$ and $C_{18:1}$, odd-chain fatty acids $C_{15:0}$ and $C_{17:0}$ and branched-chain fatty acids (C_{15Br} and C_{17Br}). KNK01 and KNK02 contained dicarboxylic acids and KNK02 had a relatively high lipid concentration than other vessels (131 μg/g) (see Chapter Six, Figure 6.7). All vessels had P/S ratios between 1 and 3. Most vessels contained unsaturated fatty acids such C20:1 and C22:1, and KNK18 contained *n*-alkanes, which were likely introduced via the burial or laboratory environment

Compound-specific isotopic data

Three samples were selected for GC-c-IRMS analysis. Despite the small sample size, the isotopic range of $C_{16:0}$ and $C_{18:0}$ fatty acids from the analysed vessels is wide (Table 7.6). The Δ^{13} C values also show variation, ranging from -3‰ to -0.5‰.

Figure 7.15: Heatmap showing percentage contribution of different fatty acids to the total lipid content of individual samples from Khanak, and presence/absence of branched-chain fatty acids, dicarboxylic acids and *n*-alkanes. FA: Fatty acid, where C_{n:x} represents the carbon chain length and the degree of unsaturation. When unspecified, unsaturation is 0, Br: branched-chain fatty acid, Diacids: dicarboxylic acids.

Figure 7.16: Partial total ion chromatogram of sample KNK11. Cn:x indicates fatty acid with n carbon atoms and x double bonds. IS: Internal Standard.

Table 7.6: Details of samples analysed for GC-c-IRMS analysis from Khanak.

Sample	Rim/	Vessel	Chronolo	Lipid	P/S	$\delta^{13}C$	$\delta^{13}C$	$A^{13}C$
ID	Base/	shape	gical	concentration	ratio	$C_{16,0}$	$C_{18:0}$	$(C_{18:0}$
	Body		period	$(\mu g/g)$	$(C_{16.0}$			$C_{16:0}$
					$C_{18:0}$			
KNK01	base	iar	LMH	18.2	1.0	-18.5	-21.6	-3.0
KNK02	rim	small jar	LMH	131	1.2	-15.3	-16.0	-0.7
KNK11	rim	iar	EMH	39.2	1.0	-28.0	-28.5	-0.5

Figure 7.17: $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values plotted against $\delta^{13}C_{16:0}$ values of vessels from Khanak, colour-coded according to context. The shaded areas in-between dotted lines indicate the regions where dairy fats and ruminant adipose fats, and ruminant and non-ruminant fats overlap respectively, indicating mixing of products.

Discussion

The lipid extracts from Khanak, just like at other sites analyses for this thesis, are not very diagnostic and may broadly be interpreted as degraded animal fats. The presence of odd long-chain fatty acids and high proportions of even long-chain fatty acids in KNK11 is unusual for a lipid residue originating from pottery and may be attributable to soil contamination. The compound-specific isotopic results reveal that two out of three lipid extracts from vessels have Δ^{13} C values that plot within the threshold of mixed ruminant and non-ruminant fats. Importantly, though, they vary in terms of their C_4 plant-input: KNK02 has a $\delta^{13}C_{16:0}$ value of -15.3‰, whereas KNK11 has a $\delta^{13}C_{16:0}$ value of -28‰, suggesting that a greater proportion of C_4 plants were indirectly (or directly) processed in KNK02 than in KNK11. KNK01, however, has a Δ^{13} C value of -3‰, which suggests that it may have been used to process both ruminant adipose and dairy fats. With a $\delta^{13}C_{16:0}$ value of -18.5‰, this vessel likely was used to process a mixture of C_3 and C_4 plants, with higher amounts of direct or indirect C_4 plant-input.

As the faunal and archaeobotanical remains from Khanak are currently under study it is not possible contextualise the results with other bioarchaeological data. However, if tooth enamel isotopic data from other Haryana sites are extrapolated, it may be possible to distinguish between the likely use of KNK11, which was possibly used to process the animals consuming higher proportions of C_3 plants (sheep/goat or pigs), and KNK02, which demonstrates relatively higher $\delta^{13}C_{16:0}$ values (and thereby high C₄ plant-input) and was probably predominantly used to process cattle/buffalo adipose fats as they were more likely to have been feeding on C₄ plants throughout the year. This vessel also has a relatively high lipid concentration (131 μg/g), which may suggest frequency of use relative to other vessels. Vessel KNK01, however, was likely used to process both sheep/goat and cattle/buffalo adipose fats, as well as dairy products from either sheep/goat or cows. Thus, the variation in δ^{13} C values from even a limited set of samples is informative and points to the diversity of products being processed in vessels. Even with the small sample size, it is possible to suggest that both meat and dairy products were being processed and consumed by inhabitants at Khanak, some of whom were also likely engaging in craft- or metalproduction.

7.2.3. Mid-sized settlements ('towns')

The results of the molecular (GC-MS) and compound-specific isotopic (GC-c-IRMS) analysis from samples from Farmana are presented below. The estimated size of this

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settlement is between 10-20 hectares, which suggests that it can be characterised as a 'smaller-than-city' settlement within the constellation of Indus settlements in the region (Petrie 2013).The contextual information from excavations suggests that vessels analysed from this site are likely indicative of household food production, possibly reflecting food choices of a small number of families over a relatively short period of time within the Early Mature Harappan (EMH) period.

7.2.3.1. Farmana (FRN)

Eighteen vessels had lipid concentrations above 5 μ g/g, the details of which are provided in Chapter Five, Section 5.1.2.4. They originated from different locations within the structural complexes exposed at the site, allowing for comparisons between vessel use across different households or buildings. Comparison between 'Haryana Harappan' and 'Classic Harappan' pottery was also possible (cf. Uesugi 2011).

Lipid composition

Nearly all analysed vessels contained mid-chain fatty acids such as $C_{14:0}$, $C_{16:0}$, $C_{18:0}$, and seven vessels also contained $C_{12:0}$. Long-chain fatty acids $(C_{20:0}, C_{22:0}, C_{24:0})$ were only present in nine vessels in very low proportions, but all samples contained odd-chain fatty acids ($C_{15:0}$, $C_{17:0}$). Most vessels contained $C_{16:1}$ and/or $C_{18:1}$ unsaturated fatty acid, however, unusually, four vessels contained unmethylated $C_{18:1}$.

Six out of eighteen vessels contained branched-chain fatty acids $(C_{15Br}$ and C_{17Br}). Only 4 out of 18 (22%) vessels contained dicarboxylic acids in very low concentrations (see Figure 7.18). A single vessel (FRN26, body sherd from a jar with mud applique surface decoration) had a P/S ratio of 3.5, while the rest had P/S ratios between 1 and 3. Six vessels contained unsaturated fatty acids such as $C_{20:1}$ and $C_{22:1}$ which were likely introduced via the burial or laboratory environment as they rarely survive in archaeological contexts, and some contained *n*-alkanes, but these may also be attributed to contamination (see Chapter Six).

Compound-specific isotopic data

Seven vessels were selected for GC-c-IRMS analysis. These included small jars (n=2), a medium jar, a large jar, a perforated vessel, a ledged jar, and a jar with an unknown rim diameter. Despite the small sample size, the range of isotopic values of the lipid extracts was wide (Table 7.7). The Δ^{13} C values also demonstrated considerable variability, ranging from -3.8‰ to -0.3‰.

Figure 7.18: Heatmap showing percentage contribution of different fatty acids to the total lipid content of individual samples from Farmana, and presence/absence of branched-chain fatty acids, dicarboxylic acids and *n*-alkanes. FA: Fatty acid, where C_{n:x} represents the carbon chain length and the degree of unsaturation. When unspecified, unsaturation is 0, Br: branched-chain fatty acid, Diacids: dicarboxylic acids.

Figure 7.19: Partial total ion chromatogram of sample FRN18. Cn:x indicates fatty acid with n carbon atoms and x double bonds. IS: Internal Standard; diacid: dicarboxylic acid; Br: branchedchain fatty acid.

Sample ID	Rim⁄ Base/ Neck/ Body	Vessel shape	'Haryana' or 'Classic Harappan'	Chronol ogical period	Lipid concen tration $(\mu g/g)$	P/S ratio $(C_{16:0}$ $/C_{18:0}$	$\delta^{13}C$ $C_{16:0}$	$\delta^{13}C$ $C_{18:0}$	$A^{13}C$ $(C_{18:0}$ $C_{16:0}$
FRN04	neck	small jar	'Haryana Harappan'	EMH	14.0	1.7	-16.0	-19.8	-3.8
FRN09	rim	medium jar	'Haryana Harappan'	EMH	41.4	1.0	-19.3	-20.2	-0.9
FRN11	body	perforated vessel	'Haryana Harappan'	EMH	23.6	1.2	-27.0	-30.0	-3.0
FRN13	rim	small jar	'Haryana Harappan'	EMH	12.3	1.1	-28.2	-27.9	0.3
FRN14	rim	large jar	'Classic Harappan'	EMH	17.6	1.1	-26.2	-25.9	0.3
FRN18	rim	ledged jar	'Classic Harappan'	EMH	45.6	1.2	-20.2	-23.0	-2.8
FRN ₂₆	body	jar - rim diam unknown	'Classic Harappan'	EMH	9.7	3.5	-25.3	-26.4	-1.1

Table 7.7: Details of samples analysed for GC-c-IRMS analysis from Farmana.

Figure 7.20: $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values plotted against $\delta^{13}C_{16:0}$ values of vessels from Farmana, colour-coded according to context. The shaded areas in-between dotted lines indicate the regions where dairy fats and ruminant adipose fats, and ruminant adipose and non-ruminant fats overlap respectively, indicating mixing of products.

Discussion

The molecular results from vessels from Farmana suggest that the lipid extracts can broadly be characterised as degraded animal fats. A single vessel (FRN26) has a P/S ratio close to 4, which may suggest it is derived from a plant product (Dunne et al. 2017). However, the rest of its molecular composition is not characteristic of plant-derived compounds; much like vessels from other sites, and it contains fatty acids ranging from C14:0 to C20:0 and has no evidence for diacids or long-chain *n*-alkanes.

The fatty acid-specific isotopic results are more informative about the diversity of vessel products processed in vessels. The range in Δ^{13} C values is indicative of different animal products processed in vessels, including dairy, ruminant adipose and non-ruminant fats. Additionally, the variability of $\delta^{13}C_{16:0}$ values indicates that the foodstuff prepared in vessels from Farmana was derived from a range of C_3 and C_4 plants.

Two vessels, FRN26 and FRN13, have Δ^{13} C values that fall within the range for ruminant adipose fats. Both vessels have $\delta^{13}C_{16:0}$ values indicating the mixing of C₃ and C₄ plants, which may suggest they were used for the processing of sheep/goat meat, as enamel carbonate values from domestic animals from Farmana suggest that sheep/goat had mixed C₃/C₄ diets compared to cattle/buffalo that were consistently feeding on C₄ plants (Tames-Demauras 2018). It is possible that many products were mixed in this vessel which makes it difficult to ascertain a single, specified use.

Conversely, two vessels, FRN14, a large jar, and FRN18, a ledged jar, have $\Delta^{13}C$ values that fall within the range for non-ruminant fats. Both vessels have $\delta^{13}C_{16:0}$ values which indicate the mixing of C_3 and C_4 plant-input. As suggested for other sites, this could represent the processing of omnivorous animals such as pigs/birds in the vessels. However, these values may also be generated via mixtures of C_3 plants and dairy products from ruminants grazing on C_3 and C_4 plants (Hendy et al. 2018), as well as other hypothetical mixtures (Section 8.2.4). As the results are ambiguous, presently, a single interpretation is not possible.

At least three vessels demonstrate evidence of processing, or mixing of dairy products: FRN04, a dark- slipped medium-sized jar, FRN11, a perforated vessel, and FRN18, a small red-slipped and black-painted jar. These vessels show variable input of C⁴ plants. FRN04, which shows clear evidence for dairy processing, has relatively higher $\delta^{13}C_{16:0}$ values, and suggests a high (likely indirect) C₄ plant input into the vessel. FRN18 and FRN11, however, have Δ^{13} C values that suggest the mixing of dairy- and ruminantcarcass fats, and demonstrate lower $\delta^{13}C_{16:0}$ values, indicating relatively less input of C₄

plants. As cattle/buffalo at Farmana were consistently feeding on C_4 plants throughout the year and sheep/goat had more variable diets (Tames-Demauras 2018), it is highly likely that FRN04 was predominantly used to process products derived from cattle/buffalo milk, whereas FRN11 and FRN18 contained mixtures of sheep/goat or wild ruminant carcass and sheep/goat and cattle/buffalo dairy products.

Farmana has stronger evidence for the processing of dairy products in ceramic vessels compared to other sites. FRN11 is the only perforated vessel from the analysed assemblage from all sites that is strongly associated with the processing of dairy, but it was likely mixed with ruminant fats. There is no clear relationship between the form of vessels and the source of the products processed inside them, as indicated by Figure 7.20. Both small jars and large storage jars were used to keep either non-ruminant fats, or, as mixing models suggest, C_3 plants and dairy products from ruminants grazing on C_3 and C_4 plants or other mixtures of products (Hendy et al. 2018; see Section 8.2.4), suggesting the multifunctionality of vessels. Furthermore, there is no observable difference between the molecular profiles or isotopic values of the locally/regionally produced 'Haryana Harappan' pottery and 'Classic Harappan' pottery found at Farmana.

Most of the vessels from Farmana were likely discarded during the same time period. The results suggest that vessels deposited within or right outside Structural Complex 3 had multiple uses. Thus, vessels deposited within or right outside the same locality appear to have been used to process a broad range of foodstuffs. As the structures at Farmana are large and contain central courtyards, dwelling rooms and antechambers for storage; they have been interpreted as residential complexes occupied by large, extended families (Shinde et al. 2011). Structural Complex 3 has been interpreted as being occupied by an influential and/or 'elite' family consisting of several households (at least eight) due to its large size and central location within the settlement (Shinde et al. 2011). Although analysis of more vessels from different structural complexes and areas across the site would enable comparisons between different households, the current evidence suggests that single household units likely processed both meat and milk products in vessels, yielding insight into family- or household-level culinary practices.

7.2.4. Large settlements ('cities')

Rakhigarhi was the only large Indus settlement from which samples were selected for analysis from known contexts (for the results of lipid and compound-specific isotopic analyses from Mohenjo-daro and Kalibangan, see Appendix F). Samples from Rakhigarhi

were selected Mound RGR-4. The role of RGR-4 within the larger settlement is unclear, but excavations suggest evidence of domestic structures, storage features and large dump/fill contexts.

7.2.4.1. Rakhigarhi (RGR)

Eighteen vessel fragments were analysed from Rakhigarhi, and their details are provided in Section 5.1.2.5. Vessel fragments were mostly collected from what were domestic structures, although some samples are associated a mudbrick platform and what was possibly a large storage feature (Yadav, pers. comm.). The samples analysed were found close to one another, and their relative position in the stratigraphy and radiocarbon dates from the site suggests they may have been contemporaneous. These samples thus provide an opportunity to examine the vessel-use strategies of a small subsection of inhabitants at Rakhigarhi in the Early Mature Harappan (EMH) period.

Lipid composition

Lipid extracts from vessel fragments from Rakhigarhi mostly contained mid-chain fatty acids such as $C_{14:0}$, $C_{16:0}$, $C_{18:0}$ (only three samples contained low proportions of $C_{12:0}$), and odd-chain fatty acids such as $C_{15:0}$ and $C_{17:0}$ (with the exception of three samples). Only 4 out of 18 vessels (22%) contained branched-chain fatty acids (C_{15Br} and C_{17Br}), and five vessels contained low proportions of C9 dicarboxylic acid.

Twelve out of eighteen samples contained even-numbered long-chain fatty acids $(C_{20:0}$ -C_{24:0}), and while most vessels had very low proportions, a single vessel (RGR24, a small-mouthed necked vessel) contained high abundances of long-chain fatty acids, higher than both $C_{16:0}$ and $C_{18:0}$, maximising at $C_{24:0}$. This vessel also had a series of odd-chain *n*alkanes upto C_{31} , maximising at C_{23} . While other vessels contained low proportions of *n*alkanes, they did not have a distinctive pattern. While all vessels had P/S ratios between 1- 3, RGR05, a large storage jar, had a P/S ratio of 3.5. Nearly all vessels contained unsaturated fatty acids such as $C_{16:1}$ and/or $C_{18:1}$. Four vessels contained unsaturated fatty acids such as $C_{20:1}$ and $C_{22:1}$ which were likely introduced via the burial or laboratory environment as they rarely survive in archaeological contexts (see Chapter Six).

Compound-specific isotopic data

Seven vessel fragments were selected for GC-c -IRMS analysis. The results from the compound-specific isotopic analysis are interesting as they demonstrate wide ranges, with Δ^{13} C values ranging from 3.4‰ to -3.4‰ (Table 7.8).

Figure 7.21: Heatmap showing percentage contribution of different fatty acids to the total lipid content of individual samples from Rakhigarhi, and presence/absence of branched-chain fatty acids, dicarboxylic acids and n -alkanes. FA: Fatty acid, where C_{nx} represents the carbon chain length and the degree of unsaturation. When unspecified, unsaturation is 0, Br: branched-chain fatty acid, Diacids: dicarboxylic acids.

Figure 7.22: Partial total ion chromatogram of sample RGR24. Cn:x indicates fatty acid with n carbon atoms and x double bonds. IS: Internal Standard; ALK: n-alkane.

Sample ID	Rim/ Base/ Body	Vessel shape	'Haryana' or 'Classic Harappan'	Chrono logical period	Lipid concent ration $(\mu g/g)$	P/S ratio $(C_{16:0}$ $C_{18:0}$	$\delta^{13}C$ $C_{16:0}$	$\delta^{13}C$ $C_{18:0}$	$\varDelta^{13}C$ $(C_{18:0}$ - $C_{16:0}$
RGR01	rim	small ledged jar	'Haryana Harappan'	EMH	21.9	2.0	-26.9	-26.7	0.1
RGR02	rim	small ledged jar	'Haryana Harappan'	EMH	16.6	1.5	-26.0	-26.8	-0.8
RGR03	rim	small ledged jar	'Haryana Harappan'	EMH	18.9	1.3	-24.7	-21.2	3.4
RGR17	rim	small jar	'Classic Harappan'	EMH	9.5	1.2	-25.8	-25.3	0.5
RGR20	rim	very small jar	'Haryana Harappan'	EMH	36.7	1.0	-16.0	-19.5	-3.4
RGR ₂₄	rim	small necked jar	'Haryana Harappan'	EMH	48.8	0.8	-28.6	-26.5	2.2
RGR ₂₉	neck	medium jar	'Haryana Harappan'	EMH	12.6	1.2	-28.9	-29.0	-0.1

Table 7.8: Details of samples analysed for GC-c-IRMS analysis from Rakhigarhi.

Figure 7.23: $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values plotted against $\delta^{13}C_{16:0}$ values of vessels from Rakhigarhi, colour-coded according to trench and context number. The shaded areas in-between dotted lines indicate the regions where dairy fats and ruminant adipose fats, and ruminant adipose and non-ruminant fats overlap respectively, indicating mixing of products.

