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# CUISINE IN TRANSITION

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Organic residue analysis of domestic containers from 9th-14th century Sicily

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*“ Now, again; the pleasures of this world are divided into six classes. They are food, drink, clothing, sex, scent, and sound. The most eminent and perfect of these is food; for food is the foundation of the body and the material of life.”* - Muhammad bin al-Hasan bin Muhammad bin al-Karīm al-Baghdadi (Translated from A Baghdad Cookery Book by Perry 2005, 1)

## Abstract

From the 9<sup>th</sup> to the 14<sup>th</sup> century AD, Sicily experienced a number of rapid and quite radical changes in regimes. From the Aghlabid and Kalbid dynasties in the 9<sup>th</sup>-11<sup>th</sup> century, then ruled by the Normans in the 12<sup>th</sup> century and then Swabians in the 13<sup>th</sup> and finally ruled by the crown of Aragon in the 14<sup>th</sup> century. This thesis used the concept of cuisine, the ways in which foods are prepared combined and consumed, as a proxy for understanding the lives of the general population of Sicily during these regimes. A multifaceted organic residue analysis approach was applied to over 240 domestic containers from different sites in western Sicily to **1)** understand what foods were selected, combined, and processed in ceramic vessels and **2)** how the contents of these ceramics compare in urban and rural socio-economic settings and between Islamic, Norman, Swabian and Aragonese contexts. Through a large-scale, multifaceted organic residue approach, a range of commodities were identified in the ceramic vessels including animal fats, vegetable products, fruit products, beeswax, and plant resins. In many cases a mixture of commodities were observed, reflecting both sequential cooking events and/or the complex mixtures reflective of medieval recipes. The identification and combination of similar products demonstrated shared culinary habits amongst populations in different sites and chronological periods. However, some discrete differences in pottery use has shed new light on the patterning of resource use between the sites studied particularly between urban and rural sites and changes in culinary traditions in line with transitions in regime. A reconciliation of the results of organic residue analysis with other archaeological evidence of food has provided further insight into foodways and lifeways of the people in Sicily that experienced these transitions.

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## **Author declaration**

I declare that this thesis is a presentation of original work. Where parts of this thesis have been published or produced in preparation for submission in peer-reviewed journals with co-authors, I declare that I was the lead author. This work has not previously been presented for an award at this, or any other university. All sources are acknowledged in text citations and references.

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# Chapter 1 Introduction

During the Middle Ages (here defined as the period from the 6th century AD to the 14th century AD), the political situation throughout the Mediterranean can be described as mercurial. After the fall of Rome, the Mediterranean as a whole experienced varying degrees of socio-political transformations in response to multiple radical changes in regime. For several reasons, particularly its central geographical location in the Mediterranean, longstanding political and economic connections, and favourable environmental and arable conditions, the island of Sicily has been a central focus of political interest and resulting conquests throughout both ancient and medieval history. As the largest Island in the Mediterranean Sea, ideally placed between the mainland of Italy, North Africa and the eastern Mediterranean, Sicily was at the epicentre of multiple transitions in regime from the Byzantine (6<sup>th</sup>-8<sup>th</sup> century) to Aghlabid (9<sup>th</sup> century) to Kalbid (10-11<sup>th</sup> century) to Norman (11<sup>th</sup>-12<sup>th</sup> century) to Swabian (12-13<sup>th</sup> century) and finally the Aragonese period (14<sup>th</sup> century).

It is without doubt that at the introduction of these ideological regimes the people of Sicily would have experienced new political, socio-economic and religious transitions, the introduction of new agricultural systems and the arrival of new migrants (Molinari 2015, 187-220). Historical records and increasing archaeological surveys have significantly added to our understanding of these regime changes, but still little is known about how these changes directly influenced the lifeways of the people who experienced them on the ground. Much of our current knowledge of these periods is based on limited, and somewhat biased, documentary evidence of the time, which give only partial insight into the lifeways of the people that experienced these transitions. Often, our understanding of these regimes from contemporary written sources is transposed onto the general Sicilian population from the viewpoint of the ruling elites and urban courts (Molinari 2021). Ongoing surveys and archaeological investigations over the last few decades have begun to largely increase our understanding of the people that lived in Sicily at this time through investigations of urban and rural settlement sequences, investigation of burial rights and increased recovery and analysis of material culture (Molinari 2010; Nef and Prigent 2018; Molinari 2013, 2016; Ardizzone and Nef 2014). It is largely agreed amongst scholars that in order to

have a better understanding of these transitions and socio-economic systems a more in depth, multifaceted approach needs to take place. It has been suggested that this approach should combine history, archaeology, and scientific methods (Molinari 2015, 215–220). The Sicily in Transition (SICTRANSIT) project, of which this thesis is part, is an archaeological project that combines different archaeological and scientific approaches focused on understanding the socio-economic impact of cultural changes as a result of these transitions (Carver, Fiorentino and Molinari 2019).

Understanding past culinary habits can yield important information about the lifeways of past populations, as these are often linked to various socio-economic factors such as food availability and foodscapes, wealth, faith, and the technologies available for their procurement and processing. This thesis, focused on the 9<sup>th</sup>-14<sup>th</sup> centuries AD, investigates cuisine, the ways in which foods are prepared combined and consumed, as a proxy for understanding the lives of the general populous of Sicily during the Islamic, Norman, Swabian and Aragonese regimes. As domestic cooking pots are utilitarian artefacts that are ubiquitous in the archaeological record, understanding their contents can provide important insight into the culinary habits of the populations that utilised them as they likely capture a significant subset of the foodstuffs combined and prepared daily. Thus, this thesis used a multi-faceted organic residue analysis approach to yield direct chemical evidence of the contents of domestic cooking wares from a range of sites covering the 9<sup>th</sup>-14<sup>th</sup> centuries AD in both urban and rural contexts. In this thesis five main research questions are addressed:

## **1.1 Research questions**

- What impact do post-firing treatments have on the identification of organic residues from ceramic containers?
- Is it possible to identify Watson's revolution plants in archaeological ceramics using organic residue analysis?
- Were the contents of domestic containers changed or maintained as a result of transitions in regime from the 9<sup>th</sup>-14<sup>th</sup> centuries?

- How does the use of domestic containers compare between rural and urban sites under the same regime and/or transition?
- What can we learn about foodways in medieval Sicily by integrating organic residue analysis with other archaeological evidence?

To address these research questions; this thesis had a number of different objectives:

## 1.2 Objectives

- To conduct an extensive literature review of the historical context of Sicily during the 9<sup>th</sup>-14<sup>th</sup> centuries and evaluate approaches to understanding past cuisines.
- To carry out an extensive literature review of organic residue analysis of archaeological ceramics.
- To extract and analyse organic residues from experimental ceramics to understand the impact of post-firing treatments.
- To extract and analyse residues from modern plant products to identify specific biomarkers and to perform cooking and degradation experiments to understand whether these markers can be identified in the archaeological record.
- To extract, analyse and interpret organic residues from a large corpus of domestic containers from 9<sup>th</sup>-14<sup>th</sup> century contexts from five key sites (both rural and urban) in Western Sicily.
- To integrate the results of organic residue analysis with other forms of archaeological and scientific evidence to build an overall understanding of cuisine and foodways in Sicily during the 9<sup>th</sup>-14<sup>th</sup> centuries.

## 1.3 Thesis structure

The introduction has identified the main research questions that will be addressed in this thesis and the core objectives set to address these.

**Chapter 2** outlines the historical and archaeological context of Sicily during the Islamic, Norman, Swabian and Aragonese transitions and discusses the present state of knowledge and research questions that are the foundation of this thesis. This chapter also provides the contexts, rationale, and approach to studying cuisine in 9<sup>th</sup>-14<sup>th</sup> century Sicily.

**Chapter 3** is a detailed literature review of organic residue analysis of archaeological ceramics and outlines the main concepts and theoretical framework of the method used in this thesis. This chapter also outlines the main challenges associated with the use of organic residue analysis in understanding past cuisines and discusses the steps taken in this thesis to optimise the study of medieval Sicilian ceramics.

**Chapter 4** presents the results of two experiments. Firstly, the results of organic residue analysis of experimental cooking pots treated with post-firing treatments in collaboration with researchers from the Monte Iato project at the University of Innsbruck are discussed. These experiments and analyses aimed to address the impact of post-firing treatments on the interpretation of organic residues of archaeological ceramics. Secondly, the results of organic residue analysis of modern plant products are presented. Here, residues from modern plant products were analysed to identify specific biomarkers that could be found in the archaeological record.

**Chapter 5** 'New insights into early medieval Islamic cuisine: Organic residue analysis of pottery from rural and urban Sicily' is an article published in PloS one (Lundy et al. 2021). This article presents the first organic residue analysis of 134 cooking pots and other domestic containers dating to the 9<sup>th</sup> -12<sup>th</sup> centuries from three sites in the urban capital of Palermo and from the rural, agricultural settlement of Casale San Pietro. The



formatting, structure and referencing style has been altered from the published version to ensure consistency in this thesis and clear references to supporting information are given in the appendices.

**Chapter 6** 'Cuisine in transition? Organic residue analysis of domestic containers from 9<sup>th</sup>-14<sup>th</sup> century Sicilian contexts' is an article prepared for submission to the journal of Archaeological and Anthropological Science. This article presents the organic residue analysis of 9<sup>th</sup>-14<sup>th</sup> century domestic containers to support an assessment of impact of the transition from the Islamic political regimes to post-Islamic Norman, Swabian and Aragonese regimes from a culinary perspective. The formatting, structure and referencing style has been altered from the published version to ensure consistency in this thesis and clear references to supporting information are given in the appendices.

**Chapter 7** is a detailed discussion that combines the organic residue analysis of domestic containers presented in this thesis with other archaeological evidence of food consumption analysed as part of the Sicily in Transition project to gain deeper insight into the foodways of populations that used the ceramic vessels.

**Chapter 8** summarises the overall results within the framework of the five main research questions addressed in this thesis. This chapter notes the limitations of this study and proposes suggestions for future work.

## **1.4 Supporting information**

All data that supports this thesis are supplied either in the text, appendices, or supplementary information.

The supplementary information are presented as the following CSV files:

**S1 Data. Organic residue analysis results for post-firing treatment samples (chapter 4).CSV.**

**S2 Data. Ceramic samples and organic residue analysis results from 9<sup>th</sup>-12<sup>th</sup> century Palermo and Casale San Pietro (chapter 5). CSV.**

**S3 Data. Ceramic samples and organic residue analysis results from 11<sup>th</sup>-14<sup>th</sup> century Mazara del Vallo and Casale San Pietro (chapter 6).CSV.**

This thesis and all supporting files and raw data files will be deposited on an online Sicily in Transition data archive.

# **Chapter 2 Historical context and rationale for studying past cuisines**

This chapter will first briefly introduce the Sicilian ruling regimes from the 9<sup>th</sup>-14<sup>th</sup> centuries and highlight some of the limitations in our current knowledge. It will then go on to discuss how cuisine can be used as an important indicator of the lifeways of the people in the past, outlining our current state of knowledge of cuisine in these periods and how we can study cuisine in the archaeological record. Finally, this chapter will highlight how we can gain insight into cuisines during these transitions by using organic residue analysis to yield direct chemical evidence of the use cooking pots and other domestic containers.

## **2.1 The transitions**

### **2.1.1 Pre- Islamic Sicily**

Historians generally summarise the beginning of the Byzantine Empire (late 6<sup>th</sup>-7<sup>th</sup> century AD) in Sicily as a period of wealth and peace on the island. As seen during the Roman Empire, Sicily's economy continued to benefit as a major exporter of grain under the Byzantine regime and it has been assumed, due to its wealth in grain, that Sicily maintained its position as one of the richest provinces in the Mediterranean (Wickham 2006, 33; Molinari 2013). However, towards the end of the 7<sup>th</sup> century economic pressure and a breakdown in political structure weakened the Byzantine Empire in Sicily. This economic downfall and breakdown in political structure became the turning point which allowed Arabs to exploit the internal difficulties in Sicily and begin to attack the Island (Finley 1968). From 642 AD, the Arabs started to take over North African territories from the Berbers and Byzantines and by 709 AD they had gained control over the region known as Ifrīqiya which covered modern Egypt, Libya, Tunisia, Morocco, and Algeria. The early 8<sup>th</sup> century saw increased militarisation of Sicily as heightened interest from the ruling Aghlabid dynasty of Ifrīqiya resulted in more regular raids from North Africa (Anderson, Fenwick and Mariam 2017, 1). Thus, the 8<sup>th</sup> century in Sicily has been defined as a period of deep crisis in comparison to the

prosperous years before, also heightened by the great plague that had a devastating impact on the population (McCormick 2001). Whilst the Byzantine Empire was able to resist the Arab conquest for some time, as a result of increased fortification and internal problems within the opposing Ifrīqiya administration, contact with Islamic North Africa and Sicily was undoubtedly prominent from as early as the 8<sup>th</sup> century (Anderson, Fenwick and Mariam 2017, 1). Therefore, although the relationship between the Christian and Muslim world during these transition phases is still not fully understood and is hindered by a general lack of archaeological material in these phases, it is possible to suppose some level of socio-economic exchange between the two worlds long before Sicily was fully under the control of the *dar al Islam* (Metcalf 2009; Davis-Secord 2010).

### **2.1.2 Islamic Sicily**

Thus, the transition from a Christian world, under the Byzantine regime, to an Islamic one was neither a sharp nor straightforward transition. According to Ibn'al-Atiir in a chronicle of military events, the Arab conquest officially began in 827 AD, when a first real attempt was made to settle on the island (Amari 1933, 394). In 827 AD the Aghlabid army landed in Mazara del Vallo, Trapani, located on the south-western coast of the island, following almost a century and a half of political unrest and regular raids from North Africa. In 831 AD Palermo was captured and afterward became the political and economic capital of Sicily (Nef 2013, 39). It has been suggested that from 839 AD onwards the *dar al Islam* had gained full control of the western part of Sicily (Val di Mazara), after a number of urban and rural sites fell under Islamic rule (Chiarelli 2018, 23–56). However, the central and eastern part of Sicily (Val di Noto and Val Demone) remained largely under Byzantine control despite regular attacks from Islamic troops until at least the end 9<sup>th</sup> century (Metcalf 2009, 36; Chiarelli 2018, 53). This thesis focuses on the western part of Sicily. Therefore, reference to chronological periods of the Islamic regimes reflects the situation in the west. However, it is important to note that experience of the Byzantine-Islamic transition was very different in central and eastern Sicily to that in the west. Despite the fact that most of the Island came under Aghlabid control at the end of the 9<sup>th</sup> century, the two regions remained distinct. The

Val di Mazara (which encompasses Palermo and the western end of the island), was largely transformed by the Aghlabid regimes, but the Val di Noto and Val Demone continued to hold resistance (Metcalf 2009; Ardizzone and Nef 2014). Figure 1 is a map which illustrates the key sites and the different regions discussed here.

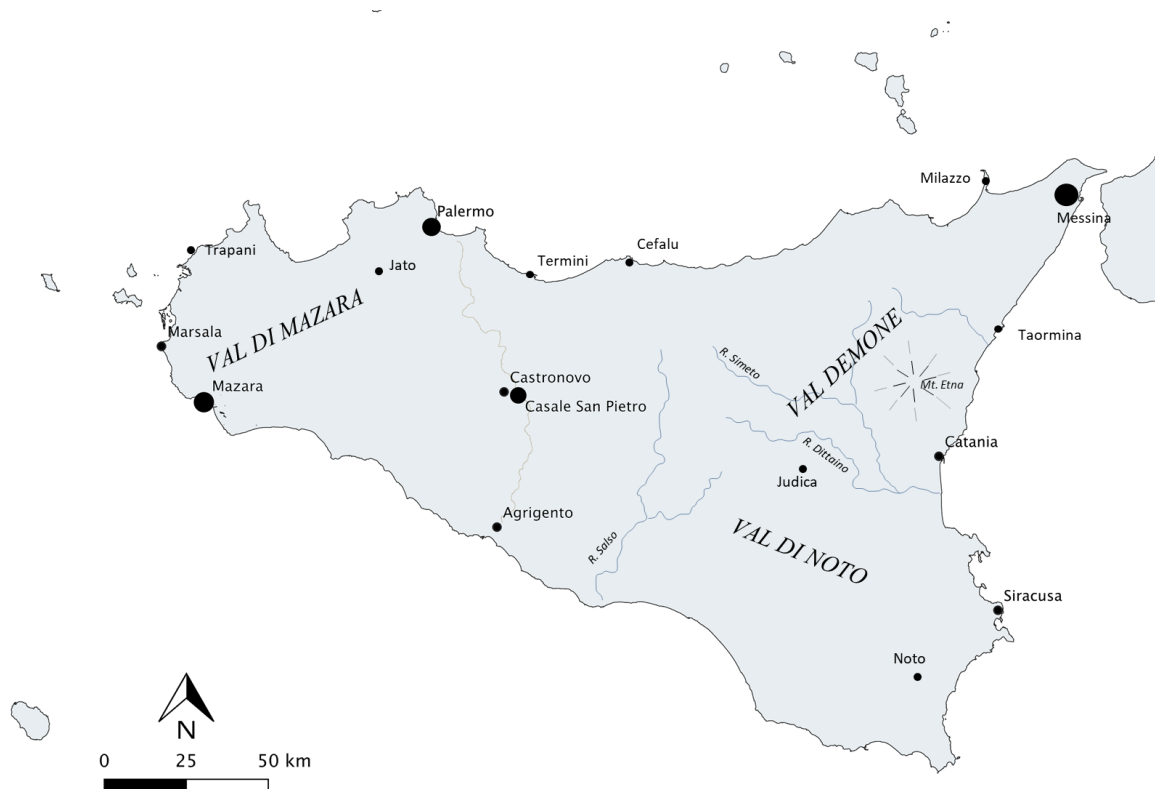


Figure 1: Map of Sicily showing the three administrative districts implemented during the Islamic periods adapted from (Loud 2000; Theotokis 2010, 384)

Over the next two centuries, Sicily was first under the control of the Aghlabid emirate (827-909 AD), then the Fatimid caliphate, firstly ruled from Ifrīqiya and then from Egypt, and finally the Kalbid emirate who ruled the island on behalf of the Fatimids from 948 AD. From an archaeological perspective, the period of initial conquest and its long period of transition is little understood due to a lack of material culture from the 9<sup>th</sup> century. However, increased surveys and ceramic typologies, which have enabled several sites in Palermo to be dated to the 9<sup>th</sup>-10<sup>th</sup> centuries (Arcifa and Bagnera 2014; Ardizzone, Pezzini and Sacco 2014; Sacco 2014; Spatafora), have begun to aid our understanding of the beginnings of Islamic political rule in Sicily. From the 10<sup>th</sup>-11<sup>th</sup> centuries an increase in material culture has been recovered that can be directly

associated with the Islamic regimes, either directly imported from Ifrīqiya or locally produced wares that have been modelled on those from Ifrīqiya. Furthermore, evidence suggests that, in Palermo, the central administrative system was likely modelled upon that of Ifrīqiya, North Africa (Johns, 2002, 29). Palermo stood as the urban capital of Sicily and was one of the richest capitals in the Mediterranean at this time. From at least the 10<sup>th</sup> century onwards, the introduction of new resources, agricultural systems and trade links generated considerable prosperity in Sicily (Watson 1974; Fiorentino, Lumaga and Zech-Matterne 2018; Carver, Fiorentino and Molinari 2019; Molinari 2021). During the period of Islamic political rule of the island it is certain that significant cultural and social change took place as Chiarelli (2018) states: *“The cultural and social changes fashioned by the Arab period are so profound that they have penetrated deeply into the island’s character”* (Chiarelli 2018, 129). However, it is not well understood how this affected the general populace that either entered Sicily at this time or already existed on the island, particularly in lesser-studied rural areas.

In less than three centuries, after a period of great prosperity in Sicily, the breakdown of the Kalbid dynasty, who ruled on behalf of the Fatimid dynasty from 948 AD, saw the end of Islamic rule in Sicily. During the Kalbid dynasty, the administration of the island became split and there were a number of internal struggles between different ruling subgroups (Booms and Higgs 2016). It was these divisions that led to the end of the Islamic rule of the island. From the 11<sup>th</sup> century, the Normans started to develop considerable interest in the southern Italian peninsula and they saw the political instability in the region as a clear advantage for their settlement (Nef 2011). These first settlements quickly developed into attempts to conquer.

### **2.1.3 Norman Sicily**

The Norman conquest was not a sudden transition and took thirty years to complete, starting in 1061 AD and not completed until 1091. It was not until 1130 AD the Norman kingdom of Sicily was formed (Metcalf 2009, 87). The Normans initiated their official invasion after an invitation from Muammad ibn Ibrāhīm ibn al-Thumna, who had

defeated the previous Kalbid ruler of the Val di Mazara and turned to the Normans in mainland Italy for their assistance in the midst of these internal divisions (Johns 2002, 33). In 1072 AD Roger Guiscard proclaimed himself Count of Sicily after the conquest of Palermo, but it took many years to conquer the Island after a long struggle against the Arabs and the final resistance in Noto, the last Muslim stronghold located in south-eastern Sicily, did not end until 1091 AD (Booms and Higgs 2016). After Roger I's death in 1112 AD, Roger II became the ruler of Sicily and in 1130 AD he proclaimed himself King of Sicily, Calabria and Puglia (Tramontana 2003). The Norman regime under Roger II embraced the economic success of the Kalbid regime and the kingdom enjoyed its benefits for much of his reign and was partly enjoyed by Roger II's successors. The Normans displayed their appreciation through elaborate Arabic inspired architecture and art, and Roger 'the Good' was well known for his imitation of Islamic customs (Britt 2007). The Normans adopted much of the administration and agricultural systems established prior to their arrival and largely profited from economic trade links that had been built with North Africa, the Middle East, and the rest of Europe. It has been said that the early collaboration with Ibn al-Thumna depicts why, when the Normans gained full control of the Island, they were able to '*treat*' with the Muslims (Johns 2002, 33). An emulation of the Arabic way of life by the Normans is often depicted in historical records. As discussed by Metcalfe (1999), Ibn Jubayr the Arab geographer on his trip to Palermo discusses the Norman rulers appreciation for Muslim culture and how they celebrated this by hiring Muslim chefs, scholars, and bodyguards and spoke Arabic (Metcalfe 1999, 28; Jubayr and Broadhurst 2001). In all ways of life, the Normans took advantage of the systems left by their predecessors and benefited from them. Thus, the Norman period in Sicily is characterised as a period of prosperity and multiculturalism, where several languages, cultures and religions existed in a single Kingdom with multiple layers and crossovers (Mallette 1998; Abulafia 2005; Metcalfe 2002; Johns 2002; Metcalfe 2003; Lomax 1996)

This evidence of 'cultural appreciation' are often used by scholars to exalt the Normans for their toleration of the existing population and way of life. However, emulating cultural ways of life and using them as their own for political gain does not necessarily mean that coexistence on the island was harmonious (Metcalfe 2002). Despite these ongoing relationships, the Norman invasion can also be seen as a turning point in

Mediterranean history. As Davis-Secord (2017) suggests, this invasion not only drew Sicily away from the Islamic world, it also tipped the balance in power between Islam and Christianity, as Christianity began to push itself into the far east and the Levant (Davis-Secord 2017, 177). Whilst written records often depict this time as a positive and accepting period, it is important to note that these portrayals often come in favour of the ruling elites at the time and the situation in Norman Sicily was likely more complex than that which is documented particularly for non-elite populations (Molinari 2021). Furthermore, to what extent this was the same in rural areas, as they are not documented equivalent to Palermo, for instance, is unknown. Into the 12<sup>th</sup> century relations between different cultural and religious groups became increasingly tense which led to a series of interethnic violence on the Island, particularly between the Arab-Muslims and Greek Christians and the Normans and Latin-Christians (Molinari 2021, 2). It was not until the rule of the Swabian King Frederick, in 1194 however, that a marked decline in the multicultural social landscape of the island is clearly seen.

#### **2.1.4 Swabian to Aragonese Sicily**

After the death of the Norman king William II in 1189 AD, the Norman period ended in Sicily with the rulership of the Hohenstaufen family of Swabia. Without a legitimate direct heir, William II agreed to the marriage of his aunt Constance de Hauteville to the Holy Roman Emperor and German Henry VI of Hohenstaufen. After William's death and multiple attempts by the couple to conquer the Island, they were finally successful five years later in 1194 AD. Their son Frederick II was crowned king of Sicily before their death and finally came to legitimate power at the age of 14 in 1208 AD. Frederick II chose the capital of Palermo as the capital of his new empire, but it has been argued that this was purely for pragmatic reasons of its central locality and multiculturalism (Booms and Higgs 2016). In fact, most of Frederick's rule and success therein occurred beyond the island. From the 12<sup>th</sup> -13<sup>th</sup> centuries the socio-economic, political, and religious situation in Sicily took a significant turn. During this time, much of the Arabic agricultural systems put into place by the Arabs disappeared in the 13<sup>th</sup> century and many Muslims and Jews were said to be expelled from the island (Metcalf 2009). As



Booms and Higgs' aptly name their chapter on this subject 'The beginning of the end', it is generally considered amongst scholars that this period saw an end to the 'Golden Age' of Norman rule (Booms and Higgs 2016, 244).

During this time, the unique multiculturalism that had defined Norman Sicily disintegrated in the rule of Swabian King Frederick II. During Fredrick's reign, there was a clear divide between Christian and Muslims who, until this point, represented the majority of the population in Sicily (Metcalf 2009, 142). However, this is a complex subject and Fredrick's intolerance for non-Christians on the island is a much-debated subject amongst historians. Fredrick's expulsion of Muslim communities has been perceived to be necessary as tensions rose and much of the Muslim community had already withdrawn from the island and/or lived in isolated communities. When Muhammad ibn Abbad created a Muslim state within Sicily located in the fort of Monte Iato (Province of Palermo, Sicily), Fredrick besieged the fort and put ibn Abbad to death after a rebellion broke out. The remaining Muslims were deported to Lucera as a safe haven to practice their faith and was considered a necessary action to restore peace on the island (Tramontana 2003; Abulafia 1992; Booms and Higgs 2016). Regardless of the reasons for this, it is undoubted that Sicily became predominantly Christianised and latinized during this time and Swabian ruled Sicily was distinct from previous reigns (Molinari 2021, 2).

As this thesis largely focuses on the Islamic, Norman and Swabian periods in Sicily and only reaches the beginning of Aragonese rule at the beginning of the 14<sup>th</sup> century, this period of rule is not discussed in full here. However, in brief, after the death of Fredrick II in 1250 AD, Sicily experienced yet another fight for power. After numerous power struggles, the island became Angevin under the rule the of Charles of Anjou, the brother of the French King Louis IX. However, the French ruler was highly disliked in Sicily and further unrest led to the rebellion known as the Sicilian Vespers in 1282 AD. The rebellion resulted in the expulsion of Angevins in Sicily. Sicily was then offered to Peter the III, King of Aragon (Booms and Higgs 2016). The Aragonese period can be seen as a time of great prosperity for the ruling Elite as the Kingdoms of Castile and Aragon united to form the Kingdom of Spain, but it is largely considered to be a time of great

decline and detriment to the general Sicilian population (Booms and Higgs 2016; Molinari 2010; Carver,Fiorentino and Molinari 2019).

### **2.1.5 Summary and rationale for research**

In summary, in the 9<sup>th</sup>- 14<sup>th</sup> centuries, at the onset of the Islamic, Norman, Swabian and Aragonese regimes we know that Sicily would have experienced significant shifts in political and social structure. New religions, people, resources, and economic links with the rest of the world undoubtedly saw change in the lifeways of the people that experienced them on the ground. However, limited historical sources and a lack of extensive archaeological studies has restricted our understanding of exactly how these people lived and were impacted by identified changes in regime. Furthermore, although relatively small in terms of land mass, it can be assumed that the impact of these transitions would have varied greatly across the island. In addition to the geographical distinctions, timings, and measured effect of the Islamic, Norman, Swabian and Aragonese conquests in different parts of the island, it is also proposed that there are likely to be considerable differences between rural and urban settlements. It is therefore of great interest to scholars not only to understand the consequences of these transitions but to also consider the micro-political climates of these regions. The SICTRANSIT project, of which this thesis is part of, was designed to investigate such changes, particularly as they affected the farmers, merchants and their families (Carver, Fiorentino and Molinari 2019).

There are a number of questions that present themselves within this gap of knowledge:

- To what extent did the people living on the ground adapt and change their way of life at the onset of these regimes?
- As these are widely underrepresented in documentary sources, what role did rural settlements play in these regimes?
- How were religious groups treated and integrated into the society of the existing regime?

## **2.2 Cuisine**

### **2.2.1 Introduction**

An assessment of resource availability leading to an understanding of diet can yield insight into the social and economic values associated with the resources available locally, and those that may have been imported. This is because culinary choices, both in the past and today, are often linked to different socio-economic and cultural factors such as food availability and foodscape, wealth, faith, and societal value (Hastorf 2016; Twiss 2012; Beaudry 2020). Cuisine can be defined as the way in which specific resources were prepared, combined, and consumed, and thus by identifying specific cuisines in the archaeological record, it is possible to gain invaluable insight into the way that people lived in the past.

Rozin (1982) reflected on the idea of food, cuisine, and society by separating the concept into four main aspects. Firstly, cuisine prioritises a set of basic foods that are selected by those who use them. In expanding this idea, depending on what is available and what can be procured, societies have either a limitation in the food they can select or a choice in the foods that they can select. This aspect becomes most apparent in wealth and social status, environment and in religion (e.g., in terms of food taboos). Secondly, specific cuisines have set ways of preparing or manipulating these selected foods. Therefore, in order to be able to assess different cuisines, a 'cultural transformation processes' must be considered. Thirdly, cuisines and those cultures associated with them acquire a certain set of protocols for eating them. This includes cultural traditions, how foods are served, techniques and tools used to consume these foods and Belasco (2008) and Rozin (1982) suggest that manners and etiquette can also define certain cuisines and culinary habits. Finally, Rozin (1982) suggests that each specific cuisine has a certain set of flavours. These flavours are not only achieved by the foods that are selected, but also how they are combined and cooked. Belasco (1987) assigns specific flavours to different 'ethnic' groups and discusses how these flavours are a significant part of the identity of cultural groups.

Belasco (2008) also adds infrastructure or a food chain as a fifth component. This can mean the immediate chain from when food is procured to when food is on the plate, but it is suggested here that the food chain involves a number of processes, including the production, procurement, processing, consumption and even the disposal of foods (Woolgar 2010; Twiss 2012; Hastorf 2016). Infrastructure of cuisines also embodies the evolution of cuisines, how they adapt over time, and how recipes have been passed on through generations and across geographical regions.

Therefore, the processes and influencing factors associated with cuisine embodies much more than just an assessment of food for nutritional and survival purposes (diet), thus dismissing the concept that food is simply used as a fuel (Beaudry 2020). Identifying cuisines in the archaeological record holds the potential to shine light not only on what foods were available to a population, but elements of choice, taste and cultural beliefs surrounding foods. Thus, shifts in cuisines are often associated with changes in socio-political structures in a society as new food traditions are imposed (by one group) and accepted or rejected (by an existing group) (Holtzman 2009; Macbeth and Lawry 1997; Powers and Powers 1984 and Vroom 2000). The five main factors of cuisine and how they are linked with socio-economic factors in society are the foundation of this thesis and are summarised in Figure 2.