Discussion

The molecular evidence from Rakhigarhi, like that from the other sites, suggest that lipid residues within vessels are broadly characterised by degraded animal fats. However, a single vessel (RGR24) has a lipid profile that is characteristic of a plant product as it contains high proportions of odd-chain *n*-alkanes, which are found in higher plants (Dunne et al. 2017) and high proportions of long-chain fatty acids, which are also found in plant products (Figure 7.22). A predominance of C_{23} and C_{25} *n*-alkanes is known to be characteristic of submerged and floating aquatic plants (Kolattukudy et al. 1970; Ficken et al. 2000; Dunne et al. 2017). Another vessel (RGR05) has a high P/S ratio relative to other vessels (3.5), and P/S ratios of 4 are suggestive of plant input (Dunne et al. 2017). The lipid profile of this vessel is, however, not characteristic of plant-derived compounds: it contains fatty acids ranging from C14:0 to C22:0 and contains no odd-long-chain *n*-alkanes. Other vessels contained *n*-alkanes, but their pattern was not distinctive, and they appeared in very low proportions. It is possible they arise from post-excavation or laboratory contamination.

Compound-specific isotopic results suggest a variety of animal products were processed in vessels from Rakhigarhi. There is a 7‰ difference in Δ^{13} C values, indicating products processed in the vessels originated from both non-ruminant fats and dairy products. The variation in $\delta^{13}C_{16:0}$ values reflects varying amounts of C_4 plant input into vessels, with a value of -16‰ indicating relatively higher input of C_4 plants (see Figure 7.23).

Five vessels from Rakhigarhi have Δ^{13} C values that fall within the non-ruminant fats range, with one vessel with a value falling in ranges that indicate the mixing of nonruminant and ruminant adipose fats (RGR02, small ledged jar) and one with a value which falls within the dairy products range (RGR20, a very small jar). RGR20 has clear evidence for the processing of dairy products with a high contribution of C_4 plant. Given that there is evidence for the diet of cattle/buffalo being almost exclusively C_4 plant-derived in this region, it is highly likely that dairy products from cattle/buffalo milk were processed in this vessel. However, as the zooarchaeological and archaeobotanical remains from Rakhigarhi are undergoing analysis, it is not possible to integrate the results obtained with other bioarchaeological evidence.

Although the number of vessels from Rakhigarhi analysed via GC-c-IRMS analysis were low, all three ledged jars have high Δ^{13} C values: one (RGR03) has the highest Δ^{13} C value from the entire assemblage of pottery studied in this thesis (3.4‰), the second (RGR01)

falls well within the established range of non-ruminant fats, and the third vessel (RGR02) falls between the non-ruminant and ruminant adipose fats range, indicating the mixing of products. As ledged jars have been linked with contemporary/modern cooking vessels (*handis*), this could suggest their use for the cooking of meat products. However, variable $\delta^{13}C_{16:0}$ values suggest differential input of C₃ and C₄ plant, indicating that animals processed in the vessels had variable diets.

Like at Farmana, the pottery from Rakhigarhi consists of a range of locally produced, regional 'Haryana Harappan' pottery, as well as 'Classic Harappan' pottery that may have been produced outside the Haryana region. Although the 'Classic Harappan' vessels that have been analysed have very low lipid concentrations relative to 'Haryana Harappan' vessels (out of 5 vessels analysed, only one has a lipid yield over 5 μg/g and is listed in Table 7.7), the molecular profiles of these vessels show no observable difference. All vessels have evidence for the processing of meat products, except for with a single small necked jar (RGR24) that contains clear evidence for plant products. When combined with isotopic analyses, the use of the vessels appears primarily associated with the processing of non-ruminant products, but the vessel with clear molecular evidence for plant products also falls within this range. It has been noted that the Δ^{13} C values of plants can be variable, ranging between -2‰ and 1‰ (Steele et al. 2010). All except one of the vessels from Rakhigarhi (and several other sites) fall within this ambiguous region. Thus, at present it is not possible to confidently state whether these vessels were predominantly used for the processing of non-ruminant fats, plant products, or mixtures of C_3 plants and dairy products from ruminants grazing on C_3 and C_4 plants (Hendy et al. 2018) or other mixtures (see Section 8.2.4).

Overall, the results from Rakhigarhi demonstrate variable ranges of $\delta^{13}C$ values from fatty acids in lipid extracts, suggesting meat, milk and possibly plant products were processed in vessels. Since vessel fragments were collected from domestic contexts that are broadly contemporaneous, they likely are indicative of the vessel-use practices of a few individuals or families at the settlement, providing a unique glimpse into culinary practices of a small subsection of the city.

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7.3. Vessel-specific results: vessel function and use

Now that the site-specific results have been assessed, this section will review the vesselspecific results, which provide insights into vessel function and use (Section 1.3). This section summarises the results from the chemical and fatty acid-specific isotopic analyses obtained from specific vessel forms to discuss actual use (Rice 1996: 140) of vessels. In this thesis, rim fragments were used to reconstruct the original shape of vessels, but except for the size of the mouth of certain jars, the size and volumetric capacity of vessels could not be determined. Broadly speaking, results from vessel forms within sites suggests that vessels were used to process different products or may have had specific uses that are not distinguishable via lipid residue analysis. Figure 7.24 demonstrates that there are no significant differences between lipid concentrations of different vessel forms. This is intriguing as forms like dishes and bowls would be less likely to be exposed to heat and would likely have lower lipid concentrations. However, vessels with specific morphological characteristics and size dimensions would have enabled or restricted the use of certain products. Additionally, vessels with high lipid yields may have been used more frequently or for the processing of fatty-rich products.

Figure 7.24: Lipid concentrations of different vessel forms across Indus sites in northwest India. Lipid concentrations are represented on a log scale for better visualisation; red diamonds are mean values.

Figure 7.25: Lipid concentrations of vessels across Indus sites in northwest India. Lipid concentrations are represented on a log scale for better visualisation; red diamonds are mean values.

Figure 7.26: $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values plotted against $\delta^{13}C_{16:0}$ values of vessel-types across Indus sites in northwest India, colour-coded according to range of lipid concentration in each vessel. The shaded areas in-between dotted lines indicate the regions where dairy fats and ruminant adipose fats, and ruminant and non-ruminant fats overlap respectively, indicating mixing of products.

7.3.1 Vessel shape, size and use

Comparisons of lipid yields across vessel forms suggest that there is no clear relationship between the shape and size of the vessel and its lipid yield (Figure 7.24), or differences in lipid yields across sites (Figure 7.25). Furthermore, Kruskal-Wallis tests show there was no significant difference between lipid concentrations across vessel forms, $\chi^2(10) = 121$, p = 0.48, or across sites, $\chi^2(6) = 121$, $p = 0.48$.

Most vessel forms have comparable mean lipid concentrations, but the ranges of lipid yield are variable. This pattern might suggest that certain vessels were used more frequently for processing of fatty or oily products or for longer periods of time. For example, only four vessels from the entire assemblage have lipid yields higher than 100 μ g/g, out of which two are small-mouthed jars (KNK02, MSD343), one is a mediummouthed necked jar (MSD3412A), and one is a large mouthed jar (LHR10) (Figures 6.5 and 7.12). Figure 7.26 demonstrates that these vessels were likely used to frequently process, or store ruminant and non-ruminant fats and mixtures of fats. The same is also true for vessels with lipid concentrations between 50-100 μ g/g. There is no clear relationship between lipid concentration and the δ^{13} C values of fatty acids in lipid extracts across vessels (Figure 7.26). There also appears to be no significant relationship between form of vessels and isotopic values obtained from fatty acids (Kruskal-Wallis tests, $\delta^{13}C_{16:0}$ values: χ^2 (10) = 66.83, p = 0.48, $\delta^{13}C_{18:0}$ values: χ^2 (10) = 69.89, p = 0.41, $\Delta^{13}C$ values: χ^2 $(10) = 70.66$, $p = 0.39$). This likely suggests the multifunctionality of vessels, or their use for processing multiple ingredients.

It is important to discuss certain vessel forms from the analysed assemblage due to their specific shape and hypotheses that have been put forward about their use in Indus contexts, specifically, ledged jars and perforated vessels.

7.3.1.1. Ledged jars

Ledged jars have a prominent carination on the shoulder. Their shape matches those of contemporaneous/modern cooking vessels in India called *handis*. Thus, they are often referred to as 'cooking jars' or 'cooking pots' in the literature (Kenoyer 1998:156; Chase et al. 2014b; Krishnan 2018) (Figure 2.4). Ethnographic data suggests that "a great majority of groups make cooking pots that are short and squat, with a large basal surface for efficient heat transfer, but usually with a somewhat restricted mouth to prevent rapid evaporation from boiling foods." (Henrickson and McDonald 1983). This definition closely matches the shape of Indus ledged jars, but the fragments analysed were not coarse

and did not have grog surface treatments that may have improved thermal insulation properties, as is observed in other 'cooking pots' in other archaeological contexts.

Ten ledged jars were analysed for this thesis, out of which eight had lipid yields above 5 μg/g. Most of these had small rim diameters (8-12cm), while two had undeterminable rim diameters due to rim preservation. The average lipid concentration of the jars was 33.8 μg/g, ranging between 12.5 μg/g and 66.7 μg/g. The lipid profiles of ledged jars were characteristic of degraded animal fats and lacked compounds indicative of possible exposure to high temperatures, such as mid-chain ketones. This pattern suggests there is little direct evidence to support the suggestion that these vessels came in contact with high temperatures.

Ledged jars from Masudpur VII had higher mean lipid yields compared to other vessel forms, which may suggest they were used more frequently, or used for cooking at Masudpur VII (Figure 7.27), but these differences were not statistically significant (oneway ANOVA test, $F(7, 20) = 0.96$, $p = 0.48$). Compound-specific isotopic results demonstrate that the ledged jars were likely used to process different products: including dairy products, non-ruminant fats or mixtures of plant and dairy products (Figure 7.28).

Figure 7.27: Lipid concentrations of fragments from different vessel-types at Masdupur VII. Ledged jars have relatively higher mean lipid concentrations, but the difference is not statistically significant. Lipid concentrations are represented on a log scale for better visualisation; red diamonds are mean values.

Figure 7.28: $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values plotted against $\delta^{13}C_{16:0}$ values of ledged jars across Indus sites in northwest India, colour-coded according to site. The shaded areas in-between dotted lines indicate the regions where dairy fats and ruminant adipose fats, and ruminant adipose and non-ruminant fats overlap respectively, indicating mixing of products.

Figure 7.29: $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values plotted against $\delta^{13}C_{16:0}$ values of perforated vessels across Indus sites in northwest India, colour-coded according to site. The shaded areas in-between dotted lines indicate the regions where dairy fats and ruminant adipose fats, and ruminant adipose and non-ruminant fats overlap respectively, indicating mixing of products.

7.3.1.2 Perforated vessels

Perforated vessels are another distinctive vessel form within the Indus Civilisation. Proposed functions of Indus perforated vessels range from vessels used for brewing or straining to braziers for storing coal and used for heating (Dales and Kenoyer 1986: 108- 109).

Perforated vessels have been used for identifying dairy activities archaeologically in European contexts (Roffet-Salque et al. 2013). For example, perforated vessels have been used for draining and separating curds during the hard cheese making process (Roffet-Salque et al. 2013). But they have not always been associated with 'cheese-straining' in South Asian contexts, because a hard cheese-making tradition has not existed historically in South Asia, and still does not today. Dairy curds and yoghurt are prepared daily in households, but cloth is preferentially used in the straining process. Ethnographic research of dairy practices in modern Punjab has, however, described the use of perforated lids used for heat regulation during dairy production (Miller 2004: 216-217). Beyond this, Bourgeois and Gouin (1995) are the only researchers to have proposed the use of perforated vessels for dairy processing on the basis of fatty acid profile of a single vessel from Nausharo (see Section 2.3.5.2.).

Figure 7.27 demonstrates that lipid concentrations of perforated vessels analysed in this thesis are comparable to those obtained for other types of jars, which is interesting as most of perforated vessels analysed were fragments of body sherds while other fragments analysed were rim sherds. Rim sherds are more likely to absorb lipids when foodstuff is boiled (Charters et al. 1997), but body sherds of perforated vessels would not be able to retain foodstuff. This morphological parameter suggests it is likely that the perforated vessels that have been analysed were used to process products that were high in fat or oil content (as this would increase the likelihood of lipid absorption into the ceramic matrix) or were very frequently used.

The lipid profiles of perforated vessels were similar to other vessels, and characteristic of degraded animal fats. Seven out of 15 perforated vessels (47%) were analysed via GC-c-IRMS analysis. The obtained δ^{13} C values of the fatty acids (Figure 7.29) fall within the established range for non-ruminant fats (n=5), with values of one vessel from Masudpur VII (MSD1873) falling in between the ranges for non-ruminant and ruminant adipose fats, and values of a vessel from Farmana (FRN11) falling within the

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range for ruminant adipose fats, close to the range for dairy fats. None of the vessels have Δ^{13} C values that fall within the established references of dairy fats, i.e. below 3.3‰.

Sample sizes are small, but there appears to be consistency between the fatty acidspecific δ^{13} C values for the analysed perforated vessels. This pattern may be indicative of a specific use for these vessels. It is possible that the vessels were used for the straining of non-ruminant fats, or that lard or animal fats were burned within the vessels, however, none of the vessel fragments display charring marks, suggesting that the latter is unlikely.

Ethnographic or historical sources may shed further light on the possible use of the vessels. Allchin (1979) discusses a set of vessels which includes a perforated vessel used for the distilling of spirits from a variety of fermented liquors in modern India (Figure 7.30). Known as a still, this apparatus includes a large pot with a smaller pot placed over its mouth having perforations in its base. A small bowl is set inside the perforated pot whose mouth is in turn closed by a third pot with a rounded or conical base and filled with cold water. The fermented liquid is boiled in the lower pot and the steam rises through the perforations, condensing on the base of the uppermost pot and dripping down into the receiving bowl (Allchin 1979: 57). Until recently, a number of liquors made from unrefined sugar, palm juice, rice and the flowers of the mahua tree (*Bassia latifolia*) were fermented and then distilled using this apparatus in the central belt of the subcontinent (Allchin 1979). Although perforated jars from Indus contexts are usually not roundbottomed, it is possible that some perforated bowls were used for steaming or distillation.

Figure 7.30: Example of a modern Indian still from Bihar (reproduced from Allchin 1979: 57). The second vessel has perforations in the bottom, but completely perforated vessels may also be used.

Another interesting historical example worth mentioning is the use of "a pot with a hundred holes" in the preparation of alcoholic beverages known as *sura* and *soma* (Oort 2002)*.* These beverages feature prominently in later ancient Hindu texts such as the *Rigveda* and are associated with healing and consumed in ritual and sacrificial contexts. The use and preparation of *sura* are described in *Satapatha Brahmana* and other Brahmanic texts with elaborate ritualization of the process, with ingredients such as malted grain, especially rice and barley; powders made of pulses and spices; milk; even the hair of exotic animals (Oort 2002).

The mention of 'a pot with a "hundred" holes' is reminiscent of the perforated vessel, however, the texts do not specify how this vessel is used in the preparation of *sura*. While it would be unwise to draw parallels between the use of a Vedic or possibly later *satatrnna* 'pot with holes' and Indus perforated vessels, given the form of the vessel, its use for the preparation of a liquid-based product is highly likely. Δ^{13} C values from the lipid extracts of the analysed perforated vessels are consistent with values obtained by mixtures of C_3 plants and dairy products from ruminants grazing on C_3 and C_4 plants (Hendy et al. 2018; see Section 8.2.4), which suggests vessels could have been used to prepare brews with these ingredients. This finding can be confirmed with the analysis of more perforated vessel fragments and the collection of reference fats from South Asia.

7.3.2. 'Classic' and 'Haryana Harappan' vessels

The difference between 'Classic' and 'Haryana Harappan' vessels were outlined in Chapter Two (Section 2.3.1.2). Although vessels from the analysed assemblage exhibiting 'Classic Harappan' characteristics were limited (n=9), the lipid and isotopic results did not reveal any visible differences between these two vessel types (see Figure 7.31, only seven 'Classic Harappan' vessels were analysed for GC-c-IRMS analysis). This pattern suggests that even though 'Classic Harappan' vessels may have been produced differently and perhaps acquired from elsewhere, they were used in similar ways as locally-produced, 'Haryana Harappan' vessels. Nonetheless, it is also possible that they were used to process foodstuff or products prepared in unique ways that are not distinguishable via lipid analysis.

Figure 7.31: $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values plotted against $\delta^{13}C_{16:0}$ values of 'Classic' versus 'Haryana Harappan' vessels across Indus sites in northwest India, colour-coded according to site. The shaded areas in-between dotted lines indicate the regions where dairy fats and ruminant adipose fats, and ruminant adipose and non-ruminant fats overlap respectively, indicating mixing of products.

Overall, the results from the lipid and isotopic analyses of different vessel forms and types across Indus sites highlights the multifunctionality of vessels and diversity of uses, except perhaps a specific function and use for perforated vessels. However, it is not possible to rule out the likelihood of more complex interactions in archaeological samples that are governing the preservation of lipids, which could be related to complex food mixtures, the physical and chemical parameters of different pottery, or variable soil chemistry (Barker et al. 2018). The results of other vessel forms are provided in Appendix G, but they do not reveal observable patterns.

7.4. Discussion: vessel-use comparison across sites and time

This section addresses questions asked by this thesis, namely: 1) are there difference in vessel-usage across settlements; and, 2) are there changes in the use of vessels over time? The lipid and isotopic results from different sites and time periods are compared and integrated here. First, a summary of results is provided, followed by discussions about the diversity of resources across settlements, variability between sites, chronological trends and continuity.

7.4.1. Summary of results

The results from the lipid analyses for vessels from sites within northwest India reveal few differences between the types of products processed in vessels across settlements. Lipid profiles from all but one vessel analysed were suggestive of degraded animal fats. However, as has been discussed previously, the degradation of both animal- and plantderived compounds into mid- and long-chain fatty acids makes it difficult to identify the source(s) of the organic residues within the vessels. Only a single vessel from Rakhigarhi contains very low concentrations of $C_{16:0}$ and $C_{18:0}$ fatty acids relative to long-chain fatty acids (maximising at C24:0), coupled with a series of odd-chain *n*-alkanes, which is typical of a residue derived from higher plants (Dunne et al. 2017). This vessel contained the only molecular evidence from the analysed assemblage for the processing of plant products, but other compounds typical of plant residues, such as campesterol, stigmasterol, β-sitosterol and cycloartenol (Steele et al. 2010), were absent from this and other samples. Biomarkers for the presence of other products, such as fish (ω -(o-alkylphenyl) alkanoic acids; APAAs), millet (miliacin) or heated products (ketones) (see Chapter Three) were also missing from the analysed assemblage.

The absence of heat 'biomarkers' is interesting as they have been previously recovered from poorly preserved lipid extracts (Craig, pers. comm.). None of the vessel fragments analysed had any sooting or charring marks which suggests that they may not have been exposed to fire for long durations of time; however, as most fragments analysed were rim-sherds, it is not possible to be sure of the degree to their direct exposure to fire.

Figure 7.32: Scatterplot of $\delta^{13}C_{18:0}$ and $\delta^{13}C_{16:0}$ values from vessels across Indus sites in northwest India, colour-coded according to site. The data is plotted against 1SD confidence ellipses from modern reference fats in the U.K. (Copley et al. 2003; Mukherjee et al. 2008).

Figure 7.33: $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values plotted against $\delta^{13}C_{16:0}$ values of vessels across Indus sites in northwest India, colour-coded according to site. The shaded areas in-between dotted lines indicate the regions where dairy fats and ruminant adipose fats, and ruminant adipose and nonruminant fats overlap respectively, indicating mixing of products.