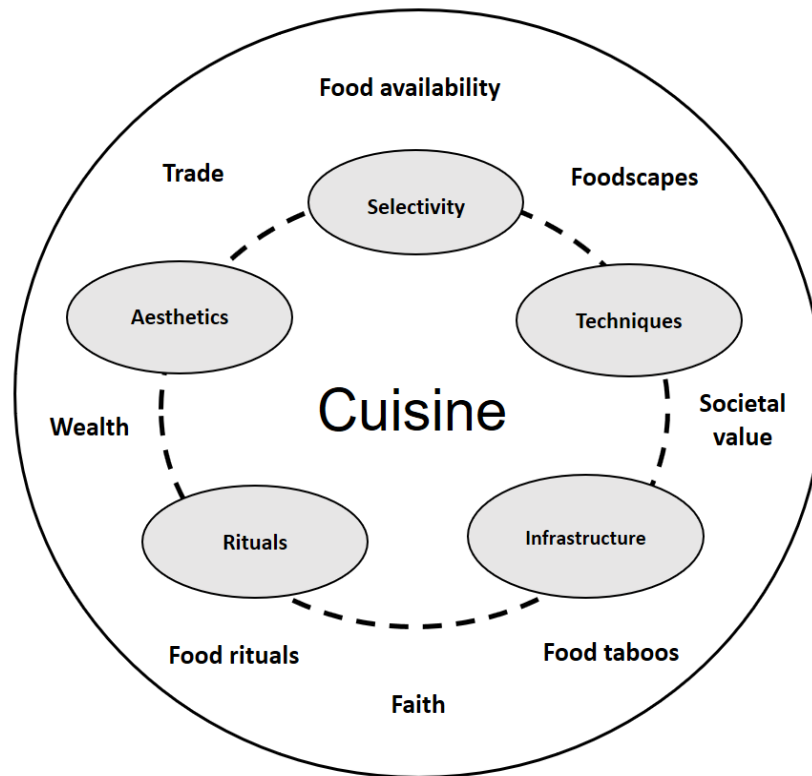


Figure 2: Summary of the five aspects of cuisine and their association with socio-economic influences. Taken from the ideas of Rozin (1982) and Belasco (1987,2008)

## 2.2.2 The impact of transitions and Sicilian cuisine

It is hypothesised here that the complexity of the impacts of the regime changes on daily life in Sicily was most likely reflected in the culinary choices of the populations that experienced them. Whilst the importance of Sicilian cuisine and its long history is unquestionable, little is known about the culinary habits of the general population during this time. The discussed history of Sicilian cuisine is widely demonstrated in modern cookbooks and often focus is drawn to the noticeable Arabic influence of food eaten in Sicily today, which has been largely attributed, although not affirmed, to the arrival of the Arabs in the 9<sup>th</sup> century (Salloum 2002; Root 1992). The noticeable difference in the flavours and dishes of Sicilian cuisine in comparison to the rest of Northern Europe and even mainland Italy has been attributed by a number of food historians to the clear influence of Arab rule (Salloum 2002; Root 1992; Truelove n.d;

da Riva 2006). There is a consensus amongst food historians that the later Norman populations, or at least the ruling elite, enjoyed Arabic influences on cuisine as evidence suggests that the Norman king celebrated much of the previous culture and even had Arabic chefs in the courts. However, to what extent the rest of the population enjoyed the same dishes as previous inhabitants is unknown and evidence is rather elusive. As da Riva (2006) suggests, this has left food historians to 'theorize' about the recipes enjoyed during this period. In the Swabian and later Aragonese periods, the evidence of culinary habits is even less known. However, it is possible to draw assumptions regarding culinary habits during the 9<sup>th</sup>-14<sup>th</sup> centuries in Sicily from our current knowledge based on written sources, influences of agriculture, trade, and animal management, as well as known cultural and religious structures of the population. The next sections of this chapter discuss some assumptions regarding culinary habits and how they may have been influenced as a result of changing political regimes in Sicily.

### 2.2.3 Written sources

At present, most of our understanding of Sicilian cuisine in the Middle Ages comes from evidence in medieval Arabic cookbooks. The idea of recording particular recipes for use in future cooking was a distinctive Arabic practice of the time, one that was not well known in the non-Islamic western world. Before the 14<sup>th</sup> century, there are more Arabic cookbooks than all of the world's languages combined (Zaouali 2009). The *Kitāb al-Tabīkh* ('Book of Cooked Food'), Ibn Sayyār al-Warrāq's Tenth-Century Baghdadi Cookbook, is the oldest surviving Arabic cookbook and it compiles the recipes of the Baghdad caliphs from the 9<sup>th</sup>- 10<sup>th</sup> centuries. Nawal Nasrallah translated this book from Arabic in *Annals of the Caliphs Kitchens* (Nasrallah 2007). This cookbook of more than 600 recipes gives insight into the cuisine of medieval Islam. *Kitāb al-Tabīkh* ('Book of Cooked Food'), a 13<sup>th</sup> century manuscript written by the scribe referred to as al-Baghdādī, is another important manuscript describing cuisines of the last Ayyubid sultan of Egypt. Charles Perry has translated the text in the book *A Baghdad Cookery Book* (Perry 2005). Whilst written from the perspective of the culinary habits of the elite class from Baghdad rather than in Sicily itself, these recipes have been used to give insight into the types of cuisines that may have been enjoyed across the medieval

Islamic world. Literature describing Arabic cuisine discusses not only the importance of new resources such as meats, vegetables and spices to cuisine, but is descriptive about the vibrant new colours, sweet taste and interesting textures that encompass and defines these new dishes, and offers insights into the technologies used to cook them (Zaouali 2009; Waines 2003; Perry 2005).

Discussions of culinary habits during the Norman, and to some extent the later Swabian rule in Sicily, are centred on the assumption that these rulers adopted much of the culinary influences of their predecessors. Whilst there are no surviving records of Sicilian cuisine from the period of Norman rule in Sicily, there are written sources which give some insight into what cuisine may have been like. One example is of the accounts written by Muslim geographer al-Idrisi who, on his visit to Palermo, composed *Kitab Rudjdjar* (The Book of Roger) (discussed in Lewis 1958, 148; Gies 1977). Furthermore, the writings of Arab geographer Ibn Jubair, who travelled to the court of William II in Palermo and wrote accounts of his travels and the Islamic world, discusses the emulation of Arabized foods in the court. Here he writes, 'The king has full confidence in the Muslims and relies on them to handle many of his affairs, including the most important ones, to the point that the Great Intendant for cooking is a Muslim.' (cited by Lewis 1958, 148). In addition, although the author is unknown, arguably one of the most famous surviving cookbooks *The Liber de coquina*, has been attributed the Angevin court of Naples in the 14<sup>th</sup> century (Mulon 1970). However, some scholars argue that, in fact, the origin of many of the recipes can be traced to the courts of Fredrick II, due to the clear Arabic influence of some recipes that had been transformed and adapted by the Norman and Swabian courts in Palermo (Martellotti 2005). Food historians have also linked the clear Arabic influence of Norman and Swabian cooking to culinary literature written outside of Sicily. For example, an extensive list of recipes written in the 14<sup>th</sup> century about culinary habits during the 12<sup>th</sup>-13<sup>th</sup> centuries in Spain, *The Anonymous Andalusian Cookbook* (Perry 1965), has many cross overs with earlier Arabic recipes, such as those in the *Kitāb al-Tabīkh*. From the 12<sup>th</sup> century onwards there was a flurry of cookbooks written in Christian Europe, which has been attributed to increased contact of the rest of the Christian world with the multi-cultural hubs of Sicily and Spain (Zaouali 2009).

Written sources can be useful as a guide to our understanding of cuisine at this time, and have been used to theorize the culinary habits of populations in the past, but they should be treated with caution. Distributed across a broad chronological period and geographic area, these writings mostly describe practices of elite consumption and do not necessarily reflect the daily foods consumed by the general population in Sicily.

#### **2.2.4 Agriculture and animal husbandry**

Fundamental to culinary activity in society is the foods that are available locally for procurement and processing. Agriculture has been at the heart of Sicilian economy since the Roman period and, as previously mentioned, the Byzantine Empire continued to profit off its prosperous grain production and export. After the arrival of the Arabs, Sicily saw a significant transformation in its agricultural economy with the introduction of new agricultural and irrigation systems and the potential introduction of new resources. The Islamic “Green Revolution”, as originally coined by Watson (Watson 1974, 1983), discusses the assumed association between the movement of Islamic communities and the introduction of new resources and agricultural innovations. In his thesis, Watson documents 17 new plants that arrived in the Mediterranean from the Middle East with the arrival of the Arabs, including spinach, cotton, durum wheat, sugar, citrus fruits and possibly broomcorn millet (Watson 1974, 1983). Figure 3 shows Watson’s proposed dispersion of different plants from the East to the Middle East to the Mediterranean. In more recent years however, aspects of Watson’s thesis have been revived both from historical research (Decker 2009; Squatriti 2014) and from the development of archaeobotanical research (Van der Veen 2010, 2011; Fuks, Amichay and Weiss 2020).



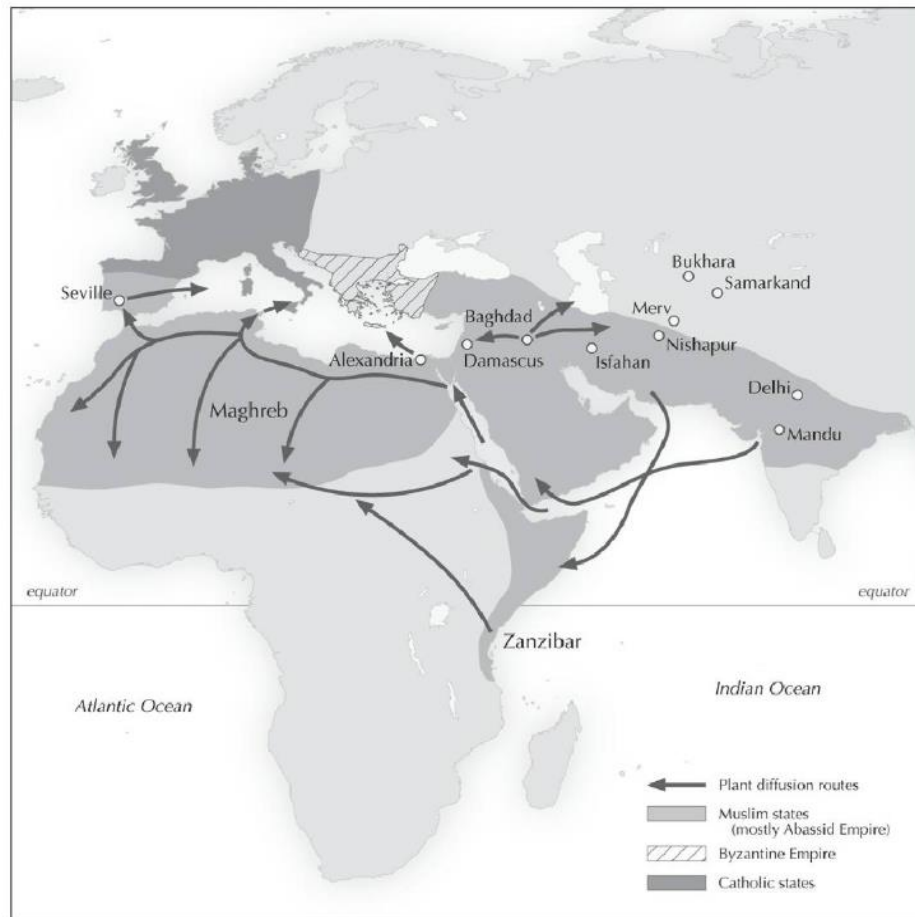


Figure 3: Watson's map of the dispersion of plants from the 'Arabic' world to the Mediterranean (Watson 1974)

Whilst evidence of these 'revolution plants' in Sicily has previously been elusive, (Sadori et al. 2013) recently archaeobotanical investigations at Mazara del Vallo (Trapani) have provided direct evidence of Watson's 'revolution plants', including citrus fruits in 10<sup>th</sup>-11<sup>th</sup> century contexts and watermelon, aubergine, cotton, durum wheat and spinach from 11<sup>th</sup>-13<sup>th</sup> century contexts (Primavera 2018a; Fiorentino, Lumaga and Zech-Matterne 2018). The work of Watson and his conclusions of the arrival of entirely new crops during the 'Arab agricultural revolution' has been subject to critique by numerous authors, who suggest that the commodities he listed did in fact exist in the Mediterranean long before the Arabs arrived (Decker 2009 and Squatriti, 2014). Without clear evidence from preceding contexts this is difficult to discern but, whether previously present or not, the Arabs did initiate new ways of growing and utilizing these products on a much larger scale than before (Decker 2009). There is no doubt that the agricultural and irrigation systems introduced by the Arabs in Sicily were revolutionary, and would have had significant impact on the types and

availability of resources in Sicily. It is generally agreed that agricultural productivity was maintained into the 12<sup>th</sup> and 13<sup>th</sup> centuries in Sicily (Molinari, Alessandra and Martin 2020). The new resources, agricultural and irrigation systems introduced during Islamic rule continued to be appreciated and expanded during the Norman rule. Evidence of these revolution plants appearing in 12<sup>th</sup> and 13<sup>th</sup> century contexts is beginning to further support their continued production (Primavera 2018a; Fiorentino, Lumaga and Zech-Matterne 2018).

Alongside evidence of Watson's revolution plants, archaeobotanical evidence from a number of sites dated to the 9<sup>th</sup>-14<sup>th</sup> centuries across Sicily have revealed a variety of other products available at this time. Various vegetables, fruits, legumes, and cereals identified in these contexts have enabled increased insight into the types of products that were available to these populations and observed trends in agricultural production over time (Primavera 2018a; Fiorentino, Lumaga and Zech-Matterne 2018; Fiorentino et al. forthcoming, forthcominga; Castrorao Barba et al. 2021). However, as indirect evidence, this does not allow us to understand how these resources were utilised by the populations on a daily basis. For example, scholars suggest that from the start of the 13<sup>th</sup> century agricultural production shifted and was mainly focused on the large-scale export of the 'Mediterranean triad' of wheat, olive oil and wine. Whilst this agricultural focus benefited the elite populations, it is said to have had significant impact on the agricultural farmers as landlords extracted surplus from the agricultural farmers for their own economic gain (Booms and Higgs 2016). A similar phenomenon is seen in Spain after the re-conquest. (Torró and Guinot 2018; Molinari, Alessandra and Martin 2020). Therefore, it is important to consider that archaeobotanical evidence is not a direct insight into the foods being procured and utilised by the general population and may in fact only reflect agriculture as an economy.

Animal husbandry was also an important factor in Sicily's farming industry during the 9<sup>th</sup>-14<sup>th</sup> centuries and animal products likely played a significant role in daily cuisines at this time. Increasing archaeological excavations and zooarchaeological investigations have given great insight into the types of animals present at both rural and urban sites in Sicily during the 9<sup>th</sup>-14<sup>th</sup> centuries. Zooarchaeological data from 9<sup>th</sup>-14<sup>th</sup> century urban contexts indicate that animal husbandry was focused on the main

domesticates: caprine (both sheep and goats), cattle and domestic fowl. Evidence shows that these animals were utilised for both their meat and secondary products (e.g., wool, leather, milk and eggs) (Arcoleo and Sineo 2014; Arcoleo 2015; Aniceti 2020; Aniceti and Albarella 2022). Research at these sites has also shown that, in most cases, pig remains were scarce in urban sites, reflective of the religious restrictions on the consumption of pork as part of Islamic hadiths (Arcoleo and Sineo 2014; Arcoleo 2015; Aniceti 2020). In contrast, zooarchaeological investigations at some rural sites across Sicily have identified the presence of pig remains in 9<sup>th</sup>-11<sup>th</sup> century contexts highlighting that some communities in Sicily continued to consume pork at these sites (Aniceti 2020; Aniceti and Albarella 2022; Castrorao Barba et al. 2021). Zooarchaeological investigations have hinted at shifts in animal husbandry practices in the later 12<sup>th</sup>, 13<sup>th</sup> and 14<sup>th</sup> centuries as biometric analysis has indicated an increase in sheep sizes from the 10<sup>th</sup> to 12<sup>th</sup> century in urban Palermo and between the 11<sup>th</sup> century and 13/14<sup>th</sup> century contexts at Mazara del Vallo. Furthermore, at urban sites, the return of pig remains in urban contexts is also noted in the 12<sup>th</sup>-14<sup>th</sup> centuries (Aniceti 2020; Aniceti and Albarella 2022).

Alongside domesticated animals, it has also been suggested that the Normans globally were 'addicted to hunting and hawking' and were somewhat fascinated by exotic animals, a tradition that was also largely celebrated by the Swabians (Metcalf 2009, 97,244). The presence of a variety of game in Sicily at this time is evident in several historical sources, royal ordinances and archaeological faunal assemblages, whereby fallow deer seems to hold most importance (Bossard-Beck 1984; Di Martino 1997; Burgio, Masseti and Sarà 1998; Bedini 1999; Sarà and Cangialosi 2005). Again, however, it is not known to what extent these hunting strategies may have been integrated in day-to-day Norman and Swabian life or even how accessible meat products were to the general population.

### **2.2.5 Restrictions, selectivity, and choice**

Religion likely played an important and complex role in shaping the economy and consumption practices of Sicily during the 9<sup>th</sup>- 14<sup>th</sup> centuries. The most obvious way in

which a change in religion at the beginning of these regimes may have affected culinary choices is due to food taboos and religious fasting. The consumption of pork and alcohol are prohibited in the Islamic hadiths, but to what extent these restrictions were observed in early medieval daily life is questionable, particularly in a pluralistic society, as was Sicily at this time. Under the Islamic political regime, Christian and Jewish communities remained in Sicily during the 9<sup>th</sup>-12<sup>th</sup> centuries, living alongside Muslims (Ardizzone and Pezzini 2014, 282). Thus, although Jewish communities share similar restrictions on the consumption of pork, for example, the observation of religious restrictions cannot be assumed for the whole population. For example, recent, zooarchaeological work by Anceti (2020,2022) has investigated the utilization of animal taxa during the Middle Ages in Sicily. In urban areas, such as Palermo and Mazara, pig remains were almost non-existent in the faunal assemblages at these sites, supporting the notion that the communities that lived at these sites practiced food taboos. On the other hand, the high occurrence of pig remains, particularly neo-natal pigs, identified at rural sites such as Casale San Pietro lends to the idea that the dietary habits of these communities were not strictly controlled by the Islamic administration (Anceti 2020; Anceti and Albarella 2022). This same phenomena has been observed in Islamicate contexts in Spain and Portugal, whereby the discovery of pig remains has been attributed to the presence of Christians or hunted wild boar (Salas-Salvadó et al. 2006; Grau-Sologestoa 2017). Furthermore, although prohibited by the hadith, wine may have been consumed by Christian and Jewish communities, or as Islamic medieval poems depict, by some members of the Muslim community as well (Branca 2003; D'Alessandro 2010). The trade of wine to and from Islamic Sicily has also recently been observed by identifying organic biomarkers associated with grape products in amphorae (Drieu et al. 2021b). A change from a predominantly Islamic faith to a Christian one may have been an important factor in defining cuisines in the later Norman and Swabian periods. For example, the loosening of prohibitions of the hadith meant that the consumption of alcohol and pork might have increased during this time. However, as Sicily, especially the west, remained largely a pluralistic society until at least the late 12<sup>th</sup> century, an increase in the consumption of these commodities remains a difficult assertion to navigate. Viniculture during the Norman rule of Sicily is said to have increased and the island seems to have remained a primary exporter of

wine until the 12<sup>th</sup> and 13<sup>th</sup> centuries when Sicily began importing most of its wine (Molinari 2018; Buccellato 1988).

Another assumption regarding the impact of the established religion is that there was an increase in the consumption of fish during the Norman and Swabian period due to meat fasting as part of the Christian religion, driving the consumption of fish as a replacement. A 'fish event horizon', in England in the 10<sup>th</sup> century AD was driven not only by population growth and urbanisation, but Christian fasting regulations that sanctioned the consumption of fish in replacement of meat (Barrett,Locker and Roberts 2004b, 2420). In Sicily, however, the consumption of fish by the general populace during the 9<sup>th</sup>-14<sup>th</sup> centuries is not well studied. The *Tonnara*, a system of tuna trapping that the Arabs in Sicily revolutionised, changed under Norman and Swabian control as the rights to these resources were under the complete control of the royal powers. The church and those loyal to the crown would receive concessions, but generally these restrictions meant that fishing communities who previously had access to these resources no longer did (Lentini 1986; Consolo et al. 2008; Longo and Clark 2012). Therefore, fish was likely an expensive commerce utilized for its economic advantage as the exportation of tuna from Sicily was the second most important export during this period (Hoffmann 2001).

However, religion is not the only factor in determining what people ate and a variety of other factors that may have implemented restrictions on the products utilized, such as economic and social access to foods, need to be considered. For example, as already touched upon, the elite populations in Sicily would likely have had access to a much wider range of resources both present locally and accessible through trade. It has been noted that those living in rural settlements, for example, were less likely to have access to the range of food products available to purchase at markets in comparison to those in urban areas (Pitchon 2020). Furthermore, temporary times of crisis be they environmental, economic or disease may also affect what people were eating. Personal choice and selectivity of products for consumption should not be, but are often, dismissed in assessing culinary choices of past populations (Holtzman 2009; Macbeth and Lawry 1997; Powers and Powers 1984 and Vroom 2000). Whilst individual choices

are difficult to assert through evidence present in the archaeological record, it is important to consider these when investigating culinary habits of past societies.

## **2.3 Ceramics**

The ubiquity and longevity of ceramics in the archaeological record makes them an extremely informative asset to the study of past populations. Distinct ceramic typologies can aid chronologies by forming chrono-type references as well as identifying trade patterns and socio-economic links. An understanding of ceramic forms and functions can provide information regarding the lifeways of people who utilised these vessels. Although increased archaeological excavations and surveys in Sicily have recovered an abundance of pottery from multi-occupational sites, until recently, extensive studies of these materials has been limited and a number of issues still persist. For example, a complete chrono-typological sequence of ceramics from Late Antiquity to the end of the Swabian period does not currently exist. This is largely owed to the fact that few extensive studies have been undertaken, and of those that have, published assemblages are rare. However, recent investigations have yielded a wealth of new material culture that has enabled an increase in ceramic studies, which have focused on identifying a chrono-typological sequence. Furthermore, due to a predominant focus on stylistic forms, such as tableware, domestic cooking vessels have received less attention in ceramic studies. Researchers agree that the study of cooking vessels from these periods can not only increase evidence to construct the typological series, but can also yield important insight into the day-to-day activities of people living on the island at the time (Kirk 2013; Testolini 2018; Pezzini, Sacco and Yenişehirlioğlu 2018).

### **2.3.1 9<sup>th</sup>- 11<sup>th</sup> century domestic containers**

Until recently, only later 10<sup>th</sup> and 11<sup>th</sup>-century ceramic typologies were well established; there have been considerable gaps in our knowledge about the latest Byzantine and earliest Islamic ceramics (8<sup>th</sup>-9<sup>th</sup> century). For this reason, most Islamic

contexts in Sicily were dated from the second half of the 10<sup>th</sup> century to the early 11<sup>th</sup> century which has restricted our understanding of Islamic occupation on the Island. The reason for this so called 'black hole' in ceramic types from these periods is a subject of much interest concerning discussions of Sicilian occupation (Ardizzone and Nef 2014, 378). Some scholars suggest that late Byzantine settlements were abandoned at the end of the 7<sup>th</sup> century and were not reoccupied again until the 10<sup>th</sup> to 11<sup>th</sup> century (Arcifa 2013, 164). However, others argue that, despite the lack of evidence, there is in fact a continued Byzantine occupation from the 7<sup>th</sup>-11<sup>th</sup> century (Molinari 2013, 108). Furthermore, the lack of defined ceramic typologies of this period does not mean that there was not Islamic presence on the Island prior to the second half of the 10<sup>th</sup>-century. What is certain, however, is that the lack of evidence from these periods, across multiple sites, has made it difficult to discern strong chronological indicators in these ceramic assemblages. In comparison to fine wares and glazed ware ceramics, of which most chrono-typologies have been based on, cooking wares and domestic containers have received much less attention. Thus, despite their abundance and importance in the archaeological record, asserting chronologies based on cooking and domestic wares is even more difficult. Scholars, in recent years, have highlighted this problem and noted the need for a far more extensive study of all types of ceramics across Sicily using a multi-faceted approach (Messina et al. 2018). Increased ceramic studies that aim to assert chrono-typologies, identify production centres, and understand the use of ceramics give great potential to our understanding of Sicily throughout the Middle Ages. Indeed, a number of production sites from the 10<sup>th</sup> century have now been identified in Sicily; Palermo (Giarrusso and Mulone 2014), Mazara del Vallo (Molinari 2010), Agrigento (Alaimo et al. 2007), Piazza Armerina (Alaimo et al. 2010), Syracuse (Amara et al, 2009) and Paterno (Messina et al., 2018). Furthermore, an increase in the number of 9<sup>th</sup>-11<sup>th</sup> century ceramics identified at a number of sites has broadened our understanding of Islamic occupation in Sicily.

In the urban capital of Palermo, at the sites of Castello San Pietro (Arcifa and Bagnera 2014), La Gancia (Ardizzone, Pezzini and Sacco 2014) and Palazzo Bonagia (Sacco 2014), ceramic surveys have enabled the identification of some of the earliest Islamic style pottery in Sicily. These findings confirm Islamic presence in Palermo as early as the 9<sup>th</sup>-10<sup>th</sup> centuries. Gragueb Chatti (2006) notes strong typological links between

tableware vessel types found in Palermo with those from Aghlabid Ifrīqiya (Raqqada) and these have been used to identify Islamic presence on the island (Gragueb Chatti 2006, 341; Arcifa and Bagnera 2014). Of the cooking pots identified at these sites, the majority of the assemblages consist of *ollae*, braziers, lids, pans and *testi/testello* (stone plates). A clear marker of Islamization, identified by Arcifa and Bagnera, is the introduction of braziers, which show strong similarity to examples found in Raqqada (Gragueb Chatti 2006; Arcifa and Bagnera 2014, 169). These sets of cooking wares appear to evolve in the Fatimid period, with distinguishable morphological features that make them reliable chronological markers for this period. In contrast, the vast majority of domestic cooking wares were produced in Palermo and appear typologically dissimilar to those found in Ifrīqiya (Sacco, Chatti and Touihri 2018). In this context we see a reliance on *ollae*, which as a general form is a continuation from antiquity and the term ‘*olla*’ has widely been used to describe a variety of closed cooking vessels from Roman times to the medieval periods in Italy and Spain (Donnelly 2016). These Palermo production *ollae* are wheel thrown and therefore stand apart from handmade examples from North Africa (Arcifa and Bagnera 2018, 388). However, the identification of specific macro groups amongst the *ollae* assemblages at the Palermo sites have unveiled a new type of *olla* that is not seen before the 10<sup>th</sup> century in Sicily, and therefore can be used as a chronological marker for the Aghlabid period. These particular *ollae* are fired with a distinct technique similar to an example found to have been imported from North Africa (Ardizzone, Pezzini and Sacco 2018). Another type of *olla* with raised rims has also been found at the sites of Vega Baja Toledo (Spain) and Istabl’ Antar (Egypt), as shown in Figure 4 (Arcifa and Bagnera 2018, 385–387). Similarly, lids that appear in these contexts may also indicate a continuation from Late Antiquity, but have also been compared to examples from North Africa and Spain (Arcifa and Bagnera 2018, 385–387).



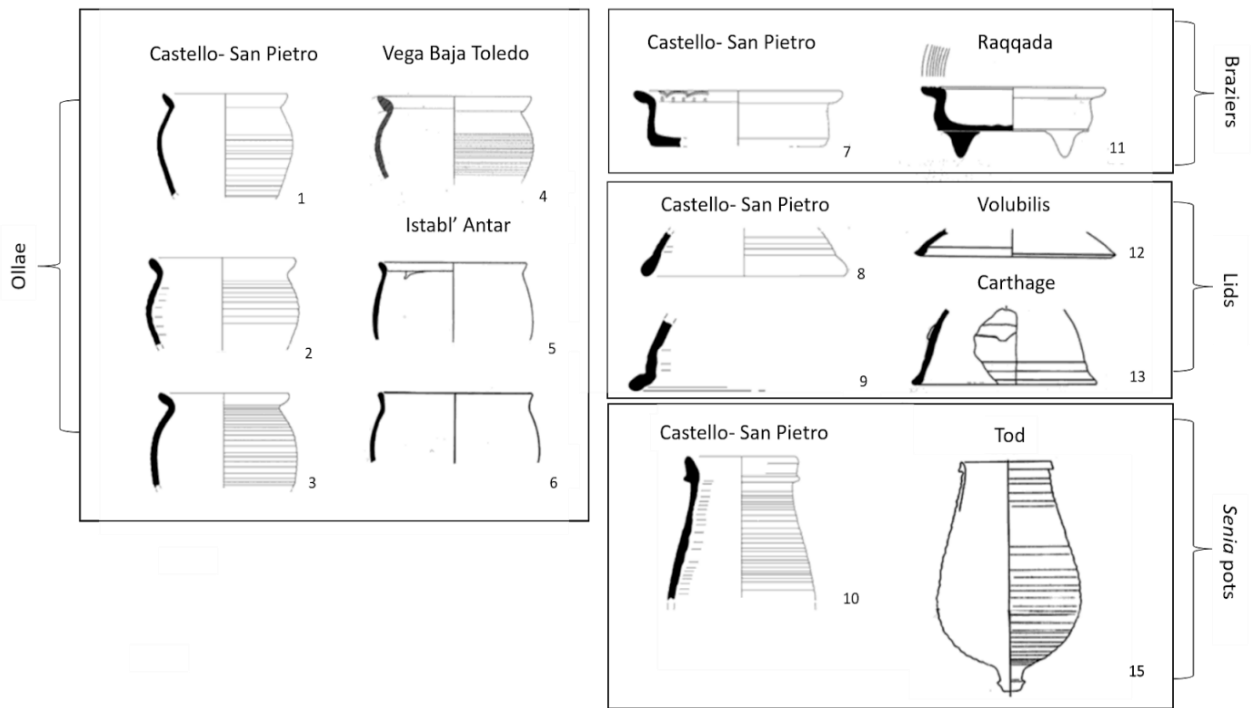


Figure 4: Comparison of ceramic forms (*Ollae*, *braziers*, *lids* and *Senia pots*) identified at the site of Castello San Pietro (Palermo, Sicily) and similar examples at other Islamic sites adapted from (Arcifa and Bagnera 2018, 385–387)

The *testello* or stone plate, increasingly occurs not only in Palermo, but other sites dated from the 9<sup>th</sup> century onwards, such as Casale San Pietro (Carver et al. 2019; Carver and Molinari 2016), Mazara del Vallo (Meo and Orecchioni 2020), Piazza Amerina (Gasparini et al. 2012) and Agrigento (Fiorilla 1990). Whilst it cannot be dismissed that these types existed in the Byzantine periods, they have yet only been found in Islamic contexts and later (Arcifa and Bagnera 2018). Increasing archaeological excavations and surveys have enabled the identification of 9<sup>th</sup>-11<sup>th</sup>-century ceramics in a number of sites across Sicily. Alongside Palermo, of interest to this thesis are the sites of Casale San Pietro and Mazara del Vallo.

At the site of Casale San Pietro, an extended agricultural settlement that lies in the rural hinterland of the Palermo province, only a small number of hand-made/slow thrown cooking pots with in-turned rims have of unknown provenance make up the assemblage of the 9<sup>th</sup> – 10<sup>th</sup> century. In the 10<sup>th</sup> – 11<sup>th</sup> century however, the pottery typology is well defined as the settlement seemed to be mostly reliant on pottery from Palermo production pottery. Palermo type pottery forms all of the tableware

assemblage and a large amount of the kitchen wares present on the site (Carver et al. 2019, Carver et al. forthcoming).

Excavations at the urban site of Mazara Del Vallo in 1997 unveiled a very rich ceramic assemblage in contexts relating to domestic residences of the Islamic town (Molinari, 1997). Current studies on this assemblage have shown that, unlike Casale San Pietro, in the 10<sup>th</sup> -11<sup>th</sup> century Mazara had independence of its pottery products and did not rely on Palermo production pottery (Meo and Orecchioni 2020; Meo forthcoming). Mazara, as a key urban centre, produced much of its own tableware and kitchenware that supplied the populations that lived there. Here, the assemblage mainly consists of cooking pots. Whilst simplistic, these cooking pots come in varying forms, such as handmade examples, including handled cooking pots, and conical and flat-bottomed pots. Indeed, there are also clear 'Islamic' examples such as the stone plate, braziers, and tripods as well as pans. Small jugs with evidence of use on the fire have also been identified in 11<sup>th</sup> century contexts. These jugs sometimes referred to as cups are widespread in North African sites and Iberian Islamic contexts and may have been used drink or serve milk or yoghurt (Rossiter, Reynolds and MacKinnon 2012, 259). Due to its coastal location, not only were typologies modelled on those from North-Africa, the site also received direct imports, such as a particular pan provenanced to Egypt (Meo and Orecchioni 2020; Meo forthcoming).

Figure 5 shows examples of the main types of domestic cooking containers that have been identified at the sites of Castello San Pietro (CSP), Palazzo Bonagia (PB), La Gancia (GA), Mazara del Vallo (MZ) and Casale San Pietro (CLESP) that have been analysed as part of this thesis' research.

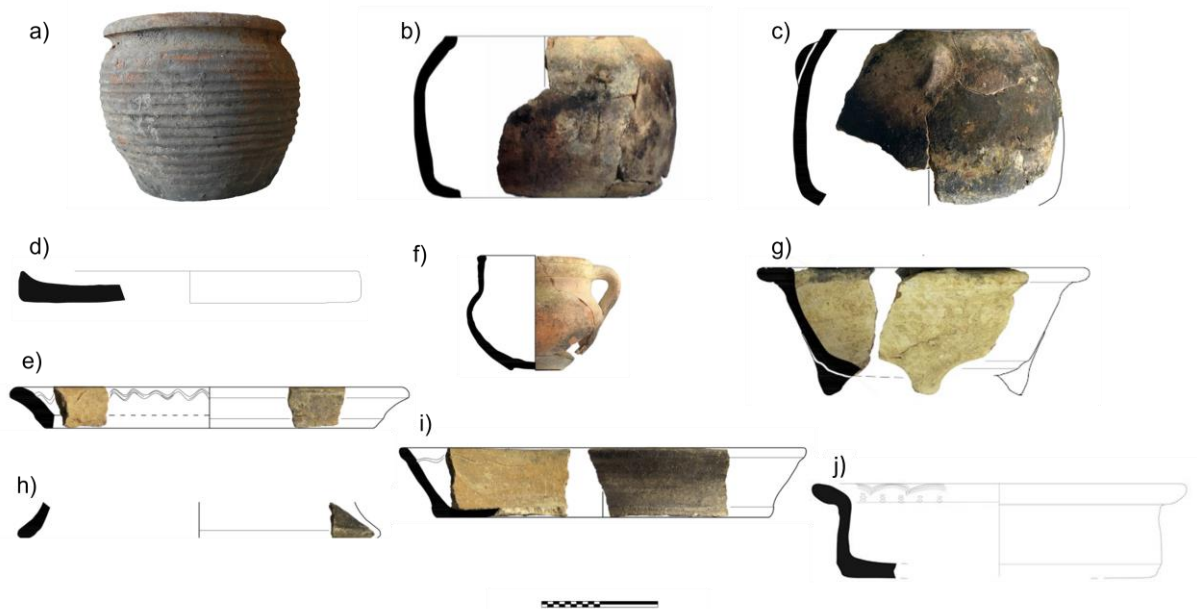


Figure 5: Examples of vessel forms from 9<sup>th</sup>-11<sup>th</sup> century contexts in Palermo, Casale San Pietro and Mazara del Vallo a) Palermo production *Olla* from Palazzo Bonagia, b) Handmade cooking pot from CLESP, c) Handmade cooking pot from Mazara, *Testello* (stone plate) from Palazzo Bonagia and e) Mazara, f) Jug from Mazara, g) Tripod from Mazara, h) Lid from Mazara, i) Pan from Mazara, j) Brazier from Castello San Pietro. (Arcifa and Bagnera 2014; Sacco 2014; Meo and Orecchioni 2020; Carver et al. 2019; Meo forthcoming),

### 2.3.2 12<sup>th</sup> century domestic containers

Although material culture of the 12<sup>th</sup> century Norman contexts in Sicily have received increased attention in recent years, the context of ceramics in this period is still not clear. This is because it is difficult to clearly distinguish ceramics of this period from the preceding Islamic contexts (Alfano and Sacco 2014; Molinari 2012). Furthermore, studies of ceramic forms in this period have predominantly focused on glazed ceramics and amphorae, and little attention has been given to domestic cooking wares. Nonetheless a number of archaeological investigations have been able to identify 12<sup>th</sup> century cooking pots at various sites across Sicily such as; Palermo (Arcifa, Lesnes and Démians D'Archimbaud 1997), Agrigento (Scuto and Fiorilla 1990), Segesta (Molinari 1997), Calathamet (Poisson and Lesnes 2013), Monte Iato (Isler 1998), Marsala (Kennet, Sjostrom and Valente 1989) and recent on-going investigations at Casale San Pietro (Carver and Molinari 2016; Carver et al. 2019). Whilst each site has its unique pottery sequence and reliance on pottery production centres, there consistently appears to be a general continuation of pottery types continued from the preceding periods such as the continuation of the most general forms such as *ollae* and cooking

pots at these sites. The remaining predominance of previous forms is not surprising given the evidence of socio-economic relations during this time. The prevalence of these types can be attributed to the fact that ceramic production largely remained in the hands of the Muslim majority (Alfano and Sacco 2014), or incoming Norman populations simply adopted the techniques and craftsmanship of their predecessors.

However, there are examples of types that do not seem to be present prior to the 12<sup>th</sup> century. For example, at Segesta, Molinari (1997) identified *Le pentole "mesinesi"* a cooking pot of Messina type that first occurs at the site in the 12<sup>th</sup> century and becomes widely in use by the second half of the century into the 13<sup>th</sup> century. These cooking pots, produced in Messina, Eastern Sicily, are considered of high craftsmanship and are recognised by the partial glazing of both the internal base and rim of the ceramics. Examples of this type were also found in Marsala (Kennet, Sjöström and Valente 1989), Calathamet (Poisson and Lesnes 2013) and Monte Iato (Isler 1998) and Mazara del Vallo (Meo and Orecchioni 2020; Meo forthcoming) (Figure 7a), depicting their use across both western and eastern Sicily.

At the site of Casale San Pietro, in the 12<sup>th</sup> century, there is a continued use of forms present in the Islamic period and the site continues to rely heavily on Palermo production pots. There is also a continued use of wheel thrown calcitic wares and slow thrown flyish wares in the 12<sup>th</sup> century. This differs to the table wares where there generally seems to be a shift in imports outside of Sicily and away from North Africa. However, towards the end of the 12<sup>th</sup> century and into the beginning of the 13<sup>th</sup> century these partially glazed cooking wares from Messina begin to enter the ceramic assemblage, in line with other sites across Sicily (Carver et al. 2019; Carver and Molinari 2016). Figure 6 depicts some of the typical pottery forms that make up the assemblage of domestic cooking wares from 12<sup>th</sup> century contexts at Casale San Pietro.

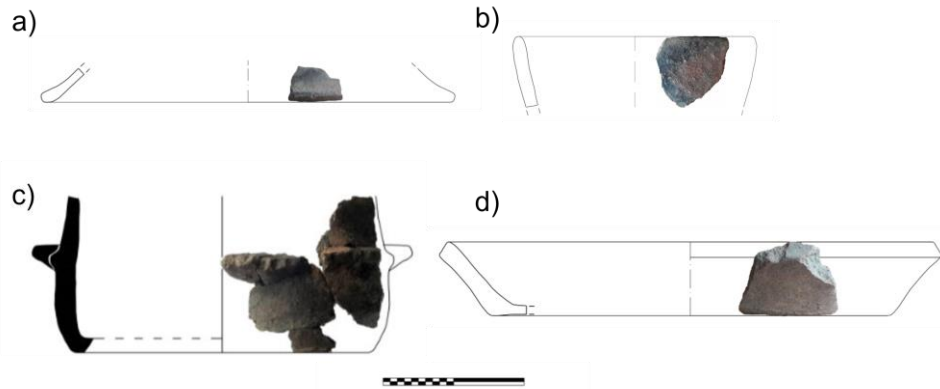


Figure 6: Examples of vessel forms from 12<sup>th</sup> century contexts at Casale San Pietro. a) Lid, b) Bowl, c) Handmade cooking pot, d) Pan (Carver et al. 2019).

### 2.3.3 13-14<sup>th</sup> century domestic containers

As noted, a significant transformation from the 12<sup>th</sup> to 13<sup>th</sup>-century cooking ware repertoire in Sicily is the high occurrence of partially glazed cooking pots from Messina. These particular wares seem to have been produced at an almost industrial scale in Messina and exported across eastern and western Sicily (Kennet, Sjoström and Valente 1989; Poisson and Lesnes 2013; Isler 1998; Molinari 2012). In the late 13<sup>th</sup> century to the 14<sup>th</sup> century AD, limited data has made it difficult to assess what happened after the end of the Swabian rule. However, it appears that the material culture reflects little of the previous transitions, including ceramic typologies. For example, Molinari (2010, 2012) suggests that the complete absence of cooking pots with handles from the late 13<sup>th</sup> century onwards in Sicily reflects an abandonment of a well rooted cultural, social, and culinary traditions from the preceding periods (Molinari 2012, 2010). A disappearance of material culture from preceding periods in the Swabian to Aragonese era seems to be in line with the shift towards the end of the 13<sup>th</sup> century from a multicultural one to a predominantly Latinized and Christian Sicily.

At the site of Mazara del Vallo, ceramic studies of 13<sup>th</sup> – 14<sup>th</sup>-century contexts indicate there is a notable shift in the types of cooking wares available at the site. Generally, there is a much-reduced variety in forms than those observed in Islamic contexts. For example, there is a reduced occurrence of pans and a total absence of stone plates and

braziers that define the Islamic ceramic assemblages. Although an example of a brazier has been identified in a 13<sup>th</sup> century context at this site, it very different from those found in preceding periods (Meo and Orecchioni 2020). Despite the reduced variability of ceramics forms, there is a coexistence of cooking pots that are both morphologically and technologically different from each other in this period (Meo and Orecchioni 2020; Orecchioni forthcoming). Of note, there is rise in glazed cooking wares, particularly partially glazed wares from Messina and later in the 13<sup>th</sup> century there is the appearance of locally produced and imported (e.g., from Spain) fully glazed wares. In contrast, unglazed hand-modelled ceramics produced locally also exist in the cooking ware assemblage (Meo and Orecchioni 2020; Orecchioni forthcoming). Figure 7 depicts some of the typical pottery forms that make up the assemblage of domestic cooking wares from 13<sup>th</sup>-14<sup>th</sup>-century contexts at Mazara.

In rural sites such as Casale San Pietro, these assemblages in the 13<sup>th</sup> century are more modest compared to those observed at Mazara del Vallo, but the transition is still distinctive. These typological distinctions are owed to the large increase of the partially glazed Messina type cooking wares and fully glazed cooking wares, generally representing a shift away from a reliance of Palermo production pottery during this phase (Carver et al. 2019; Carver and Molinari 2016).

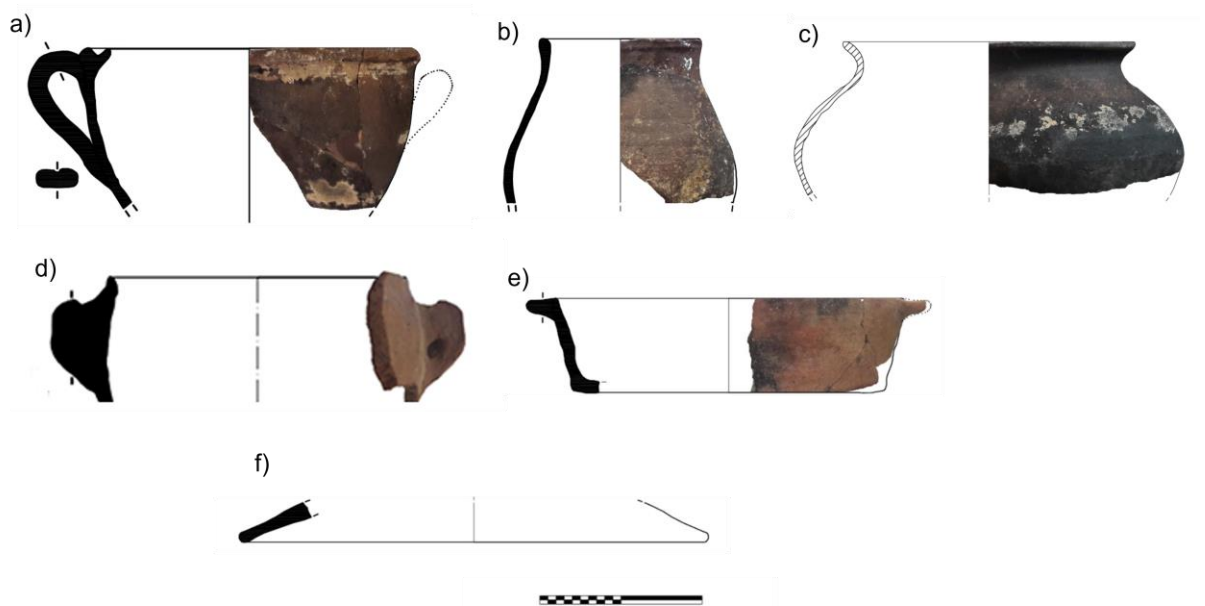


Figure 7: Pottery forms from 13<sup>th</sup>-14<sup>th</sup> century Mazara del Vallo. a) Partially glazed Messina type cooking pot, b) Partially glazed Messina type small cooking pot c) Unglazed cooking pot imported from Spain, d) Handmade cooking pot, e) Pan, f) Lid. (Meo and Orecchioni 2020; Orecchioni forthcoming).

## 2.4 Identifying the use of ceramics

As highlighted, the recovery of a large number of ceramics and increased investigations of vessel forms and typologies across Sicily has enabled clear chrono-type references to be identified as well as insight into trade patterns and socio-economic links from the 9<sup>th</sup>-14<sup>th</sup> centuries. However, recent reviews of these ceramic investigations in Sicily highlight that a major gap in our knowledge still exists in the understanding of how these ceramic containers were used, stating that this is essential for our understanding of the populations that utilised them (Testolini 2018; Kirk 2013). By understanding how these cooking vessels were used, we are able to directly link resource use and ideas of cuisine to assigned periods and, as utilitarian artefacts, ceramics are key to understanding daily consumption practices of populations. To date, the large majority of hypotheses about the use of ceramic vessels in Sicily have been formed on the basis of typological associations and any suggestions of use have been assumed based on the most logical use based on the form, shape, and size of the ceramic. However, these approaches usually rely on several assumptions and often supposed uses of objects in the archaeological record are based on contemporary analogies which can lead to misinterpretations (Allison 2013). Some insight can be gained about the use of ceramic vessels from recipes in contemporary cookbooks, but often this is limited due to the wide variety of names used for the same type of pot or the same name given to a wide variety of different types such as the *ollae* for example (Donnelly 2016). Furthermore, use wear analyses have also been used to understand how ceramics might have been used. For example, evidence and placement of soot can indicate their use as cooking vessels as opposed to storage vessels (Skibo 2015; Meo and Orecchioni 2020). However, these types of studies offer little information about the types of commodities that were processed in these vessels and therefore should be used in conjunction with other approaches.

The development of organic residue analysis of ceramic vessels has greatly improved our understanding of the use of archaeological vessels and is one of the few ways in which to systematically investigate their contents (Reber et al. 2019a) Organic residue

analysis (ORA) of absorbed residues in ceramics vessels can give direct chemical evidence of their contents and thus offers a unique insight into the types of foodstuffs that were prepared and combined by the communities that utilised them. If the ceramic typology and chronology is well defined, ORA offers an opportunity to link material culture of a defined chronological period with consumption patterns of the populations at the time. Significant developments in ORA studies have enabled the identification of numerous organic products in archaeological vessels and has yielded important insight into culinary habits in a broad range of geographical and chronological contexts. Whilst a number of studies have used ORA to assess the use of medieval pottery vessels, large scale studies have mainly focused on ceramic studies in the UK (Craig-Atkins et al. 2020b; Dunne et al. 2019, 2020). Those applied in medieval Mediterranean contexts remain small and focus on Northern and Central Italy (Giorgi,Salvini and Pecci 2010; Salvini,Pecci and Giorgi 2008; Buonincontri et al. 2017; Pecci and Grassi-Munibe 2016; Pecci et al. 2016) and only one study has used ORA to assess the use of a small sample of stone plates from 10<sup>th</sup> century Sicily (Lucejko et al. 2018). To the best of the knowledge of this study, there have been no large-scale organic residue studies of medieval Sicilian ceramics and no large-scale studies of cuisine in the medieval Mediterranean as a whole. Therefore, this thesis presents the first large scale study of organic residues from medieval Sicilian cooking pots to understand their use during the 9<sup>th</sup>-14<sup>th</sup> centuries.

## **2.5 Summary of chapter**

This chapter has outlined the historical context of the 9<sup>th</sup>-14<sup>th</sup> century as it relates to the focus of this thesis. It has introduced the concepts of cuisine, its importance to the study of past populations and the current state of knowledge of cuisine in medieval Sicily. A discussion of ceramic studies of domestic containers in 9<sup>th</sup>-14<sup>th</sup> century contents in Sicily has been given and this chapter has highlighted the rationale for the study of their use. The benefit of organic residue analysis to the study of pottery use has been briefly outlined and the following chapter will provide a detailed discussion of organic residue analysis of archaeological ceramic containers.



# **Chapter 3 Organic residue analysis of ceramic containers**

## **3.1 Introduction to chapter**

This chapter outlines the main concepts of the study of organic residues of archaeological ceramics, which is the focus of this thesis. Firstly, the definitions and characteristics that underlie organic residues in archaeological contexts are outlined, followed by specific definitions of lipids associated with archaeological ceramics. The fundamental molecules that are identified through organic residue analysis of archaeological ceramics are defined and the methods for extracting and analysing these are discussed, with focus on the methods and techniques used in this study. Furthermore, the common contents in ceramic containers that are interpretable, based on molecular and isotopic compositions using organic residue analysis, are outlined. Finally, this chapter discusses some of the interpretive limitations and important considerations in the study of organic residue analysis of absorbed residues in archaeological ceramics, particularly in medieval Mediterranean contexts.

## **3.2 Introduction to organic residues**

In archaeological studies, the term organic residue has been used to describe a wide variety of amorphous organic remains (Evershed 1993). 'Organic' refers to anything derived from living organisms and, in chemical terms, refers to material composed of carbon and hydrogen (whereby most 'organic' compounds contain at least one carbon-hydrogen bond) as well as oxygen, nitrogen, sulphur, silicon, phosphorus, and halogens. Organic compounds therefore encompass a variety of structures with different functions such as DNA, proteins, carbohydrates and lipids (Evershed 1993; Regert 2007). The term amorphous separates organic residues from other organic remains found at archaeological sites, such as bone, wood, leather, and plant remains, in that these describe organics that do not have morphological features that can be discerned visually. Organic residues are preserved in various contexts in the

archaeological record and include a range of unaltered and manufactured natural products such as animal fats, waxes, plant oils, fermented beverages, pigments and tars (Heron and Evershed 1993; Regert 2011, 2007). Organic residues can occur on their own, but in the archaeological record are most commonly associated with organic (e.g., bone, wood, leather, sediments, and coprolites) and inorganic matrices (e.g., ceramics, lithics, and metals). The development of archaeological science in the last 40 years has enabled the identification and analysis of a wide range of organic residues in the archaeological record by extracting and analysing a variety of amorphous structures. Studies of organic residues in archaeological contexts have greatly expanded the breadth of archaeological questions that can be answered by unveiling information previously invisible, but highly important to our understanding of the past. In this chapter, due to the scope of this project, only organic residues associated with ceramics vessels are discussed and, from this point forward, the term organic residues refers only to those extracted and analysed from ceramic vessels unless otherwise stated.

### **3.3 Organic residues and ceramic vessels**

During the processing of foods and other organic commodities such as those for tars, glues and waterproofing agents, organic residues (the remnant of organic commodities in the form of DNA, proteins, carbohydrates and lipids) can adhere to ceramic matrices in the form of surface residues (also known as visible residues or food crusts) and/or absorbed residues (Evershed 2008b). Surface residues are deposited on the surface of the ceramic wall as carbonised crusts, usually through the processing of organic products using heat (cooking) or as visible sealants, adhesives or organics used as decorations. Surface residues are best preserved when charred as they are protected from microbial degradation by a carbonized layer. Studies of visible residues associated with ceramics have yielded important insight into the use of ceramics, in particular they are more likely to be related to a single use and can provide information about intentional mixtures or recipes (Miller et al. 2020). However, visible surface residues do not always survive in the archaeological record as they are prone to degradation and contamination in the burial environment or can easily be lost or contaminated during post-excavation processes such as cleaning and handling (Roffet-

Salque et al. 2016). Due to the porous nature of ceramic matrices, residues can also be absorbed into the ceramic vessel as they are released from the organic material through processes such as cooking, storing, serving, and crushing. Researchers have aimed to understand the complex processes of how these residues are absorbed into the porous ceramic matrix and how they can remain there for thousands of years. Whilst still not fully understood, it has been shown that the residues can either chemically bind to the ceramic matrix or be trapped in the pores, of which both scenarios likely exist (Evershed 1993, 2008b; Heron and Evershed 1993; Heron, Evershed and Goad 1991; Drieu et al. 2019). The bonds formed between these residues reduces migration of the residues from the ceramic and the ceramic matrix itself protects them from microorganisms (Evershed 1993). Thus, absorbed residues are representative of the original contents of the vessel. Significant development in archaeological sciences has meant that absorbed organic residues associated with ceramic vessels are most commonly analysed. As stated previously, organic residues embody a range of amorphous structures, such as DNA, proteins, carbohydrates, and lipids. However, lipids are the most commonly targeted group studied in organic residue analysis of archaeological artefacts as they are less susceptible to post burial loss and alteration than other structures (Evershed, Heron and John Goad 1990). As no visible residues were identified in this study, this thesis focuses only on absorbed lipid residues.

### **3.4 Lipid residues**

Lipids are most commonly referred to as fats, but also include oils, waxes, sterols, glycerol, phospholipids, and terpenes, and are a group of naturally occurring organic molecules and compounds that are insoluble in water, but soluble in organic solvents. Lipids occur naturally in a variety of foodstuffs and other organic materials such as animal fats (adipose and dairy), marine and freshwater aquatic, plant, and insect products. Lipids mainly consist of carbon, hydrogen and oxygen and carry different chemical properties dependant on their structure and the arrangement of these atoms around the carbon core, into either linear, branched or cyclic (Heron and Evershed 1993). Due to the high proportions of hydrocarbon moieties, the most defining

chemical property of lipids is their nonpolar properties and hydrophobic nature, which aids their survival in the archaeological record as they are insoluble in water and are therefore less prone to leaching and exchanges with surrounding sediment in the burial environment (Heron, Evershed and Goad 1991; Evershed 1993). Lipids are made up of a variety of compounds with different functional groups, such as triacylglycerols, wax esters, fatty acids, alcohols, and alkanes. Triacylglycerol and wax esters, although dominant in modern organics, are susceptible to degradation and alteration, through the chemical processes of hydrolysis or saponification, breaking these compounds down into their simpler constituents: fatty acids, alcohols, and alkanes, which are more commonly observed in the archaeological record. A brief summary of the structure of these lipids is detailed in the following sections (3.3.1-3.3.7).

### **3.4.1 Fatty acids**

Fatty acids (FAs) are carboxylic acids with either saturated (Figure 8a and b) or unsaturated (Figure 8c) aliphatic chains, constituting a straight hydrocarbon chain and a terminal carboxyl group. Naturally, they occur predominantly as even, unbranched carbon chains, but odd-chain and branched fatty acids do exist naturally, as they are synthesised by microorganisms (Christie 1989). FAs are often depicted in the shorthand nomenclature  $C_{x,y}$ , in which  $x$  refers to the number of carbons in the aliphatic chain and  $y$  indicates the number of unsaturation. FAs are classified into short, medium, or long-chain dependent on the number of carbons that make up their aliphatic chain. As the carboxyl group at the end of the carbon chain is polar and reacts with water, the shorter the fatty acid chain, the higher the degree of solubility of the fatty acid.

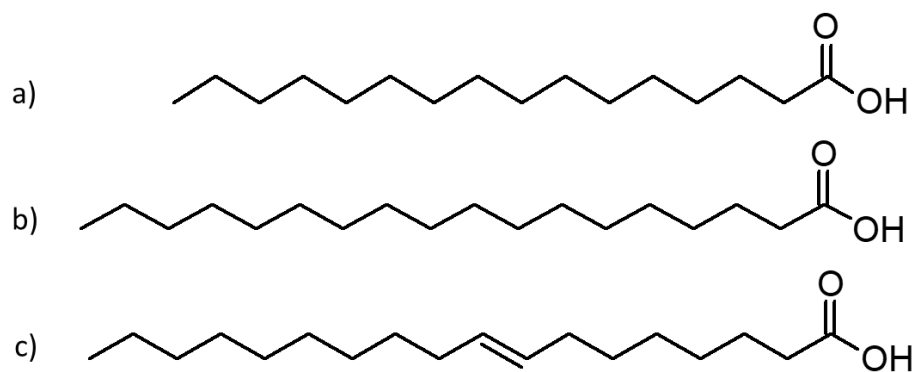


Figure 8: Examples of fatty acids (FAs). The structure of two saturated fatty acids a) palmitic acid ( $C_{16:0}$ ) and b) stearic acid ( $C_{18:0}$ ) and a monounsaturated fatty acid c) oleic acid ( $C_{18:1}$ ) are shown.

FAs are the most commonly occurring lipids identified in archaeological ceramics. However, these rarely occur freely in nature and are instead usually bound to other molecules, existing as bigger structures such as triacylglycerols or wax esters (3.4.2 and 3.4.5).

### 3.4.2 Triacylglycerols

Triacylglycerols (TAGs) are major components in plants and animal organisms, representing over 95% of the lipids in our diet (Oudemans 2007). TAGs are esters that are derived from a glycerol 'backbone' with three hydroxyl groups. These hydroxy groups are attached to three fatty acids, either saturated or unsaturated, of different carbon chain lengths, via an ester bond (Christie 1989). The nature of these fatty acid chains, which can either be the same or different in the same TAG structure, can indicate the TAGs original source. This is because the structure and nature of TAGs is dependent on various metabolic processes of different organisms (Evershed 2008b). TAGs are hydrolysed when exposed to water and this process is catalysed when TAGs are exposed to heat and an alkaline or acidic medium. Therefore, TAGs are susceptible to hydrolysis of the ester bonds as a result of the processing of the organics in which the constituents derive predominantly through heating during the cooking process or during burial (Aillaud 2001). These reactions result in the formation of free fatty acids (FFAs), monoacylglycerols (MAGs) and diacylglycerol (DAGs). This happens in a chain reaction whereby the TAGs first lose a FA chain, yielding a diacylglycerol (DAG) and a free fatty acid (FFA), the DAGs then degrade further to yield a monoacylglycerol (MAG)

and a FFA and finally degrading further leaving glycerol and a FFA (Evershed et al. 2002)(Figure 9).

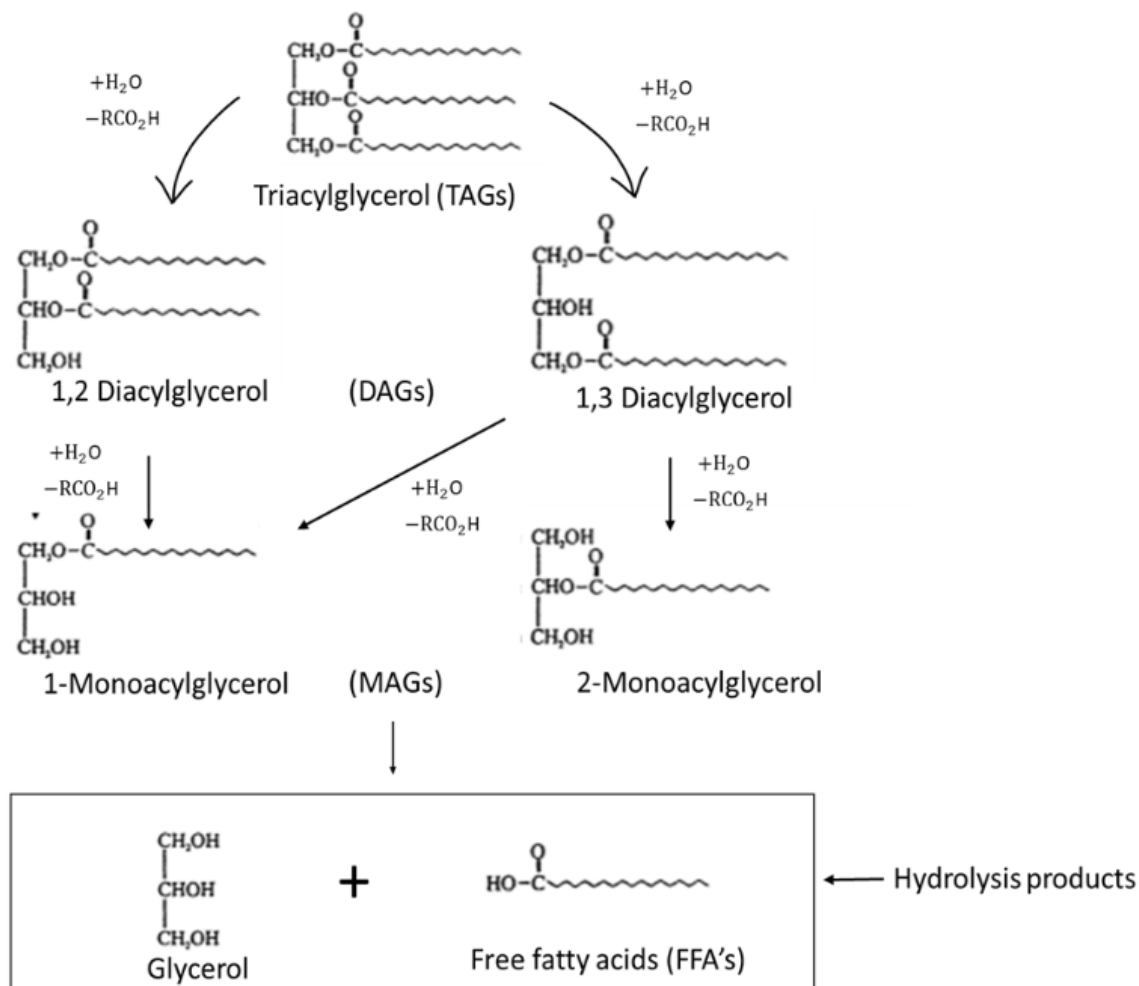


Figure 9: Pathway of the hydrolysis of TAGs to FFAs adapted from (Evershed et al. 2002)

### 3.4.3 Alcohols

Linear alcohols (*n*-alcohols), sometimes referred to as *n*-alkanols, are composed of a straight aliphatic chain with a terminal hydroxyl group. Often alcohols are bonded to other molecules via an ester bond to form other molecules such as wax esters, but they also occur freely in low abundances in tissues of living organisms (Christie 1989).