Compound-specific isotopic analysis of fatty acids from lipid extracts provided a means to distinguish between different sources of animal fats. Figure 7.32 demonstrates that the δ^{13} C values obtained from fatty acids in vessels from the study region cannot be meaningfully compared to reference fats obtained from U.K. or Europe. When plotted using the Δ^{13} C proxy that removes environmental variation (Figure 7.33), most of the vessels analysed from Indus sites in this thesis have Δ^{13} C values ranging from -1‰ to 3.4‰ (n=44; 60.3%), which places them within the global range for non-ruminant products. Ten vessels (15%) from Alamgirpur, Masudpur VII, Lohari Ragho I, Khanak, Farmana and Rakhigarhi, fall in-between the thresholds for non-ruminant fats and ruminant adipose fat values, suggesting a mixing of the two types of fats in those vessels. Five vessels (8%, from Lohari Ragho I, Farmana and Masudpur VII) fall within the range for ruminant adipose products, and three vessels (5%, from Masudpur VII, Masudpur I and Farmana) fall within the range for dairy products. None of the vessels showing evidence for dairy fall near previously published dairy references from India (Craig et al. 2005), which include a modern milk pot used to process milk from a C_4 plant fed cow from Gujarat (West India) and cow's milk from Tamil Nadu (South India) that was fed c. 65 per cent rice bran and 35 per cent sorghum (Craig et al. 2005: 886). Comparison with these reference fats suggests that the archaeological vessels with evidence for dairy-processing were likely used to process milk products from ruminants that was entirely C_4 fed. Three vessels (5%, from Khanak, Farmana and Rakhigarhi) have values that overlap between ruminant adipose and dairy fats ranges, indicating the mixing of products.

7.4.2. Diversity of resources across settlements, processing similar foodstuffs?

The molecular and isotopic evidence from residues within vessels suggests that similar types of products were processed across settlements of different sizes. No significant differences for isotopic values from lipids in vessels across sites were observed (Kruskal-Wallis tests, $\delta^{13}C_{16:0}$ values: $\chi^2(67) = 64.6$, $p = 0.6$, $\delta^{13}C_{18:0}$ values: $\chi^2(68) = 71.6$, $p = 0.4$, Δ^{13} C values: χ^2 (68) = 70.3, p = 0.4). Three settlements within the study area, namely, Masudpur VII (small village), Masudpur I (large village) and Rakhigarhi (city) exhibit a similar means and spread for δ^{13} C values for fatty acids within vessels, which may indicate that similar types of products were processed in vessels across these settlements (see Figures 7.33 and 7.35). The seasonal movement of animals to different pastures could explain the wide range of δ^{13} C values observed in adipose fats, but this is more difficult to

justify for non-ruminants as they are rarely grazed across large distances (see Chapter Eight).

Figure 7.34: Boxplots of $\delta^{13}C_{160}$ and $\delta^{13}C_{180}$ values across Indus sites in northwest India discussed in this chapter.

7.4.3. Variability between sites

Most sites demonstrate similar vessel-usage practices, but Lohari Ragho I, Alamgirpur and Farmana sites appear to show some degree of variance in terms of the fatty-acid specific δ^{13} C values within vessels. This may be reflective of unique settlement-specific practices. For example, even though Lohari Ragho I lies within the hinterland of Rakhigarhi, samples from this site demonstrated the widest range for $\delta^{13}C_{16:0}$ values, indicating variable degrees of C_3 , mixed C_3 and C_4 , and dominantly C_4 plant input within the vessels analysed. Some of these values may also be indicative of freshwater fish. Despite small sample sizes; the wide range of C_3 and C_4 plant-input into the vessels is unique to Lohari Ragho I, indicating that its inhabitants may have practised a uniquely diverse subsistence strategy or animal management practices.

Farmana is the only site that has three vessels with evidence for processing or mixing of both sheep/goat and cattle/buffalo dairy products, which contributes to nearly half of the total vessels with evidence for dairy/dairy product processing from all of the sites investigated (n=7). While this may be attributable to random chance, it could also be

indicative of a feature of Farmana's pastoral economy, as evinced by the high percentages of adult cattle/buffalo and sheep/goat found at the site (Channarayapatna 2014, 2018).

Alamgirpur also demonstrates a unique pattern in vessel usage. Alamgirpur is located over 200 km away from Rakhigarhi and lies in a very different environmental context. The δ^{13} C values from fatty acids within vessel extracts cluster tightly between -27.8‰ and -29.5‰ for $\delta^{13}C_{16:0}$, and -28‰ and -29.8‰ for $\delta^{13}C_{18:0}$ (Figures 7.3 and 7.34) The range for Δ^{13} C values is restricted between 0.4‰ and -0.7‰, suggesting a limited range of foodstuff was processed in vessels (Figure 7.35). The values fall within modern ranges for nonruminant and ruminant adipose fats, including their mixtures; but as suggested by mixing models the results may also indicate the mixing of C_3 plants with dairy products from ruminants grazing on C_3 and C_4 plants (Hendy et al. 2018), or other mixtures explored in Section 8.2.4. The lack of clear evidence for dairy processing, and the absence of ruminant adipose fats with relatively higher $\delta^{13}C_{16:0}$ values in vessels (i.e. C_4 plant-input) is notable and may be indicative of a regional or site-specific animal management strategy, food choice or constraint. However, δ^{13} C vales from enamel carbonate from animal teeth at Alamgirpur indicate that both cattle/buffalo and sheep/goat had high input of C_4 plant in their diet (Lightfoot et al. in prep.) It is possible that the moderate climate, with cooler summers, warmer winters, and higher moisture availability compared to the other study sites in the Haryana, even during the likely failure of monsoons in the post-urban period, rendered it ideal for the growing of C_3 crops and pulses for human consumption, while domestic ruminants were fed C_4 plants and/or millet. Conversely, the exclusive processing of a narrow range of ingredients in vessels may also suggest that there was limited availability of resources.

Figure 7.35: Boxplots of $\delta^{13}C_{16:0}$ and $\delta^{13}C_{18:0}$ values of vessels from Indus sites in northwest India divided by chronological time period. EMH: Early Mature Harappan, LMH: Late Mature Harappan, LH: Late Harappan.

Figure 7.36: $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values plotted against $\delta^{13}C_{16:0}$ values values of vessels across Indus sites in northwest India, colour-coded according to site and divided by chronological time period. The shaded areas in-between dotted lines indicate the regions where dairy fats and ruminant adipose fats, and ruminant adipose and non-ruminant fats overlap respectively, indicating mixing of products. EMH: Early Mature Harappan, LMH: Late Mature Harappan, LH: Late Harappan.

Figure 7.37: Boxplots of $\delta^{13}C_{16:0}$ and $\delta^{13}C_{18:0}$ values of vessels from Indus sites in northwest India divided into pre-, during and post-4.2 ka BP.

Figure 7.38: $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values plotted against $\delta^{13}C_{16:0}$ values of vessels across Indus sites in northwest India, colour-coded according to site and divided into pre-, during, and post-4.2 ka BP. The shaded areas in-between dotted lines indicate the regions where dairy fats and ruminant adipose fats, and ruminant adipose and non-ruminant fats overlap respectively, indicating mixing of products.

7.4.4. Chronological trends

The diversity of plant and animal products grown and managed by Indus populations, particularly in the urban period, have been highlighted by previous scholars (e.g., Weber 2003; Petrie et al. 2016). The results that have been obtained do indeed demonstrate that a wide variety of food resources were likely processed within vessels during the urban period (EMH and LMH), including different types of animal meat and milk. The dominance of non-ruminant animal fat-processing in vessels across these Indus sites cannot be clearly explained and is discussed in detail in Chapter Eight. Although evidence for the direct processing of plant products is not present, the δ^{13} C values for C_{16:0} fatty acids within lipid extracts suggest the input of a range of C_3 and C_4 plants, highlighting the likely mobility of animals and diversity of animal management and feeding strategies, particularly in the Early Mature Harappan period (EMH), but possibly continuing in the Late Mature Harappan (LMH) and Late Harappan periods. Statistical tests revealed no significant difference between isotopic values from fatty acids and chronological periods (Kruskal-Wallis tests: $\delta^{13}C_{16:0}$ values: $\chi^2(2) = 70.51$, p= 0.36, $\delta^{13}C_{18:0}$ values: $\chi^2(2) =$ 71.36, $p = 0.37$, $\Delta^{13}C$ values: χ^2 (2) = 68.12, $p = 0.47$) or climatic periods ($\delta^{13}C_{16:0}$ values: $\chi^2(2) = 70.25$, p= 0.368, $\delta^{13}C_{18:0}$ values: $\chi^2(2) = 71.34$, p = 0.368, $\Delta^{13}C$ values: $\chi^2(2) =$ 68.57, $p = 0.458$).

Although changes across cultural periods can be explored at some settlements, a nuanced assessment of possible temporal changes related to the 4.2 ka BP 'event' is challenging as evidence coming from the period that spans 4.2 ka BP is limited to a small set of samples from Alamgirpur, Masudpur VII, Masudpur I, Lohari Ragho I and Khanak. Samples that can be confidently dated after 4.2 ka BP could only be accessed from two sites: Alamgirpur and Masudpur VII. Figures 7.35 and 7.37 show that Alamgirpur has narrow ranges for δ^{13} C values from fatty acids in vessels, suggesting that vessels were used for a limited number of similar foodstuff, which may either include ruminant adipose and/or non-ruminant fats, mixtures of both, or, alternatively, mixtures of C_3 plants with C_4 based dairy products. The samples analysed were all dated to the post-urban period, and several samples date after the 4.2 ka BP 'event'. This pattern suggests that there is no observable change in products processed in vessels during and after the period of climatic instability. Although this pattern appears unique to Alamgirpur and might represent a regional or site-specific food practice, it may also be a unique chronological development.

It is not possible to be certain of this interpretation as samples dating to earlier periods at Alamgirpur were not analysed due to logistic and time restrictions.

Four vessels from Masudpur VII date between *c*.2500-2300 BC and twenty-one vessels date between *c*.1900-1700 BC, representing diachronic vessel-usage pre-, during, and post 4.2 ka BP (see Figures 7.34 to 7.37). Although the sample sizes are small, vessels pre- and during 4.2 ka BP fall within the range for non-ruminant fats, suggesting they were used for processing meat of omnivorous animals, or alternatively, mixtures of C_3 plants and dairy products from ruminants grazing on C_3 and C_4 plants (Hendy et al. 2018), or other mixtures (see Section 8.2.4). The range of δ^{13} C values of the fatty acids from vessels from after 4.2 ka BP is wider, indicating evidence for the processing of a broader range of products including ruminant adipose fats and dairy products. It is possible that the differences observed are due to uneven sample sizes, or reflect the fact that inhabitants at Masudpur VII diversified the products processed in vessels after 4.2 ka BP. Although there is considerable difficulty in reconciling climate 'events' and archaeological evidence as they operate on unique temporal scales, the evidence appears to suggest that specific culinary practices seem to have been practised for hundreds of years at Alamgirpur, with minor evidence for some change at Masudpur VII.

7.5. Chapter Summary

This chapter has provided the results for lipid and isotopic analysis of vessel residues from very small settlements (small villages), small settlements (villages), a medium-sized settlement (town) and a large settlements (city) in the Indus civilisation in northwest India. The results obtained from vessels from each site are provided along with a short discussion and interpretation. The results are also discussed at a broader, vessel-specific level, with salient findings highlighted. Finally, a discussion integrating the results across settlements is provided, which addresses two key questions framed by this thesis. Chapter Eight will provide a broader discussion, integrating the results presented in both Chapters Six and Seven with zooarchaeological remains and discussing the implication of results for understanding Indus foodways.

Chapter Eight

Synthesis: Vessel-use and foodways in the Indus Civilisation

The analyses presented in this thesis has made it possible to assess a range of topics, including preservation of lipid residues in Indus vessels, inter- and intra-site variation in ceramic lipid residues, vessel-specific results, and spatial and chronological patterns. This chapter synthesises the results provided in previous chapters, contextualising the findings with bioarchaeological evidence available from northwest India in the Indus period and organic residue analyses from other regions in the world. Section 8.1 discusses the extent to which lipid yields inform us about vessel usage in the Indus Civilisation, and Section 8.2 discusses the different types of products that have been detected in different Indus vessels, setting out to answer the main question posed by this thesis, 'what was cooking in Indus vessels?' Section 8.3 addresses how this thesis contributes to our understanding of Indus foodways, and Section 8.4 expands upon our understanding of urban and rural food practice in Indus sites in northwest India. Finally, potential issues influencing interpretation of the results are discussed, along with future research avenues.

8.1. Lipid yields and vessel use

The first objective of this thesis was to test, as proof of concept, whether lipid residues are preserved in a range of Indus pottery from different sites (Chapter Six). As hypothesised, results demonstrated that lipid concentrations obtained from vessel fragments were low, ranging between trace amounts to 215 μg/g, with mean lipid concentration of 23.5 μg/g and median of 13.8 μ g/g (n=122). This is likely due to the location of the study sites in arid environments with alkaline sediments, experiencing seasonal variations in temperature and rainfall, factors that are detrimental to the survival of organic remains (Cramp 2008; Evershed 2008a). Lipid yields from sediment were similarly low, ranging between trace amounts to 38.6 μg/g, (mean of 11.2 μg/g, median of 8.6 μg/g; n=7) suggesting that it is unlikely lipid yields from vessels were influenced by surrounding sediment organic content, but this must be assessed on a case-by-case basis. Thus, preservation of lipids is strongly contingent on burial conditions at sites. However, results from this thesis also

indicate that certain sites, or locations within sites may have unique micro-environments that enable the preservation of organic residues (Barker et al. 2018), for example, Masudpur VII and Khanak in northwest India. Thus, lipid residue studies in South Asia should not be neglected due to assumptions about poor organic preservation.

Despite low yields of organic residues, 74% of the analysed vessels contained lipid concentrations above 5 μ g/g, which is the threshold by which lipid yields are considered archaeologically meaningful (Heron and Evershed 1991; Reber et al. 2019). Vessels recovered from the same archaeological context had varying ranges of lipid yields. As it is likely these vessels were buried at similar times and experienced similar post-depositional conditions, their lipid yields are likely to be indicative of their use in antiquity, with higher lipid concentrations indicating more frequent use, or their use for the processing of fattyrich products. Conversely, although difficult to conclusively suggest, low lipid yields may indicate brief or ephemeral usage of vessels. Given the vast quantities of vessels recovered from Indus sites occupied for relatively short periods of time; the sheer volume of ceramic production may suggest that vessels were transient and not intended for long-term usage. Previous studies in lipid residue analysis have suggested that there is a relationship between lipid yields and the life-use of ancient vessels (Evershed 2008a; Budja 2014; Correa-Ascencio and Evershed 2014; Cramp et al. 2014; Reber et al. 2019). Other studies have demonstrated how specific types of food processing in vessels, such as boiling or roasting, would lead to higher concentrations of lipid in specific parts of vessels (e.g. Charters et al. 1993, 1997). No significant differences between lipid concentrations across different type of fragments such as rims, bases, neck- and body fragments were observed in the samples analysed in this thesis. Additionally, the body fragments of perforated vessels had mean lipid concentrations comparable to rim fragments, which might indicate they were used for the processing of fat-rich products. However, as different parts of the same vessel were not analysed, it was not possible to make interpretations about the use of vessels for specific types of food-processing, except with samples from Stone Tower I, Salut, Sultanate of Oman (Section 6.1.3.1). Additionally, no significant relationship between lipid yields within sites and vessel-types were observed, even at Masudpur VII, where ledged jars, that have previously been associated with cooking (Kenoyer 1998; Chase et al. 2014b), had higher lipid concentrations relative to other vessel forms (but this was not significant).

This thesis was also able to address questions related to the use of Indus perforated vessels, which has been much debated (e.g. Dales and Kenoyer 1986). The results suggest that perforated vessels were not primarily associated only with dairy processing, which was previously suggested (Bourgeois and Gouin 1995). Instead they have ambiguous, yet consistent fatty acid-specific δ^{13} C values that links them either to the processing of the meat of ruminant and/or non-ruminant animals, or mixtures of C_3 plants and dairy products. Although not conclusive, these results are exciting and have implications for how the Indus perforated vessel is interpreted in future Indus research.

8.2. Food choice: products in Indus vessels in northwest India

This thesis has conducted the first large-scale ceramic lipid residue analysis on vessels in the Indus Civilisation to address questions about the cultural use of vessels by Indus populations. While studies have investigated Indus subsistence strategies by examining crops and agricultural strategies via macrobotanical and phytolith studies (e.g. Vishnu-Mitre and Savithri 1982; Fuller and Madella 2002; Weber 2003; Madella and Fuller 2006; Weber et al. 2010; Wright 2010; Fuller 2011; Petrie and Bates 2017), or animal consumption via zooarchaeology (Meadow 1991; Belcher 1991; Joglekar et al. 2013; Chase 2014; Chase et al. 2014a, 2018) few studies have considered both (for an exception, see Fuller 2005; García-Granero et al. in prep). Such combined approaches were advocated by Weber and Kashyap (2010), but their study only investigated plant-use.

An integration of zooarchaeological, archaeobotanical and other bioarchaeological evidence with food-related artefacts such as ceramics and grinding stones is necessary to provide a deeper understanding of how Indus food systems operated in the past. This has been attempted for the Neolithic in India (Fuller 2005) but has not been discussed in terms of the Indus Civilisation. Furthermore, it is important to consider how both plants and animals were grown/raised, prepared and consumed, and how these processes related to social life and ideas of identity or power. The study of cultural, social and economic practices concerning food production and consumption, or 'foodways' (Staller and Carrasco 2009; Twiss et al. 2009; Peres 2017) have been largely neglected for the Indus Civilisation.

This thesis contributes to our understanding of how Indus populations processed organic products in ceramic vessels and suggests new means by which we can reconstruct daily *habitus* practices of inhabitants of Indus settlements. The organic residue analyses presented here has revealed a range of degraded animal fats in vessels, including dairy fats, ruminant adipose fats, non-ruminant adipose fats, and their mixtures. Evidence for plantprocessing is limited, as only a single vessel from Rakhigarhi provides direct evidence for

degraded plant matter, however, mixing models suggest the input of mixtures of C_3 plants and C4 dairy products in vessels (Hendy et al. 2018). Each of these products are discussed in detail below, providing further understanding of food choices of Indus inhabitants.

8.2.1. Dairy products

Table 8.1 provides a comparison of the results of organic residue analyses from Indus pottery and the frequency of faunal remains (when available) from study sites. The relative proportions of animal remains allow an assessment for whether the standard interpretations from the lipid residues match the zooarchaeological evidence.

Table 8.1: Comparison of the results of organic residue analysis from Indus pottery and frequency of faunal remains. Na: not available; nd: not detected; nr: not reported.