### 3.4.4 Alkanes

Alkanes are composed of a straight chain or cyclic saturated hydrocarbon chain with no functional group. Acyclic alkanes (*n*-alkanes) with an odd number carbon chain are major constituents in waxes and they occur widely in plant and insect (i.e. beeswax) waxes (Eglinton and Hamilton 1967; Roffet-Salque et al. 2015; Tulloch 1971).

### 3.4.5 Wax esters

Wax esters are esters formed of fatty acids and long-chain alcohols bonded through an ester bond (Figure 10). Both the alcohol and fatty acid chains are predominantly straight chain, saturated or monounsaturated, but wax esters also exist as branched or hydroxyl chains. Through hydrolysis the ester bond is broken to release FFAs and alcohols.

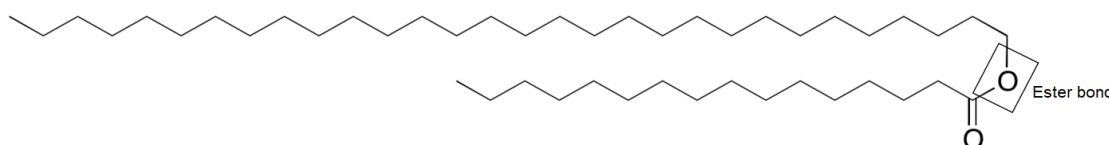


Figure 10: Triacontanyl palmitate an example of a typical wax ester showing the ester bond.

### 3.4.6 Sterols

Where animal and plant lipids are mainly composed of TAGs, sterols are considered a minor component of animal (zoosterol) and plant (phytosterol) lipids. Sterols are naturally occurring unsaturated steroid alcohols and can be specific to each commodity and perform different functions in the organism. Cholesterol is the most common zoosterol observed in archaeological ceramics.  $\beta$ -Sitosterol, stigmasterol and campesterol are the predominant phytosterols, whereas ergosterol comes from bacteria or fungi in various contexts (Figure 11).

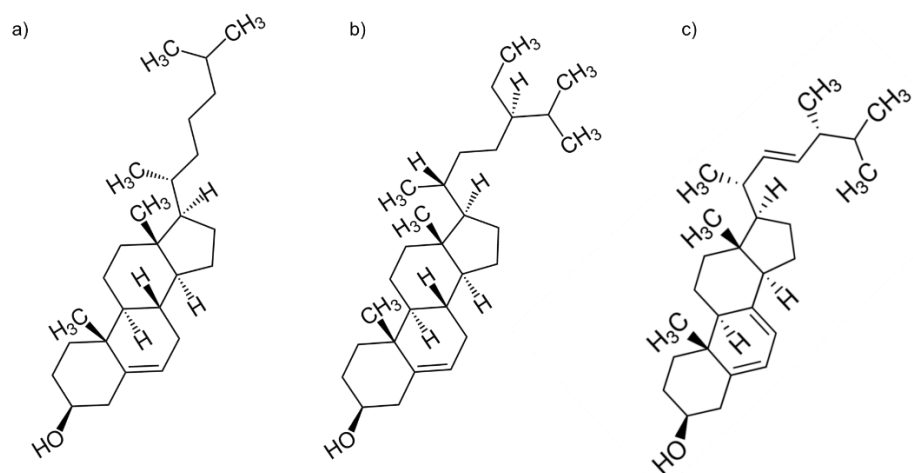


Figure 11: Structures of sterols found in a) animal tissue, cholesterol, b) plant tissue,  $\beta$ -Sitosterol and c) bacteria or fungi, ergosterol.

### 3.4.7 Terpenes

Terpenes can be described as non-saponifiable lipids or 'simple lipids' (Christie 1989). Terpenes are formed from the polymerization of isoprene and are classified into mono-, di-, ester- and triterpenes according to the number of carbon atoms presents or the number of isoprene units it has (2, 3, 4, 5 and 6 respectively) (Connolly and Hill 1991). Terpenes are a wide class of natural products found extensively in higher plants and exist in various arrangements and cyclisations that dictate their various functions (Evershed 1993).

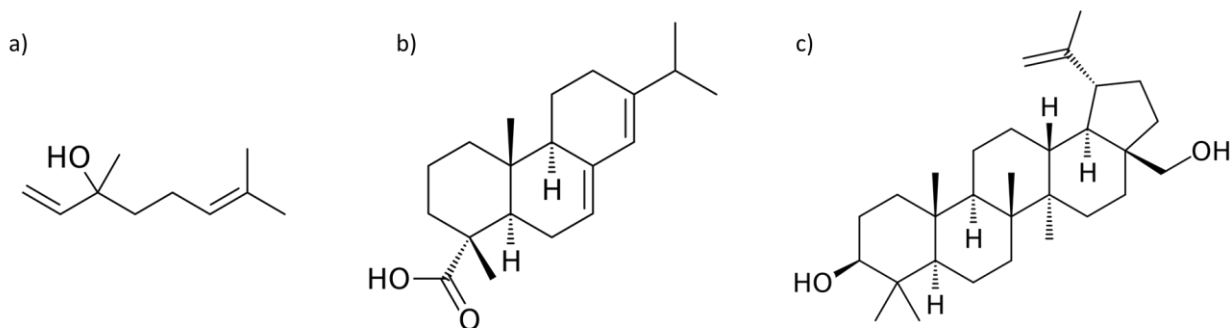


Figure 12: Examples of terpenoid structures a) the monoterpene structure linalool, b) the diterpene structure abietic acid and c) the triterpene structure betulin



## **3.5 Recovering absorbed lipids from archaeological ceramics**

As mentioned, lipids are soluble in organic solvents and a number of methods have been employed to remove these lipids from the ceramic matrix using different organic solvents. The way that lipids bind to the ceramic matrix differs depending on their structure. As a result, lipids have various levels of protection from the ceramic matrix and solubility in organic solvents (polar or non-polar and bound). Thus, the use of different solvents and extraction methods are required dependent on the lipids targeted.

### **3.5.1 Sample preparation**

To prepare the sample, conventionally the top layer (~1mm from the surface) is first removed to avoid exogenous contamination from the burial environment and post-burial contamination such as handling. However, some studies have avoided removing this outer surface layer as important information may be lost (Stern et al. 2000). To increase the surface area and contact between the solvent and the organic residue, usually a sample of ~1g of ceramic powder is collected from the ceramic sherd by drilling into the matrix, although the mass of ceramic powder used has varied in studies from as high as 100g (Condamin et al. 1976) to a little as 0.1 g (Papakosta et al. 2015; Stern et al. 2000). Furthermore, in some extraction methods, the powdering of the ceramic is not necessary and can be analysed with lesser degrees of destruction (Devièse et al. 2018; Gerhardt, Searles and Biers 1990). However, it is important to note that any alteration of the ceramic matrix is always, to some degree, considered as destructive.

### **3.5.2 Extraction methods**

Total lipid extraction (TLE) or solvent extraction (SE), as it is commonly referred to, conventionally uses a chloroform/methanol or dichloromethane solution as the solvent to remove organics from the ceramic matrix. Extracts need to undergo derivatization before they are analysed through gas-chromatographic techniques in

order to decrease the polarity of the functionalised molecules and to increase their volatility (Řezanka, Pádrová and Sigler 2016). Since the method was first proposed by Bligh and Dyer in 1959, and later applied to archaeological ceramics (Evershed, Heron and John Goad 1990; Bligh and Dyer 1959; Condamin et al. 1976), it has remained relatively unchanged for decades. However, now dichloromethane is the preferred solvent as it is less toxic than chloroform (Lucquin et al. 2016a). TLE enables the extraction of major components such as TAGs, DAGs and MAGs, FAs, wax esters, *n*-alkanes, *n*-alcohols, terpenes, and sterols. However, TLE can be limited as the solvent is unable to extract very polar, such as diacids and dihydroxy acids and strongly 'bound lipids' such as FAs and alkanols that have bound to the ceramic matrix or each other through mechanisms that are not yet well understood (Correa-Ascencio and Evershed 2014; Regert et al. 1998). This can result in low lipid yields which cannot be reliably interpreted (Evershed 2008b; Evershed et al. 1999). Furthermore, the SE method can be time consuming and requires further steps to be analysed using Gas Chromatography- combustion-Isotope Ratio Mass Spectrometry, as discussed later in this chapter (GC-c-IRMS).

A method for extracting strongly bound lipids was developed and widely used in the early 2000's (Regert et al. 1998). However, this extraction method required SE prior, which was time consuming. Correa-Ascencio and Evershed (2014) have since developed the direct acidified methanol extraction, commonly referred to as acid extraction (AE), as a more efficient extraction technique of organics from archaeological potsherds (Correa-Ascencio and Evershed 2014). The use of an acidic reagent such as sulphuric acid enables intermolecular bonds to be broken between lipids (even those strongly bound polar bonds) and the ceramic matrix. By applying AE method, yields recovered from ceramic powder using this technique were much higher than those previously extracted using conventional solvent extraction as these more polar compounds and bound lipids (e.g., diacids, dihydroxy acids, and bound FAs and alkanols etc.) could be recovered. Neutral compounds such as terpenes, sterols and alkanes are also recovered either comparably or in some cases more effectively than TLE (Reber 2021). Furthermore, due to the direct methylation of compounds, further treatment to derivatize the extracts before GC analysis is not required for FAs, but due to the polar nature of alcohols and dihydroxy fatty acids, these extracts need to be

further derivatized to analyse them. Thus, due to its ability to extract higher lipid yields and high-throughput, acidified methanol extraction has been widely applied in ORA studies of archaeological ceramics (Miller et al. 2020; Correa-Ascencio and Evershed 2014; Hammann et al. 2020; Craig et al. 2007; Reber 2021; Craig et al. 2013a). Importantly, the use of AE has enabled the extraction of appreciable lipid yields from areas that commonly report poor lipid preservation such as the Mediterranean (Fanti et al. 2018; Drieu et al. 2021a) and North Africa (Dunne et al. 2018). However, due to hydrolysis and methylation of more complex lipids such as TAGs, MAGs and DAGs and wax esters into their constituent acids and alcohols, important compositional information is lost. Therefore, it is advised that AE and TLE, where possible, should be used in combination to ensure that the maximum information is extracted from the ceramic samples (Hammann et al. 2020).

Researchers, with varying success, have explored several other attempts to create a more efficient and effective extraction technique of lipids. These include, Soxhlet (Condamin and Formenti 1978), sonication and saponification (Mukherjee, Gibson and Evershed 2008), and accelerated solvent extraction of sediments (Hughen et al. 2004; Jansen et al. 2006). Furthermore, Gregg and Slater (2010) investigated the use of microwave-assisted extraction to increase the yield of lipids extracted (Gregg et al. 2009; Gregg and Slater 2010). The use of supercritical fluids as an alternative extraction method to conventional solvent extraction has also been investigated (Devièse et al. 2018). This method yielded higher concentrations of lipids in comparison to conventional solvent extraction methods and can be applied to whole sherds (Devièse et al. 2018). Other studies have also investigated the potential for 'non-destructive analysis' of ceramic sherds, for example by using chloroform followed by methanol in a complete vessel (Gerhardt, Searles and Biers 1990). However, there is doubt surrounding these types of extractions due to possible exogenous contaminations on the surface of the vessel and as mentioned previously, the addition of solvents to the ceramic matrix involves some degree of destruction although the vessel might remain structurally intact.

Small organic compounds, such as tartaric acid and malic acid, are not easily extracted using conventional solvent extraction methods or acidified methanol extraction as

they are highly polar and strongly bound to the ceramic matrix. Several methods have been employed to aid the extraction of these small organic compounds, in particular tartaric acid which is widely used as a biomarker of grapevine/wine products. These include, various different alkaline treatments (Condamin and Formenti 1978; Pecci et al. 2016; Pecci, Cau Ontiveros and Garnier 2013; Guasch-Jané et al. 2004, 2006; Pecci et al. 2013), acid extractions (Garnier and Valamoti 2016) and water extractions (Zhang et al. 2018), albeit the latter with little success. With minimal consensus amongst researchers regarding which methodology should be applied, Drieu et al., (2020) compared the two most commonly applied methods- ‘alkaline fusion’ (Guasch-Jané et al. 2004; Pecci et al. 2013) and acidified butanol extraction (Garnier and Valamoti 2016). It was concluded that the latter of these two techniques was far more successful in the extraction of tartaric acid (Drieu et al. 2020)(Table 1).

Table 1: Comparison of alkaline fusion (Guasch-Jané et al. 2004; Pecci et al. 2013; Garnier and Valamoti 2016) and acidified butanol extraction (Garnier and Valamoti, 2016) of tartaric acid. Taken from (Drieu et al. 2020)

	Quantification by GC-MS	
	KOH (1M) treatment Extraction in Ethyl acetate TMS derivatisation <sup>1</sup>	BF <sub>3</sub> /BuOH in hexane Extraction with DCM <sup>2</sup>
Derivatives	TMS	Bu
Pure tartaric acid 400 µg	0.01 µg	308.03 µg
White wine (Pinot Grigio, 2017 Trentino, Italy), 100 µL	0.00 µg	103.85 µg
Red wine 1 (Shiraz, House by Sainsbury's, South Africa), 100 µL	0.12 µg	162.65 µg
Red wine 2 (Torretta di Modelli, Sangiovese, Puglia, Italy), 100 µL	1.24 µg	163.07 µg
Red wine 3 ( <i>vino da tavola</i> , personal production, Castronovo di Sicilia, Italy), 100 µL	0.00 µg	134.2 µg

The acidified butanol extraction requires lipids to first be removed from the ceramic powder using TLE. Thus, although using this method requires further extraction, which can be time consuming; it is highly beneficial if performing TLE. The recovery of small acids can provide important information about the contents of ceramic containers not observable using other extraction methods. This is particularly important in medieval Mediterranean contexts where grapevine and other fruits

products might have been processed in ceramic vessels and incorporated into cuisines.

To maximise the information obtained from the ceramic samples in this study, a multifaceted extraction method was used, whereby solvent extraction, acid extraction and acidified butanol extraction was applied, where possible, to all samples. These methods are summarised in Appendix A and the full standard operating procedures and protocols used in this thesis' research are outlined in Appendix B.

## **3.6 Identifying and interpreting lipids in archaeological ceramics**

After extraction, the amorphous organic residues need to be analysed to identify the molecular structures. Multiple chromatographic techniques have been used to separate the complex mixtures of compounds extracted from the ceramic matrix and mass spectrometry is commonly used to identify the structure of these compounds. Once these structures have been identified, two main approaches are used to interpret the residues and assign them to an original organic product. These approaches are the 'Biomarker approach' and the 'Compound specific isotope approach', which are outlined in the following sections.

### **3.6.1 Biomarker approach**

To identify molecules in the organic residues, the extracts are commonly analysed using Gas Chromatography (GC) and Gas chromatography- Mass Spectrometry (GC-MS) techniques. The GC separates different compounds as they interact with the stationary phase (that coats the column). As they react differently, this allows them to elute at different times, known as retention times. By analysing standards of known retention time, it is possible to identify the compound, based solely on the retention time (Evershed, 1992b). More commonly, the structure of the compound is identified by coupling gas-chromatography with mass spectrometry (GC-MS). This allows the

compounds to separate as they interact with the stationary phase and then to be identified based on their molecular mass and fragmentation pattern. Different methods and columns can be used depending on the types of molecules that need to be analysed and the resolution required. For example, TAGs have a high molecular weight and are not very volatile, and therefore need a high temperature programme to ensure that the TAGs are volatilized. Furthermore, Selected Ion Monitoring (SIM) acquisition methods can be used to identify specific compounds at higher sensitivity. These methods are often used in conjunction with a longer more polar column to allow for better separation of certain compounds (Whelton et al. 2021). Numerous studies, over the last decades, have successfully identified and defined lipids found within archaeological ceramics and related them back to their original sources. This can only be done by understanding how these molecules have been altered during the processing of products and as a result of dispositional degradation.

The determined structures of these compounds or collection of compounds which originate from plants or animals are known as biomarkers which are essentially molecular 'fingerprints' that can be directly related to the source that they derived from (Philp and Oung 1988; Evershed 2008b). Biomarkers refer to native molecules that can be directly related to their natural sources, but in archaeological contexts organic residues often undergo modifications through natural processes in the burial environment or anthropogenic activities which can alter their original chemical composition. These markers are referred to as degradation markers (either encompassing anthropogenic transformation markers or natural degradation markers). In archaeological samples it is also important to consider contamination markers and to be able to distinguish these from markers of the original source of content. These markers can be defined as:

- Natural degradation markers: Natural decay can lead to the transformation of the initial molecular structure of biomarkers. Transformations occur through chemical and biochemical processes during the use of the pottery, in the burial environment or post- excavation lifetime. These molecules can still inform us of the original content of the ceramic vessel through an understanding and

consideration of these processes and can also yield important information about the conditions the pottery vessel has been subject to.

- Anthropogenic transformation markers are formed through chemical transformations induced through anthropogenic conditions such as thermal treatments. These markers can inform us not only about the original content of the ceramic vessel, but also about the treatment of these products such as cooking practices or manufacturing processes.
- Contamination markers are exogenous compounds that can be transferred to the ceramic matrix during burial or during post-excavation activities and handling. Exogenous compounds can migrate from the burial environment to the archaeological record. However, migration of lipids during burial has been found to be negligible (Rottländer, 1990; Heron et al., 1991a) and, as mentioned previously, it is general practice to remove the outer layer as it is a possibility that close contact with organic materials such as animal tissue may have an effect. If transferred to the matrix, these contaminants can cause issues as exogenous contaminants can be similar to those related to original contents of the vessel. Exogenous contamination can also be transferred to the ceramic through handling and packaging of samples during post excavation. Lipids from human skin such as cholesterol and squalene are the most commonly identified. However, as cholesterol is also common in animal fats it can be difficult to distinguish between content of the vessel and exogenous contamination. As squalene is highly sensitive to degradation and unlikely to survive in archaeological samples, when found together with cholesterol, this is often attributed to contamination. Phthalate plasticisers are also commonly found, and these come from storing samples in plastic. Furthermore, post excavation treatments of ceramics such as, adhesives, inks or varnishes can have a significant impact to the molecular signals observed. It is of upmost importance to carefully examine the sherd prior to sampling and analysis and avoid parts of the ceramic that may have been treated.



### 3.6.2 Compound specific isotope approach

A major advance in the analysis of organic residues from ceramics comes from the development of Gas Chromatography-combustion-Isotope Ratio Mass Spectrometry (GC-c-IRMS) (Matthews and Hayes 1978). Since the 1990's this technique has been developed and applied to archaeological samples (Evershed et al. 1994, 1997a). GC-c-IRMS first separates individual molecules from complex mixtures and enables the  $^{13}\text{C}/^{12}\text{C}$  stable isotope ratios from specific molecules to be measured. In brief, stable isotopes are atoms of the same element (e.g., carbon) with the same number of protons, but a different number of neutrons. A difference in the number of neutrons means that an isotope of the same element will have a different mass. The mass of an isotope influences the way in which it behaves during both chemical and physical processes. This effect is called isotope fractionation, where the relative proportions of isotopes (isotope ratios) of the initial substrate are different in the product of a reaction (DeNiro 1987). Isotope ratios are expressed as delta ( $\delta$ ) in parts per mil (‰) and are calculated by measurement of the heavier to lighter isotope with reference to an international standard specific to the element being measured. Isotope ratios are measured and expressed using a universal standard to create a notation that is easy to use and was first created by geochemists. In archaeological ceramics, stable isotopes of carbon are predominantly targeted. The ratio of  $^{13}\text{C}/^{12}\text{C}$  is calculated relative to the internal standard V-PDB (Vienna Pee Dee Belemnite) (Rieley 1994; Woodbury et al. 1995). This expression is defined by the following equation (Equation 1) where  $R = \frac{\delta^{12}\text{C}}{\delta^{13}\text{C}}$ .

Equation 1

$$\delta^{13}\text{C}_{\text{‰}} = \frac{R_{\text{Sample}} - R_{\text{Sample}}}{R_{\text{standard}}} \times 1000$$

Compound specific isotope analysis can be used in organic residue analysis of archaeological ceramics to calculate isotope ratios of different compounds to distinguish between original food sources in the vessels. This is done by utilising basic



isotopic principles of plant photosynthesis and animal digestive systems, which result in differential isotope ratios. Differential mechanisms of photosynthesis in plants results in different degrees of fractionation of atmospheric CO<sub>2</sub> (Smith and Epstein 1971). These mechanisms differ according to the type of plant C<sub>3</sub>, C<sub>4</sub> and Crassulacean Acid Metabolism (CAM) (O’Leary 1981). C<sub>3</sub> plants include trees, grasses and crops like wheat, barley and rice and make up around 85% of all plant species. In contrast, C<sub>4</sub> plants are better adapted to more arid climates and include crops like millet, sorghum, maize, and sugar cane. Different mechanisms, or isotopic fractionation, results in distinct carbon stable isotope ratios between C<sub>3</sub> and C<sub>4</sub> plants(O’Leary 1981) (Figure 13).

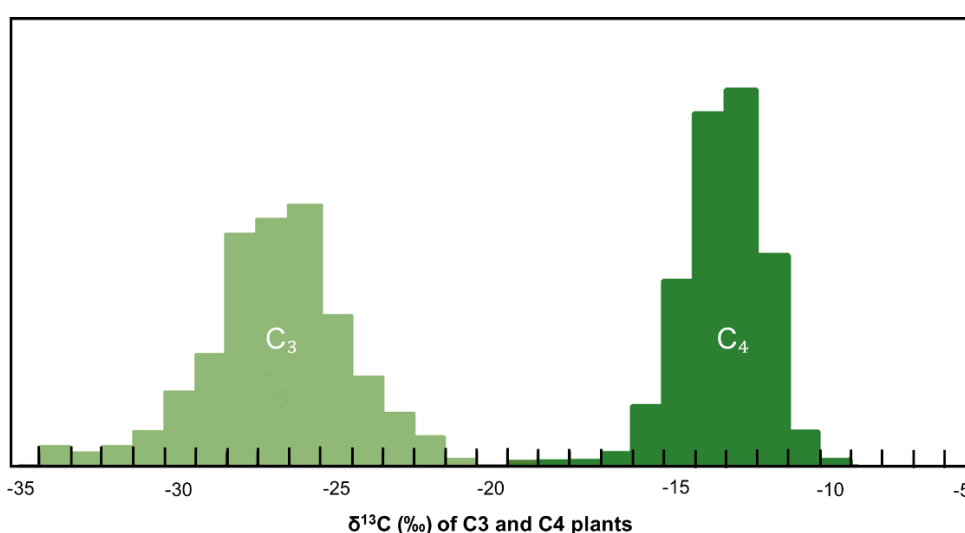


Figure 13: δ<sup>13</sup>C (‰) values of modern terrestrial C<sub>3</sub> and C<sub>4</sub> plants adapted from (Evershed 2009) whereby original values for the chart were taken from (O’Leary 1981)

In the tissues of the consumers of plants, carbon isotopic fractionation is small (~1-2%), and therefore can be directly linked to the plants consumed (DeNiro and Epstein 1978; Regert 2011). However, differences in the digestive mechanisms of ruminants and non-ruminant animals means that they exhibit different carbon isotope ratios. Therefore, GC-c-IRMS can help to determine the organic source of residues from a ceramic by measuring carbon isotope ratios. GC-c-IRMS analysis has been used to distinguish freshwater and marine animals, terrestrial animal fats and plant fats. Furthermore, ruminant, non-ruminant adipose fats and ruminant dairy products can be differentiated based on these values, as discussed in section 3.7.3.

## **3.7 Organic products identified in archaeological ceramics**

The interpretation of organic residues and the assignment of the original, specific resources processed in archaeological ceramics using the biomarker approach and compound specific isotope analysis has yielded direct chemical evidence for the contents of archaeological ceramics from various geographical and chronological contexts. However, the direct comparison of archaeological organic residues and contemporary products is not straightforward. Complex mixtures and alteration of organic residue structures by natural decay, anthropogenic processes and possible exogenous contamination all need to be considered. Here, a number of organic products routinely identified in archaeological ceramics, with particular focus to those that may be expected to be found in Mediterranean medieval contexts, as is the focus of this thesis, are discussed. How these commodities can be identified through organic residue analysis using both the biomarker and isotope approach are discussed.

### **3.7.1 Degraded terrestrial animal fats**

The most commonly observed product in the archaeological record is degraded animal fats. This is predominantly because animal adipose tissues contain high concentrations of fats, but also owes to their importance as a food source in past human societies. Several studies have investigated the exploitation of animal products in ceramic containers, either by processing of the carcass or the utilisation of secondary products such as dairy products (Dudd and Evershed 1998; Evershed 2008b; Copley et al. 2005c; Dunne et al. 2012; Spiteri, Gillis and Roffet-Salque 2016). A key question in archaeological culinary studies is to determine what types of animal fats were selected for processing in the ceramic vessels.

Saturated, even number TAGs are the main components of non-degraded animal fats constituting > 90% of the total lipid content (Regert 2011). Non-degraded adipose fats of different animal origin (e.g., non-ruminant and ruminant adipose) can be distinguished based on the distribution of TAG profiles as well as dairy fats (Dudd 1999; Dudd and Evershed 1998; Kimpe, Jacobs and Waelkens 2002; Regert 2011).

Figure 14 shows the TAG distributions that have been obtained from a variety of modern reference fats for both ruminant (cow, lamb, sheep) and non-ruminant (pig, goose, chicken) adipose fats. Generally ruminant fats exhibit a broad and 'smooth' TAG distribution ranging from T<sub>42</sub> to T<sub>54</sub>, centred around T<sub>50</sub> or T<sub>52</sub>, with no clear dominance of either one (Figure 14) (Evershed et al. 1997b; Dudd and Evershed 1998; Dudd 1999). Conversely non-ruminant adipose fats have a narrower TAG distribution ranging between T<sub>46</sub> to T<sub>54</sub> and often have a clear predominance of either T<sub>50</sub> or T<sub>52</sub> (Figure 14).

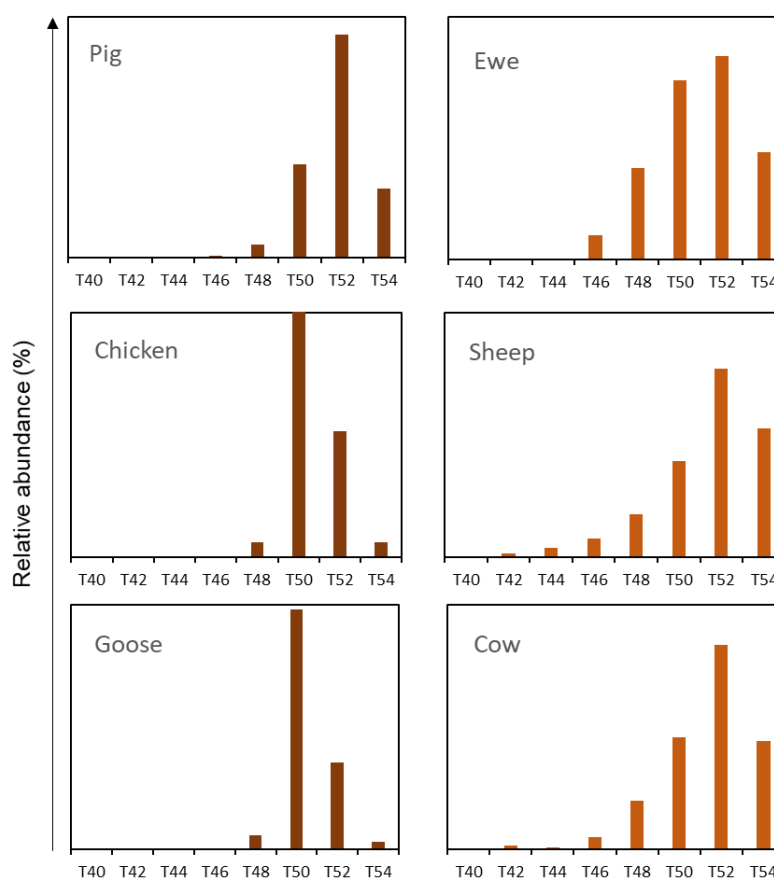


Figure 14: TAG distributions (relative abundance of TAG of different carbon number) of ruminant and non-ruminant adipose fats. Values taken from (Dudd,Regert and Evershed 1998a; Dudd 1999).

TAGs are also the main components of fresh dairy products, such as milk and processed products such as butter and cheese. The TAG distribution of fresh dairy products can be differentiated from adipose fats as they exhibit a high proportion of

low molecular weight TAGs and display a much broader profile ranging from T<sub>26</sub> or T<sub>44</sub> (Dudd and Evershed 1998). However, lower molecular weight TAGs are more susceptible to degradation than higher molecular weight TAGs and thus are preferentially lost. The resulting profile therefore of degraded dairy products can be difficult to distinguish from adipose fats in the archaeological record (Dudd and Evershed 1998; Regert et al. 1998) (see Figure 15).

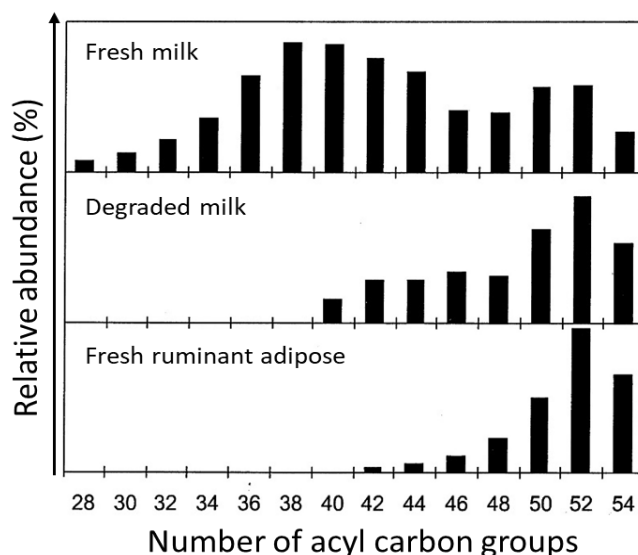


Figure 15: TAG distribution of fresh milk, degraded milk and fresh ruminant adipose fat adapted from (Dudd, Regert and Evershed 1998a; Dudd and Evershed 1998).

TAG distributions are most commonly depicted in bar chart form where the range of TAG acyl carbon groups and the dominant TAG can be visually discerned as shown in figures 14 and 15 above. Mirabaud et al., 2007 used a statistical approach to separate the TAG profiles obtained from archaeological ceramics into three groups. Here, they calculated the average carbon number ( $M$ ) using the formula shown in Equation 2 whereby  $P_i$  is the relative percentage of each TAG and  $C_i$  is the number carbon atoms in each TAG (CN) and calculated to a percentage. They then plotted this against the dispersion factor, which was calculated using the formula shown in Equation 3:

Equation 2

$$M = \frac{\sum (P_i C_i)}{\sum P_i}$$

$$DF = \frac{\sqrt{\sum [(C_i - M)^2 C_i P_i]}}{\sum P_i}$$

It has been shown that by plotting the average carbon number of the TAGs (M) and the dispersion factor (DF), ruminant adipose and dairy products can be broadly distinguished as the lower the CN and higher the DF, the larger the TAG distribution. (Mirabaud, Rolando and Regert 2007) (Figure 16).