Site	Cattle/ buffalo	Sheep/ goat	Pig	Wild rumi- nants	Sherds analyse d via $GC-c-$ IRMS	Dairy fats	Rumina nt adipose fats	Mixed dairy/ ruminant adipose fats	Non- rumin ant fats	Mixed ruminant/ non- ruminant fats
ALM	80.5	9.3	3.8	5.2	9	nd	nd	nd	89	11
MSDVII	79.5	17	nr	1.9	21	5	10	nd	62	24
MSDI	83	10.4	2.0	3.0	14	7	nd	nd	93	nd
LHRI	na	na	na	na	12	nd	17	nd	75	8
KNK	na	na	na	na	3	nd	nd	33	33	33
FRN	78.3	11.1	0.6	0.7	7	14	14	14	29	29
RGR	na	na	na	na	7	14	nd	nd	71	14

Faunal evidence (NISP%) from relevant periods Organic residue evidence (% of analysed vessels)

Four vessels (3.3%) from four different sites have Δ^{13} C values that fall within the established range for dairy products, specifically, below -3.3‰. A relatively limited number of samples have been analysed, but this pattern suggests that vessels were not commonly used for this purpose. As mentioned previously (Section 7.3.1.2), none of these vessels are perforated vessels, which have been previously linked to dairy processing (contra Bourgeois and Gouin 1995) but are all jars with differences in shape and surface treatment (see Figure 8.1). Significantly, these vessels have $\delta^{13}C_{16:0}$ values ranging between -16% and -14.7% , which suggests that the input of C_4 plants within the vessels was very high. Given the evidence from the Haryana region for Indus cattle/buffalo consistently foddering on C⁴ plants throughout their lifetimes (Jones 2017; Lightfoot et al. in prep.), and comparison with previously published dairy reference fats from India (Craig et al. 2005), it is likely these vessels were predominantly used for the processing of

cattle/buffalo dairy products. The vessel from Farmana (FRN04) may, however, have also had the input of goat/sheep dairy products (see Chapter Seven, Figure 7.32).

Figure 8.1: Vessels with fatty acid-specific δ^{13} C values that indicate dairy processing. Top left: MSD329, top right: MSD3586, bottom left: FRN04 and bottom right: RGR20.

Figure 8.2: Examples of representations of bulls and related material culture in the Indus Civilisation. Top left: bull seal from Harappa, top right: unicorn seal from Mohenjo-daro, bottom left: terracotta bull figurine with mould and bottom right: toy cart from Nausharo. Source: www.harappa.com.
Cattle provide resources that have been intricately involved in the early urban economies (Zeder 2006). These large animals yield great output in both meat and milk per animal compared to sheep and goat and serve as beasts of burden in agricultural production (Zeder 2006). The importance of cattle in the Indus Civilisation has long been emphasised (e.g. Fairservis 1967, 1986), but zooarchaeologists have mostly focused on early cattle domestication and breed differentiation (Meadow 1981, 1989, 1993). The use of cattle for secondary-product exploitation in the Indus Civilisation has also been assumed (Fairservis 1967; Gouin 1991; Thomas and Joglekar 1994; Wright 2010; Chase et al. 2014a, 2018), but the precise timing for when this might have begun is unknown. Few other faunal assemblages have been as well-studied as those from Harappa (e.g. Miller 2004), but the high proportion of adult cattle found in Indus zooarchaeological assemblages generally is often used as evidence for their role in secondary products utilisation (e.g. Channarayapatna 2014, 2018; Chase et al. 2014a, 2018).

However, few researchers have discussed the complexities involved in managing cattle for maintaining a dairy economy in the Indus River basin. Cattle have higher water requirements than sheep and goats, and more selective pasturing preferences. They require shade and rest for almost eight hours a day to ruminate food from one stomach to another, limiting their mobility (Bhattacharya and Bhattacharya 2002: 165). Additionally, although nowadays we have come to rely on milk as a staple commodity, without human management, milk is a seasonal product. A cow must give birth every year in order to continue producing milk. The amount of milk cows produce depends on how much fodder they have access to, which changes throughout the year. Producing milk for any animal is demanding and requires suitable nutrition, which requires adding grain to the animal's diet, and extensive foddering may have been needed to maintain cattle within areas under intensive cultivation (Zeder 2006: 166), especially in summer months (Miller 2004).

As mentioned in Section 2.3.3., herd management is another vital part of maintaining a dairy economy. It requires the survival of a high proportion of female cattle and early slaughter of all but the few males kept for breeding purposes (Chase et al. 2014a). In contrast, keeping cattle for draft animals requires that a high proportion of castrated males be allowed to live a good deal longer than would be the case in herds managed for meat and milk. It is likely that multiple, possibly conflicting, cattle management strategies were needed to produce different resources of traction, meat and milk (Zeder 2006: 165). As it is yet not possible to distinguish cow, bulls, and castrates among cattle and buffalo in Indus contexts, making nuanced assessments of different management strategies based on age

and sex profiles of faunal remains alone difficult (Chase et al. 2014a: 9). At Harappa, Miller suggested that since 90% of the bovine animals were kept alive into adulthood, it was likely that cows were used for dairying and bulls were used for draft/traction, but evidence for dairying at Harappa is ambiguous (2004: 625).

The storage of milk is an additional practical aspect of managing a dairy economy. As milk spoils quickly and may be difficult to digest in its various raw forms, it must be quickly consumed or converted into products like butter, clarified butter or cheese. DNA studies of modern Indian populations indicate that the lactase persistence -13910*T allele has highest frequency among observed mutations as well as the widest distribution throughout the Indian subcontinent, but only one out of eighteen individuals in India can digest lactose (Gallego Romero et al. 2011; Gerbault et al. 2011). This statistic suggests that populations may have developed strategies to convert milk into more easily digestible products (such as yoghurt or ghee) from an early period.

Notably, scholars have noticed an emphasis on male cattle across the Indus Civilisation. Increase in use of cattle for traction is documented by their increased size from late pre-urban to urban period in Harappa, a change suggestive of an emphasis on males and castrates (Miller 2004: 619; Wright 2010; 204). Miller suggests there is evidence for the development of special cattle breeds for traction, such as for plow agriculture and harnessing carts (2003: 304; see also Wright 2010: 207). The most common engraving on Indus stamp seals consists of a male bovid (often called 'unicorn') in profile that faces the left side of the seal (Rissman 1989), and other bovids usually include male zebu cattle, bison and water buffalo (Ameri 2013: 361). These representations may suggest a requirement for bulls (Figure 8.2). Cows are noticeably missing from the visual repertoire of the Indus Civilisation, which may suggest that they were not as highly valued as bulls.

The results of this thesis confirm that cattle/buffalo milk, and possibly goat/sheep milk in some contexts, was processed in some Indus vessels, either stored in its raw form, or used to produce different types of dairy products, including yoghurt, clarified butter (ghee), or cream. However, the percentage of vessels that can be directly linked to dairy processing is very small, which raises questions about how widespread the practice of dairying may have been. A comparison of these results with ceramic residues found in prehistoric contexts around the world suggests that the minimal presence of dairy processing in Indus vessels from northwest India is highly unusual. Direct evidence for extensive dairy processing has been found at sites in northwest Anatolia as early as the

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seventh and sixth millennia BC (Dudd and Evershed 1998; Evershed et al. 2008), in the sixth millennium BC in eastern Europe (Craig et al. 2005), and in Britain by the fourth millennium BC, with increased dairy processing in the Bronze and Iron Ages (Copley et al. 2005a, 2005b, 2005c). Even in arid regions such as Libyan Sahara, the independent inception of dairying practices by mobile pastoral groups has been dated to the fifth millennium BC (Dunne et al. 2012, 2018).

In contrast, evidence from Neolithic northern Greece and western Turkey suggests that the incidence of dairy products was low, further decreasing in the Late Neolithic period (Whelton et al. 2018). It has been suggested that these results are consequence of the masking of the dairy lipid signal by the processing of greater than 50% non-ruminant fat in vessels. But as non-ruminants contribute a low percentage to the faunal assemblage in northwest India in the Indus period (Table 8.1) it is unlikely that Δ^{13} C values higher than 3.1‰ are false negatives. It is also possible that dairy consumption was limited to fewer groups or was not as widely practised in these specific Indus sites. Preliminary evidence from Chalcolithic and an Indus-period sites in Gujarat (García-Granero et al. in prep.) suggest a similar trend in Gujarat with possible exceptions at certain village sites (Chakrobarty, pers. comm.). The possibility that dairy products were rare or considered 'special' at certain settlements opens up new ways of understanding the relationship between Indus pastoral groups and communities and the ruminant animals they herded.

Alternative suggestions include the possibility that wooden vessels (that have not survived) were more widely used for processing milk products, of which ethnographic examples exist in Nepal and South India (Madella, pers. comm.). It is also possible that vessels used for processing dairy were re-used for several years, thus constraining the likelihood of other vessels demonstrating a strong dairy signal from contemporaneous contexts. Although lipid concentrations for the vessels with evidence for dairying are not very high (ranging between 14 μg/g and 66.8 μg/g), three out of the four vessels have relatively higher lipid concentrations compared to other analysed vessels from similar contexts, which might suggest their prolonged use.

Additionally, three vessels fall in between the thresholds for dairy and ruminant carcass processing. These include two vessels from Farmana: a small ledged jar (FRN18), a perforated jar (FRN11) and the base of a red-slipped jar (KNK01) from Khanak. It is possible these vessels were used for processing both dairy- and meat-products.

8.2.2. Ruminant carcass fats

Five vessels (4%) from the studied assemblage fall within the range of global references of domestic and wild ruminant carcass fats (with Δ^{13} C values between -3.3‰ and -1‰). One vessel (LHR40) has $\delta^{13}C_{16:0}$ value of -15.5‰, indicating increased amounts of C₄ plant input. Given the high input of C_4 plants into cattle/buffalo diet at Lohari Ragho I (Tames-Demauras 2018; Lightfoot et al. in prep.), it is likely that this vessel was predominantly used to process cattle/buffalo adipose fats. Other vessels demonstrate the processing of either sheep/goat or wild deer meat mixed with cattle/buffalo meat. These include two vessels from Masudpur VII (MSD3402 and MSD3788) and one vessel from Lohari Ragho I (LHR11).

Nine vessels (7.4%) have Δ^{13} C values falling where references for mixtures of ruminant and non-ruminant products meet. This suggests that these vessels were used for processing the meat of small ruminants/large ruminants and non-ruminants. These include one vessel from Alamgirpur (ALM119-370), three vessels from Masudpur VII (MSD3602, MSD3392, MSD3794), one vessel from Lohari Ragho I (LHR10), two vessels from Farmana (FRN09) and one vessel from Rakhigarhi (RGR02).

In total, eighteen vessels (28%) from the studied assemblage were used to process some proportion of ruminant adipose fats. Out of these, two vessels from Lohari Ragho I, (LHR40, a large dark-slipped jar and LHR10, a red-slipped jar of unknown rim diameter that resembles a Classic Harappan vessel) and one vessel from Khanak (KNK02, a small burnished jar) have high $\delta^{13}C_{16:0}$ values, indicating increased amounts of C₄ plant input. Although feeding patterns of fauna from Khanak are unknown, cattle/buffalo from Lohari Ragho I were consistently feeding on C⁴ plants (Tames-Demauras 2018; Lightfoot et al. in prep.), suggesting it is likely those vessels were used to process some proportion of cattle/buffalo adipose fats.

The overall low proportion of Indus vessels showing evidence of ruminant adipose fats are surprising given the high percentage of ruminant faunal remains at sites (Table 8.1). Cattle/buffalo generally make up 70-80% NISP of faunal remains at Indus sites, and proportions of sheep/goat range between 20-40% NISP. Available evidence from the study sites indicate similar patterns; but lower proportions of sheep/goat, and a small, but consistent presence of wild ruminant remains such as *Antilope cervicapra*, (blackbuck) *Tetracerus quadricornus* (four-horned deer), and *Bosephalus tragocamelus* (nilgai) (Channarayapatna 2014, 2018; Joglekar et al. 2016, 2017). Cut-marks have been found on

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all these animals, indicating they were likely hunted for primary products, i.e., meat (Channarayapatna 2014, 2018; Joglekar et al. 2016, 2017).

Evidence for the processing of ruminant adipose fats in vessels is very common across the world from prehistoric contexts. Residue analysis from early pottery from the Near East (Gregg et al. 2009), Anatolia (Evershed et al. 2008), Libyan Sahara (Dunne et al. 2012), and prehistoric Britain (Copley et al. 2005a, 2005b, 2005c) indicate that the meat of small and large ruminants (wild and domestic) was routinely processed in vessels in antiquity. Evidence from Neolithic northern Greece indicates an increased preference for the cooking of ruminant and non-ruminant meat in vessels among populations compared to the Levant and Anatolia (Whelton et al. 2018). However, given the limited evidence of ruminant adipose processing in vessels from Indus sites in northwest India, it is possible ruminant meat was consumed in other ways, such as via roasting. This is discussed in Section 8.3.

8.2.3. Non-ruminant fats

A high percentage (60%) of analysed vessels from all sites investigated from northwest India have Δ^{13} C values that match with modern reference fats from non-ruminant fats, implying they origin from mono-gastric, omnivorous animals such as domestic/wild pig or birds. This suggests most vessels were used to process the meat of such animals. However, as mentioned in Chapter Seven, there is a clear inconsistency between faunal assemblages from sites and isotopic values from the fatty acids within vessel residues, as pigs (domestic and wild) average only between 0.5-3% of the total NISP at the study sites (Table 8.1) (Singh et al. 2013; Channarayapatna 2014, 2018; Joglekar et al. 2013, 2016, 2017). Similarly, while the presence of birds such as peafowl, duck, and other omnivorous animals is attested, they make up a very small portion of the animal bones found (less than 1%) (Singh et al. 2013; Channarayapatna 2014, 2018; Joglekar et al. 2013, 2016, 2017; Joglekar and Channarayapatna 2018).

8.2.3.1 Pigs?

Zeder (1996:298) notes that pigs have higher reproductive rates and a greater per capita yield of fat-rich meat than any other domestic livestock species, and would have likely provided "a low-cost, low-labour intensive, highly reliable and highly productive resource" within household-based sty management, providing a different set of opportunities and obstacles for provisioning in early urban contexts (also Zeder 1991:30-32, 1996, 2003). In her opinion, within the Near Eastern urban context, pig-raising likely gave the urban

household considerable autonomy in an otherwise highly specialised interdependent economy (Zeder 1996: 298). This is supported by zooarchaeological evidence that suggests that pigs were a common staple in the diet in 'poor urban residential areas' in early Near Eastern cities (Zeder 2003, 2006). Extensive processing of porcine fats has also been found at sites in Neolithic Britain, associated specifically with Grooved Ware vessels (Mukherjee et al. 2007). However, pigs are less heat resistant than domesticated bovids and have different forage requirements. They are unsuited to being driven over long distances – and large herds of free-foraging swine raised around sedentary settlements can damage agricultural fields (Redding 1991; Zeder 1996). They also provide only one non-renewable resource, meat, compared to ruminants, which also provide milk and other secondary products. The importance of pigs within the Indus food economy is difficult to argue. Other possible non-ruminants that may have been processed in Indus vessels are birds such as peafowl or chickens.

8.1.3.2 Chicken?

As omnivorous animals, chickens route their dietary fats differently from ruminants, however, given the broad range of food sources consumed by them, the fatty acids derived from their carcass can exhibit considerably larger isotopic variability (Colonese et al. 2015). The Δ^{13} C values of most the analysed vessels in this thesis are consistent with established values of chicken adipose fats (Colonese et al. 2015), but the presence of the domesticated chicken in the Indus period is contested, and bones have not been recovered from the sites that have been investigated (Table 3.1).

Sewell and Guha (2004[1931]) and Prasad (1936) first attributed bird bones found in Mohenjo-daro and Harappa respectively to *Gallus gallus domesticus*, domesticated chicken. Its presence was also attested at Kalibangan (Fairservis 1967). Since then, it is a popular notion that Indus populations domesticated the chicken and that poultry farming was practised in the Mature Harappan period at Indus sites. It has also been suggested that chicken was cooked in *tandoors* (underground ovens) in the Indus period, as it is today (e.g. Lawler 2013, Bhattacharya 2016). At present, however, the chicken's domestication history within the subcontinent remains uncertain. Studies on the domestication of the chicken have revealed that the red junglefowl (*Gallus gallus*) is the primary wild ancestor of the domestic chicken and that there were possibly multiple, independent domestication events in southern China, South Asia and Southeast Asia (Liu et al. 2006; Kanginakudru et al. 2008; Miao et al. 2013). Modern genetics suggest that the Indian

subspecies *Gallus gallus murghi* contributed to the domestic gene pool (Kanginakudru et al. 2008; Miao et al. 2013) but the timing of this domestication is debated (West and Zhou 1988; Sykes 2012). There are also suggestions that the Mohenjo-daro and Harappa samples have been misidentified as bone size suggests the samples are intrusive (Sykes and Larsen, pers. comm.). *Gallus* bones have not been identified at other Indus sites; rather, blue peafowl (*Pavo cristastus*) and other types of birds such as ducks are more commonly identified (Joglekar et al. 2013).

The idea of the chicken being a bird unique to the Indus in the ancient world also comes from Mesopotamian texts dated to the Ur III period (During-Caspers 1989). Two Sumerian myths refer to a 'Meluhhan bird', described as the '*dar-musen Me-luh-ha'* that wears a 'beard' made of carnelian, and another reference mentions the '*dar-dar*' cries of the *dar* bird (During-Caspers 1989). It is suggested that this description refers to a cock, the red carnelian 'beard' possibly being the red wattles underneath the beak of this bird, and the sound of its cries indicating the crowing of a cock (During-Caspers 1989). While it is possible the bird in question was a cock, it is also possible the texts are referring to another bird or a even mythical bird. Meluhha is a toponym for the region of the Indus Civilisation from where Mesopotamia appears to have acquired several raw materials and products, as listed in administrative texts (Potts 1993, 1999; Magee 2014; Laursen and Steinkeller 2017).

Thus, despite Δ^{13} C values for vessels falling within the range for porcine or chicken adipose fats, at present it is not possible to be certain if either of them is the likely source for products processed within Indus vessels. The inconsistency between the faunal assemblage and isotopic results from vessel residues (Table 8.1) may also be due to other factors. These include factors influenced by archaeological practice, such as: i) taphonomic conditions privileging the survival of large animal bones, making large bones of cattle and buffalo dominate the assemblage; or ii) incomplete recovery practices that may reduce the chances of collection of small bones of pigs or birds. It also possible that: iii) animals may have been prepared away from sites and brought in for consumption, or bones may have been discarded away from sites (Mukherjee et al. 2008); iv) some animals were roasted on spits as whole carcasses, and not cooked in vessels; v) the potsherds selected may not be representative of the whole pottery assemblage, or lastly; vi) mixtures of products were processed in Indus vessels, which makes the isotopic values difficult to interpret.

Some of these factors may be ruled out. While taphonomic conditions no doubt privilege the survival and identification of large animal bones, the fact that cattle bones dominate Indus zooarchaeological assemblages cannot be explained by coincidence or taphonomy alone. Similarly, although archaeological recovery methods have focused on the recovery of material culture and paid less attention to bioarchaeology, several excavations in the region have dramatically improved sampling strategies for archaeobotany and zooarchaeology and collect small remains such as fishbones and charcoal during excavation. Furthermore, we have evidence for the butchering of a range of different animals from several sites including Masudpur I, Masudpur VII and Farmana (Joglekar et al. 2013, 2016, 2017; Channarayapatna 2014, 2018), including the presence of a variety of skeletal elements, such as axial, forequarter, hindquarter and extremities such as phalanges, within fills and pits inside settlements (Channarayapatna 2014). For example, at Farmana, wild and domestic animals were butchered on-site, with no evidence for disarticulation and removal of low meat-bearing parts of wild animals at a separate kill-site (Channarayapatna 2014, 2018). It is thus unlikely that animals were prepared away from sites and brought in for consumption or discarded outside sites. It is possible, however, that animals were processed for consumption in different ways, or the potsherds selected were not representative of the whole pottery assemblage, or that mixtures of products were processed in Indus vessels, which makes the isotopic values difficult to interpret.