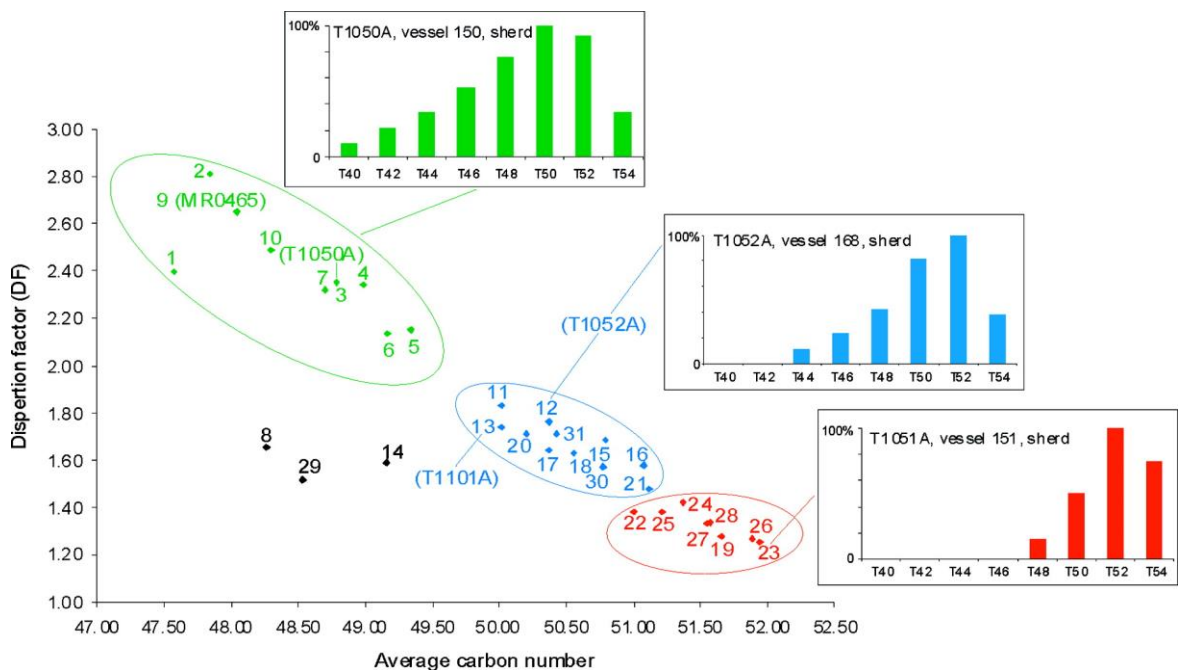


Figure 16: Plots of TAG information based on HT-GC data of individual pottery samples. The dispersion factor (DF) and average carbon number (M) were calculated using statistical equations Mirabaud et al. (Mirabaud, Rolando and Regert 2007).

TAG profiles can be analysed using high temperature GC-MS (HT-GCMS) either by comparing retention times with known standards, assessing the order of elution, or by interpreting the mass spectra, of which a combination results in the most successful identification (Evershed, Heron and John Goad 1990). However, caution needs to be taken when analysing TAGs in this way, as it is difficult to distinguish between saturated and unsaturated TAGs. Furthermore, analysing the structure of TAGs has been proposed to distinguish between adipose and dairy products as well as to differentiate between animal species. However, conventional GC-MS techniques do not

allow for targeted analysis and therefore requires additional instrumentation such as MS/MS, high performance liquid chromatography (HPLC) or nano electrospray ionization MS (Nano ESI MS) (Mirabaud, Rolando and Regert 2007).

Whilst non-degraded animal fats commonly consist of TAGs, degraded animal fats can be recognised in the archaeological record by the distribution of relative abundance of MAGs, DAGs and TAGs and most commonly, their corresponding FAs. FAs arise from the hydrolysis of TAGs due to natural decay or anthropogenic activities such as cooking animal fats in the vessel (Evershed et al. 1997b; Regert 2011; Hammann et al. 2018). FAs palmitic (C<sub>16:0</sub>) and stearic acid (C<sub>18:0</sub>) dominate the spectrum of animal fats. Assessment of the relative abundance of C<sub>16:0</sub> and C<sub>18:0</sub>, reported as a ratio (P/S), has been used to determine the original source of these fats. When C<sub>18:0</sub> is in higher abundance than C<sub>16:0</sub> (P/S <1) this has been used as an indication of ruminant adipose animal fat origin, whereas a P/S ratio >1 has been attributed to non-ruminant fats and dairy fats (Romanus et al. 2007; Baeten et al. 2013). However, multiple criteria for non-terrestrial animal fats have also been proposed for plant oils and aquatics which all use a variety of P/S ratios above 1. Furthermore, caution must be taken when assigning origin of lipids based on relative ratios, as shorter chain FAs are more susceptible to degradation and solubility in the burial environment, thus the preferential loss of palmitic acid needs to be considered (Dudd, Regert and Evershed 1998a; Evershed et al. 2002; Steele, Stern and Stott 2010; Colombini and Modugno 2009; Whelton et al. 2021). The P/S ratio can also be impacted through alterations in the cooking process and due to mixtures of different products (Heron and Evershed 1993; Mottram et al. 1999). Additionally, it may be possible to distinguish modern adipose fats and dairy fats through the presence of short chain FAs with carbon chain lengths between C<sub>4:x</sub> - C<sub>14:x</sub> in dairy fats (McDonald 2002). This, however, is rarely possible in archaeological residues due to the preferential dissolution of short molecules through degradation and hydrolysis (Laakso 1996).

Monomethyl C<sub>15:0</sub> and C<sub>17:0</sub> and branched chain isomers can be indicative of ruminant fats, both adipose and dairy products, as these branched chain FAs are formed through bacterial activity which occurs in the rumen of a ruminant animal. However, it should be noted that they are also synthesised in the hindgut of horse and therefore are

present in horse adipose fats (Christie 1989; Evershed 1993; Mileto et al. 2017). Furthermore, although the presence of branched chain fatty acids are commonly used to support the presence of animal fats, they do also occur widely in nature as they are present in many bacterial membranes (Evershed 1993; Dudd and Evershed 1998; Oudemans, Boon and Botto 2007). Unsaturated fatty acids are also detected in adipose fats, but in lower abundance than saturated FAs. Nonetheless, the position of the unsaturation can provide information about the origin of the fat. Fats deriving from ruminant products present a broad range of positional isomers of monounsaturated fatty acids C<sub>18:1</sub>, whereas non-ruminant fats only display one isomer C<sub>18:1Δ9</sub> (Evershed et al. 1997b; Regert 2011; Mottram et al. 1999). Unsaturated fatty acids are susceptible to degradation processes, such as oxidation and therefore may not be identified in the archaeological record. However, through the oxidation of unsaturated fatty acids, dicarboxylic acids (diacids), hydroxy and dihydroxy fatty acids (DHYAs) can be formed (Regert et al. 1998; Copley et al. 2005a; Regert 2011). These can be used to identify the original position of isomerisation of monounsaturated fatty acids as explained in section 3.6.2.

As noted previously, zoosterols such as cholesterol are essential but minor compounds in animal tissues. Whilst sterols are present in almost every food commodity (Hammann et al. 2018), and cholesterol in animal fats, their presence in the archaeological record is rare. Research has suggested that sterols are relatively resistant to post-burial degradation (Hammann and Cramp 2018). Therefore, thermal alteration through the process of cooking has been demonstrated as the predominant reason for the absence of sterols in archaeological cooking vessels, which is further enhanced through mixtures of sterols with fatty acids and triacylglycerols (Hammann et al. 2018; Kim and Nawar 1991). Although, when present, cholesterol can be used to infer an animal fat origin of residues in archaeological ceramics, this identification needs to be treated with caution considering the general rarity of sterols in the archaeological record. Cholesterol is present in fingerprints; thus, exogenous contamination needs to be considered. When cholesterol is present alongside squalene, another common sterol present in fingerprints, contamination from post-excavation handling is likely (Evershed 1993).

Further to this, the identification of compounds in extracted residues are not only used as biomarkers to identify the original organic source but can also be used to inform how these resources were processed. For example, Evershed et al., (1995), investigates the presence of ketones in archaeological ceramics. It has been suggested that odd-numbered mid-chain ketones of carbon chain lengths 29 to 35 can signify free radical- induced dehydration and decarboxylation of fatty acids, and subsequent self and cross condensation when heated to temperatures above 300°C (Evershed et al. 1995; Raven et al. 1997; Evershed et al. 2002; Baeten et al. 2013) (Figure 17). The presence of odd numbered mid-chain ketones have therefore been used to indicate the heating of animal fats in archaeological ceramics. However, ketones present in absorbed lipids can also occur as a result of pyrolysis of acyl lipids and through the process of biosynthesis of plants (Evershed et al. 1995; Charters et al. 1997).

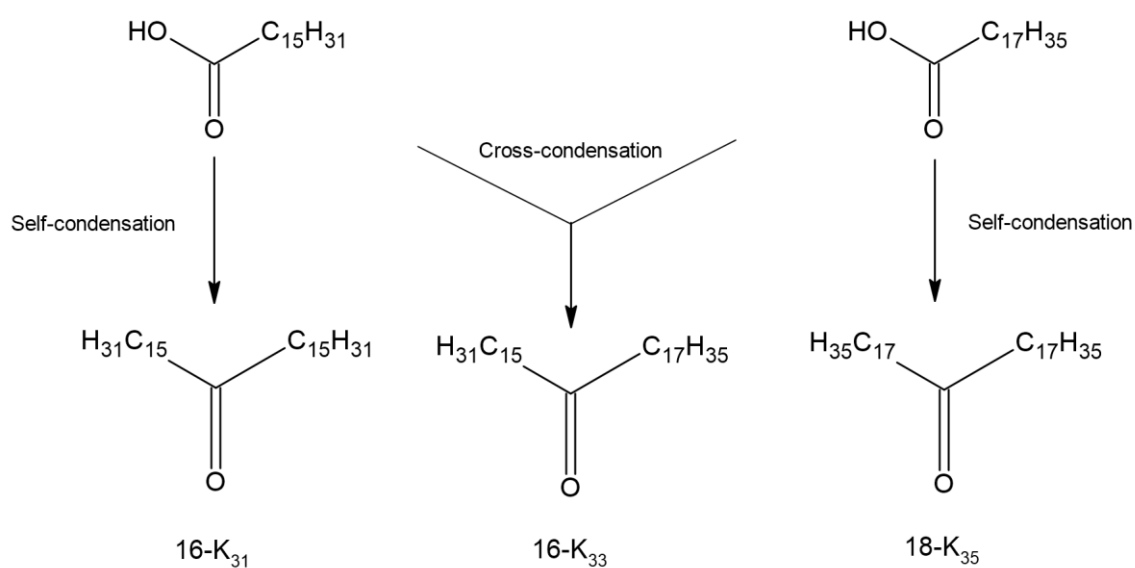


Figure 17: Formation of mid- chain ketones induced by fatty acid pyrolysis as a result of intense heating of animal products >300°C adapted from (Raven et al. 1997).

Furthermore,  $\omega$ -(*o*-alkylphenyl) alkanolic acids (APAAs) do not occur naturally, but can be formed through thermal alteration of mono- and polyunsaturated fatty acids (Hansel et al. 2004; Evershed, Copley and Dickson 2008; Cramp and Evershed 2014; Bondetti et al. 2021). Although mono- and polyunsaturated fatty acids are minor in terrestrial animal fats, APAA C<sub>16</sub> and APAA C<sub>18</sub> can be formed via thermal alteration of unsaturated fatty acids with 16 and 18 carbon lengths, respectively. However, these are not only formed through heating of terrestrial animal fats, but plant, starchy



products and aquatic products also (Hansel et al. 2004; Evershed, Copley and Dickson 2008; Cramp and Evershed 2014; Bondetti et al. 2021).

### 3.7.2 Aquatic products

Molecules present in aquatic sources (both marine and freshwater) are well known in modern sources. Modern aquatic fats and oils consist of saturated fatty acids (predominantly C<sub>16:0</sub>), a high abundance of mono- and long-chain polyunsaturated fatty acids as well as isoprenoid fatty acids.

Whilst a dominance of C<sub>16:0</sub> over C<sub>18:0</sub> can characterise aquatic products in the archaeological record, this profile is also observed in porcine fat, dairy products (Regert 2011) and plant products. As already discussed, the ratio of these fatty acids may be altered by the preferential degradation of C<sub>16:0</sub> and by mixing of products in the vessel (section 3.6.1). Therefore, C<sub>16:0</sub> should not be used as a sole inference for the presence aquatic products. Monounsaturated fatty acids (predominantly, C<sub>16:1</sub>, C<sub>18:1</sub>, C<sub>20:1</sub>, C<sub>22:1</sub>,) and long-chain polyunsaturated acids (C<sub>20:5</sub>, C<sub>22:6</sub>) are highly abundant in modern aquatic resources (Ackman, Hooper and Others 1970). However, in the archaeological record unsaturated acids rarely survive due to the weak single bonds that are susceptible to oxidation through the use of the vessel and burial (Heron, Evershed and Goad 1991; Evershed, Copley and Dickson 2008; Heron and Evershed 1993). Through oxidation processes of unsaturated fatty acids, diacids and DHYAs can be formed (Regert et al. 1998; Copley et al. 2005a; Regert 2011). The presence of vicinal dihydroxy acids directly reflects the number and original position of the double bond in fatty acids as they are formed through the free radical dihydroxylation of double bonds (Copley et al. 2005a; Hansel and Evershed 2009; Hansel, Bull and Evershed 2011). 9,10-dihydroxypalmitic and 9,10-dihydroxyarachidic and 11,12 dihydroxydocosanoic acid, formed through the oxidation of monounsaturated C<sub>16:1</sub> C<sub>18:1</sub> and C<sub>20:1</sub>, respectively, are indicative of aquatic origin as they directly reflect the original presence of monounsaturated fatty acids abundant in aquatic organisms (Hansel, Bull and Evershed 2011; Hansel and Evershed 2009).

Conversely, isoprenoid fatty acids are less susceptible to degradation than unsaturated fatty acids due to their high branching level (Cramp and Evershed 2014). Isoprenoid fatty acids are common in aquatic resources. Through the synthesis of phytol, a constituent of chlorophyll which is ubiquitous in aquatic organisms produces, 4,8,12-trimethyltridecanoic acid (4,8,12-TMTD or TMTD as referred to as from here), 3,7,11,15-tetramethylhexadecanoic acid (phytanic acid) and 2,6,10,14-tetramethylpentadecanoic acid (pristanic acid) can be formed (Ackman, Hooper and Others 1970; Cramp and Evershed 2014). However, isoprenoid fatty acids are not unique to aquatic resources as they also occur in terrestrial animals (3.6.1). For example, phytanic acid is also present in high concentrations in ruminant adipose and dairy fats and in lower abundances in rabbit adipose fats (Brown et al. 1993). A new criterion for distinguishing the origin of phytanic acid in archaeological samples has been proposed (Lucquin et al. 2016c). Here, Lucquin et al. (2016) show that by calculating the ratios of the two phytanic acid diastereomers (3S, 7R, 11R, 15-phytanic (SRR) and 3R, 7R, 11R, 15-phytanic (RRR)) it is possible to distinguish sources of phytanic acid in archaeological samples. This is because SRR is proportionately higher than RRR in aquatic products. It has been shown that aquatic products yield an SRR% ( $\text{SRR}/(\text{SRR}+\text{RRR}) \times 100$ )  $>75.5\%$ , whereas a SRR%  $<75.5\%$  is difficult to distinguish between ruminant and aquatic origin (Lucquin et al. 2016c).

As previously explained,  $\omega$ -(*o*-alkylphenyl) alkanolic acids (APAAs) can be formed through thermal alteration of mono- and polyunsaturated fatty acids (Hansel et al. 2004; Evershed, Copley and Dickson 2008; Cramp and Evershed 2014; Bondetti et al. 2021). Mono- and polyunsaturated fatty acids are highly abundant in modern aquatic fats, but do not generally survive in the archaeological record. However, thermal alteration of these fatty acids can lead to the formation of APAA C<sub>16</sub> to APAA C<sub>22</sub>. Whilst APAA C<sub>16</sub> and APAA C<sub>18</sub> can be formed by heating of terrestrial animal fats, plant and starchy products and aquatic products also, APAA C<sub>20</sub> and APAA C<sub>22</sub> APAA on the other hand are highly indicative of heating aquatic products (Hansel et al. 2004; Evershed, Copley and Dickson 2008; Cramp and Evershed 2014; Bondetti et al. 2021). This is because they are formed from polyunsaturated fatty acids C<sub>20</sub> and C<sub>22</sub>, which can only be found in significant quantities in aquatic organisms (Cramp and Evershed 2014). Thus, the presence of APPAs with carbon chain lengths of 18, 20 and 22 in

addition to one of the three isoprenoid fatty acids that commonly occur, is indicative of the processing of aquatic resources (Craig et al. 2007; Cramp and Evershed 2014; Hansel et al. 2004).

### **3.7.3 Identifying terrestrial and aquatic products using compound specific isotopes**

The biomarker approach has proved a powerful tool to identify the presence and transformations of animal fats, both terrestrial and aquatic, in archaeological ceramics. However, as stated, degradation processes and loss of compositional information can hinder our ability to be more assertive about animal fat origins. In particular, whilst TAG distributions can to some extent help to distinguish between ruminant adipose and dairy products, as well as non-ruminant animal products, TAGs are often broken down into their constituent fatty acids and are not always observed in archaeological samples.

To further aid the identification of animal products, compound specific isotope analysis through GC-c-IRMS is widely applied to archaeological ceramics. By measuring the  $\delta^{13}\text{C}$  values of the most ubiquitous and stable fatty acids,  $\text{C}_{16:0}$  and  $\text{C}_{18:0}$ , these can be compared and plotted against  $\delta^{13}\text{C}$  values of modern reference fats to determine the origin of the fatty acids. The most widely used application of compound specific analysis, since its first applications at the end of the 1990s in archaeological ceramic studies, has been to distinguish between ruminant adipose fats and terrestrial non-ruminant adipose fats (e.g., porcine) (Evershed et al. 1997b, 2002; Dudd and Evershed 1998; Dudd 1999; Mottram et al. 1999; Copley et al. 2003). Ruminant adipose fats yield significantly lower  $\delta^{13}\text{C}$  values than non-ruminant adipose fats as a result of differential incorporation of carbon from plant lipids into their tissues, due to distinguished digestive systems between the two species. In ruminant animals, only carbon from acetates from foods consumed is incorporated to biosynthesise  $\text{C}_{16:0}$  and  $\text{C}_{18:0}$  in the rumen, whereas non-ruminant animals incorporate carbon from both acetate and glucose from the foods they consume to biosynthesise the fatty acids (Vernon 1980).

As glucose is more enriched in  $^{13}\text{C}$  than in acetate, non-ruminants display significantly higher  $\delta^{13}\text{C}$  values than ruminants (Dudd 1999).

Furthermore, the ability to distinguish ruminant adipose fats from dairy products through GC-c-IRMS analysis has contributed significantly to the understanding of secondary product resource use in the archaeological record (Dudd and Evershed 1998; Copley et al. 2003; Evershed 2009; Evershed et al. 2002). Dairy fats, in comparison to adipose fats, yield significantly depleted  $\delta^{13}\text{C}_{18:0}$  values. This is because the biosynthesis of  $\text{C}_{18:0}$  in milk and adipose fats are distinct as a result of different biochemical pathways. Whilst  $\text{C}_{16:0}$  is synthesised in the mammary glands and from acetates deriving from the foods consumed by the animal,  $\text{C}_{18:0}$  cannot be synthesised in the mammary gland. Instead  $\text{C}_{18:0}$  in milk derives from dietary lipids formed from bacterial reduction in the rumen and from the mobilization of adipose fatty acids in the rumen. This results in the preferential routing of dietary fatty acids during lactation and therefore yields depleted  $\delta^{13}\text{C}_{18:0}$  values similar to dietary lipids, whereas the  $\text{C}_{16:0}$  values are closer to that of carbohydrates (Dudd and Evershed 1998; Copley et al. 2003; Evershed 2009; Evershed et al. 2002).

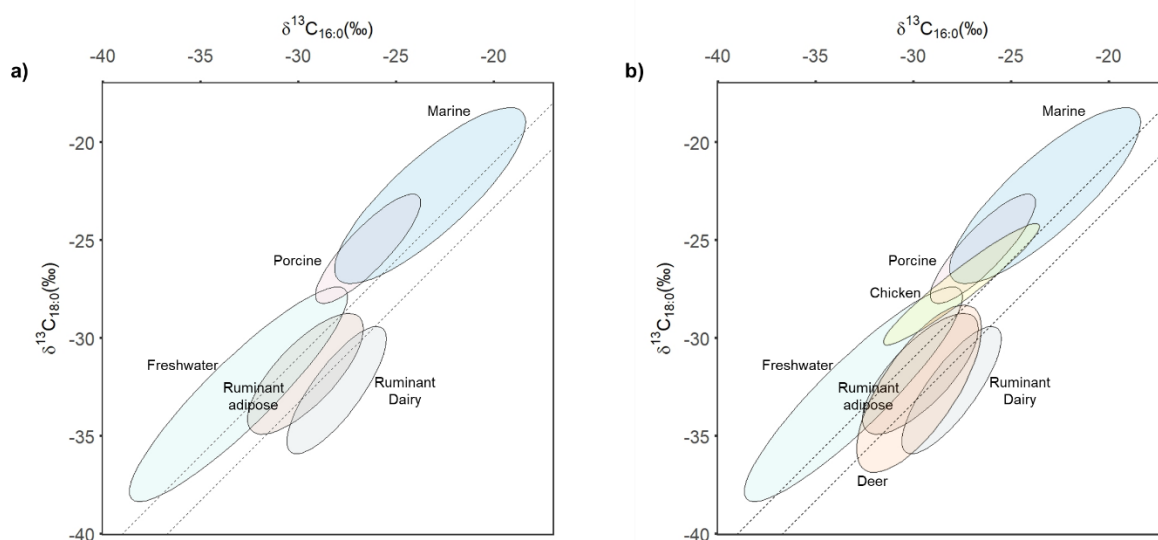


Figure 18: a) Plot of  $\delta^{13}\text{C}_{16:0}$  against  $\delta^{13}\text{C}_{18:0}$  ranges (68% confidence) of 269 modern authentic reference products are shown of fresh water, porcine, ruminant adipose, ruminant dairy and marine products. These values and the original studies in which they were obtained are reported in (Cubas et al. 2020). b) plots  $\delta^{13}\text{C}_{16:0}$  against  $\delta^{13}\text{C}_{18:0}$  ranges (68% confidence) of modern authentic reference of chicken (Colonese et al. 2017b) and deer (Craig et al. 2012).

Marine resources can also be readily distinguished from terrestrial ruminant animal products by their  $\delta^{13}\text{C}$  values, as marine organisms yield highly enriched  $\delta^{13}\text{C}$  values

due to differential sources of CO<sub>2</sub> in comparison to their terrestrial counterparts (Schoeninger and DeNiro 1984). However, it must be noted that there is some overlap in marine  $\delta^{13}\text{C}$  values and non-ruminant adipose values such as porcine (Figure 18a). Thus, when extracts of archaeological samples fall within this range, a combination of both biomolecular evidence and isotopic values is important. Marine  $\delta^{13}\text{C}$  values are also distinct from freshwater  $\delta^{13}\text{C}$  values, of which the latter tends to be more depleted in <sup>13</sup>C. Despite the fact that freshwater organisms live in a variety of environments which can directly impact the  $\delta^{13}\text{C}$  values of the organism resulting in a wide range of  $\delta^{13}\text{C}$  values, they tend to be more depleted than marine organisms and fall close to the range of terrestrial C<sub>3</sub> consuming animals (Robson et al. 2016; Evershed 2009; Craig et al. 2007). This is predominantly due to the direct increase of  $\delta^{13}\text{C}$  in correlation with an increase in water salinity in marine environments (Gladyshev 2009; Hobson and Wassenaar 1999).

Increasingly, focus on ORA studies utilising GC-c-IRMS analysis seek to gather more comprehensive modern reference ranges for a wide variety of products that may have been processed in archaeological ceramics. For example,  $\delta^{13}\text{C}$  values for chicken have been obtained from both modern tissues and in-situ bone remains (Colonese et al. 2017b). However, distinguishing between non-ruminant monogastric animals and omnivorous animals is a complex issue, predominantly due to the wide range of  $\delta^{13}\text{C}$  values that omnivorous animals (e.g., chickens) yield due to their varied diet. As shown in Figure 18 b, the reference  $\delta^{13}\text{C}$  values for chicken adipose and bone fats overlap the values of freshwater, porcine and ruminant adipose. Furthermore, wild ruminant adipose fats such as deer have been shown to yield diverse  $\delta^{13}\text{C}$  values which fall in the range of both ruminant fats and dairy products (Figure 18 b) (Copley et al. 2003; Mukherjee, Gibson and Evershed 2008; Dudd and Evershed 1998; Gregg et al. 2009; Roffet-Salque et al. 2017). In archaeological studies where a variety of different animal products may have been incorporated in ceramic vessel recipes, large overlaps and reference samples can hinder the possibility of unambiguously assigning a single animal fat origin.

When considering  $\delta^{13}\text{C}$  values of modern reference fats it is important to note that these are influenced by the diet of the animal and environmental factors (Copley et al.

2003; Mukherjee, Gibson and Evershed 2008; Dudd and Evershed 1998; Gregg et al. 2009; Roffet-Salque et al. 2017). Therefore, regionally specific references provide a better means of interpretation of archaeological sources as the  $\delta^{13}\text{C}$  values are comparative. To date, however, the majority of reference  $\delta^{13}\text{C}$  values come from Northern European contexts. Some  $\delta^{13}\text{C}$  values of modern animal fats have been obtained from Africa (Dunne et al. 2012), the Middle East (Gregg et al. 2009) and Mediterranean contexts (Drieu et al. 2021a; Debono Spiteri 2012). However, these are still relatively limited in comparison to the extensive database of references from the United Kingdom, for example (Dudd and Evershed 1998; Dudd 1999; Evershed et al. 1997b, 2002). Furthermore, in the case of reference samples from the Middle East for instance, these references come from modern markets and are unlikely to reflect the original diet of the animals (Gregg et al. 2009). The difficulty in obtaining reliable  $\delta^{13}\text{C}$  values from modern references ultimately lies in limitations of replicating historic feeding practices. Although a controlled  $\text{C}_3$  diet best represents dietary habits in the past (when we know  $\text{C}_4$  is absent from these contexts) it is difficult to replicate the dietary and environmental conditions of the animal. Furthermore, Roffet-Salque et al. (2017) shows that feeding supplements and silage practices have a strong impact on  $\delta^{13}\text{C}$  values of modern animals even with a predominantly  $\text{C}_3$  diet (Roffet-Salque et al. 2017). Therefore, caution needs to be taken when directly comparing  $\delta^{13}\text{C}$  values from archaeological ceramic residues with modern reference fat  $\delta^{13}\text{C}$  values.

Compound specific stable isotope values can also be reported as  $\Delta^{13}\text{C}$ , which corresponds to the differences in isotope values between  $\text{C}_{18:0}$  and  $\text{C}_{16:0}$  ( $\delta^{13}\text{C}_{18:0} - \delta^{13}\text{C}_{16:0}$ ). Unlike the  $\delta^{13}\text{C}$  values,  $\Delta^{13}\text{C}$  are not influenced by dietary and environmental conditions of the consumer as isotopic fractionation of  $\text{C}_{18:0}$  and  $\text{C}_{16:0}$  is similar (Copley 2002, 75–83). By plotting the  $\Delta^{13}\text{C}$  against  $\delta^{13}\text{C}_{16:0}$ , it is possible to discriminate between non-ruminant, ruminant, and ruminant dairy sources (Figure 19). Generally, studies report  $\Delta^{13}\text{C} < -3.3\text{‰}$  are typically associated with ruminant dairy, values between  $-3.3\text{‰}$  and  $-1.0\text{‰}$  are associated with ruminant adipose and above  $-1.0\text{‰}$  can be considered as non-ruminant (Craig et al. 2012). However, in some studies of the Mediterranean and Africa values of  $< -3.1\text{‰}$  have been associated with dairy products, whilst non-ruminant fats fall above  $\Delta^{13}\text{C}$  of  $0.3\text{‰}$ , whilst ruminant adipose fats fall between the two values (Copley et al. 2003; Craig et al. 2007; Evershed et al. 2008;

Evershed 2009; Cramp and Evershed 2014). However, studies of animals from different regions have shown that values can overlap and therefore whilst these ranges can act as a guide, consideration of overlapping ranges and mixing of products always needs to be considered (Copley et al. 2003; Craig et al. 2007; Evershed et al. 2008; Evershed 2009; Cramp and Evershed 2014).

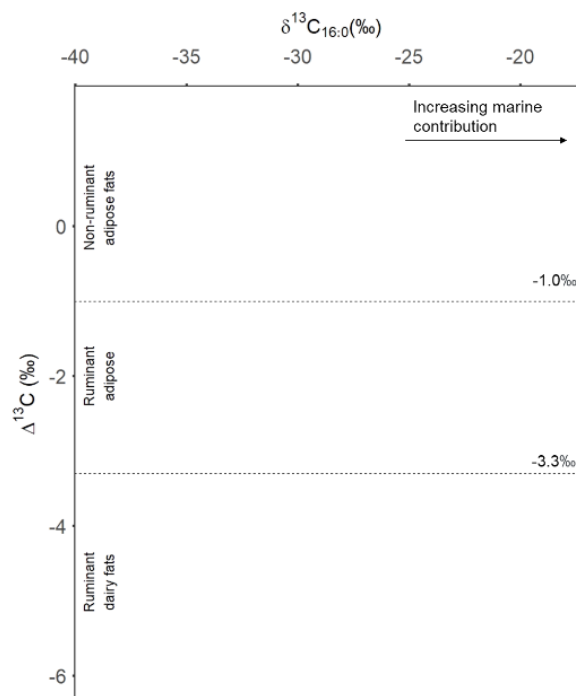


Figure 19: Plot of  $\Delta^{13}C$  against  $\delta^{13}C_{16:0}$  values of modern references where  $<-3.3$ ‰ are typically associated with ruminant dairy, values between  $-3.3$ ‰ and  $-1.0$ ‰ are associated with ruminant adipose and above  $-1.0$ ‰ can be considered as non-ruminant. Arrow represents the more positive  $\delta^{13}C_{16:0}$  values consistent with marine contribution (Copley et al. 2003; Craig et al. 2007; Evershed et al. 2008; Evershed 2009; Cramp and Evershed 2014).

### 3.7.4 Plant oils and waxes

In contrast to animal fats, the ability to assess the presence of plant products in archaeological ceramics can be somewhat limited by their lower lipid yield compared to animal products (Evershed 2008b, 2008a). In archaeological ceramics, plant products are often masked by animal fats. Evershed (2008b) showed that after boiling leaf waxes ten times in experimental cooking events, the total lipid concentration of plant waxes was significantly lower than that of animal fat boiled only once in the same

vessel (Figure 20) (Evershed 2008a). Nonetheless, plant products have been routinely identified in archaeological ceramics.

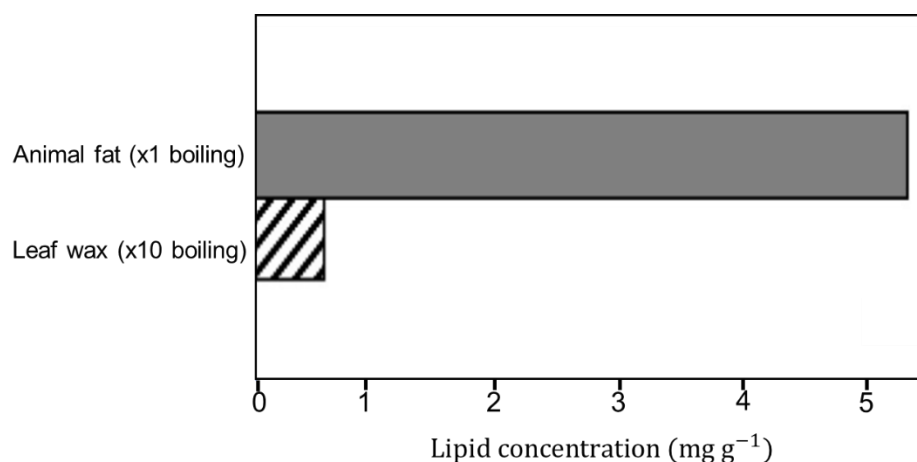


Figure 20: Mean lipid concentrations (mg g<sup>-1</sup>) of three vessel rims used to boil Brassica 10 times followed by the single boiling of lamb meat adapted from (Evershed 2008a)

The plant sterols most predominantly found in archaeological ceramics are  $\beta$ -sitosterol, stigmasterol and campesterol. These sterols can indicate the presence of plant products in the ceramic but are not specific to plant type, as they exist ubiquitously across the plant kingdom.