8.2.4. Mixtures of products

As highlighted in Chapter Three, Section 3.8.3., significant interpretative challenges exist in resolving compound-specific isotopic data from contexts where both plants and animals were processed in vessels. Hendy and colleagues (2018) demonstrated that mixtures of ruminant adipose products and C₃ plants could create Δ^{13} C values similar to non-ruminant fats. Given the diversity of resources that were available to Indus populations, it is highly likely that individuals used vessels to process mixtures of plant and animal products to create foodstuff, and that vessels were multi-functional throughout their life-histories.

The availability of C_3 and C_4 plants, and freshwater resources to Indus populations in northwest India further adds to the complexity in resolving isotopic mixtures. Mixing plots adapted from Hendy and colleagues (2018) are given below (Figure 8.2) that demonstrate hypothetical Δ^{13} C values created by mixing various animal and C₃ and C₄ plant products using published references.

A) provides hypothetical Δ^{13} C values generated by mixing C₃ plants and C₄ fed ruminant dairy products, and B) provides hypothetical Δ^{13} C values generated when C₄ plants are mixed with C_3 plant-fed ruminant carcass fats. A) demonstrates that equal

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mixtures of C_3 plants with C_4 dairy products would generate values that fall between -1‰ and 3‰, which correspond to those obtained from most Indus vessels and those that plot within ranges for ruminant and non-ruminant adipose fats. Conversely, B) demonstrates how an equal mixture of a C_4 plant, for example, millets, with ruminant meat fed on C_3 plants would generate Δ^{13} C values the same as those created by dairy fats.

A) offers an alternative potential explanation for the sources of the Δ^{13} C values obtained from most of the lipid extracts of Indus vessels. This suggests that mixtures of C_3 plants and C_4 dairy products (or mixtures of C_3 plants and ruminant adipose fats, as in Hendy et al. 2018) could have been processed in Indus vessels. Alternatively, B) suggests that Δ^{13} C values falling within the 'global' dairy range should no longer be considered as equivocal evidence for the processing of dairy in vessels. In environments where there is minimal evidence for availability/ consumption of C_4 plants or for ruminant animals grazing on C_3 plants, however, Δ^{13} C values under -3.3‰ are still likely to demonstrate dairy processing. Within the Indus context, although there is evidence for the cropping of C⁴ plants such as millets at multiple sites, enamel carbonate isotopic values from ruminants suggests that both domestic and wild ruminants had at least some input from C_4 plants in their diet (Jones 2017; Lightfoot et al. in prep). As it is unlikely that ruminants were entirely C₃ plant-fed, Indus vessels with Δ^{13} C values under -3.3‰ can probably be more confidently associated with dairy processing, but they are few in number (Section 8.2.1).

In summary, the present evidence from lipid residues suggests that Indus vessels were used to process a range of products. Unfortunately, most of the vessels have isotopic values that are ambiguous, with equifinal interpretations. The current options include:

- 1) Non-ruminant fats (porcine or other omnivorous taxa),
- 2) Mixtures of C_3 plants and dairy products from ruminants grazing on C_3 and C_4 plants (Hendy et al. 2018)
- 3) Mixtures of C_3 plants and adipose fats from ruminants grazing on C_4 dairy products (Figure 8.2, A), or,
- 4) Mixtures of C_3 and C_4 plants with C_3 and C_4 fed ruminant adipose fats,
- 5) Mixtures of C_4 plants and C_3 ruminant adipose fats, as C_4 plants are high in $C_{16:0}$ and C_3 fed ruminant adipose fats are high in $C_{18:0}$, which cause an offset as observed in the ruminant adipose/non-ruminant fats range.

Out of all these options, options 2), 3) and 4) match the available bioarchaeological evidence. Option 5) does not match the molecular evidence as there is minimal evidence for direct plant-processing in the lipid extracts (except for a single vessel, RGR24). There

Figure 8.3: Density distributions of Δ^{13} C values obtained by theoretical mixtures of plant and animal products. A) Theoretical mixtures of C_4 dairy products with increasing C_3 plant contribution which create high positive Δ^{13} C values, similar to ruminant adipose fats and non-ruminant fats, and B) Theoretical mixtures of C_3 ruminant adipose fats with an increasing amount of C_4 plant lipids, which create negative Δ^{13} C values similar to dairy fats (Courtesy Oliver Craig).

is also an absence of purely C_3 -fed animals according to the available isotopic evidence from animals. Without further knowledge of the isotopic end-members for different food products, which may include C_3 - and C_4 -fed ruminant and non-ruminant animals and a wide range of plants, further interpretation or more accurate quantification using stable isotope analysis is challenging at present. A dedicated programme involving the creation of theoretical models of mixtures of different products, experimental research, and the collection of modern reference fats within South Asia may help resolve such interpretational challenges.

The interpretations offered by theoretical mixtures also dramatically alter traditional interpretations offered by the Δ^{13} C proxy. Crucially, these mixtures challenge interpretations for sources of lipids in vessels in *all* environments with C_3 and C_4 plants. This includes regions such as Africa and Anatolia, where assertions about early evidence for dairying (Evershed et al. 2008; Dunne et al. 2012) may now be contested.

8.3. Food preparation in the Indus Civilisation

The results obtained from this thesis provide new ways to imagine a fundamental part of the broad realm of culinary activity, or foodways, in the Indus Civilisation: the preparation of food. Cooking or preparing foodstuff requires skills to coordinate the preparation and input of ingredients in a particular order, the heating source and manipulation of vessels to obtain the desired dish and taste, it also takes a considerable amount of time (D'Anna and Jauss 2014; Nell 2014)*.* This aspect of time and skill involved in cooking is rarely discussed in archaeological literature. The complexities involved in cooking mean it is usually assigned to certain persons who develop special skills in this activity (D'Anna and Jauss 2014: 65). In many past societies and even today, the labour of cooking, especially in domestic contexts, is deeply gendered and mostly carried out by women (Hastorf 1991, 2016; Bray 2003; Twiss 2012). Although we do not know if cooking was a gendered practice in the Indus Civilisation, the preparation of food in the household in contemporary Indian societies is often the sole responsibility of women. The gendered nature of household labour is a vital characteristic of most agricultural or sedentary societies and is important to keep in mind when thinking about ancient practice.

Some material aspects of the preparation of food may provide insight into the social dimension of food preparation in the Indus Civilisation. The presence of open hearths and ovens in architectural complexes (for example, at Farmana), suggests that cooking took place within the household or in their close vicinity in open spaces like courtyards. As an openly visible practice, people who cooked would have formed a loose community of practice exchanging skills, techniques and recipes (after Wenger 1999; D'Anna and Jauss 2014: 75). Perhaps people not practicing cooking themselves would have been involved in other ways and smelled the food being prepared (D'Anna and Jauss 2014:75). Small rooms with hearths are found in some contexts (for example, at Alamgirpur, Masudpur VII, Masudpur I and Farmana) (Shinde et al. 2011; Singh et al. 2013; Petrie et al. 2017). Indoor

cooking may have been a more restrictive activity or may have been practiced seasonally, as evinced via ethnographic studies (Jordan 2003).

Cooked food, or specifically, food heated, stewed, or boiled in vessels was probably one of many facets of Indus diet. Other techniques such as drying, fermenting, roasting, or baking, may also have played a major part of Indus foodways. Although evidence for all of these forms of food-processing is fragmentary, some examples have been found and may have social implications. As noted in Section 2.2.3.3, a large proportion of animal bone at Farmana, and small proportions at Masudpur I and Masudpur VII were charred or partially charred (Channarayapatna 2014; Joglekar et al. 2016, 2017). At Farmana, it was observed that all anatomical elements of cattle bones were charred to different degrees, whereas specific elements (cranial fragments, ribs, scapula, vertebrae, and phalanges) of sheep/goat were charred (Channarayapatna 2014). The extensive charring of ruminant bones might suggest that their meat was processing via spit-roasting. The charring of bone may also occur through its use as fuel (Théry‐Parisot 2012; Costa 2016) or through post-depositional processes (Costa 2016), but the high percentages of charred bones in the assemblages of these three sites suggests it may be connected to how animal carcasses were being processed.

Finally, eating is, in all cultures, a social activity and commensality is undeniably one of the most important articulations of human sociality (Twiss 2012; Kerner et al 2015; Hastorf 2017). Levi Strauss (1966) has famously suggested that roasted food belongs to "exo-cuisine" that which one offers to guests or large groups of people, whereas boiled foods can most often be ascribed to what might be called "endo-cuisine", prepared for domestic use, destined to a small, closed group. Although not much is known about Indus commensality, and explicit evidence for 'feasting' has not been discovered, it is possible that the meat of small and large ruminants was preferentially prepared via spit-roasting in Indus settlements in northwest India and consumed by larger groups of people, as the meat produced would likely be too much to feed a small household. A distributive system of meat-sharing was in place at Bagasara (Chase et al. 2014a), and possibly at Farmana (Channarayapatna 2014), which suggests meat may have both been prepared within and outside the household. As food preparation was perhaps executed in both public and domestic settings, it constitutes a starting point to think about diverse spheres of Indus commensality.

The consumption of unleavened breads from cereals such as wheat, barley, and millet, and pulses such as chickpea was also probably common in the Indus Civilisation (Weber 2003; García-Granero et al. 2017). Today, cereal grains are de-husked, cleaned, and stone-ground to produce flour, and lentils are usually soaked, then dried and stoneground with the use of '*sil batta'* (grinding stone and pestle). Tools similar to those found in modern South Asian kitchens, such as grinding stones, mortars and pestles have been located within many Indus settlement contexts (e.g. Shinde et al. 2011; Singh et al. 2013; Joshi 2015), which have led to assumptions that flatbreads like modern-day *rotis* were prepared in the Indus period (Nath 2014; Granero-Garcia et al. 2017). Starch-grains of barley, mango, and small millet adhering to the surfaces of pounders and grinding stones were found from Farmana (Kashyap and Weber 2010), and starch grains and phytoliths of small millet and tropical pulses (horse gram and mung bean) have been found on grinding stones from Indus period (and earlier) sites in Gujarat (García-Granero et al. 2017). The available evidence thus suggests the use of grinding stones for the processing of cereals and pulses for flour, and possible fruit (e.g. mango) for the creation of pastes. This realm of plant-processing was likely to be more catered towards individual, or household consumption rather than for large groups, but very large grinding stones have been found in Rakhigarhi (Yadav, pers. comm.).

Organic residue analysis has shown the absence of millet biomarkers within the ceramic vessels (Chapter Seven), which may suggest that millet seeds were more likely to be processed as flour than boiled or cooked. Given the high C_4 plant input in the diet of ruminant animals, especially cattle/buffalo (Jones 2017; Lightfoot et al. in prep), it is possible that millet was used primarily as fodder, although it is also possible that these animals foraged on wild C_4 plants. It is also probable that plants were cooked or boiled in vessels, as suggested by mixing models. However, only a single vessel analysed for Rakhigarhi in this thesis provides evidence for plant processing.

While it is not possible to be certain of the dishes prepared or preferred by Indus populations, some broad suggestions can be made. Given the evidence, it appears that either dairy-based stews mixed with pulses, cereals and vegetables, or alternatively, porcine or avian meat were regularly processed in a range of different vessel-types. The meat of cattle/buffalo, sheep/goat or wild deer may also have been prepared in vessels but may have been preferentially prepared via spit roasting over fire. Finally, dairy products were also processed in vessels, but only four vessels demonstrated clear evidence for dairy processing.

8.4. Rural and urban food practice in northwest India

Chapter Two highlighted how studies on Indus agriculture and food production in the urban period demonstrate that farmers inhabited diverse environments and grew a range of crops across different regions of the Indus Civilisation (e.g. Vishnu-Mittre and Savithri 1982; Possehl 1992; Weber 2003, 2010; Petrie et al. 2016; Petrie and Bates 2017). Evidence confirms regional diversity in crop choices and relative proportions/reliance on winter and/or summer crops in different regions (Petrie et al. 2016; Petrie and Bates 2017), but the only urban settlement that has been studied in detail is Harappa. At Harappa, strategies such as intensification and diversification of agricultural and pastoral resources have been used as models to suggest how large urban settlements were sustained during the Indus urban period (Miller 2004; Wright 2010). These strategies have also been discussed in terms of their suitability for mitigating risks associated with changing environmental conditions or food stress in the post-urban period (Miller 2006, 2015; Petrie 2017). These models imply that diversity and diversification played an important role in Indus agricultural practices (Petrie 2017: 51). Importantly, though, it appears that while there is clear evidence of diversity in terms of plant products used, there is only a limited amount of archaeobotanical evidence that provides clear insight into *diversification of Indus subsistence practices over time*. Similarly, the evidence available from faunal assemblages across the region suggests that Indus populations used a limited suite of domestic animals, primarily cattle/buffalo, with limited evidence of variability across regions or change over time. Our knowledge about subsistence practices is gradually increasing, especially in northwest India and Gujarat, but nuanced characterisation of regional diversity (or uniformity) of practices requires more dedicated studies and analysis.

Results from ceramic lipid analysis presented in Chapter Seven suggest there were different types of products being processed in vessels in urban and rural settlements, specifically in the Early Mature Harappan period (EMH), but with evidence of continuity in the Late Mature Harappan (LMH), and Late Harappan period in northwest India. This thesis thus provides us with an understanding of food practices in a whole range of settlements: small villages, villages, towns, and cities in the region, and also suggests evidence for homogeneity in terms of food choices.

Systematic surveys and calculations of site densities around Rakhigarhi in the urban period have revealed linear concentrations of settlements extending towards the southwest (Singh et al. 2010; Petrie et al. 2017; Green and Petrie 2018). Additionally, comprehensive multi-sensor and multi-temporal approaches using remote sensing and computational methods have revealed a vast network of palaeo-rivers and complex fluvial history in this region (Orengo and Petrie 2017). It has been suggested that the location of these settlements along water channels and their high density may have been a significant feature, or a 'signature landscape' of the urban Indus period in this region (cf. Wilkinson 2003; Green and Petrie 2018). 'Signature landscapes' manifest differences in land use, production, and cultivation practices that can reveal trends in subsistence practices and social processes (Wilkinson 2003). It is thus possible the common environmental landscape and shared network of possibly ephemeral watercourses, especially between Rakhigarhi, Lohari Ragho I, Masudpur I, and Masudpur VII created access to similar plant and animal resources, thus resulting in broadly similar culinary choices across settlements.

Additionally, fatty acid-specific isotopic values are likely indicative of animal feeding patterns (Dunne et al. 2018; Whelton et al. 2018), which appear to be wide-ranging within single sites in northwest India. Large variations in $\delta^{13}C$ values at Masudpur VII (small village), Masudpur I, Lohari Ragho I (villages) and Rakhigarhi (city), may be indicative of the mobility of animals and diverse feeding strategies. Petrie (2017: 55) suggests that settled populations may have been relatively mobile in order to survive a constantly shifting hydrology. These results have implications for how we imagine rural *and* city inhabitants managing and moving their animals in their surrounding landscape. However, the precise mechanisms and management of different animals and crops at these sites is yet not understood. As archaeobotanical and zooarchaeological analyses from Rakhigarhi are ongoing, questions about how residents of Rakhigarhi sourced their food, and whether they were reliant on their surrounding hinterland settlements for food provisioning are yet to be answered.

The evidence presented in this thesis suggests that rural and urban inhabitants had equally complex and varied animal management strategies and processed similar products in vessels. Evidence from rural Indus settlements in northwest India already indicates the presence of a unique, rural ceramic industry, which is influenced by, but visually distinct from, the urban pottery repertoire (Parikh and Petrie 2017, 2018; Ceccarelli in prep.; Parikh in prep.) This suggests that rural craftspeople were actively engaged in production and consumption of pottery that set them apart from urban inhabitants in some way. This is matched with further evidence of complex crop-processing choices made by rural inhabitants, for example, at Masupdur I and VII (Bates 2016; Petrie and Bates 2017). To what extent was the food they processed in vessels seen as 'different', or representative of

a rural identity? Did inhabitants at different settlements have distinctive concepts of culinary identity? Were these linked with their rural character, or their proximity or distance from urban settlements? Although at present these questions cannot be answered, they are vital to broadening how the somewhat nebulous concept of 'identity' in the Indus Civilisation can be conceptualised (e.g. Chase et al. 2014b).

The results from Chapter Seven also inform us about food and vessel usage practices in the post-urban period/after 4.2 ka BP. The evidence from Alamgirpur suggest continuity in vessel-use practices from 4.2 ka BP and onwards, whereas Masudpur VII has evidence for the use of a wide range of products in vessels dating after 4.2 ka BP and also in the Late Harappan period, with possible evidence of diversification of products compared to the urban period. The results fit with other evidence from northwest India that demonstrates the general continuity of cropping practices and watering regimes at rural settlements during this period of cultural change (Petrie et al. 2016; Jones 2017; Petrie and Bates 2017). It is possible that the diversity of products used in the urban period in rural settlements allowed populations to compensate for potential reduction in resources or cultural changes in the post-urban period. If rural populations were "optimising their economic activities in a diverse ecology" (Wright 2010: 214) and were also well-versed with the potential risks they faced due to changing environmental conditions, they may have adapted to the hydrological unpredictability that was likely experienced during and after 4.2 ka BP (Petrie 2017). No Late Harappan occupation has been reported from Farmana or Rakhigarhi (Shinde et al. 2011; Vahia et al. 2016; Nath 2018), suggesting that larger urban settlements may not have survived in the post-urban period, but such occupation may underlie the large parts of Rakhigarhi that are buried under the modern village that covers a large portion of the site (Vahia et al. 2016; Petrie 2017). While concrete evidence is lacking, it is possible that urban settlements were unable to adequately provision food for populations in the late urban period, resulting in abandonment of largerscale settlements. These results reiterate the importance of investigating rural, small settlements across the Indus Civilisation and testing the relationship between large, medium-sized and small rural settlements, particularly in terms of food-production. They also highlight that regional patterns must be examined independently, and generalised relationships between climate, subsistence and society in the region must be interrogated.

This thesis addresses gaps in previous research by studying food production and vessel usage within large, medium-sized, *and* small, rural settlements in northwest India. Vessels from the urban and post-urban periods have been examined via organic residue

analysis to examine how quotidian commensal practices were constructed within settlements in the region, and if they changed over time. Importantly, this thesis also focuses on addressing the interaction between Indus material culture and foodstuff, on a regional, and local, site-specific scale.

8.5. Cautionary tales: preservation and interpretation

Chapter Three highlighted issues surrounding preservation of organic residues in hot, arid and seasonally-wet environments. Low lipid recovery from vessels create significant challenges and make interpretations about archaeological vessel-use difficult. Similarly, the possibility of contamination from sediments, post-excavation practices, or during laboratory processing add further complications to interpretation.

In this thesis, predicting the preservation of lipids was not possible. Mean lipid concentrations across analysed samples were very low, creating interpretational challenges. The similarity of results across multiple contexts, however, assert the reliability of the results, and the range of lipid concentrations within vessel fragments recovered from the same context suggest that the connection between lipid yield and archaeological use of the vessel is valid. Combining chemical date with compound-specific isotopic analysis also enables a way to cross-reference the results obtained. Attempts have been made to control for contamination wherever possible, but these must be assessed at a case-by-case level.

It is highly likely that results from organic residue analysis are missing several nuances of meal preparation as it is not possible to get down to details of 'ingredients' processed in vessels. For example, it was not possible to achieve species-specificity of animal fats and confidently detect direct plant-processing in vessels. Thus, at present it is not possible to know precisely how quotidian activities occurred in practice. It is also possible that not all of these vessels were used to process food products; organic products processed in vessels may have had other economic functions. This conceptual challenge is similar to others faced by archaeologists called the 'utilisation fallacy', in which everything recovered is interpreted into terms of some anthropogenic meaning. However, as most vessel fragments analysed for this thesis were recovered from domestic contexts, it is likely they were used on a day-today basis.

Finally, and critically, studies on Indus lifeways sometimes suggest an unchanging connection between ancient and present-day practices of the subcontinent, particularly in rural settlements. Ethnographic analogies can be highly relevant and informative (e.g. Bhattacharya and Bhattacharya 2002), but scholars often uncritically apply ethnographic or contemporary examples to explain archaeological data, implying long-term historical continuities between the past and the present (e.g. Allchin and Allchin 1973; Kenoyer 1998). These discussions continue to exist and may be used to promote specific agendas. In some cases, this is also true for studies on Indus food production and cuisine (e.g. Achaya 1998; Kashyap and Weber 2010; Lawler 2012, 2013). For example, anachronistic references to 'curry' and 'proto-curry' (e.g. Lawler 2012, 2013; Bhutia 2018) are common, but they are typically reductionist and irrelevant within the context of the Indus Civilisation. Although it is well-documented that contemporary Indian communities exhibit conservatism in food production and taste preferences (e.g., Appadurai 1998), several of these food traditions have extremely complex historical trajectories and are inextricably linked to religious and caste identity. Similarly, a number of ingredients have been introduced to South Asia in recent historical periods and via colonial processes (e.g. tomatoes, potatoes, chillies), which have irrevocably transformed South Asian cuisine (Nandy 2004). Thus, while this thesis refocuses attention on Indus foodways, this is done in order to centre investigations into the complex relationship between environment, plants, and animals in the Indus Civilisation. A concerted effort has been made to *not* fall into the conceptual 'trap' of drawing connections between modern South Asian cuisine and how we imagine Indus food. While several ingredients important to South Asian cooking today existed in the Indus period (e.g. summer and winter pulses), how they were brought together to create specific recipes is not the focus of this thesis.

8.6. Chapter Summary

This chapter brings together the results from the organic residue analysis to conceptualise food choice and vessel usage in the Indus Civilisation. It refocuses attention on foodways in the Indus Civilisation, a topic that has been neglected in the past.

The results of the thesis provide empirical, direct evidence for the processing of a variety of products in vessels from rural and urban Indus settlements in the urban period, and rural settlements in the post-urban period in northwest India. Evidence points to the multiple products being processed in vessels. However, a substantial quantity of the vessels have compound-specific isotopic values that may have equivocal interpretations caused by the challenges in resolving mixtures using organic residue analysis. Additionally, there was limited evidence for the use of vessels for dairy processing, although the possibility of vessels being used both for mixing dairy and other products cannot be excluded. The similarity of the types of products processed in vessels at rural and urban settlements

indicate similarity of culinary choices in the region; while the lack of change in products processed over time indicates diachronic continuity in food and vessel-use choices.

The results of this thesis have added to the existing evidence collected during the *Land, Water and Settlement* and *TwoRains* projects (e.g. Bates 2016; Jones 2017; Ceccarelli in prep. Lightfoot et al. in prep.; Parikh in prep.) that suggest the complexity of rural food and ceramic production strategies in northwest India, and their continuity (with small changes) in the post-urban period. The results of this thesis are encouraging and create new avenues with which to imagine Indus food and culinary identity, but there is inevitably a need for more bioarchaeological data from Indus settlements to explore potential regional variations and make more nuanced interpretations about changes in access to food sources over time. At present, the existence of entangled ecological and cultural considerations perpetuates circular reasoning, 'correlation-equals-causality' arguments, and uncritical connections from contemporary South Asian lifeways. It is clear that there is a need for more archaeological, geoarchaeological, archaeobotanical, zooarchaeological and palaeoclimatic data from a broad range of environments across the greater Indus region to test the relationship between climate, agriculture, pastoralism, urbanism and foodways in the Indus Civilisation.

The next chapter summarises the aims, scope and results obtained from this thesis, and discusses future research avenues within Indus archaeology and organic residue analysis in South Asian archaeological contexts.

Chapter Nine

Towards an integrative approach for studying Indus food: implications for Indus archaeology

9.1. Thesis Summary

This thesis sets out to test differences and/or similarities in vessel-use across settlements in the Indus Civilisation, specifically in northwest India, by using ceramic lipid residue analysis as a proxy for investigating food preparation in vessels. It also investigates whether there was continuity or change in vessel-use practices in the post-urban period. This research is rooted in questions critical to Indus archaeology that are centred on: 1) understanding the nature of urbanism and rural-urban relationships, 2) understanding Indus rural and urban food production, and 3) characterising the degree of change to Indus lifeways in the post-urban period. This thesis particularly focused on investigating foodstuff and food processing because as essential and quotidian practices, they are a valuable lens by which to investigate the degree of variability in the urban period and extent of change in the post-urban period.

Chapter Two demonstrated that there is still much to learn about the nature of urbanism in the Indus Civilisation, as well as the degree of regional variability across the greater Indus region in terms of ecological zones, crop availability, and access to rainfall and water (Petrie 2013, 2017; Petrie and Bates 2017; Petrie et al. 2016, 2017, 2018). Characterising regional variability affects how well we can understand and explain cultural changes in the post-urban period. A 'bottom-up' consideration of evidence of local climatic and agricultural strategies suggests sustainability and resilience of practices, but there is simultaneous evidence for economic simplification through reduction in settlement scale and reduced interaction networks (Petrie 2017). The current hypothesis proposes that large urban settlements embedded in large networks may have become unsustainable in the post-urban period while rural settlements were able to continue to thrive due to preexisting food diversity and sustainable practices, particularly in northwest India (Petrie 2017). Chapter Two also reviews current knowledge about agricultural and pastoral practices in the Indus Civilisation in the urban period, with an emphasis on what it suggests about social practice and foodways. At present, knowledge about food processing and food

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choice when it comes to animal and/or secondary products is inadequate. Much more highresolution data is required to reach a comprehensive understanding of food procurement, processing and consumption in the Indus urban and post-urban periods.

Chapter Three reviews the basic principles related to ceramic organic residue analysis, including different types of lipids encountered in archaeological contexts, issues about preservation and degradation, contamination, analyses and interpretation. Interpretational challenges that are encountered later in the thesis are highlighted in Section 3.6.3.

Chapters Four and Five lay out the necessary archaeological and methodological background information for the analyses conducted in this thesis, setting out key palaeoecological, contextual and practical information that are necessary for the interpretation of data.

Chapters Six and Seven present the results obtained from GC-MS and GC-c-IRMS analyses and provide discussions of the results. As most study sites are located in environments not particularly conducive to the survival of organic remains, it was necessary to test the viability of organic residue analysis within the South Asian context. Chapter Six presents the results of lipid yields obtained from Indus vessels collected from 1) collections with little to no contextual information (Group I), 2) recently-excavated material with variable degrees of spatial contextual information (Group II), and finally, 3) freshly-excavated material with detailed contextual information and adhering burial soil for control (Group III). The results suggest that while lipids are preserved and extractable from vessels collected from different types of contexts, overall lipid yields are relatively low, and the degree to which one can confidently link lipid yield to archaeological use of the vessel and adequately interpret the results is dependent on available contextual information and post-excavation treatment details. Lack of local or regional modern reference fat material also presents challenges to interpretation, as explored in Chapter Seven. Thus, while preservation conditions and methodological challenges are an impediment to the widespread use of organic residue analysis in Indus archaeology, the degradation of lipids is not a predictable, linear process and should not discourage future researchers. Future studies would benefit from more experimental work coupled with modern reference studies to address methodological and interpretational uncertainties.

Chapter Seven presents the site-specific and vessel-specific results of organic residue analysis. Combined with available contextual, archaeobotanical and zooarchaeological evidence, they provide interesting new data on food production and processing from small, medium-sized and large settlements in northwest India. Despite interpretational challenges, the data provide the first clear evidence of secondary-products utilisation (although minimal) at certain settlements, as well as clear evidence for meat-processing in vessels. The results also hint at complex animal feeding practices at rural and urban settlements, suggesting that organic residue analysis provides a practical avenue with which to develop or test models of social and ecological change. The results demonstrate some variability in vessel-usage across settlements, but there are no clear differences in products processed in vessels between urban and rural settlements in the urban period, and rural settlements do not demonstrate measurable change in vessel-usage in the post-urban period. A larger sample, together with detailed, well-dated archaeological contextual information from different sites located in different regions of Indus Civilisation would likely provide a more refined understanding of regional diversity in food choice and processing in the Indus context.

Chapter Eight provides a synthesis of the results obtained from the thesis to problematise and more broadly consider their implications for Indus archaeology. It demonstrates how concepts of food choice and foodways are productive avenues with which to approach Indus environment, plants, and animals and their relationship with space, sociality and identity.

The following sections discusses the specific ways in which this thesis opens up new avenues to contribute to new understandings of the connections between environment and society in Indus archaeology and biomolecular approaches in South Asia and other parts of the world.

9.2. Developing new perspectives and approaches in Indus archaeology

9.2.1. Theorising food in Indus archaeology

This thesis discusses how developing stronger theoretical approaches to Indus food are relevant to Indus archaeology, South Asian history and contemporary ideas about ancient cuisine and gastro-politics in South Asia. Moving away from static ideas of 'subsistence', this thesis focuses on food production and processing as active and meaningful, driven by choices made by inhabitants at different settlements. It takes a holistic approach by looking at how both plant and animal products may have been conceptualised and consumed as food.

An evidence-based, 'bottom-up' foodways approach in Indus archaeology would require a great deal more of regionally nuanced data coupled with high spatial and chronological resolution. This would include greater specificity about food acquisition, such as how a variety of ingredients are grown, reared, collected and/or caught; food processing and preparation, such as data on butchery (Chase 2010, 2012, 2014; Chase et al. 2014a), microbotanical and chemical evidence available from starch grains and phytoliths (García-Granero et al. 2015, 2016) and organic compounds associated with tools and in vessels; and finally food consumption via isotopic evidence from humans and animals (Chakraborty et al. 2018; Chase et al. 2018; Lightfoot et al. in prep.). At present different elements of the available data do not neatly fit into a single narrative and come with their own methodological uncertainties and interpretational limitations. This suggests that this approach will likely reveal many new aspects of Indus lifeways that are ill-defined or unknown.

9.2.2. Refining models of Indus agriculture and pastoralism

The review of present evidence on Indus archaeobotany, and specifically, animal use and pastoralism highlight that concepts of diversification, intensification and extensification in the urban period and their possible transformation in the post-urban period in the Indus Civilisation must be archaeologically tested. At present there are several assumptions about change over time that have not been systematically investigated.

9.2.3. Biomolecular archaeology in South Asian archaeology

Already well-established in archaeology in Europe (and increasingly in other regions of the world) as a means by which to investigate the relationship between material artefacts and organic products (Evershed 1998, 2008a; Craig et al. 2011; Dunne et al. 2017; Whelton et al. 2018), biomolecular techniques in Indus archaeology have seen limited application (e.g. Bourgeois and Gouin 1995). As the first large-scale investigation into lipid residues from Indus vessels, this thesis demonstrates the viability of organic residue analysis as a technique by which to investigate food and the use of vessels in South Asian archaeological contexts. The results from this thesis also provide a critical way to examine previously published results of lipid residues from environments with isotopically wideranging plant and animal resources. The possibility of mixtures of animal and plant resources in vessels must be considered as alternative option for vessel-use in diverse prehistoric and historic contexts.

However, it is important to acknowledge that certain limitations, such as poor lipid preservation, the absence of reference fats, and interpretational challenges related to stable isotopic analyses must be addressed for the success of any future study in South Asia. Going forward, lipid residue research in South Asia would benefit from: 1) a dedicated reference-collection programme of modern fats from a variety of ruminant and nonruminant animals in South Asia, following precautions detailed by Roffet-Salque and others (2017); 2) a focus on regions in the area demonstrating good organic preservation (such as regions with high aridity) for comparison with other regions; 3) experimental research with modern cooking/mixing experiments as well as degradation experiments to clarify the parameters that might affect future interpretation.

Current methodological advancements in the field of organic residue analysis may also help problems related to low lipid recovery and contamination in South Asia be addressed. For example, the use of supercritical fluids for higher rates of extraction of lipids from ceramics (Deviese et al. 2018) or the use of vibrational spectroscopy techniques to discriminate between natural and synthetic organic compounds used in conservation (Casanova et al. 2016) could be instrumental in improving lipid yields and issues related to the analysis of pottery from collections.

Future lipid residue analysis could also investigate a myriad of topics that would contribute to a better understanding of major developments in South Asian prehistory and protohistory. These include the origins of dairying in the subcontinent and the process of neolithisation, as well as changes in culinary practices with the advent of religions with varying food taboos in later historic periods. Other exciting developments in the field, such as the direct compound-specific radiocarbon dating of residues in pottery vessels (Berstan et al. 2008; Casanova et al. 2018) may be able to resolve long-standing debates about chronologies for various types of pottery in the Indus Civilisation and later historic periods in South Asia. Additionally, δ^2 H values of animal fat residues in vessels may reflect variations in precipitation, providing a unique, extremely localised climate proxy (Roffet-Salque et al. 2018). If such an approach could be utilised for the South Asian context, it could potentially revolutionise our understanding of not only how Indus communities adapted to climate change but also investigate human response to climate change in a wide range of contexts.

The application of other biomolecular methods such as starch-grain analysis has already been utilised within Indus archaeology to investigate Indus processing and artefact use (e.g. García-Granero et al. 2015, 2016). Recent research suggests that proteins may

also be recovered from ceramics, providing species-specificity and extremely highresolution data about vessel-use (Cappellini et al. 2018; Hendy et al. 2018). If viable in South Asia, the combination of starch-, lipid- and proteomics techniques would also yield fascinating insights into the processing of organic products and human-artefact relationships.

9.3. Final conclusions

This thesis poses questions about foodstuff processed in the Indus Civilisation, specifically addressing how issues about food acquisition and processing are linked to concepts about urbanism, ruralism, and our present knowledge about agricultural and pastoralism, environment and climate change. This study uses a novel approach in South Asian archaeology that focuses primarily on lipid residues in pottery, but combines the evidence with archaeobotanical, faunal, and isotopic data to test, across a range of different Indus settlements, what kinds of organic products were processed by inhabitants in quotidian settings. Crucially, in using these discrete set of data, this study was able to assess the wide range of commensal products and management strategies used by Indus populations on a site-specific and chronologically-diverse scales.

Overall, this thesis provides important methodological and archaeological insights. Firstly, the results suggest that ceramic lipid residue analysis is challenging to conduct in arid, hot and seasonally-wet environments, and that low lipid preservation creates a serious impediment to interpretation of data. This is an important finding for future researchers conducting biomolecular analyses in South Asia. Crucially, however, this does not mean that such analyses should not be undertaken in the region: rather, it is vital that we develop more methodological means to investigate organic remains in challenging environments. The obtained lipid and isotopic results also raise a number of important questions about the degree of confidence with which we can interpret stable isotopic values from fatty acids in lipid extracts. Secondly, the results provide a new means by which to investigate quotidian practices of inhabitants at South Asia's first urban civilisation. They provide new insights into different organic products processed at a diverse range of Indus settlements. Although some of the results are ambiguous and have equivocal interpretations, they raise crucial questions about the scale and extent of dairying in the Indus Civilisation; the likelihood of mixtures in vessels and complexities involved in resolving them; and the possibility of specific culinary preferences for certain types of foodstuff, for example, roasting of meat and boiling for vegetables/pulses and dairy. These are important findings and open up new

avenues for research into secondary product utilisation, foodways, and biomolecular approaches across ancient South Asia.

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Appendix A

Supplementary information and analyses from Stone Tower I, Salut

A.1. Supplementary site background

The emergence of Bronze Age cultures in southeastern Arabia in eastern Arabia (presentday United Arab Emirates and Sultanate of Oman) can be traced back to local Neolithic societies in the sixth millennium BC. These cultures developed extensive interaction networks across the Persian Gulf, beginning with interactions with Mesopotamia and south-eastern Iran, and extending to the Indus region by the third millennium BC (Edens 1992; Cleuzio and Tosi 2007; Magee 2014). Characteristic features of the Umm an-Nar period include settlements centred around oases focused on date-palm cultivation and other cultivars; stone and mud-brick architecture coupled with large stone towers with an as yet unclear function; thousands of monumental stone cairns; and the extensive development of metallurgy of copper and its alloys (Magee 2014; Frenez et al. 2016).

Excavations at Stone Tower I, Salut have revealed evidence of complex water management systems designed to support the inhabitants of the settlement in an arid, marginal environment. The 22m circular stone tower, like other Bronze Age tower in southeastern Arabia at this time, had a central stone-lined well within it, but no other structures were preserved on the tower (Frenez et al. 2016). The ditches and channels around the tower are hypothesised to be related to water management, storage and agricultural activities (Frenez et al. 2016). The later use of the main ditch contained a range of Indus pottery and local Umm an-Nar pottery, as well as Indus-inspired pottery that appears to have been produced locally (Frenez et al. 2016). Indus vessel forms found include Indus Black-Slipped Jars, ledged jars, globular jars, dish-on-stands and perforated vessels, and pottery with distinctive red slip and black-painted Indus motifs (Frenez et al. 2016). Other Indus materials such as terracotta figurines, carnelian beads, and Indus stamp seal and fragment of a pottery vessel with an Indus sealing (Frenez et al. 2016). The discover of a diverse range of utilitarian and high-value Indus objects at a site located far inland in Oman reveals that the nature of interaction between the Indus Civilisation and southeastern Arabia are possibly more complex than previously understood and require new models of investigation (Frenez et al. 2016).

A.2. GC-MS and GC-c-IRMS analyses of Arabian vessels and Indus Black-Slipped Jars

A.2.1. Lipid composition

The lipid profiles of all vessels from Stone Tower I, Salut do not appear compositionally different from one another except for differences in the quantities of specific fatty acids. All eleven fragments have very high concentrations of $C_{14:0}$, with ST101, ST1022 containing equal concentrations of $C_{14:0}$ and $C_{18:0}$ or higher concentrations of $C_{14:0}$ than any other fatty acid. ST102, ST1018, ST1078 and ST1079 contain higher concentrations of $C_{18:0}$ relative to $C_{16:0}$ and $C_{14:0}$, whereas ST1021 and ST1040 contain higher concentrations of $C_{16:0}$ relative to $C_{18:0}$ and $C_{14:0}$. Chromatograms also display little or no evidence of longchain fatty acids, odd-chain fatty acids, or dicarboxylic acids.

A.2.2. Compound-specific isotopic results

GC-c-IRMS analyses were conducted on 5 samples from ST1; on two Arabian vessels (ST1018 and ST1021), and three fragments of BSJ vessels (ST109, ST1074 and ST1079). $\delta^{13}C_{16:0}$ values from the local Arabian vessels were -26.2‰ and -23.7‰ and $\delta^{13}C_{18:0}$ values were -27.9‰ and -27.7‰ for ST1018 and ST1021 respectively. The $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values of the samples were -1.7‰ and -4‰ respectively. $\delta^{13}C_{16:0}$ values from samples from the BSJ fragments (n=3) ranged between -29.0‰ and -26.6‰, and $\delta^{13}C_{18:0}$ values ranged between -29.2‰ and -27.5‰. $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values of the samples ranged between -0.0‰ and -1‰.