### 3.7.5 Plant oils

TAGs are the main constituent of modern plant oils (Evershed 1993). However, as mentioned previously, TAGs are susceptible to degradation and their identification in archaeological records can be limited. Further to this, our ability to identify plant products based on their TAG distributions is restricted as they are largely dominated by unsaturated TAGs, which are more rapidly degraded than their saturated homologues. Unsaturated TAGs are difficult to identify, even if present, using basic GC-MS. Resulting from the hydrolysis of TAGs, FAs are the main constituents of degraded plant oils. Unlike in terrestrial animal fats where saturated fatty acids dominate the profile, unsaturated fatty acids are more predominant in plant oils (Heron and Evershed 1993; Romanus et al. 2008; Baeten et al. 2013). High concentrations of unsaturated FAs in plant oils lowers their melting point, which makes them liquid at



room temperature. Modern plant oils have specific unsaturated FA profiles that can be used in the archaeological record to distinguish between them. A high dominance of oleic acid ( $C_{18:1}$ ), for example, can indicate the presence of olive or rapeseed oils. Additionally, when a relatively high amount of linoleic acid ( $C_{18:2}$ ) is present, plant oils such as cotton, soya, grapeseed, or sesame may be inferred. In addition to  $C_{18:1}$ ,  $C_{18:2}$ , the presence of  $C_{18:3}$  has been used to indicate the presence of linseed oil in archaeological ceramics (Copley et al. 2005a). However,  $C_{18:2}$  and  $C_{18:3}$  degrade faster than  $C_{18:1}$ , and therefore all oils can yield profiles that look similar to olive oil. A  $C_{18:1}/C_{18:0}$  ratio  $>2$  has been used to identify the presence of olive oil, but it should be used only to prescribe a plant oil of unspecified origin more generally. The susceptibility of unsaturated fatty acids to degradation through oxidation processes means that degraded plant oils, regardless of species, exhibit similar profiles making them difficult to distinguish. Mixing of plant oils and other products can further limit the use of unsaturated fatty acids ratios for distinguishing species (Heron and Evershed 1993; Romanus et al. 2008; Baeten et al. 2013; Regert et al. 1998; Whelton et al. 2021).

The identification of short chain fatty acids and  $\alpha,\omega$ -dicarboxylic acids (diacids) can be indicative of plant oils as they are products of these oxidation processes (Regert 2011; Copley et al. 2005a; Regert et al. 1998). Diacids are formed by different oxidation mechanisms of the double bond in unsaturated FAs (Passi et al. 1993). The chain length of a diacid directly reflects the position of the double bond of the unsaturated fatty acid it derived from. The high abundance of  $C_9$  diacid therefore, is indicative of the  $\Delta^9$  unsaturation of the precursor fatty acid. Furthermore, vicinal dihydroxy acid directly reflects the original position of the double bond of the unsaturated fatty acids, as they are formed through the process of free radical dihydroxylation of the double bond. 9, 10- dihydroxy octadecanoic acid is formed through the free radical dihydroxylation of  $C_{18:1\Delta^9}$ . Although a high dominance of  $C_{18:1\Delta^9}$  and its oxidation products can be indicative of plant oil, particularly olive oil, as  $C_{18:1\Delta^9}$  is ubiquitous in other sources it can be difficult to be specific. However, there are some uncommon fatty acid structures that, if identified, can be highly indicative of specific plant oils. For example, dihydroxyl acids (11,12 dihydroxyeicosanoic acid and 13,14 dihydroxydocosanoic acid) are formed through oxidation of  $C_{22:1}$  ( $\Delta$  -13),  $C_{20:1}$  ( $\Delta$  -11) and  $C_{24:1}$  ( $\Delta$  -15) which are abundant in *Brassicaceae* (inc., turnip, mustard, and radish) seed oil (Copley et al. 2005; Romanus

et al. 2007). Additionally, 12-hydroxyoctanoic acid, formed through the oxidation of C<sub>18:1</sub>( $\Delta$ -12), can indicate the presence of castor oil (Copley et al. 2005a).

Saturated FAs, less prone to degradation, are also present in plant oils and differ from animal fats as they typically exhibit a predominance of palmitic acid (C<sub>16:0</sub>) over (C<sub>18:0</sub>) (Steele, Stern and Stott 2010; Copley et al. 2005a). In archaeological ceramics, C<sub>16:0</sub>/C<sub>18:0</sub> fatty acid ratio is often used to distinguish between plant oils and animal fats, where a high C<sub>16:0</sub>/C<sub>18:0</sub> ratio >4 strongly suggests the presence of plant origin. However, in many cases a ratio between the values of 2 and 4 have been used to distinguish plant oils from terrestrial animal fats in archaeological ceramics (Debono Spiteri 2012; Taché and Craig 2015). It should be noted, as discussed previously, that aquatic products, dairy products and porcine fats also display a high prevalence of C<sub>16:0</sub> over C<sub>18:0</sub> and mixing of products may affect these ratios (Regert 2011).

### 3.7.6 Plant waxes

Plant waxes or epicuticular waxes are a complex mixture of molecules and display a wide diversity of structures and chemical components (Eglinton and Hamilton 1967). Their role in the plant is to protect the surfaces of the plant that are exposed to the atmosphere. Leaf waxes are the most studied and identified in the archaeological record, but the stems, fruit and petals of a plant may also be covered by wax (Eglinton and Hamilton 1967). Leafy vegetables can be identified in archaeological vessels through the presence of long-chain odd *n*-alkanes, *n*-alkanol and ketones of specific distributions. Long-chain odd *n*-alkanes from C<sub>21</sub>-C<sub>37</sub>, where the distribution is centred around C<sub>29</sub> and C<sub>31</sub>. Ketones with odd number carbon length, with the carbonyl group in the middle position are also indicative of plant waxes (Evershed, Heron and Goad 1991; Evershed 1993; Baeten et al. 2013; Charters et al. 1997).

In some cases, it is possible to offer greater taxonomic resolution regarding the origin of the plant waxes based on long-chain odd *n*-alkanes, *n*-alkanol and ketones profiles. Brassicas have been identified in archaeological ceramics through a specific *n*-alkane,

*n*-alcohol and *n*-ketone distribution of *n*-nonacosane; *n*-hexacosanol; *n*-heptacosanol; nonacosane-15-one; nonacosane-15-ol; *n*-hentriacontane; and *n*-octacosanol (Evershed, Heron and Goad 1991; Evershed 1993; Baeten et al. 2013; Charters et al. 1997). Furthermore, the presence of hentriacontane-16-one (*n*-ketone C<sub>31</sub>) and the presence of *n*-hentriacontane (*n*-alkane C<sub>31</sub>), has been attributed to the presence of *Allium porrum* (leek) in archaeological ceramics (Evershed et al. 1992a, 1995; Evershed, Heron and Goad 1991; Raven et al. 1997). However, the distribution and abundance of ketones needs to be taken into consideration, as *n*-ketones can also be formed through condensation of fatty acids present in animal fats through intensive heating (Evershed et al. 1995; Raven et al. 1997; Evershed et al. 2002; Baeten et al. 2013). The presence of ketone C<sub>31</sub> on its own is more likely to indicate the use of plant products, but should be treated with caution and *n*-alkanes and *n*-alkanols should also be present in order to assert a plant wax origin (Raven et al. 1997).

Modern plant waxes are also comprised of aliphatic long-chain wax-esters with predominantly even number carbon lengths with chain length C<sub>30</sub>-C<sub>56</sub>, comprised of *n*-alcohols ranging from C<sub>26</sub>-C<sub>28</sub> and fatty acid moieties of carbon lengths C<sub>18</sub> and C<sub>22</sub> (Walton 1990). Associated with odd-numbered *n*-alkanes, wax esters C<sub>18:0</sub>, C<sub>20:0</sub> and C<sub>22:0</sub> can indicate a plant origin. This is in contrast to wax esters associated with beeswax as these are almost entirely comprised of C<sub>16:0</sub> wax esters (3.7.11) (Ribechini et al. 2008; Rageot et al. 2019c).

### 3.7.7 Cereals and millet

The ability to identify the processing of cereals in archaeological ceramics has, to date, proved difficult due to their low lipid content and lack of species-specific biomarkers. Furthermore, cereals have high contents of carbohydrates, which rapidly degrade through anthropogenic and microbial processes. However, biomarkers have been identified in some grain plants. Miliacin (olean-18-en-3 $\beta$ -ol methyl ether) is a pentacyclic triterpene methyl ether (PTME). Miliacin occurs in a variety of plants, but specifically occurs in high concentrations, making up ~99% of the total PTMEs in broomcorn millet (*Panicum miliaceum*) (Heron et al. 2016; Bossard et al. 2013).

Therefore, when miliacin is identified as the predominant PTME it can be used as a reliable biomarker for the presence of broomcorn millet. Initially, miliacin was used to identify the presence of millet in sediment samples (Bossard et al. 2013) providing important information for the cultivation of millet and has since been used to identify the presence of millet in ceramic containers (Heron et al. 2016; Ganzarolli et al. 2018). Identifying millet in ceramic containers has given direct evidence for preparation of millet for human consumption.

Additionally, alkylresorcinols are present in high concentrations in wheat and rye and can be found in lesser quantities in barley, maize, millet and bran (Ross et al. 2003; Chen et al. 2004; Landberg et al. 2008; Ross et al. 2001; Ross 2012). Colonese et al., (2017) identified alkylresorcinols in an Early Bronze Age wooden container from Switzerland and concluded that the compounds were derived from cereal grains (Colonese et al. 2017a). The distribution of alkylresorcinols homologues of different alky chains from C<sub>17</sub> - C<sub>25</sub>, as shown in Figure 21, can inform about the species of grain. Wheat and rye display a notably different C<sub>17</sub> / C<sub>21</sub> ratio of 0.1 and 1.0, respectively (Chen et al. 2004; Ross 2012). Chen et al., (2004) showed that these ratios were not altered during the processing of these cereals. This has recently been disputed by Hamman and Cramp (2018), who showed that cooking cereals can impact the distribution of alkylresorcinols. Therefore, if identified in archaeological ceramics, alteration through cooking processes needs to be considered if distinguishing between grains. (Chen et al. 2004; Hammann and Cramp 2018). However, to date, no study has been able to unambiguously identify alkylresorcinols in archaeological ceramics. This is partly due to the low levels of cereal lipids that are transferred to the ceramic matrix when processed in vessels as shown by Hamman and Cramp (2018) in a series of experiments. Furthermore, they are highly susceptible to degradation in anoxic conditions in the burial environment resulting in low recovery (Ross et al. 2003; Hammann and Cramp 2018; Colonese et al. 2017a).

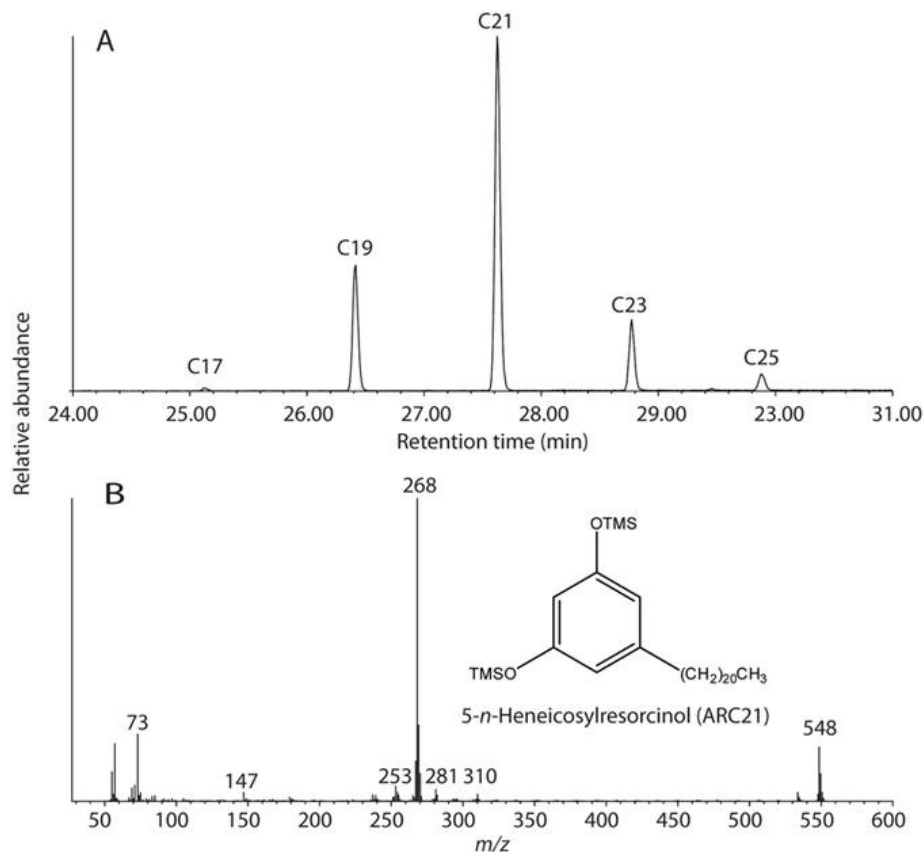


Figure 21: A) Partial TLE chromatogram of ion m/s 268 depicting the homologues of alkylresorcinols ranging from alky chain lengths 17-25 and B) a selected mass spectra of 5-n-heneicosylresorcinol alkylresorcinol of 21 carbon atoms (Colonese et al. 2017a).

Recent works have identified the presence of ergot estolides in ceramics from Sardinia, Calabria and Sicily (Lucejko et al. 2018). Ergot is a group of fungi of the genus *Claviceps* that grows on rye, related plants and other grasses such as wheat. Ergot fungi metabolically produces lipids such as TAGs and estolides. Lucejko et al., (2018) identified secondary lipid metabolites produced by ergot in the archaeological vessels and depicted their importance in identifying the presence of cereals and cereal derivatives in archaeological contexts. However, the identification of ergots relies on high performance liquid chromatography (HPLC/ESI-Q-Tof) as these compounds elute at the same time as TAGs, which are in high abundance in animal fats. Coelution of these compounds means that they are undetectable through conventional GC-MS techniques.

### 3.7.8 Identifying plant products using compound specific isotopes

Compound specific isotope analysis can distinguish between C<sub>3</sub> plants and C<sub>4</sub> plants as they have distinct carbon isotope fractionations as a result of different photosynthetic pathways between the two plant types (O'Leary 1981). C<sub>3</sub> plants use the Calvin-Benson cycle for CO<sub>2</sub> fixation and tend to have bulk  $\delta^{13}\text{C}$  values that fall around the range of  $-27\text{‰}$  (Marshall, Brooks and Lajtha 2007), whereas C<sub>4</sub> plants use the Hatch-Slack cycle for CO<sub>2</sub> fixation and have bulk  $\delta^{13}\text{C}$  values that fall around the range of  $-24\text{‰}$  (Marshall, Brooks and Lajtha 2007). Measuring the  $\delta^{13}\text{C}$  values of fatty acids C<sub>16:0</sub> and C<sub>18:0</sub> can help to distinguish between residues of C<sub>3</sub> and C<sub>4</sub> plant contribution as well as animal products that have a predominantly C<sub>3</sub> or C<sub>4</sub> diet. Figure 22a shows the ranges of  $\delta^{13}\text{C}_{16:0}$  and C<sub>18:0</sub> values obtained from a range of modern C<sub>3</sub> and C<sub>4</sub> plant products corrected for the post-industrial effect. However, in archaeological studies there are limitations to identifying pure C<sub>3</sub> and C<sub>4</sub> plant signals based on these values. This is because the ranges of C<sub>3</sub> plants fall well within the range of freshwater aquatic products and, in part, covers the range of ruminant adipose fats. Similarly, the ranges of C<sub>4</sub> plant products overlap ranges of porcine and marine products.

Steele et al., (2010) depict the inherent complications with interpreting the contributions of plant products based on  $\delta^{13}\text{C}$  values from archaeological ceramics. Here they show that when modern vegetable oils are plotted by  $\Delta^{13}\text{C}$  against  $\delta^{13}\text{C}_{16:0}$  values, plant oils can easily be misinterpreted as porcine adipose or ruminant adipose fats (Steele, Stern and Stott 2010) (Figure 22b). Furthermore, mixtures of plant products and animal products may affect the  $\delta^{13}\text{C}$  values obtained from archaeological ceramics and causes complications in interpreting these values. For example, Hendy et al., (2018), through a mixing model suggest that the mixing of C<sub>3</sub> plants, such as barley, with dairy can yield isotopic values similar to those of ruminant adipose fats (Hendy et al. 2018). Mixing models can help to address what contributions plants may have had based on simulated  $\delta^{13}\text{C}$  values, but plant products can have varying  $\delta^{13}\text{C}$  values and therefore it is difficult to determine their contributions (Steele, Stern and Stott 2010). As Steele et al., (2010) explain, this is not a problem in archaeological

contexts where plant products are not expected to be processed in ceramic vessels (Steele, Stern and Stott 2010), but in medieval Mediterranean contexts this is certainly an issue that needs to be considered when using compound specific isotope analysis. Whelton et al., (2021) recently suggested that residues that yield plant biomarkers should be excluded from GC-c-IRMS analysis to avoid the complications of mixing (Whelton et al. 2021). However, this approach does not account for cases where plant products are present, but cannot be identified.

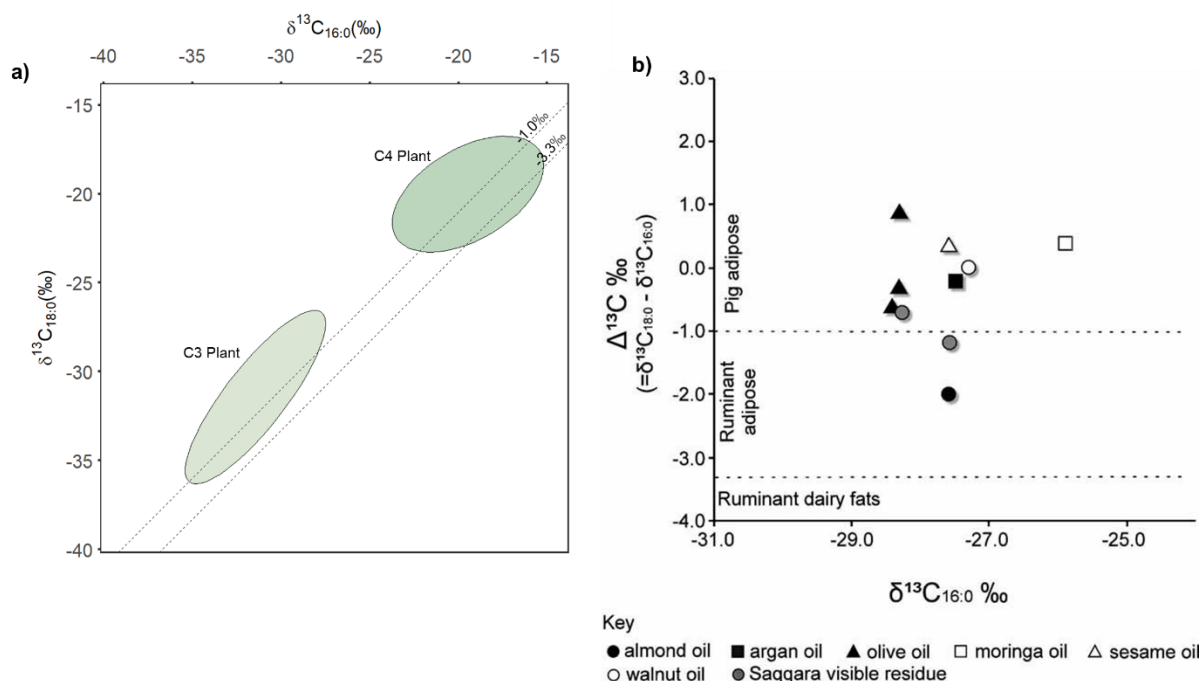


Figure 22: a) Plot of  $\delta^{13}\text{C}_{16:0}$  against  $\delta^{13}\text{C}_{18:0}$  ranges (68% confidence) of modern authentic reference products of C<sub>3</sub> plant and C<sub>4</sub> plant products all samples and values are presented in Appendix C. b) Plot of  $\Delta^{13}\text{C}$  against  $\delta^{13}\text{C}_{16:0}$  values of modern oil references and archaeological visible residue taken directly from (Steele, Stern and Stott 2010).

Alternatively,  $\delta^{13}\text{C}$  values have been determined for specific plant biomarkers to further confirm the presence of plants in archaeological ceramics and eliminating problems associated with the food web effect (Reber and Evershed 2004). For example, one of the first uses of compound specific isotope analysis of archaeological ceramics measured  $\delta^{13}\text{C}$  values of alkanes associated with plant wax contribution and showed a clear C<sub>3</sub> signal. Furthermore,  $\delta^{13}\text{C}$  values have been obtained from *n*-dotriacontanol (OH C<sub>32</sub>), a biomarker that has been used to indicate the presence of maize in archaeological samples (Reber and Evershed 2004).

### 3.7.9 Fruits including grape products

Modern fruits, including both citrus and vine, are depleted in lipids and are mainly comprised of sugars which are highly soluble in water and small organic acids and phenolic compounds. The advantage of small organic compounds is that, although they are soluble in water, they are also more polar and bond strongly to the ceramic matrix. Whilst these bonds are likely broken during conventional AE, it is difficult to solubilise the small acids into an organic solvent, but they can be extracted and analysed using acidified butanol extraction method which makes the molecules less polar and soluble into the organic solvent. Short chain carboxylic acids (fumaric, succinic, malic, and tartaric) can be used to indicate the presence of different fruit products (Garnier and Valamoti 2016; Drieu et al. 2021b).

Tartaric acid is one of the main small acids in grapes, alongside malic acid and citric acids (Hale 1962; Ribéreau-Gayon 2006). Therefore, its presence in ceramics vessels is commonly interpreted as a biomarker of grapevine products, and in particular wine (Guasch-Jané et al. 2004, 2006; McGovern 2009). A number of studies, particularly those focused on the Mediterranean, as wine is an important beverage in the Mediterranean, have aimed to identify the presence of wine biomarkers in archaeological ceramics, most often amphorae, but in some cases cooking pots as well (Salvini, Pecci and Giorgi 2008; Buonincontri et al. 2017). However, the mere presence of tartaric acid in ancient ceramics is not enough to formally identify the presence of grapevine products, much less wine. This is because tartaric acid also exists in other plants such as tamarind, star fruit and yellow plum (Drieu et al. 2020, 2021b; Barnard et al. 2011; Garnier 2007), and low amounts may be attributed to contamination from the burial environment (Drieu et al. 2020). A comparison of the proportions of malic and tartaric acids in the pots has been proposed to distinguish between the presence of grapevine products and that of other plants (Drieu et al. 2021b, 2020; Jaeggi, Wittmann and Garnier; Linger-Riquier et al. 2016; Cherel et al. 2018). A ratio of tartaric acid to the sum of tartaric and malic acids (%TA) of greater than 35% was proposed to be characteristic of ripe grapes and their products (wine, juice, vinegar), as well as tamarind and some pomegranate cultivars (Drieu et al. 2021b, 2020).



The presence of malic acid, succinic and fumaric acid with low concentrations of tartaric acid (<35% TA) in archaeological ceramics can indicate the presence of fruit products other than grapevine products. Malic acid, for example, is ubiquitous in the plant kingdom and is present in large quantities in fruits such as apple, plum, cherry or peach (Drieu et al. 2021b; Jaeggi, Wittmann and Garnier; Linger-Riquier et al. 2016; Cherel et al. 2018; Walker and Famiani 2018). However, malic acid is also present in vegetable products such as brassica and alliums, and therefore caution needs to be taken when using malic acid as a marker for fruit products, especially when evidence of plant waxes also exist in the same vessel (Ruhl and Herrmann 1985a). The analysis of small organic acids to identify the presence of fruit products has been largely neglected in routine organic residue analysis studies, partially those focused on prehistoric pottery where most large-scale analyses take place. However, the identification of fruit products in ceramics has had its beginnings in a number of small scale studies on ceramics from medieval contexts (Salvini, Pecci and Giorgi 2008; Buonincontri et al. 2017; Pecci and Grassi-Munibe 2016; Notarstefano et al. 2011) as well as Early Celtic ceramic samples (Rageot et al. 2019c, 2019b).

### **3.7.10 Resins and tars**

Resins and tars have a long history of use in ceramic containers predominantly for technological purposes, such as adhesives for repair and waterproofing agents for the ceramics themselves, but they are also processed in the ceramics for secondary uses for other materials. Several studies have used organic residue analysis to identify a variety of resins and tars in ceramic vessels from various archaeological and geographical contexts. Resins, tars, and pitches are made up of a complex mixture of terpenoid compounds of which di- and triterpenes are the most commonly identified in the archaeological record. These di- and triterpenes can be specific to resins of different plant origin as they are never jointly synthesised in the same resin and undergo little alteration (Evershed 1993). For example, specific triterpenoids can be indicative of birch-bark tars (Morandi, Porta and Ribechini 2018), whereas specific diterpenoids have been considered strong indicators for conifer (Pinaceae) resins (Colombini et al. 2005; Modugno and Ribechini 2009; Regert and Rolando 2002).

Pinaceae resin or pitches are the most commonly identified resin in the archaeological record. Pinaceae resin in archaeological ceramics can be identified by the presence of diterpenoids, mainly, abietic, primaric and isoprimary acids, which constitute the non-volatile fraction of the resin. Through the process of oxidation and dehydrogenation in the burial environment, characteristic molecules are formed, including dehydroabietic acid (DHA), 7-oxo-DHA and 15-hydroxy-DHA (Colombini et al. 2005; Modugno and Ribechini 2009; Regert and Rolando 2002). Pine tar and pitch can also be identified in archaeological ceramics through the identification of various compounds formed via thermal treatment of pine resins and woods needed to produce the tar/pitch. Furthermore, the presence of methyl dehydroabietic acid indicates that the resin has been intensively heated with wood and is formed through methylation (Mills and White 1989; Hjulström, Isaksson and Hennius 2006). There are a number of examples in the archaeological record where Pinaceae resins have been used as waterproofing agents to line porous ceramics, mainly in amphorae to store liquids (Dimitrakoudi et al. 2011; Izzo et al. 2013; Heron and Pollard 1988). Pinaceae products may also be used for medicinal or culinary purposes as flavouring for foods, and in particular wine, as noted in ancient Greece and Rome as well as contexts from the Middle East and Egypt (Reber and Hart 2008; Beck et al. 2008; McGovern 1997; McGovern, Mirzoian and Hall 2009). Pitch or pine tar can be used as a sealant, adhesive or to waterproof boats as observed in a number of medieval contexts (Pecci and Grassi-Munibe 2016; Salvini, Pecci and Giorgi 2008; Buonincontri et al. 2017).

Birch-bark tar has been identified in archaeological ceramics as an adhesive, sealant and as surface decoration. Its presence may also be attributed to the reuse of this cooking pot for the storage, production or transportation of birch-bark products (Urem-Kotsou et al. 2002; Charters et al. 1993a; Morandi, Porta and Ribechini 2018; Stacey et al. 2020; Rageot et al. 2019a; Robson et al. 2019). The presence of pentacyclic terpenoids, betulin and lupeol are characteristic of birch bark (O'Connell et al., 1988; Hayek et al., 1989; Hayek et al., 1990; Cole et al., 1991; Hua et al., 1991., Charters et al., 1993 Aveling and Heron, 1998, Binder et al., 1990, Charters et al., 1993, Regert et al., 1998). During thermal treatment of birch bark to make birch bark tar, two characteristic molecules are formed, lupan-2, 20(29)-dien-28-ol and lupan-2, 20(29)-

diene resulting from the dehydration of betulin and lupeol respectively (Aveling and Heron, 1998, Regert, 2004). Other derivatives may also be present including naphthalene, lup-20(29)-en-3-one;  $\beta$ -amyrin, lupeol and lup-20(29)-en-3-ol (Regert, 2004) and allobetul-2-ene is formed via natural degradation in the burial environment. The archaeological record shows that birch-bark tar has been used as an adhesive since prehistory to the beginning of the Roman period when it is believed there was a shift away from birch tar when pine products became a common source of resin (Pollard and Heron, 2008). However, the discovery of birch-bark tar in Western Europe from the 1<sup>st</sup>-6<sup>th</sup> centuries A.D (Regert et al. 2019) and in early medieval contexts in England (Stacey et al. 2020) has shown that birch-bark tar may have been in use longer than first expected.

### **3.7.11 Beehive products**

In the past, beehive products, such as honey and wax had numerous uses. Evidence of beeswax has been identified in a number of archaeological ceramic samples and has been used a proxy for the identification of honey or pure beeswax used as a commodity for sealing and waterproofing vessels or as an adhesive (Garnier et al. 2002; Heron et al. 1994; Kimpe, Jacobs and Waelkensaa 2002; Roffet-Salque et al. 2015). Fresh beeswax can be characterised by a complex mixture of compounds with specific distributions and profiles. Beeswax profiles consist of predominantly odd-chain alkanes ranging from chain lengths C<sub>23</sub> – C<sub>33</sub> where C<sub>27</sub> is dominant; even chain fatty acids ranging from C<sub>22</sub> – C<sub>36</sub> where C<sub>24</sub> is dominant; and a series of long chain palmitate esters ranging from C<sub>40</sub> – C<sub>52</sub> where C<sub>46</sub> prevails (Aichholz and Lorbeer 1999; Regert et al. 2001; Garnier et al. 2002) . Monohydroxy esters with long chain-alcohols ranging from C<sub>24</sub> – C<sub>38</sub> esterified to hydroxy palmitic acids with chain lengths from C<sub>40</sub> – C<sub>54</sub>, are also characteristic of beeswax (Aichholz and Lorbeer 1999).

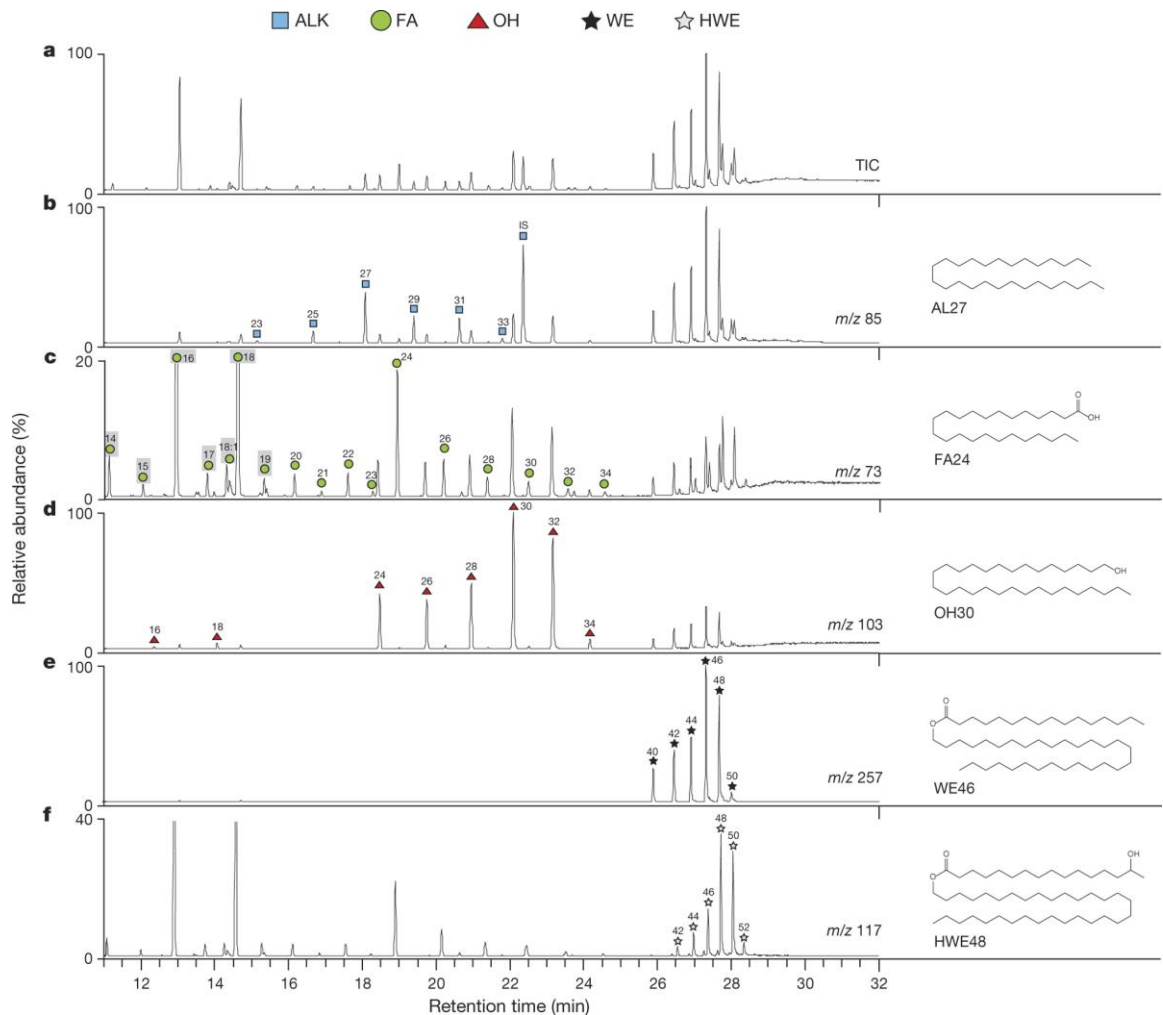


Figure 23: Partial total ion chromatogram (TIC) (a) and mass chromatograms (b-f) of a total lipid extract of a ceramic sherd containing beeswax. Chromatograms showing ion masses of b) *n*-alkanes ( $m/z$  85), c) *n*-fatty acids ( $m/z$  73), d) *n*-alcohols ( $m/z$  103), e) wax esters ( $m/z$  257) and f) hydroxy-wax esters ( $m/z$  117) alongside the molecular structure of the prevailing compound for each class (Roffet-Salque et al. 2015)

Whilst beeswax is relatively resistant to degradation due to its hydrophobic nature, limiting microbial degradation and anthropogenic transformations, partial hydrolysis of palmitic esters gives rise to *n*-alcohols with even number chain length ( $C_{24} - C_{34}$ ) (Heron et al. 1994; Evershed et al. 1997a; Regert et al. 1999, 2001). These degradation markers have been identified in a number of archaeological samples. Furthermore, palmitic acid is formed through the hydrolysis of palmitic esters, but this does not stand as a strong biomarker for degraded beeswax alone. It must be noted that wax esters, *n*-alkanes and *n*-alcohols are also present in degraded plant waxes, and therefore it is important to assess the distributions of compounds to distinguish between plant waxes and beeswax. Figure 23 shows the distributions of *n*-

fatty acids, *n*-alcohols, wax esters and hydroxy- wax esters identified in Neolithic ceramic containing beeswax (Roffet-Salque et al. 2015).

### **3.8 Challenges of ORA and considerations for study of medieval Mediterranean ceramics**

Organic residue analysis studies of archaeological ceramics have succeeded in identifying a number of different resources in various chronological and geographical contexts. The ability of ORA to provide direct chemical evidence for the contents and use of ceramic containers has enabled a broad range of archaeological questions to be addressed. This chapter has outlined a variety of methods for the extraction and interpretation of organic residues from ceramic vessels and has discussed the wide range of products that can be identified. However, before undertaking an ORA study of archaeological ceramics, a number of factors that can complicate the interpretation of organic residues need to be considered. These challenges include the complex formation processes of organic residues and their preservation and degradation through the use, depositional and post excavation lifetimes of the ceramic vessel. Furthermore, limitations in the ability to identify specific products, the complexities involving mixtures of different products and the lack of modern references are also challenges that need to be considered. Additionally, ORA studies must, with these complexities and overall archaeological questions in mind, carefully consider the sampling strategies employed. Careful consideration of the sampling strategy is essential to plan the correct methodological approach that will most benefit the aims of the investigation. This section outlines some of the key challenges faced in the study of organic residues from archaeological ceramics, paying particular focus to problems encountered in medieval Mediterranean contexts, as is the focus of this thesis. This section discusses how this thesis has considered these challenges and gives a rationale for the methodological approach and sampling strategy employed.

### 3.8.1 Preservation and degradation

Although it has been suggested that lipid residues survive in more than 80% of assemblages of domestic ceramic cooking wares worldwide (Evershed 2008b), the conditions in which a vessel is buried can greatly impact the survival of the residues. Lipid preservation in ceramics is considerably better in waterlogged or anaerobic conditions as well as frozen or very arid environments (Regert et al. 1998; Malainey, Przybylski and Sherriff 1999; Colombini et al. 2005; Copley et al. 2005c; Evershed and Connolly 1994; Gülaçar, Buchs and Susini 1989; Corr et al. 2008). Conversely, variable conditions of alternating wet and dry conditions can greatly affect the survival of lipids in the burial environment. This is due to increased microbial activity and possible leaching of more soluble lipids due to water moving through the burial environment (Dudd, Regert and Evershed 1998a; Evershed 2008b). The processes of lipid degradation can also be impacted by the pH of the soil, where, although the direct association between pH of the soil and lipid preservation is far from straightforward, acidic soils are considered the best conditions for lipid preservation (Evershed 1993, 2008b; Gregg and Slater 2010; Debono Spiteri 2012; Spiteri, Gillis and Roffet-Salque 2016). However, Drieu (2020) compared the levels of lipid yields recovered from archaeological ceramics from across Europe to the pH levels of the soil in those areas and showed that predicting the levels of preservation in different geographical areas is a complex issue. This is because there are several varying factors that influence the preservation of lipids in ceramics, including porosity and mineralogical composition of the clay, and the possibility that micro-environments may exist in different areas (Drieu 2020). Furthermore, it has been suggested that 99% of lipids in ceramics are lost through microbial degradation and the majority of this degradation occurs in the first year after burial (Dudd, Regert and Evershed 1998a; Aillaud 2001). Thus, whilst we can make assessments based on the burial conditions when excavated, these may have been very different when the majority of degradation took place.

Sicily experiences very dry periods followed by high rainfall and therefore the environmental conditions may negatively affect the level of lipid survival in the ceramics. Indeed, investigations have shown that lipids are less well preserved in

Mediterranean and Middle Eastern contexts in comparison to those from Northern Europe (Gregg et al. 2009). In this thesis, potential low lipid yield was an important consideration when designing the ORA study. Ceramic samples were obtained from sites that showed generally good preservation of other organic materials such as bone and archaeobotanical remains. Whilst it is difficult to discern the survival of lipids based on this, the survival of organic material indicated some level of preservation in the burial environment. Furthermore, a large corpus of samples were selected for analysis in this study to account for the potential of low lipid yields from some of the samples studied, whilst still ensuring that a representative sample size could still be interpreted. Additionally, it was important to consider the extraction methods used to enhance the recovery of interpretable lipid concentrations from the samples studied. Three extraction methods were used: acidified methanol extraction (AE), solvent extraction (SE) and acid butylation extraction (ABE). AE has been shown to yield higher concentrations of lipids from archaeological ceramics in comparison to conventional solvent extract and AE has enabled the recovery of appreciable lipid yields from areas from Mediterranean contexts (Drieu et al. 2021a; Breu et al. 2021; Tarifa-Mateo et al. 2019) and North African contexts (Dunne et al. 2018). Finally, to enable the greatest recovery of lipids it was important ensure that enough ceramic powder could be obtained (~2g) and thus ceramic samples were selected on this basis.

### **3.8.2 Specificity of products and lack of modern references**

As shown in this chapter, there has been great success in the variety of products that have be interpreted from degraded lipid profiles and related back to the original source processed in the ceramic vessel. These products include animal fats, plant products, beeswax, and resins. In some cases, it is possible to be more specific about these commodities by distinguishing species and taxon level through the unique biomarkers associated with these products, and through the development of compound specific isotope analysis. For example, leaf waxes of brassicas can be identified from specific markers and animal fat origin distinguished between non-ruminant, ruminant, marine and dairy. However, there still exists limitations in the ability to identify certain products, due to the lack of specific biomarkers, loss of compositional information

through degradation and limitations in analytical sensitivity etc. In the study of medieval Sicilian cuisine, it is hypothesised that a diverse range of products were processed in ceramic vessels. In this thesis, it was important to understand the wide range of products that may have been utilised in Sicily during the 9<sup>th</sup>-14<sup>th</sup> centuries to guide the analysis of organic residues. To do this, evidence was drawn from historical records, archaeobotanical and zooarchaeological remains and stable isotope analysis from previously published works and analysis carried out by the wider Sicily in Transition research project. Furthermore, based on this evidence, this thesis investigated organic residues of a variety of vegetable products with the aim to extract and identify specific profiles that might present in archaeological ceramics (Chapter 4).

### **3.8.3 Formation process and mixing of products**

The formation process of absorbed lipid residues is complex, and it is important to note that without the presence of visible residues it is not possible to distinguish whether the lipid profiles observed reflect a single cooking event or an accumulation of residues of multiple cooking events throughout the vessels lifetime. Through a yearlong experiment, Miller et al. (2020) confirmed that the lipid profiles observed are a mixed signal of products from a palimpsest of cooking events. Therefore, mixtures of products can be the result of both mixtures of products cooked together as part of a recipe, but also from sequential cooking of different products. This makes it difficult to interpret mixtures of products identified in ceramic vessels and understand whether these signals reflect certain cuisines- the combination of certain food products, or not. Furthermore, as mentioned previously in this chapter, mixtures of commodities can cause difficulties in identifying products based on their lipid profiles. For example, mixtures of animal fats with plant products can hinder the ability to identify these individually as the higher lipid content of the former can mask the signal of the lower lipid signal of the later. Additionally, as explained in section 3.6.3, mixing of different products in a single vessel can have a significant impact on the on the  $\delta^{13}\text{C}$  isotopic values obtained. Furthermore, it is also important to consider the impact of the use of organic products for technological purposes such as sealing or polishing ceramic



vessels as this may interfere with the identification of the culinary contents of the ceramics and could lead to a mixed signal of both products.

In this thesis, it was not only important to be able to identify specific products in the ceramic vessels analysed, but also to identify how products were combined in order to yield insight into specific cuisines. In the absence of food crusts, this presents challenges to the interpretation of mixtures in the ceramics analysed as part of this thesis. However, by analysing a large corpus of ceramics it can be possible to assess trends in the selection of certain products and distinguish recipes because of intentional mixing from differential uses of the ceramic. Furthermore, applying a range of extraction and analytical methods for the extraction and identification of different compounds can aid in untangling complex mixtures. Additionally, this thesis aimed to understand the impact of post-firing surface treatments on our interpretation of the original culinary contents of the ceramic by undertaking organic residue analysis of experimental pottery (Chapter 4).

### **3.8.4 Sampling strategies**

When undertaking an organic residue analysis study, it is important to carefully consider the sampling strategy of materials being investigated to ensure that viable and representative samples are selected. To enable the archaeological research questions, aims and objectives to be addressed. It is agreed that, as a rule, no less than 20 ceramic samples representative of each group should be studied (Dunne 2017). Groups include chronological period, geographical area or vessel type and should be scaled dependant on the area of study whether it regional or intra-site analysis that is being performed. It is important that the nature of samples including their typology, date and contexts are well understood. Furthermore, it is of best practice to sample ceramics where their post-excavation history is known so that post- excavation contamination can be avoided or at least, well understood. On this note, those performing organic residue analysis should have open communication with those that have excavated or studied the ceramic material and close visual examination of the sherd prior to sampling is crucial. Visual examination of the sherd can help to identify

glues or other post excavation materials (pen marking, varnishes etc.,) that should be avoided, but can also help to aid the interpretation of the use of the ceramic by identifying charred remains and/or soot marks.

Set within the scope of the wider ERC funded project Sicily in Transition, sampling acquisition and availability was somewhat limited by the wider aims of the project. To address the main research questions of the Sicily in Transition project, a variety of different analyses (ORA of ceramics, archaeobotanical analysis, zooarchaeological etc.,) were being carried out at the same time as this thesis. Therefore, sites and samples were selected, not only for the best interest of the aims of this thesis, but also to align with the multiple areas of investigation of the wider project. Whilst the main Sicily in Transition project investigated a number of sites across Sicily, dated from the 6<sup>th</sup>-14<sup>th</sup> centuries AD, this thesis focused only on 3 main areas in Western Sicily (three sites in the urban capital of Palermo, the urban centre of Mazara del Vallo and rural settlement Casale San Pietro). These sites offered the largest sample size of ceramics of known date and typology to be sampled for organic residue analysis and were best suited to answer the main aims and objectives of this thesis to understand cuisine in 9<sup>th</sup>-14<sup>th</sup>-century Sicily. Here, at least 20 samples that had been studied typologically by ceramic specialists in Rome for Vegata as part of the Sicily in Transition project or were previously studied and the ceramic typologies fully published (Palermo sites) were selected. It was ensured that each sample set represented the range of domestic cooking vessels utilised at these sites. Whilst close collaboration with the ceramic specialists was maintained and guidance was given on the sampling strategies for this thesis, initial choice of site and sample selection was undertaken by other members of the Sicily in Transition project. Full details of the sample sizes, typologies and chronologies are outlined in chapters 5 and 6 of this thesis.

### **3.9 Methodological rationale**

The application of a multifaceted organic residue approach to a large number of ceramics was essential to identify a wide range of products and begin to unravel complex mixtures in the domestic cooking pots. Three different extraction methods

were applied to ensure that a wide range of compounds were recovered from the ceramic matrix. As outlined in previously in this chapter, the application of acidified methanol extraction enabled a high lipid recovery from the ceramic matrix, solvent extraction enabled the extraction compositional information that is otherwise lost by using AE by keeping TAGs, MAGs, DAGs and wax esters intact. A further acid butylation extraction was applied to enable the extraction and recovery of very polar, small organic acids such as tartaric acid and malic acid for the identification of grapevine and other fruit products. As previously described, both a biomolecular and isotopic approach was applied for the analysis of the compounds extracted by using a range of gas chromatographic and mass spectrometry techniques. The full details of these methods are outlined in the relevant chapters (Chapters 4, 5 and 6) and in the appendices of this thesis (Appendix A and B). Only by applying a multifaceted organic residue analysis approach to a large corpus of ceramics can complex cuisines, as expected in medieval Sicilian ceramics, begin to be untangled and insight into the culinary habits of populations be gained.

### **3.10 Summary of Chapter**

This chapter has introduced the concepts of organic residue analysis of absorbed residues in archaeological ceramics. Here, the techniques required to extract and analyse absorbed residues have been outlined and the analysis and interpretation of different commodities found in archaeological ceramics has been discussed in detail. These approaches are applied to the analysis and interpretation of ceramic samples analysed in this study. This chapter has also outlined the cautionary steps required in the analysis and interpretation of commodities in residue studies particularly those applied to medieval Mediterranean ceramics. This chapter has also discussed how these considerations have shaped the research design of this thesis and guided the sampling strategies, experimental designs and organic residue analysis undertaken in this research. The next chapter discusses the results of two experiments designed to 1) understand the impact of post-firing treatments such as sealants and polishes on organic residue interpretation and 2) to identify specific biomarkers of plant products expected to be present in medieval Sicily.

# Chapter 4 Experiments and modern products

## 1.1 Introduction

This chapter aims to address two major issues in organic residue analysis of absorbed residues from archaeological ceramics and more importantly to those from medieval Mediterranean contexts. As outlined in chapter 3, the complex formation process of absorbed residues in archaeological ceramics can make interpretation of the commodities and use of vessels difficult. One reason is because mixed signals can be difficult to untangle, and it is not possible to understand whether mixed products are present from a single cooking event or multiple sequential cooking events without the presence of food crusts (Miller et al. 2020). One important issue is that organic products are not only used for processing in ceramic vessels for culinary purposes, but also for technological purposes. Thus, it is hypothesised that mixed signals may also come from a mixture of products that were used for technological purposes such as sealing and polishing as well as from products that were cooked in the vessel. Furthermore, sealants and polishes are used to waterproof the porous ceramic matrix and therefore further examination is required to understand how these may impact the absorption of lipids from the contents of the vessel and subsequently affect our ability to identify these in the archaeological record. Another significant limitation is the specificity of products that can be identified using organic residue analysis. Here the results of organic residue analysis, undertaken on modern experimental ceramic containers to understand what impact organic sealants and polishes have on our interpretation of the vessel contents, are discussed. This chapter also discusses the results of ORA of modern plant products with the aim of identifying specific biomarkers or lipid profiles that can be applied to archaeological ceramics.

## **4.1 What impact does post-firing treatments have on the interpretation of organic residues?**

### **4.1.1 Introduction and rationale**

In organic residue analysis of archaeological ceramic containers, it is not uncommon to identify mixed chemical signatures, as a result of the mixture of different commodities. However, it is difficult to assert whether these mixtures are present due to; 1) an intentional mixing of products as part of a single recipe, 2) an accumulation of sequential uses of the vessel for different products (Miller et al. 2020), or 3) a mixed signal reflecting both technological uses (sealing or polishing the vessel with organics) and the organic products that were cooked in the vessel for culinary enjoyment (Drieu, Lepère and Regert 2020). The latter has recently been addressed by Drieu et al., (2020) who investigated the use of post-firing technologies to understand how these may be identified in archaeological ceramics (Drieu, Lepère and Regert 2020). Here the researchers treated replica clay cups after firing, by rubbing with lard. Some vessels rubbed with lard were then used to simulate olive oil cooking and a number of ceramics were used just to cook lard with no treatment. In these experiments, they found that post-firing treatment had an impact on the molecular composition of the lard, which was more degraded than lard used just for cooking, likely because of contact of the lard with the surface of the ceramic at different temperatures. They also found that whilst the post-firing treatment had some impact on the absorption of the contents (olive oil), these samples yielded profiles indicative of fresh olive oil and a molecular signal from the lard rubbing. This showed that only partial waterproofing was achieved by the post-firing treatment and in archaeological samples a mixed signal may be attributed to the mixture of the post-firing treatment and the contents of the vessel (Drieu, Lepère and Regert 2020). However, this investigation only focused on one product used for post-firing surface treatment (lard) and two contained products (olive oil and lard). It is of great interest therefore to test these questions with a wider range of organic products that may have been used for post-firing treatments and/or processed for culinary purposes.

As part of the Monte Iato projects "Between Aphrodite Temple and Late Archaic House I-II" and "New wine in old bottles or old wine in new bottles?" researchers carried out organic residue analysis to understand the contents of ceramic containers found at the site of Monte Iato, Sicily. The analysis yielded interesting results and mixed products were identified in the same vessels. For example, a locally made Greek wine mixing vessel yielded biomarkers indicative of wine, resin and ruminant adipose fats. Whilst it is possible that this vessel had multiple uses, researchers hypothesised that this mixed signal could have been the result of the use of resin and animal fats used as a sealant and the subsequent use for the contents of wine (<https://www.uibk.ac.at/projects/monte-iato/ex-inhalt/>). To test this, researchers at the University of Innsbruck carried out a series of cooking experiments where they applied post-firing treatments (as sealants or polishes) of a variety of different organic products such as milk, beef tallow, beef tallow and resin, and then cooked a variety of foodstuffs in the same vessel. The details of these cooking experiments are summarised in Appendix D of this thesis and described in full by Hass (2018). This question is of great interest to the study and interpretation of organic residues from medieval Sicilian ceramics as part of this thesis' research. Therefore, in collaboration with the University of Innsbruck, the organic residue analysis of the experimental cooking pots was carried out as part of this research. Some of the results presented here from part of a report produced for the University of Innsbruck, Monte Iato project authored by Jasmine Lundy (author of this thesis) and Lea Drieu (Drieu, Lundy and Craig unpublished).

#### **4.1.2 Methods**

As mentioned, the cooking experiments were performed as part of the Monte Iato experimental project funded by the Nachwuchsförderung der Universität Innsbruck. The full details of these experiments are outlined in Haas (2018) and have been summarised in Appendix D. In brief, replica cooking pots were made from clay collected near the site of Monte Iato, Sicily. A number of these replica vessels were then sealed or polished with different commodities immediately after placing in a field fire at different temperatures (290- 494 °C). A number of products were then cooked over a period of two days (pork, beef, fish and vegetables) or poured into the ceramics over

a period of two weeks (wine, olive oil and milk). Some vessels were used to cook or pour commodities in without the addition of a post-firing treatment for comparison. The difference between sealing and polishing is that sealants were applied immediately after being in the field fire by either submerging in the sealant product or generously coating the ceramics, whereas polishing was done by rubbing the product into the internal surface of the vessel with a rose quartz stone (Haas 2018).

To assess the impact of different post-firing treatments and products, the ceramic vessels were analysed using high-throughput organic residue analysis techniques. The full methods are described in Appendix A of this thesis and these experimental ceramics were analysed using the same methods and analytical techniques applied to archaeological ceramics. In brief, the ceramic sample was first 'cleaned' by removing the outer layer of the ceramic surface (approx. 1 mm deep) to avoid exogenous contamination. Approximately 2g of ceramic powder was obtained overall for analysis by drilling into the pot and collecting the powder. Three extraction methods were applied: acidified methanol extraction method (AE) (Craig et al. 2013a), now the most commonly used method in archaeological samples that favour both high extraction yield and direct methylation for stable carbon isotopes measurements; solvent extraction method (SE/TLE) (Charters et al. 1993b), to ensure the preservation of the most-informative molecules in fatty and waxy products (triacylglycerols and wax esters); and acid butylation extraction method (ABE) (Garnier and Valamoti 2016) for the extraction of highly polar compounds (i.e. highly soluble in water), such as tartaric acid, a molecule of wine. A summary of all the samples, the treatment applied as part of the experimental analysis and the ORA methods applied is presented in Table 2.

Table 2: Summary of all experimental samples and the commodities used to seal/polish the ceramics before cooking or pouring various contents. The analysis applied to each sample is also shown. RAF= ruminant adipose fats, NRAF= non-ruminant adipose fat,

Extraction method				Acidified methanol extraction			Solvent extraction		Acidified butanol extraction
Sample	Sealing	Content	Use	GC/MS	AQUA SIM	GC-C-IRMS	GC/MS	HT GC-FID / HT GC-MS	GC/MS
1	milk	-		x	x		x	x	x
2	milk	RAF	Cooking	x	x	x	x	x	
3	milk	NRAF	Cooking	x	x	x	x	x	
4	milk	Fish	Cooking	x	x	x	x	x	
5	milk	Veg	Cooking	x	x	x	x	x	
6	milk	Wine	Pouring	x	x		x	x	x
8	Beef tallow	-		x	x		x	x	x
9	Beef tallow	Milk	Cooking	x	x	x	x	x	
10	Beef tallow	NRAF	Cooking	x	x	x	x	x	
11	Beef tallow	Fish	Cooking	x	x	x	x	x	
12	Beef tallow	Veg	Cooking	x	x		x	x	
13	Beef tallow	Wine	Pouring	x	x		x	x	x
15	Beef tallow	Oil	Pouring	x	x		x	x	x
16	Resin	-		x	x		x	x	x
18	Resin	Milk	Cooking	x	x	x	x	x	
19	Resin	NRFA	Cooking	x	x	x	x	x	
20	Resin	Fish	Cooking	x	x		x	x	
21	Resin	Wine	Pouring	x	x		x	x	x
23	Resin	Oil	Pouring	x	x		x	x	x
31	Beef tallow		Polished	x	x		x	x	
32	Oil		Polished	x	x		x	x	
33	None	Wine	Pouring	x	x		x	x	x
35	None	Milk	Cooking	x	x		x	x	
36	None	RAF	Cooking	x	x	x	x	x	
37	None	NRAF	Cooking	x	x	x	x	x	
38	None	Fish	Cooking	x	x	x	x	x	
39	None	Veg	Cooking	x	x		x	x	
40	None	Wine	Pouring	x	x		x	x	x
42	None	Oil	Pouring	x	x		x	x	x
43	None	Milk	Pouring	x	x		x	x	x



## **4.1.3 Results and discussion**

### **4.1.3.1 Lipid yield**

Out of 34 acidified methanol extracts, 27 of these yielded appreciable concentrations of lipids ranging from 9.3 mg g<sup>-1</sup> to 55 mg g<sup>-1</sup>. Although a value of 5 µg g<sup>-1</sup> is deemed the lowest lipid concentration that can be reliably interpreted in archaeological samples (Evershed 2008b) it must be noted that much higher concentrations are expected in modern samples. As explained in Chapter 3, an important caveat is that 99% of lipids are degraded through microbial action in the burial environment and a number of different factors influence the rate and process of degradation after burial. Thus, it is not possible to directly compare lipid yields of modern experimental cooking pots to those obtained from archaeological containers. The lipid yields obtained were very variable, even between vessels that had undergone the same treatment. This variability is likely due to multiple parameters that are not easy to control in the experiments, such as variable surface temperatures, non-homogeneous minerals in the ceramic paste, and biases in sampling different parts of the vessel, all of which may impact the lipid concentrations that are obtained (Charters et al. 1997).

### **4.1.3.2 Animal products versus plant products**

Here, the lipid yields depict, in agreement with other published literature, that even in non-degraded samples plant products yield very low concentrations of lipids in contrast to animal fats (Evershed 2008b, 2008a; Steele, Stern and Stott 2010; Hammann and Cramp 2018). Vessels used to cook vegetables without the addition of post-firing treatments yielded very low concentrations of lipids (<5 µg g<sup>-1</sup>) in comparison to vessels used to cook milk, ruminant and non-ruminant products (ranging from 3254-13227 µg g<sup>-1</sup>). These results can be attributed to the known low concentrations of lipids in plant products compared to animal products (Evershed 2008a). Previous experiments have shown that plant biomarkers identified in archaeological ceramics are the result of multiple or prolonged cooking events

(Evershed 2008b, 2008a; Steele, Stern and Stott 2010; Hammann and Cramp 2018). However, olive oil poured into the ceramic vessel yielded a relatively high lipid yield > 4000  $\mu\text{g g}^{-1}$ . This is likely due to the higher fat content in olive oil than in leafy vegetable products and its higher permeability into the ceramic as it is in liquid form. Furthermore, it has been previously shown that, for lipids from leafy vegetables to permeate the ceramic, repeated cooking events need to take place (Charters et al. 1997). In this experiment, only two cooking events took place, which is not sufficient for the release and subsequent absorption of lipids from vegetable products. Conversely, olive oil was repeatedly poured into the ceramic vessel over a period of 2 weeks, likely increasing the chance of absorption into the ceramic matrix. The ceramics used to contain just wine with and without the addition of a post-firing treatment also yielded low concentrations of lipids (<3  $\mu\text{g g}^{-1}$ ). However, these products contain low concentrations of fatty acids and small organic acids present in fruit products are not easily extracted using acidified methanol extraction and require another extraction method as described below. Interestingly, the vessel used to cook fish products (Red fish, sea bass, coalfish and salmon) yielded a lipid concentration almost 10 times lower than other animal products (441.7  $\mu\text{g g}^{-1}$ ). This could be the result of a low-fat content of the fish used.

#### **4.1.3.3 Impact of the sealant**

Products used for sealing have a mixed impact on the overall lipid yield. This is unsurprising as a number of different factors influence in the absorption of lipids into the ceramic matrix and their subsequent survival after cooking. In some cases, the addition of a sealant increased the lipid content and in other cases, it had limited impact on the overall yield. Vessels used to cook vegetables were slightly impacted by the addition of post-firing sealant. Here, the addition of beef tallow had little effect on the yield (3.5  $\mu\text{g g}^{-1}$ ), but the addition of milk as a sealant produced a slightly higher yield (9.3  $\mu\text{g g}^{-1}$ ). The low lipid yield observed despite the addition of beef tallow is somewhat surprising due to the high fat content of beef tallow. However, it is possible that, during the process of the post-firing treatment, high temperatures will have led to the degradation of lipids of the sealant. Furthermore, during the cooking of vegetable

products with the addition of water, there could have been some migration of lipids out of the ceramic matrix.

On the other hand, vessels that were used to contain wine and sealed with beef tallow or resin and beef tallow combined yielded much higher concentrations of lipid, showing that the sealant had a significant impact (4239 and 3053  $\mu\text{g g}^{-1}$ , respectively). This further strengthens the observation that the cooking mode could have a significant impact on the sealant. The use of milk as a sealant in this case, however, still produced low lipid yields for wine contents (2.9  $\mu\text{g g}^{-1}$ ). This is likely due to the lower fat content of fresh milk in comparison to beef tallow. However, additional molecules present in fresh milk could also influence this. A high abundance of proteins in fresh milk, for example, could plug the pores of the matrix, which would affect the ability of the lipids to absorb and bind to the ceramic matrix. Again, of interest, the lipid yields obtained from vessels used to cook fish products were variable when treated with a post-firing sealant. Whilst the addition of milk and beef tallow as a sealant had minimal impact on the lipid yields obtained, high lipid yields were obtained with the addition of beef tallow and resin together depicting the clear impact of the sealant (1052  $\mu\text{g g}^{-1}$ ). These results further highlight the complexities associated with products used as a sealant. The higher yields obtained with the addition of beef tallow and resin as a sealant suggests that the resin has a role in either aiding the absorption of lipids into the ceramic matrix or acts as a barrier, reducing the migration of lipids out of the ceramic matrix during the cooking process.

#### **4.1.3.4 Impact of polishing**

All samples that were polished with beef tallow and oil yielded low concentration (less than 10  $\mu\text{g g}^{-1}$  after acidified methanol extraction). This is likely due to the minimal absorption of these products into the ceramic matrix due to the small quantity of organic product. Treatment of ceramic vessels through the process of polishing is therefore unlikely to be identified in archaeological vessels.

#### 4.1.3.5 Molecular composition

In general, the molecular profiles obtained are in accordance with the data published in the literature for animal adipose fats, milk, olive oil and resin as previously outlined in full in Chapter 3. For vessels that were not treated with a sealant or polishing and solely used to cook unmixed products, the detail of these lipid profiles are not described in full here as there is already sufficient literature of these products and their detail is described in the previous Chapter 3. For those that yielded appreciable lipid concentrations, these extracts were dominated by free fatty acids, where palmitic (C<sub>16:0</sub>) and stearic (C<sub>18:0</sub>) acids were most dominant. Linear and branched odd carbon-numbered fatty acids (mainly C<sub>15:0</sub> and C<sub>17:0</sub>) were detected in the majority of samples. The presence of branched chain fatty acids are due the bacterial degradation in the gut of ruminants (Evershed 1993), and were observed in all samples that contained ruminant products; ruminant adipose, milk or tallow, regardless of whether these products were used for sealing or cooking. Furthermore, branched chain fatty acids were also observed in the vessel used to cook non-ruminant adipose, this could be explained by the fact that these animals may have consumed dairy products and highlights that caution needs to be taken when interpreting ruminant adipose fats based on the presence of branched chain fatty acids (Dudd, Evershed and Gibson 1999). Fatty acids were also extracted using solvent extraction methods, but generally in lower concentrations than acidified methanol extraction as shown by previous works (Correa-Ascencio and Evershed 2014). Diacids (resulting from the degradation of unsaturated fatty acids) were present in a number of samples ranging from carbon atom lengths C<sub>7</sub> to C<sub>14</sub>. These smaller chain acids are also more susceptible to degradation in the burial environment than longer chain acids, and therefore these distributions may not reflect what would be observed in the archaeological record.