Lipid

Table A.1. δ¹³C values from fatty acids from vessels from Stone Tower I, Salut

Figure A.1: $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values plotted against $\delta^{13}C_{16:0}$ values of local Arabian Umm an-Nar vessels and Indus Black-Slipped Jars (BSJs) from Stone Tower I, Salut. The shaded areas in-between dotted lines indicate the regions where dairy fats and ruminant adipose fats, and ruminant adipose and non-ruminant fats overlap respectively, indicating mixing of products.

A.2.3. Discussion

The lipid profiles from the examined vessels are intriguing. Examples with comparable proportions of $C_{14:0}$ within lipid extracts could not be found in the literature, however, lipids from the arid site of Qasr Ibrim in Egypt have revealed high concentrations of $C_{12:0}$ and $C_{14:0}$ that are have been linked to the processing of palm-fruit lipids in vessels (Copley et al. 2000). $C_{14:0}$ has also been linked to the presence of seed oils (Dunne et al. 2017). Thus, although high proportions of $C_{14:0}$ may be indicative of the remains of plant or fruit oils, this alone is not conclusive evidence for the processing of plant products in vessels. Additionally, there appears to be no characterisable difference between the lipid profiles of local Arabian vessels and BSJs.

The isotopic data presents a difference picture. Notably, the fatty acid-specific isotopic values of the BSJs cluster closely together, falling within the range of nonruminant products and mixtures of non-ruminant fats and ruminant adipose fats. These values could also be indicative of mixtures of dairy products and plant oils, as demonstrated by mixing models in Chapter 8. The local Orange Sandy Ware Umm an-Nar jar (ST1018) has a Δ^{13} C value that falls within the range for ruminant adipose products, whereas the local Umm an-Nar suspension vessel (ST1021) has a value consistent with

reference ruminant dairy products. $\delta^{13}C_{16:0}$ values of the lipid extracts suggest that the (likely indirect) plant input in the vessels comes primarily from C_3 plants, however, ST1021 demonstrates evidence of a mixed C_3 and C_4 plant input. Comparison with forthcoming archaebotanical and faunal data, and collection of reference fats from the region may reveal further insight into what products were processed in these vessels.

Appendix B

Details of analysed vessels per site

B.1. Kalibangan (KLB)

Table B.1: Details of all analysed vessels from Kalibangan.

B.2. Mohenjo-daro (MD)

Table B.2: Details of all analysed vessels from Mohenjo-daro.

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B.3. Alamgirpur (ALM)

Table B.3: Details of all analysed vessels from Alamgirpur. Vessel fragments marked with * had lipid concentrations lower than 5 μg/g and were excluded from analysis. Drawings courtesy Alessandro Ceccarelli.

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Table B.4: List of analysed samples from Alamgirpur with lipid concentrations <5 μg/g. Details of the vessel fragments, chronological period, lipid concentration, lipid composition, P/S ratio and $\delta^{13}C$ values of the C_{18:0}, and C_{16:0} fatty acids for every sample are provided. FAs: saturated fatty acids, UFAs: unsaturated fatty acids, Br: branched-chain fatty acids, Diacids: dicarboxylic acids. Fatty acids in bold reflect the compound with the highest abundance within the lipid abstract.

B.4. Masudpur VII (MSDVII)

Table B.5: Details of all analysed vessels from Masudpur VII. Vessel fragments marked with * had lipid concentrations lower than 5 μg/g and were excluded from analysis. Drawings and photos with blue blackground courtesy Danika Parikh.

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Table B.6. List of analysed samples from Masudpur VII with lipid concentrations <5 μg/g. Details of the vessel fragments, chronological period, lipid concentration, lipid composition, P/S ratio and $\delta^{13}C$ values of the C_{18:0}, anission to scientific reportsd C_{16:0} fatty acids for every sample are provided. FAs: saturated fatty acids, UFAs: unsaturated fatty acids, Br: branched-chain fatty acids, Diacids: dicarboxylic acids. Fatty acids in bold reflect the compound with the highest abundance within the lipid abstract

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B.5. Masudpur I (MSDI)

Table B.7: Details of all analysed vessels from Masudpur VII. Vessel fragments marked with * had lipid concentrations lower than 5 μg/g and were excluded from analysis. Drawings courtesy Danika Parikh.

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Table B.8: List of analysed samples from Masudpur I with lipid concentrations <5 μg/g. Details of the vessel fragments, chronological period, lipid concentration, lipid composition, P/S ratio and $\delta^{13}C$ values of the C_{18:0}, and C_{16:0} fatty acids for every sample are provided. FAs: saturated fatty acids, UFAs: unsaturated fatty acids, Br: branched-chain fatty acids, Diacids: dicarboxylic acids. Fatty acids in bold reflect the compound with the highest abundance within the lipid abstract.

B.6. Lohari Ragho I (LHRI)

Table B.9: Details of all analysed vessels from Lohari Ragho I. Vessel fragments marked with * had lipid concentrations lower than 5 μg/g and were excluded from analysis. Photos and drawings are in the process of being finalised.

Table B.10: List of analysed samples from Lohari Ragho I with lipid concentrations <5 μg/g. Details of the vessel fragments, chronological period, lipid concentration, lipid composition, P/S ratio and $\delta^{13}C$ values of the C_{18:0}, and C_{16:0} fatty acids for every sample are provided. FAs: saturated fatty acids, UFAs: unsaturated fatty acids, Br: branched-chain fatty acids, Diacids: dicarboxylic acids. Fatty acids in bold reflect the compound with the highest abundance within the lipid abstract

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B.7. Khanak (KNK)

Table B.11: Details of all analysed vessels from Khanak. Vessel fragments marked with * had lipid concentrations lower than 5 μg/g and were excluded from analysis

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Table B.12: List of analysed samples from Khanak with lipid concentrations <5 μg/g. Details of the vessel fragments, chronological period, lipid concentration, lipid composition, P/S ratio and $\delta^{13}C$ values of the C_{18:0}, and C_{16:0} fatty acids for every sample are provided. FAs: saturated fatty acids, UFAs: unsaturated fatty acids, Br: branched-chain fatty acids, Diacids: dicarboxylic acids. Fatty acids in bold reflect the compound with the highest abundance within the lipid abstract.

B.8. Farmana (FRN)

Table B.13: Details of all analysed vessels from Farmana. Vessel fragments marked with * had lipid concentrations lower than 5 μg/g and were excluded from analysis

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Table B.14: List of analysed samples from Farmana with lipid concentrations <5 μg/g. Details of the vessel fragments, chronological period, lipid concentration, lipid composition, P/S ratio and $\delta^{13}C$ values of the C_{18:0}, and C_{16:0} fatty acids for every sample are provided. FAs: saturated fatty acids, UFAs: unsaturated fatty acids, Br: branched-chain fatty acids, Diacids: dicarboxylic acids. Fatty acids in bold reflect the compound with the highest abundance within the lipid abstract

B.9. Rakhigarhi (RGR)

Table B.15: Details of all analysed vessels from Rakhigarhi. Vessel fragments marked with * had lipid concentrations lower than 5 μg/g and were excluded from analysis

Table B.16: List of analysed samples from Rakhigarhi with lipid concentrations <5 μg/g. Details of the vessel fragments, chronological period, lipid concentration, lipid composition, P/S ratio and $\delta^{13}C$ values of the C_{18:0}, and C_{16:0} fatty acids for every sample are provided. FAs: saturated fatty acids, UFAs: unsaturated fatty acids, Br: branched-chain fatty acids, Diacids: dicarboxylic acids. Fatty acids in bold reflect the compound with the highest abundance within the lipid abstract

B.10. Stone Tower I, Salut (STI)

Table B.17: Details of all analysed vessels from Stone Tower I, Salut.

Appendix C

Protocol: Lipid extraction with acidified methanol

Bioarch, University of York

PRINCIPLE:

Acid extraction of lipid residues from pottery sherds.

SAMPLE TYPE:

Ceramic powder from pottery sherds

CAUTION:

Sulphuric acid and Methanol are toxic, use fume extraction at all times. Wear eye protection, laboratory coat and gloves at all times when using sulfuric acid. Sulfuric acid will degrade gloves over time, always monitor the condition of your gloves if splashing occurs. All users of the Nitrogen blow down must be trained to use the gas cylinder and blow down equipment before use.

MATERIALS REQUIRED:

Aluminium foil, Hach tubes, scintillation vials, auto-sample vials, Methanol (HPLC grade), DCM (HPLC grade), Sulphuric acid, Hexane (HPLC grade), C16/C18 fatty acid standard, C36 alkane standard, Pasteur pipettes, sterile glass wear, potassium carbonate (extracted 3x with DCM and baked at 350°C), glass wool (extracted 3x with DCM).

1.0 PREPARATION PROCEDURES:

- 1.1 Make sure all glassware and tools are solvent rinsed (3x rinsing in DCM) between samples, or sterile.
- 1.2 No more than 20 samples to be processed in one batch (18 samples $+ 1 C_{16}/C_{18}$ STD + 1 method blank).

2.0 LABELLING:

2.1 Label both vial and lid with unique sherd identifier followed by I for interior surface or E for exterior surface.

3.0 SAMPLE RETRIEVAL:

- 3.1 Drill if possible at least 1g of sherd from the interior/exterior surface using a modelling drill with a tungsten carbide bit.
- 3.2 Drill to a depth of 2 to 4mm.
- 3.3 Collect sherd powder on aluminium foil and transfer to labelled Hach tube.

4.0 ACID EXTRACTION:

- 4.1 Accurately weigh about 1g sherd powder into a clean, labelled Hach tube, leaving a portion of the sherd powder as a reserve sample if possible. Reserve sample should be stored in freezer at −20°C.
- 4.2 Using syringe add 100µl of isotopically measured C_{16}/C_{18} fatty acid standard to a clean, labelled Hach tube and evaporate under nitrogen to dryness.4.3 Using Pasteur pipette add approximately 4ml of MeOH to pottery samples $+ C_{16}/C_{18}$ standard + method blank.
- 4.4 Sonicate for 15 minutes.
- 4.4 Using Pasteur pipette add 800μl of pure sulphuric acid (under fumehood, wearing eye

protection).

- 4.5 Heat at 70ºC for 4 hours on the heating block.
- 4.6 Prepare a Pasteur pipette by sample packing glass wool enough to plug the pipette and

 adding cleaned potassium carbonate (about 5 mm). Clean it passing 1-2ml of DCM through

it.

- 4.7 Centrifuge the samples at 3000rpm for 5 minutes and carefully pipette off the liquid extract into a clean, labelled Hach tube.
- 4.8 Add 2ml hexane and use vortex to mix.
- 4.9 Allow the hexane layer (top layer) to separate out and pipette off carefully through the prepared Pasteur pipette pack with potassium carbonate into a clean, labelled Hach tube.
- 4.10 Repeat steps 4.8 and 4.9 twice more, combining the extracts.
- 4.11 Add 1ml of hexane through the pipette.
- 4.12 Evaporate to dryness under a very gentle stream of nitrogen with gentle warmth.
- 4.13 Add 1ml of hexane, mix with vortex and transfer to a clean hydrolysis vial.
- 4.14 Repeat the previous step combining in the same vial and vortex.
- 4.15 Evaporate very gently to dryness.
- 4.16 Store extracts in a freezer at −20°C until required for analysis.

BEFORE ANALYSIS:

4.16 Using syringe Add 90ul of Hexane to re-suspend the sample, roll the vial in order to make sure the whole extract is suspended (including the neck).

4.17 Add 10µl of the C36 alkane standard $(1\mu g.\mu l^{-1})$ to a clean, labelled auto-sampling vial with 0.1ml conical insert.

- 4.18 Transfer the 90µl of hexane + extract to the auto-sampling vial using Pasteur pipette or syringe. If using syringe clean the needle 10 times with hexane between each sample.
- 4.19 Analyse by GC/GC-MS and/or store in a refrigerator at 4°C (short-term) or in a freezer at −20°C (long term).

5.0 BLANKS:

5.1 For every run a method blank should be included.

5.2 GC/GC-MS analysis of blanks will provide a measure of contamination introduced during the extraction of organic residues from sherds.

Appendix D

Protocol: Lipid extraction with solvent method

Bioarch, University of York

PRINCIPLE:

Solvent extraction of lipid residues from pottery sherds.

SAMPLE TYPE:

Pottery sherds, ceramics.

CAUTION:

DCM and Methanol are toxic; use fume extraction at all times. Wear eye protection, laboratory coat and gloves at all times when using DCM. DCM will degrade gloves over time, always monitor the condition of your gloves if splashing occurs. All users of the Nitrogen blow down must be trained to use the gas cylinder and blow down equipment before use.

MATERIALS REQUIRED:

Aluminium foil, scintillation vials, Dichloromethane (HPLC grade), Hexane (HPLC grade), C34 alkane standard, Methanol (HPLC grade), Pasteur pipettes, sterile glass wear.

PROCEDURE:

All members of BioArCh wishing to use the GC in this way are responsible for ensuring that the procedures detailed in the SOP are followed when carrying out solvent extraction of organic residues from pottery sherds.

PREPARATION PROCEDURES:

1.1 Make sure all glassware and tools are solvent rinsed (3x rinsing in DCM) between samples, or sterile.

1.2 No more than twelve samples to be processed in one batch (11 samples + 1 method blank/10 samples $+ 1$ method blank $+ 1$ pottery blank).

2.0 LABELLING:

2.1 Label both vial and lid with unique sherd identifier followed by I for interior surface or E for exterior surface.

3.0 SAMPLE RETRIEVAL:

3.1 Drill if possible at least 1g of sherd from the interior surface using a modelling drill with a tungsten carbide bit.

3.2 Drill to a depth of 2-4mm.

3.3 Collect sherd powder on aluminium foil and transfer to labelled scintillation vial.

4.0 SOLVENT EXTRACTION:

4.1 Accurately weigh about 1g sherd powder into a clean, labelled scintillation vial, leaving a portion of the sherd powder as a reserve sample if possible. Reserve sample should be stored in freezer at −20°C. Add 10-100ul 1ul/g of the C34 alkane standard to the powder.

4.2 Add approximately 5ml of DCM:MEOH 2/1 v/v.

4.3 Sonicate for 15 minutes at 25ºC.

4.4 Centrifuge at 3000rpm for 10 minutes.

4.5 Carefully pipette off the liquid extract into a clean, labelled (TLE) scintillation vial.

4.6 Repeat steps 4.2 to 4.5 twice more, combining the extracts.

4.7 Reduce volume of extracts to about 2ml under a stream of nitrogen with gentle heat.

4.8 Transfer to a clean, labelled, small vial and continue to evaporate under nitrogen to dryness.

4.9 Store in a refrigerator at 4°C (short-term) or in a freezer at −20°C (long term).

5.0 EXTERIOR SURFACES:

5.1 For the extracting the residue from the exterior surface of the sherd repeat steps 1.0 to 4.0.

6.0 BLANKS:

6.1 For every run a method blank should be included. Eleven samples may be processed with a method blank.

6.2 GC/GC-MS analysis of blanks will provide a measure of contamination introduced during the extraction of organic residues from sherds.
Appendix E

Protocol: Silylation of organic residues fo GC/GC-MS

Bioarch, University of York

PRINCIPAL:

The derivatisation of organic residues of archaeological origin by silylation.

SAMPLE TYPE:

Pottery sherds, ceramics.

CAUTION:

DCM and Methanol are toxic; use fume extraction at all times. Wear eye protection, laboratory coat and gloves at all times when using DCM. DCM will degrade gloves over time, always monitor the condition of your gloves if splashing occurs. All users of the Nitrogen blow down must be trained to use the gas cylinder and blow down equipment before use.

MATERIALS REQUIRED:

Aluminium foil, C36 alkane standard, scintillation vials, Dichloromethane (HPLC grade), Hexane (HPLC grade), Methanol (HPLC grade), Pasteur pipettes, sterile glass wear.

1.0 PRE-PREPARATION PROCEDURES:

1.1 Make sure all glassware and tools are solvent rinsed (3x rinsing in DCM) or sterilised. 1.2 Samples should be processed in batches of no more than twelve (11 samples + 1 method blank/10 samples $+ 1$ method blank $+ 1$ pottery blank).

2.0 LABELLING:

2.1 Make sure that labels are still legible on both vials and lids before starting this procedure. Residues for silylation will be extracts A, B2 or C and should still be identified by a unique sherd identifier, a letter indicating the origin of the residue and one of the letters above.

3.0 SILYLATION PROCEDURE:

3.1 Add 50ul of Hexane to re-suspend the sample, roll the vial in order to make sure the whole extract is suspended (including the neck), add four drops of *N,O*-bis(trimethylsilyl) trifluoroacetamide (BSTFA) with 1% trimethyl-chlorosilane (TMCS) to each residue using a sterile Pasteur pipette. Alternatively add 100μl BSTFA using a micro-syringe. If you touch the needle onto the sample vial during this stage, dispose of the BSTFA and clean 10 times with hexane.

3.2 Heat at 70ºC for 60 minutes on the heating block.

3.3 Evaporate off excess BSTFA under nitrogen with gentle heat.

3.4 Add 10ul of the C36 alkane recovery standard to a clean auto-sampling vial with 0.1ml conical insert, and 50ul of hexane to the sample vial, again rolling the vial to re-suspend the material. Transfer the 50ul of hexane + extract to the auto-sampling vial. If you touch the needle onto the sample vial during this stage, clean the needle times with hexane before moving to the next sample.

3.5 Analyse by GC/GC-MS within 48 hours. If analysis cannot be performed within that time, repeat steps 3.1 to 3.3.

4.0 BLANKS:

4.1 Blanks from the extraction of residues should be silylated with the same batch of residues.

4.2 GC/GC-MS analysis of blanks will test for contamination introduced during the preparation of samples.

Appendix F

GC-MS and GC-c-IRMS analyses from Kalibangan and Mohenjo-daro (Group I)

F.1. Lipid composition

The lipid extracts from 3 out of 5 sherds from Kalibangan (KLB01, KLB02, KLB05) comprised of a range of medium- and long-chain fatty acids from $C_{12:0}$ to $C_{24:0}$, with high concentrations of $C_{16:0}$ and $C_{18:0}$. Two samples had unusual lipid profiles. KLB03 contained small quantities of short-chain fatty acids ranging from $C_{8:0} - C_{10:0}$, maximising at $C_{9:0}$, along with relatively higher concentrations of $C_{16:0}$. KLB04, which had high lipid concentrations, consisted of α -ω dicarboxylic acids ranging from C_7 to C_9 , maximising at C_9 (azelaic acid), which was more abundant than the peaks for $C_{16:0}$ and $C_{18:0}$ fatty acids. All extracts contained unsaturated fatty acids $C_{16:1}$ and $C_{18:1}$, odd-chain fatty acids $C_{15:0}$ and $C_{17:0}$ and branched-chain fatty acids (C_{15Br} and C_{17Br} , iso and ante-iso). Lipid extracts from Mohenjo-daro contained medium- and long-chain fatty acids from C12:0 to $C_{26:0}$, with higher concentrations of $C_{16:0}$ relative to $C_{18:0}$. Two vessels from Mohenjodaro (MD02 and MD05) contained high concentrations of short-chain carboxylic acids ranging from $C_{6:0} - C_{9:0}$, maximising at $C_{9:0}$. Less abundant concentrations of short-chain fatty acids were also present in MD03. All extracts contained unsaturated fatty acids $C_{16:1}$ and $C_{18:1}$, odd-chain fatty acids $C_{15:0}$ and $C_{17:0}$ and branched-chain fatty acids (C_{15Br} and C_{17Br} , iso and ante-iso).

F.2. Compound-specific isotopic data

Eight out of ten samples were analysed via GC-c-IRMS (Figure I.3). These included all five samples from Kalibangan and three samples from Mohenjo-daro (Table F.1). $\delta^{13}C_{16:0}$ values from samples from Kalibangan had a mean value of -25.5‰, and $\delta^{13}C_{18:0}$ values had a mean value of -27.2‰. The $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values of the samples ranged between -3.6‰ and -1.6‰. $\delta^{13}C_{16:0}$ values from samples from Mohenjo-daro had a mean value of -28.2‰, and $\delta^{13}C_{18:0}$ values had a mean value of -29.1‰. The $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values of the samples ranged between -0.1‰ and -0.3‰.

S. No	Sample No.	Vessel Type	Rim/ Base/	Lipid concentration	$\delta^{13}C$	$\delta^{13}C$	$\varDelta^{13}C$ $(C_{18:0}$ -
			Body	$(\mu g/g)$	$C_{16:0}$	$C_{18:0}$	$C_{16:0}$
	KLB01	Jar	Rim	68.4			
2	KLB ₀₂	Perforated jar	Rim	178	-26.8	-27.7	-1
					-25.9	-27.5	-1.6
3	KLB03	Jar	Rim	3514.2	-25.6	-26.4	-0.8
$\overline{4}$	KLB04	Jar	Rim	290.4	-27.3	-28.8	-1.5
5	KLB ₀₅	Dish	Rim	64.1	-22.2	-25.8	-3.7
6	MD01	Perforated jar	Body	82.1	-28.7	-29.2	-0.4
$\overline{7}$	MD02	Small pot	Rim	41.4	-26.6	-27.5	-0.9
8	MD05	Unknown	Body	36.5	-28	-29.1	-1.2

Table F.1. δ^{13} C values from fatty acids from vessels from Kalibangan and Mohenjo-daro.

Figure F.1: $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values plotted against $\delta^{13}C_{16:0}$ values of vessels from Kalibangan. The shaded areas in-between dotted lines indicate the regions where dairy fats and ruminant adipose fats, and ruminant adipose and non-ruminant fats overlap respectively, indicating mixing of products.

Figure F.2: $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values plotted against $\delta^{13}C_{16:0}$ values of vessels from Mohenjodaro. The shaded areas in-between dotted lines indicate the regions where dairy fats and ruminant adipose fats, and ruminant adipose and non-ruminant fats overlap respectively, indicating mixing of products.

Figure F.3: Δ 13C (δ ¹³C_{18:0} - δ ¹³C_{16:0}) values plotted against δ ¹³C_{16:0} values of vessels from Group I. The shaded areas in-between dotted lines indicate the regions where dairy fats and ruminant adipose fats, and ruminant adipose and non-ruminant fats overlap respectively, indicating mixing of products.

F.3. Discussion

The obtained lipid profiles from Kalibangan and Mohenjo-daro are ambiguous. The presence of short-chain fatty acids in KLB04, MD02 and MD05 is interesting as shortchain fatty acids rarely survive in archaeological contexts and are found in dairy fats, fruits and flowers (Craig 2005; Rageot 2019). In this context, however, it is unclear what their origin might be. Additionally, the high concentration of dicarboxylic acids in KLB04, MD02 and MD05 is interesting. As is well-known, the predominance of azelaic acid implies the precursor fatty acid bare a double-bond at the 9-position, suggesting the vessel once contained unsaturated fatty acids that are common in plants (Regert et al. 1998; Regert 2011). However, the absence of plant-derived 'biomarkers' or other compounds characteristic of aquatic or marine products make it difficult to be certain of the origins of the residue within these vessels. Overall, the lipid profiles from Kalibangan and Mohenjodaro were indicative of degraded animal fats, except for KLB04, MD02 and MD05, which may have also contained plant-derived products.

The compound-specific isotopic data from fatty acids within extracts from Kalibangan and Mohenjo-daro are clustered; with a difference of 4‰ between $\delta^{13}C_{16:0}$ values and 3‰ between $\delta^{13}C_{18:0}$ values from Kalibangan, and just 1‰ for both $\delta^{13}C_{16:0}$ and $\delta^{13}C_{18:0}$ values for samples from Mohenjo-daro. The range in the values from vessels from Kalibangan can be attributed to a single outlier, KLB05, a medium-sized bowl, which demonstrates evidence for dairy processing based on its Δ^{13} C value (-3.6‰) and evidence of a relatively higher input of C_4 plant based on its $\delta^{13}C_{16:0}$ value (-22.1‰). Notably, this vessel's lipid extracts did not contain short-chain fatty acids, which are generally associated with milk fats. The rest of the vessels from Kalibangan have Δ^{13} C values that fall within the reference range for ruminant animal adipose fats, with $\delta^{13}C_{16:0}$ values indicating mixed C₃- C_4 plant input. Two samples from Mohenjo-daro (MD02 and MD05) have $\Delta^{13}C$ values that fall within the range for ruminant adipose fats, however one sample (MD01, a perforated vessel) has a Δ^{13} C value that suggests it was used to process mixtures of non-ruminant and ruminant adipose fats. The $\delta^{13}C_{16:0}$ values of the samples suggest a mixed input of both C₃ and C⁴ plants. In the absence of faunal data or solid archaeobotanical evidence from both sites, however, the interpretation of these values is limited. Thus, preliminary interpretations suggest that ruminant and non-ruminant products were dominantly processed in the vessels from Kalibangan and Mohenjo-daro, with evidence for dairyprocessing in a single vessel from Kalibangan (KLB05, a medium-sized bowl). Notably, the perforated vessels from both Mohenjo-daro and Kalibangan do not fall within the range for dairy fats.

These results are comparable with those found from other sites in northwest India in this thesis. It is possible the obtained isotopic results are also indicative of mixtures in vessels (Chapter 8). Unfortunately, the lack of palaecological, archaebotanical and faunal data from these sites makes interpretations challenging.

There are other problems associated with the interpretation of these lipid profiles and compound-specific isotopic results. High concentrations of synthetic compounds within the samples increase the likelihood of the coelution of peaks, making the identification of peaks difficult. Furthermore, the uncertain collection history of these potsherds also renders them unreliable, as their context is unknown, and so are the details of how they were processed, or if they were exposed to any other organic compounds after they removed from the site. Thus, the organic compounds and their respective fatty acid specific isotopic composition may not be products of the use of the vessels in antiquity.

Appendix G

Vessel-specific GC-c-IRMS results

Results from the ranges and average concentrations of lipid yield presented in Chapter Six suggest that certain vessels may have been used more frequently, or for the processing of fatty- or oil-rich products. This section will discuss the results from the chemical and fatty acid-specific isotopic analyses obtained for different vessel forms. In combination with lipid yields, these reveal insights about the purported use of vessels in antiquity.

G.1. Small jars

Small and very small jars (ranging between 5-12cm rim diameters) with lipid quantities above 5 µg/g made up 19.7% (n=27) of the total analysed assemblage. Lipid concentrations averaged 25 μg/g, ranging between 6.1 μg/g to 131 μg/g.

Molecular evidence revealed that small jars contained medium and long-chain fatty acids such as $C_{12:0}$ up to $C_{26:0}$, with high concentrations of $C_{16:0}$ and $C_{18:0}$ fatty acids relative to other compounds. These compounds are generic to both plant and animal products; and due to the lack of characteristic plant-derived compounds in the vessels, the lipid profiles suggest that they are signatures of degraded animal fats.

Figure G.1: $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values plotted against $\delta^{13}C_{16:0}$ values of small jars across Indus sites in northwest India

Eighteen vessels were selected for GC-C-IRMS analysis. The δ^{13} C values of the fatty acids within the vessels demonstrate a wide range, suggesting they were used to process a diverse number of products (Figure I.1). These include Δ^{13} C values that fall within the global references for non-ruminant fats, ruminant fats, dairy fats, their mixtures, and possible mixtures of plant and dairy products. The $\delta^{13}C$ values for C_{16:0} fatty acids also suggest variable direct or indirect contribution of C4 plants into the vessels. Figure G.1 also demonstrates there is no clear correlation between site and the δ^{13} C values of fatty acids from the residues of vessels, suggesting that small jars had varied functions within a single settlement. However, it is possible that their small mouth size and limited volumetric capacity would have restricted their use for food-processing, and rather enabled the storage of oils or other condiments.

G.2. Medium-mouthed jars

Medium-mouthed jars were classified as jars that had rim diameters ranging between 13- 17cm. Although it is not possible to predict the volume of the vessels based on the rim diameters; this measurement gave an idea of the size of the mouth of the vessel, which may be indicative of the vessel's role in food processing.

Nine vessels were excluded from analysis due to low lipid concentrations. The remaining 18 vessels vessel made up 14.4% of the analysed assemblage. Lipid concentrations ranged between 5 μ g/g and 79 μ g/g, averaging 23.4 μ g/g.

Like other vessels, the molecular evidence revealed that medium-mouthed jars contained fatty acids ranging from $C_{12:0}$ up to $C_{26:0}$, with high concentrations of $C_{16:0}$ and $C_{18:0}$ fatty acids relative to other compounds. As mentioned previously, these compounds are generic to both plant and animal products, however the lack of characteristic plantderived compounds in the vessels suggest that they are more likely derived from degraded animal fats.

Figure G.2: $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values against $\delta^{13}C_{16:0}$ values of medium-mouthed jars across Indus sites in northwest India

Eight out of eighteen medium-mouthed jars (44.4%) contained relatively higher lipid concentrations, which made them suitable for GC-c-IRMS analysis. As Figure G.2 indicates, all but one of the analysed vessels fall within the established non-ruminant fat range, which, as discussed above, is challenging to interpret within the Indus context. Only a single vessel from FRN (FRN09, a red-slipped black-painted jar) falls within ranges indicative of the mixing of ruminant and non-ruminant adipose products. The vessels also

show a mixed input of C_3 and C_4 -plant sources, with no correlation between the site location and the likely use of the vessel or source of the vessel residue.

G.3. Large jars (storage jars or vats)

Large jars were classified as jars that had rim diameters from 18-30 cm. Although it is not possible to predict the volume of the vessels based on the rim diameters; rim sizes above 18 cm would only be supported by large vessels. These vessels possibly functioned as storage vessels or large food-processing vessels. A total of 20 large jars were analysed. Out of these, 11 vessels (7.6% of the analysed assemblage) contained above 5 μg/g of lipid. Lipid concentrations ranged between 5.3 μg/g to 57.7 μg/g, averaging 16.8 μg/g.

GC-MS results from large jars indicated the presence of fatty acids ranging from $C_{12:0}$ up to $C_{26:0}$, with high concentrations of $C_{16:0}$ and $C_{18:0}$ fatty acids relative to other compounds, which were indicative of degraded animal fats.

Fig G.3: $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values against $\delta^{13}C_{16:0}$ values of large jars across Indus sites in northwest India

Seven of the ten large jars were selected for compound-specific isotopic analysis based on their lipid concentrations. Five of the jars (ALM114-252, FRN14, MSD228, MSD1602 and MSD2209, from Alamgirpur, Farmana, Masudpur I and Masudpur VII respectively) fall within the established range for non-ruminant adipose fats with evidence of predominantly C_3 plant input, whereas two (LHR11, a red-slipped large jar, and LHR40 a chocolate-slipped large jar from Lohari Ragho I) fall within the range for ruminant adipose fats, demonstrating a higher input of C_4 plant within them, possibly indicating the processing of cattle/buffalo fats.

G.4. Jars

Certain vessels within the analysed assemblage had rim diameters that could not be determined due to the state of preservation of the pottery fragment, however, their form could still be ascertained. Thus, these vessel fragments were simply classified as 'jars', and further inferences about the size of the vessel was not possible.

Out of 25 jars, 23 contained above 5 μ g/g of lipid, making up 16% of the analysed assemblage. Lipid concentrations ranged between 5.5 μg/g to 214.7 μg/g, with an average of 29.8 μg/g.

Lipid profiles of jars were similar to those of other vessels, containing a range of medium- and long-chain fatty acids, maximising at saturated C16:0 and C18:0 fatty acids. These profiles are not diagnostic and are likely characteristic of degraded animal fats. A single vessel stood out as an exception. LHR10 from Lohari Raho I, a red-slipped jar possibly produced at an urban centre, contained short chain fatty acids from C9:0 and very long-chain fatty acids up to $C_{28:0}$. Short-chain fatty acids are found in dairy products but are prone to degradation are rarely survive in archaeological contexts. Very long-chain fatty acids are derived from plant remains but may also indicate contamination from sediments. Thus, compound-specific isotopic analysis was required to further determine the potential source of the lipid residues.

Figure G.4: $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values against $\delta^{13}C_{16:0}$ values of jars (unknown rim diameter) across Indus sites in northwest India

Twelve out of eighteen vessels were selected for GC-c-IRMS analysis. As Figure G.4 demonstrates, the δ^{13} C values of the fatty acids from jars are variable and have a wide range; five of the vessels have Δ^{13} C values that fall within the accepted range of nonruminant fats, four vessels fall between the ranges for non-ruminant and ruminant meat products, two vessels fall within the range for ruminant adipose products, and one vessel falls between the ranges for ruminant adipose and dairy products. The $\delta^{13}C_{16:0}$ values for the jars are also variable, indicating mixed input of C_3 and C_4 plants into the vessels, with two jars demonstrating a higher input of C_4 plants than the others. One of the vessels is LHR10, which has relatively positive $\delta^{13}C_{16:0}$ values relative to other vessels as well as a unique molecular profile. The fatty acid-specific δ^{13} C values from the vessel suggest it was used for processing ruminant adipose products, more specifically cattle/buffalo carcass fats, as suggested by high C_4 plant input. The other vessel is KNK01, which falls within the range for ruminant adipose fats but lies close to the threshold of dairy fats, which may suggest the mixing of ruminant and dairy products in the vessel from both sheep/goat and cattle/buffalo.

G.5. Necked jars

Eighteen necked jars of different rim-sizes were analysed but grouped into a single category. Out of the 18 vessels analysed, two large-necked jars had very low lipid

concentrations (below 5 μ g/g) and were excluded from analyses. Of the 16 vessels remaining, six vessels were small necked jars (with rim diameters between 8-12cm), seven vessels had medium-sized mouths (with rim diameters between 13-17cm), one vessel was a large necked jar (rim diameter of 20cm) and one jar had an undeterminable rim diameter due to incomplete preservation of the rim. In total, these vessels made up 11.1% of the analysed assemblage. The shape of these vessels likely constricted cooking or food processing as the neck is generally smaller than the rim; however, they were possibly used for the pouring or storing of liquid-based foodstuff.

The average lipid concentration of the vessels was 29.6 μg/g, with small necked jars averaging 15.8 μg/g of lipid and medium-mouthed necked jars averaging 44 μg/g of lipid. A medium-mouthed (rim diameter = 15cm) necked jar from Masudpur VII contained 214 μ g/g, which was the highest concentration of lipid obtained from these vessels.

The lipid profiles of analysed necked jars included medium- and long-chain fatty acids, with higher concentrations of $C_{16:0}$ and $C_{18:0}$ fatty acids. Since these molecular profiles are largely undiagnostic except for the fact that they suggest that vessels were used to process animal fats, GC-c-IRMS analysis was conducted.

Figure G.5: $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values against - $\delta^{13}C_{16:0}$ values of necked jars across Indus sites in northwest India

Eleven out of sixteen necked jars contained enough lipid suitable for compoundspecific isotopic analysis. As Figure G.5 indicates, the δ^{13} C values of the fatty acids fall within the range for non-ruminant adipose products, except for a single outlier, MSD329, a medium-mouthed, wheel-finished, red-slipped necked jar from Masudpur I, which falls within the range for dairy fats. The $\delta^{13}C_{16:0}$ values indicate that C_3 plant input in the vessels was predominant; however, two vessels (LHR13 from Lohari Ragho I and MSD3412 from Masudpur VII) demonstrate both C_3 and C_4 plant-input, whereas MSD329 has highly enriched $\delta^{13}C_{16:0}$ values, suggesting a very high C₄ plant input. It is likely that MSD329 was predominantly used to process dairy products from cattle/buffalo milk, however, the other results are not straightforward to interpret. It is possible they were used to process non-ruminant products, plant products, or mixtures of plant and dairy products.

G.6. Globular jars

Only three examples of globular jars, all from Lohari Ragho I, were analysed. Their classification as globular jars was possible due to their semi-complete preservation. The average lipid concentration of globular jars was 28.34 μg/g, ranging between 5.8 μg/g and 44.6 μg/g.

The lipid profiles of the globular jars were characteristic of degraded animal fats, with medium- and long-chain fatty acids between $C_{12:0}$ - $C_{24:0}$. These included odd- and branched-chain fatty acids such as $C_{15:0}$, C_{15Br} , $C_{17:0}$ and C_{17Br} . GC-c-IRMS analysis was conducted on two vessels (LHR03 and LHR07) with relatively higher lipid concentrations to determine the potential source(s) of the fat input into the vessels.

Figure G.6: $\Delta^{13}C$ (δ¹³C_{18:0} - δ¹³C_{16:0}) values against δ¹³C_{16:0} values of globular jars from Loharo Ragho I.

The fatty acid-specific $\delta^{13}C$ values for the two analysed globular jars fall within the range for non-ruminant adipose products, with $\delta^{13}C_{16:0}$ values indicating higher input of C₃ plants. It is possible these vessels were used to store or process lard or animal fats, however, as mixing plots suggest, these δ^{13} C values may also represent mixtures of products in vessels.

G.7. Serving vessels: bowls and dishes

Eight bowls and six dishes comprised of the vessels that likely had a serving function or were used to display foodstuff rather than be used for food preparation. Of these, only four bowls, four dishes and the goblet fragment had lipid yields above 5 μg/g: FRN25, a medium-sized, red-slipped, black-painted bowl from Farmana, MSD2209 and MSD3392, large- and medium-sized bowls from Masudpur VII. Lipid concentrations of bowls averaged 10.3 μg/g. The three dishes (ALM119-363, a medium-sized dish, ALM125-494, a large dish, and RGR16, a medium-sized, red-slipped, black-painted dish) also had low lipid concentrations, ranging from 6.6 μg/g to 9.5 μg/g, averaging 8.3 μg/g.

GC-MS analysis demonstrated that the lipid profiles of bowls and dishes were similar to those of other vessels, containing even medium-chain fatty acids such as $C_{14:0}$, $C_{16:0}$, and $C_{18:0}$, and low quantities of long-chain fatty acids such as $C_{20:0}$, $C_{22:0}$, and $C_{24:0}$, indicating the presence of degraded animal fats.

Figure G.7: $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) value against $\delta^{13}C_{16:0}$ value of a dish from Alamgirpur.

Only a single dish from Alamgirpur (ALM125-494) had sufficient lipid concentrations to be analysed for GC-c-IRMS. As Figure G.7 indicates, the fatty acidspecific δ^{13} C values for the dish fall within the range for non-ruminant products, with $\delta^{13}C_{16:0}$ values indicating higher input of C₃ plants. This suggests the dish was used to serve or display other non-ruminant animal fats, however, as mixing plots suggest, these δ^{13} C values may also represent mixtures of products.

Appendix H

Details and lipid yields of vessels excluded from analysis

Table H.1: Details and lipid concentrations and composition of vessels excluded from analysis.