Diterpenoids (primaric and isoprimary acids, abietic acid, dehydroabietic acid (DHA), diDHA, and 7-oxo-DHA, and pimara-8,15-dien-18-oic acid, were present, as TMS or methylated derivatives, in all vessels where resin was used to seal the vessel. Interestingly, traces of dehydroabietic acid were also observed in the TLE extracts of samples 37 and 38 used only to cook non-ruminant adipose and fish products

respectively, and not sealed with resin. It is possible that these compounds could have absorbed into the pottery matrix from the fire (Reber et al. 2019a).

#### 4.1.3.5.1 Wine

Another type of acidified method, developed by Garnier and Valamoti (2016) (Appendix A), was used to extract small organic acids from 17 samples to specifically detect tartaric acid, a biomarker of fruit, generally used to identify wine in archaeological samples (Garnier and Valamoti 2016). Tartaric acid was successfully extracted in high quantities ( $>400 \mu\text{g g}^{-1}$ ) in 5 samples all of which contained either red or white wine. The concentration of tartaric acid varied from  $519.4 \mu\text{g g}^{-1}$  to  $4442.7 \mu\text{g g}^{-1}$ , agreeable with studies that suggest high quantities of this compound observed in archaeological samples can be indicative of the use of wine (Guasch-Jané et al. 2004, 2006; McGovern 2009). However, it is important to mention that tartaric acid cannot be used as definitive evidence of the presence of wine in an archaeological vessel, as this compound is also present in other fruits (Drieu et al. 2020, 2021b).

Of note, sample 40 was not treated with a post-firing sealant, prior to the addition of wine to the vessel, and yielded the lowest concentration of tartaric acid ( $519.4 \mu\text{g g}^{-1}$ ). This is contrary to what may be expected, whereby it might be expected that the prior addition of the sealant will prevent absorption of wine into the ceramic matrix. However, these results suggest that the sealant instead had a positive impact on the absorption/preservation of tartaric acid inside the ceramic matrix. The highest concentration of tartaric acid was observed in samples that were sealed with resin and beef tallow. This shows that only partial waterproofing of the vessel was achieved with the sealant as previously shown (Drieu, Lepère and Regert 2020), and indicates that the resin may actually help to protect the tartaric acid inside the ceramic matrix. Tartaric acid can bind to the ceramic matrix, but may also bind to the resin or, as previously described (section 4.2.3.1.2), the resin bound to the surface could act as a barrier limiting the migration of tartaric acid out of the ceramic matrix. Thus, the addition of a sealant could aid survival of tartaric acid in archaeological samples, as tartaric acid is soluble in water and can be lost in the burial environment via leaching unless it is

chemically bound to the ceramic matrix. However, more experiments are required to test this hypothesis.

Furthermore, a number of sugars were detected in samples of vessels that contained wine. However, sugars are difficult to precisely identify by GC-MS as the column used in GC is not adapted to correctly separate the sugars, and the electron ionisation source in the MS does not allow the production of sufficiently specific mass spectra to differentiate the multiple isomers of sugars. It is important to note that such compounds are unlikely to be preserved in archaeological ceramics due to their solubility in water and their rapid consumption by microorganisms, and therefore are not discussed further here.

#### **4.1.3.5.2 Thermal transformation markers**

Thermal transformation markers: ketones (K<sub>31</sub>, K<sub>33</sub> and K<sub>35</sub>), produced by condensation of saturated fatty acids (Raven et al. 1997; Evershed et al. 1995), and APAA (C<sub>18</sub>) resulting from the cyclisation of unsaturated fatty acids that are present in animal fats (Hansel et al. 2004; Bondetti et al. 2021), were detected in samples sealed with animal fats (milk and tallow). However, these were absent from the pots that have been used to cook these commodities. The formation of these ketones in vessel samples that have only been sealed with organic products may be due to the higher temperatures used for sealing these products and their absence in vessels used to cook these products due to the lower cooking temperature < 300 °C (Evershed et al. 1995; Raven et al. 1997; Drieu, Lepère and Regert 2020). Therefore, caution should be taken when ketones are observed in archaeological samples as they can reflect technological processes rather than cooking processes. However, it is important to note that multiple cooking events in the same container can lead to the formation of these ketones, and therefore it is not probable to simply exclude cooking of animal products as the reason for these formations in archaeological samples. Such formation of ketones through the process of sealing has already been observed in previous experiments, whereby cups that were rubbed with lard yielded odd mid-chain ketones, whereas those used to simply cook lard did not (Drieu, Lepère and Regert 2020).

The formation of APAAs has not yet been attributed to heating during the sealing process. These observations are interesting, as such thermal transformation markers are generally used to identify cooking of commodities in pots in the published literature (Hansel et al. 2004; Shoda et al. 2018; Bondetti et al. 2021). It is possible to suggest that APAAs were not formed in the vessels only used for cooking commodities due to lower temperatures in the cooking process as opposed to the sealing process. New research has indicated that, in contrast to previous experiments that show APAAs are formed under long heating processes (>17 hours at >270 °C), APAAs can in fact be formed relatively easily after 1-5 hours, but still at temperatures > 200 °C (Bondetti et al. 2021). However, the same experiment by Bondetti et al., (2021) did confirm that APAAs are more likely to form when in direct contact with the vessel, which would explain the effect observed in this research, as the sealant is in more direct contact with the vessel than commodities processed through boiling. These experiments therefore lead us to reconsider the archaeological interpretations that have been made so far and might offer new insights into the identification of sealants applied to archaeological ceramics.

#### **4.1.3.5.3 Triacylglycerol profiles**

Several samples extracted using solvent extraction yielded intact triacylglycerols (TAGs) which have variable profiles typical of the fats used in the experiments (Figure 24). All samples used to contain milk yielded a wide TAG profile (T<sub>40</sub> to T<sub>54</sub>) characteristic of dairy products, despite being sealed with other products. Vessels used to cook ruminant animal fat (samples 2 and 36) yielded profiles typical of ruminant animal fat despite the addition of milk as a sealant with a shorter profile (T<sub>46</sub> to T<sub>54</sub>) centred on T<sub>52</sub>. Non-ruminant fats are distinct from ruminant fats by the clear predominance of T<sub>52</sub> and all samples that contained non-ruminant adipose fat clearly displayed this profile (Evershed et al. 2002). Furthermore, the TAGs of fish are made up of a large proportion of unsaturated fatty acids and no TAG profiles for fish oils and vegetables in archaeological contexts are available in the literature. Here, TAGs observed in vessels used to contain fish may be interpreted as ruminant adipose fats based on their short profile, centred around T<sub>52</sub>, even when no sealant has been applied. These samples therefore show that sealant products had little impact on the

TAG profile and are in this case invisible, as shown previously by Drieu, Lepère and Regert (2020).

On the other hand, samples that were sealed with these products (milk, beef tallow and resin), but had no content cooked or poured in the vessels yielded the relative TAG profiles reflective of the commodities used as sealants. Here, both samples that used beef tallow and beef tallow with the addition of resin, but had no commodities cooked in the vessels yielded clear TAG profiles indicative of ruminant adipose fats. Furthermore, as wine contains no TAGs it is clear that the ruminant TAG profiles obtained for samples that contained wine and were sealed with either beef tallow or resin and beef tallow together, clearly come from the sealant of the vessel. Additionally, the sample sealed with milk and not used for cooking other products also yielded a profile indicative of dairy products.

Furthermore, the sample that was used to contain mixed vegetable products did not yield TAG profiles when cooked on its own or with ruminant adipose products as a sealant. However, it did when sealed with milk, but this profile reflects non-ruminant adipose fats rather than dairy products. It is possible that this sample was either contaminated during the cooking process or, as plant products contain predominantly unsaturated TAGs, these were not identified properly using standard GC-MS techniques. Additionally, the TAG profiles of samples used to contain olive oil are difficult to interpret and would likely be interpreted as degraded animal products.

These results show that in most cases the subsequent cooking or pouring of commodities into the ceramic completely masks the profile of the original sealant if the lipid content of the commodity is higher than the sealant. However, in the case of wine and vegetable products the TAG profiles were indicative of the sealant used instead. This may suggest that profiles of ruminant adipose products and wine together found in archaeological ceramics may in fact represent the sealing of the ceramic with animal products such as beef tallow and then used to contain wine. However, these results may also come from intentional mixing of products for recipes or through sequential uses of the ceramic for different purposes. Therefore, it has been shown that TAG



profiles would not clearly distinguish between commodities used to seal vessels in the archaeological record.

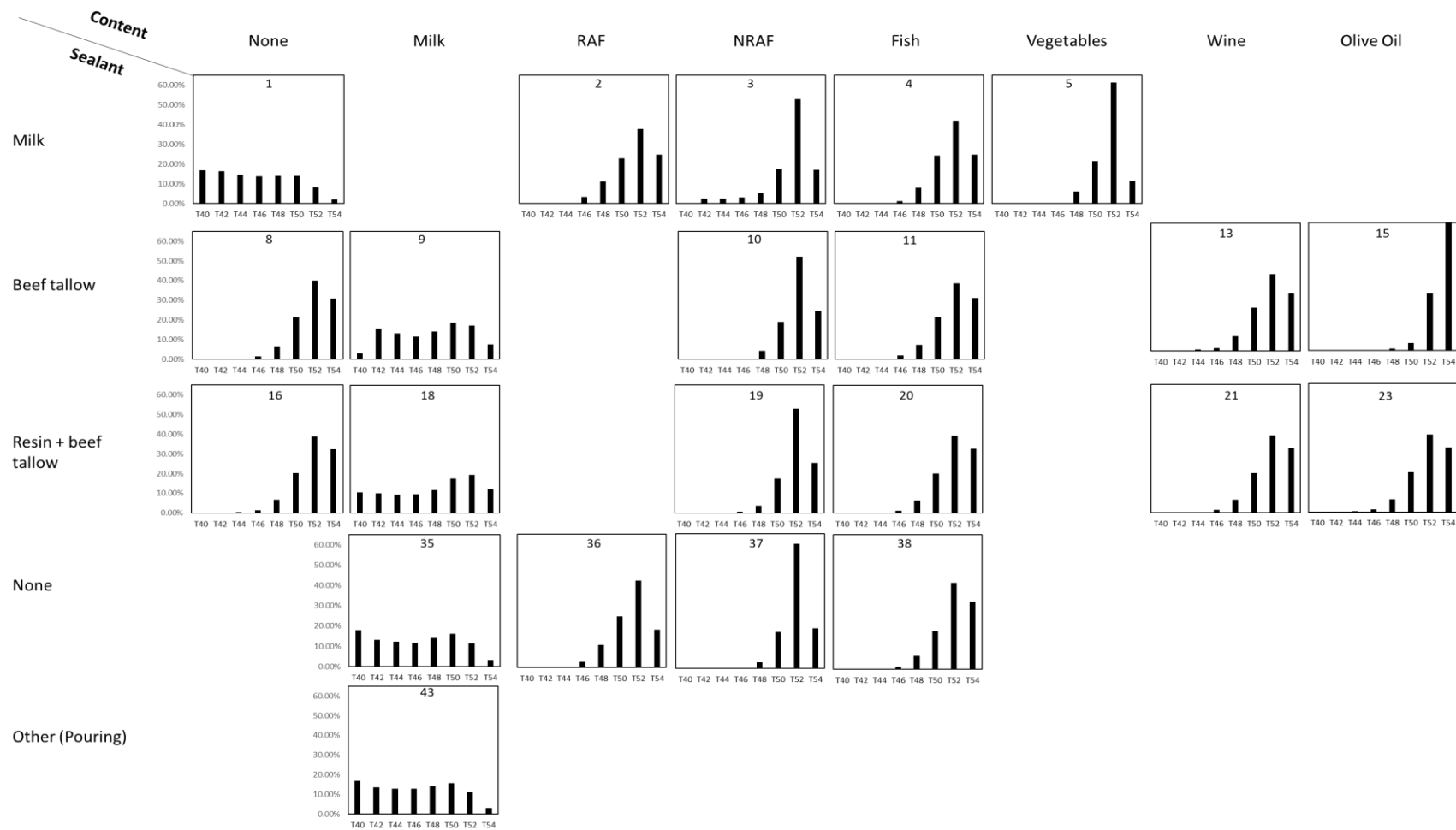


Figure 24: Triacylglycerol (TAG) distribution graphs showing the percentage distribution of each TAG from T40-T54 from experimental cooking pots sealed with different products and contained different contents

#### 4.1.3.6 Compound specific isotope analysis

Gas Chromatography-combustion-Isotope Ratio Mass Spectrometry (GC-c-IRMS) techniques were applied to 12 of the samples analysed here to further assess the ability to differentiate the sealant from the commodity cooked or poured inside the vessel. It was not possible to analyse all samples, either because of low lipid yields or due to the difficulty in breaking TAGs through acidified methanol extraction due to the high concentrations in modern samples. GC-c-IRMS is used to determine the compound specific  $\delta^{13}\text{C}$  values of the most ubiquitous molecules identified in archaeological residues ( $\text{C}_{16:0}$  and  $\text{C}_{18:0}$ ). Here, less depleted  $\delta^{13}\text{C}$  values seen in non-ruminant fats compared with those in ruminant fats are due to differences in diet and the metabolic and biochemical processes, as explained in full in Chapter 3. GC-c-IRMS can also help to identify whether these lipids originate from an adipose (animal tissue) or dairy source (Copley et al. 2005; Dudd and Evershed 1998). It must be noted that the modern meat samples used in this study cannot be directly compared to archaeological samples as the meat was not obtained from animals with a known controlled diet (i.e. Roffet-Salque et al. 2017). The effect of these modern diets to the  $\delta^{13}\text{C}$  values cannot be measured, but here the compound specific isotope values have been plotted in graphical form as  $\Delta^{13}\text{C}$  against  $\delta^{13}\text{C}_{16:0}$ .  $\Delta^{13}\text{C}$  are not influenced by dietary and environmental conditions of the consumer as isotopic fractionation of  $\text{C}_{18:0}$  and  $\text{C}_{16:0}$  is similar (Copley 2002). Generally,  $\Delta^{13}\text{C}$  values  $<-3.3\text{‰}$  are typically associated with ruminant dairy, values between  $-3.3\text{‰}$  and  $-1.0\text{‰}$  are associated with ruminant adipose and values above  $-1.0\text{‰}$  can be considered as non-ruminant (Craig et al. 2012) (Figure 25).

For samples that were used to cook fish products with no sealant these values generally fall within the range expected for marine products with  $\delta^{13}\text{C}_{16:0} >-27\text{‰}$ . However, in the cooking experiments performed, these vessels were used to cook a mixture of different fish with the addition of a commercial fish paste, and therefore these values are not directly comparable to archaeological contexts. Nonetheless, the addition of milk as a sealant had little impact on these values, but the addition of beef tallow as a sealant yielded values more depleted than would be expected for marine products and would be interpreted as ruminant adipose fats or a mixture of ruminant adipose and

non-ruminant products. This was likely due to the low lipid content of the fish products processed in the vessel. Thus, it is important to note that in archaeological contexts the small addition of ruminant products to marine products in the same vessel could impact the compound specific values. Therefore, it is important to consider both biomolecular data and compound specific isotope values together.

For samples that were used to contain milk these values fell well within the range of other modern dairy ranges  $< -3.3$  ‰, the addition of beef tallow as a sealant did not have any impact on these values. Thus, we might expect archaeological vessels used to process milk can be clearly distinguished despite the small addition of other products used to seal the vessel. For the vessels that contained non-ruminant products, the use of milk as a sealant had no impact on the isotopic values, as these were similar to the values obtained from the vessel containing non-ruminant products and no sealant and fell well within the expected range of non-ruminant products ( $> -1.0$  ‰). These results are also consistent with the TAG profiles observed where the use of milk as a sealant had no impact on the distribution. However, the use of beef tallow and resin with beef tallow as a sealant did influence the isotopic values which fell within the range of ruminant adipose fat ( $< -1.0$  ‰). Thus, although the  $\delta^{13}\text{C}$  modern values cannot be compared to those in archaeological contexts, it is possible that the use of beef tallow as a sealant would affect the interpretation of the contents of archaeological vessels. The influence of beef tallow on the  $\delta^{13}\text{C}$  values of these samples is likely due to the higher lipid content of this product as demonstrated in the higher lipid yields obtained from samples sealed with beef tallow in comparison to vegetable products, milk and even the non-ruminant meat and fish.

For samples that were used to cook ruminant adipose fat, these yielded values that fell within the range of ruminant adipose, although with slightly enriched  $\Delta^{13}\text{C}$  values falling  $\sim -1.0$  ‰, and the use of milk as a sealant did not have a significant impact on these values. However, the isotope values obtained from the vessel used to cook vegetables and sealed with milk show that the milk sealant dominated the molecular profile and therefore the isotope values obtained point to a sole dairy source ( $< -3.3$  ‰). These results are expected as vegetables contain very low concentrations of lipids in

comparison to animal fats and the milk was readily absorbed into the ceramic matrix via the sealing process.

These results show that the impact of sealants on the isotopic values are rather variable. In cases where ruminant and non-ruminant products are mixed together it may not be possible to distinguish between the two based on the isotopic values. Furthermore, the use of milk products as a sealant clearly masked the presence of vegetable products cooked in the vessel and therefore could lead to a misinterpretation of the vessel use. It is concluded that the use of compound specific isotope data would not help to distinguish between the commodities processed in the vessel and those used to seal the vessel, but it does highlight the importance of combining both isotopic and molecular data together when complex mixtures are expected, as would be the case in medieval Sicily.

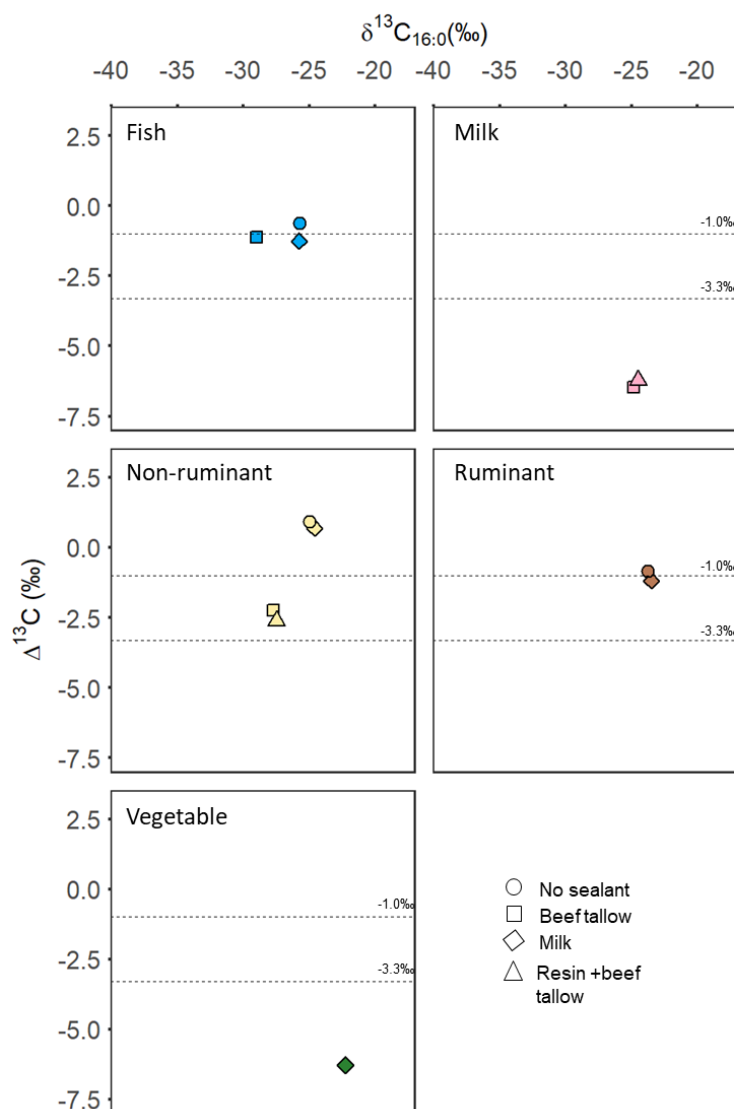


Figure 25: Plot of  $\Delta^{13}\text{C}$  against  $\delta^{13}\text{C}_{16:0}$  of experimental pot sherds. Each graph is labelled with the commodity cooked inside the vessel and each shape represents the product used to seal the vessel. Values  $<-3.3\text{‰}$  are typically associated with ruminant dairy, values between  $-3.3\text{‰}$  and  $-1.0\text{‰}$  are associated with ruminant adipose and above  $-1.0\text{‰}$  can be considered as non-ruminant based on other modern reference values (Craig et al. 2012).

#### 4.1.4 Overview and conclusion

The interpretation of the molecular signal of each sample is presented in Table 3. The interpretation is based on the addition of all the molecular and isotopic indices identified by all the analytical methods, which are presented in full in the supplementary information file (S1. data) of this thesis. These final interpretations were made blind to ensure that there was no bias in the interpretation by knowing the original contents and sealing of the vessels. In the majority of cases, it was possible to

identify the commodities contained in the vessel when there was no other commodity used as a sealant. It should be noted that the interpretation is quite easy, due to the absence of degradation of the organic signal. Triacylglycerols, in particular, allow the precise identification of the type of fat, but these are likely to have completely disappeared in an archaeological context. The contents of sample 39 could not be identified as a low quantity of lipids were obtained. This further highlights the difficulty in identifying vegetable products as they need to be processed for long periods or multiple times in the same vessel for lipids to be absorbed due to their low lipid content (Hammann and Cramp 2018; Evershed 1993). For samples that were sealed with different products the interpretation was of variable success. For samples that were sealed with milk generally there was no impact of the sealant on the identification of the contents of the vessel, apart from sample 5 which contained vegetable products. Here, the profile of milk could be clearly identified. For those that were sealed with beef tallow, in most cases the contents of the vessels were correctly identified by using a combination of molecular and isotopic data. However, fish products could not be correctly identified in this case and a mixture of ruminant and non-ruminant products were observed. Furthermore, the addition of resin was clearly identified as a sealant.

Table 3 Summary of the interpretation based on ORA of all experimental vessels. RAF= ruminant adipose fats, NRAF= non-ruminant adipose fat,

Sample				Interpretation	Thermal transformation marker
Sample	Sealing	Content	Use		
1	milk	-		Dairy product	Heated fat (APAAs)
2	milk	RAF	Cooking	Ruminant fat	
3	milk	NRAF	Cooking	Non-ruminant	
4	milk	Fish	Cooking	Aquatic fat	
5	milk	Veg	Cooking	Undetermined fat	Heated fat (APAAs)
6	milk	Wine	Pouring	Fruits (possibly grapes)	Heated fat (APAAs)
8	Beef tallow	-		Animal fat (probably ruminant)	Heated fat (ketones, APAAs)
9	Beef tallow	Milk	Cooking	Dairy product	Heated fat (APAAs)
10	Beef tallow	NRAF	Cooking	Animal fat, possibly a mixture of ruminant and non-ruminant	Heated fat (APAAs)
11	Beef tallow	Fish	Cooking	Animal fat (maybe a mixture including ruminant fat)	Heated fat (APAAs)
12	Beef tallow	Veg	Cooking	No signal	
13	Beef tallow	Wine	Pouring	Animal fat (probably ruminant) + fruits (possibly grapes)	Heated fat (ketones, APAAs)
15	Beef tallow	Oil	Pouring	Plant oil	Heated fat (ketones, APAAs)
16	Resin	-		Resin + Animal fat (probably ruminant)	Heated fat (ketones)
18	Resin	Milk	Cooking	Dairy product + resin	Heated fat (APAAs)
19	Resin	NRFA	Cooking	Animal fat (possibly a mixture of ruminant and non-ruminant) + Resin	Heated fat (APAAs)
20	Resin	Fish	Cooking	Resin + Animal fat	
21	Resin	Wine	Pouring	Animal fat (probably ruminant) + Resin + fruits (possibly grapes)	Heated fat (ketones)
23	Resin	Oil	Pouring	Plant oil + Resin	Heated fat (ketones, APAAs)
31	Beef tallow		Polished	Undetermined fat	
32	Oil		Polished	Undetermined fat	
33		Wine	Pouring	Fruits (possibly grapes)	
35		Milk	Cooking	Dairy products	
36		RAF	Cooking	Ruminant fat	
37		NRAF	Cooking	Non-ruminant fat	
38		Fish	Cooking	Aquatic fat	
39		Veg	Cooking	No signal	
40		Wine	Pouring	Fruits (possibly grapes)	
42		Oil	Pouring	Plant oil	
43		Milk	Pouring	Dairy products	



The main conclusion of these experiments is that post-firing treatments had a variable impact on the interpretation of organic residues, and it is concluded that in the archaeological record the ability to distinguish between post-firing treatments and the culinary contents of the vessel would be unlikely. However, these results have highlighted the problems that may be encountered when analysing archaeological vessels. It needs to be considered that the commodities identified through lipid analysis could indicate the sealant rather than those processed in the vessels for culinary purposes or a mixture of both. Furthermore, the impact of the sealant on the absorption and survival of lipids in the ceramic matrix is complex and can vary dependant on the organic substance used as the sealing property. As previously shown, some sealants offer only some protection of the absorption of lipids into the ceramic matrix such as lard (Drieu et al., 2020). Conversely, other molecules present in the sealants, such as proteins, likely impact the absorption of lipids into the ceramic matrix, but this cannot be easily understood through organic residue analysis alone. It is important to be aware of these mixtures when interpreting archaeological ceramics and to consider the various factors which play a role in the absorption, survival and subsequent extraction of lipids into and from the ceramic matrix. These experiments have highlighted the importance of applying a multifaceted organic residue approach with multiple extraction methods and analytical techniques to enable the identification of multiple products in the same vessel. Different extraction methods enabled a wide range of molecules to be recovered and the use of different GC techniques enabled their identification, as in a number of cases products were masked by other products and not identifiable with some techniques, but were with others. Furthermore, the presence of heated animal fats can be identified thanks to the presence of ketones and APAAAs. However, they can be formed during the use of the pot or the treatment of surfaces to modify their properties (colour, permeability, etc.). Future analysis of homologous vessels after burial should be carried out in order to evaluate the degradation of the organic signal and the stability of molecular markers (sugars, TAGs, unsaturated fatty acids). Further research should investigate the complexity of lipid absorption, survival and extraction and how these are impacted by the use of different post-firing treatments.

## 4.2 Organic residue analysis of modern vegetables

### 4.2.1 Introduction and rationale

As discussed in Chapter 3, a number of different plant products have been successfully identified in archaeological ceramics including specific profiles of plant oils and plant waxes. However, low lipid yields in comparison to more fatty products such as animal fats or the high susceptibility to degradation of plant oils, and our inability to be more specific about plant taxa due to a lack of specific biomarkers has somewhat limited our identification and interpretation of the use of certain plant products in the archaeological record. Plant products were undoubtedly an important aspect of medieval Sicilian cuisine and historical sources regularly document the uses of vegetables such as turnips, cabbage, cauliflower, onion, garlic and leek in early medieval Islamic cuisine (Lewicka 2011; Riḍwān 1984, 132–137). A major consideration when investigating cuisine in medieval Sicily is the Islamic ‘Green revolution’ first proposed by Watson (1974). Here Watson (1974) documents the introduction of 17 plants that arrived in the Mediterranean from the Middle East, associating the movement of Islamic communities with the introduction of these resources. These plants include spinach, cotton, durum wheat, sugar, citrus fruits and possibly broomcorn millet (Watson 1974, 1983). Watson’s thesis has since been debated in the scholarly community, and understanding the extent of this ‘green revolution’ in Sicily is important to our understanding of its history. Some aspects of Watson’s thesis have been revived through both historical and archaeobotanical research (Van der Veen 2010; Fuks, Amichay and Weiss 2020; Primavera 2018a; Fiorentino et al. forthcoming; Fiorentino, Lumaga and Zech-Matterne 2018), but to what extent these plants only appeared in Sicily at the onset of the Islamic political regimes, or were already present in the region, has been difficult to ascertain (Decker 2009). However, it is without doubt that with the Islamic green revolution, certain vegetables, fruits and cereals gained new importance. It is therefore important to understand how these may have been incorporated into cuisines, of which their identification in ceramic vessels would greatly aid our understanding.

To aid the research set out in this thesis, it was important to investigate whether it was possible to identify these ‘revolution plants’ and a variety of other plants in archaeological ceramics. If these plants could be identified, this would aid our understanding of the presence of these plants in medieval Sicily and enable a crucial understanding of their importance as part of local cuisines. To do this a variety of modern plants were extracted and analysed using the same well-established methods in organic residue analysis as the archaeological ceramic samples (Craig et al. 2013a; Correa-Ascencio and Evershed 2014; Garnier and Valamoti 2016; Craig et al. 2007; Dudd and Evershed 1998) (Appendix A). The aim was to identify possible biomarkers or characteristic molecular profiles to understand how these commodities may have been processed in the ceramic vessels. Here this research analysed aubergine, spinach and lemon, all of which are thought to have appeared/developed in Sicily as part of the Islamic ‘Green revolution’ (Watson 1974, 1983). Other plant products were not analysed for various reasons, such as broomcorn millet, as a biomarker for this is already well known in archaeological ceramics (Heron et al. 2016; Bossard et al. 2013) and watermelon, because the high water and sugar content was unlikely to yield any usable information. Additional products including, *Giri di Campania* (leaves from a local turnip), *Cavoli* (Italian for cabbage), kale, leek, onion and carrot were also analysed as hypothesized products that may have been processed in the vessels. The samples were either bought at the local market in Castronovo, Sicily or at the local supermarket in the UK (Table 4).

#### **4.2.2 Results**

All samples delivered mainly saturated ( $C_{16:0}$ -  $C_{30:0}$ ) and unsaturated (i.e.,  $C_{16:1}$ ,  $C_{16:3}$ ,  $C_{18:1}$ ,  $C_{18:2}$ ,  $C_{18:3}$ ) fatty acids, sugars and sterols. Since sugars are soluble in water and rapidly exposed to microbial degradation in the burial environment, they were not considered here as they are unlikely to survive in the archaeological record. Furthermore, as discussed in Chapter 3, unsaturated fatty acids are also rapidly degraded in the burial environment and can be altered through the use of the pot. Unsaturated acids do not survive well due to the weak single bonds that are susceptible to oxidation (Heron, Evershed and Goad 1991; Evershed, Copley and Dickson 2008;

Heron and Evershed 1993). Thus, unsaturated fatty acids and their relative abundances in modern samples cannot be directly compared to archaeological samples. Therefore, the fatty acid profiles are not discussed further here but are summarised in Table 4.

#### **4.2.2.1 Sterols**

$\beta$ -sitosterol, a ubiquitous phytosterol across the plant kingdom, was present in all samples except spinach. In addition to  $\beta$ -sitosterol, stigmasterol was also identified in aubergine, carrot and both the waxy peel and pulp of the lemon. Lemon also yielded campesterol, another ubiquitous phytosterol in the plant kingdom. Furthermore, the onion, in addition to  $\beta$ -sitosterol, also yielded cycloartenol.  $\beta$ -sitosterol, stigmasterol, campesterol and cycloartenol are present in a variety of plants and are not distinctive sterols and therefore cannot be used as a specific biomarker for these vegetables. However, their presence in archaeological ceramics can help to indicate the presence of non-specific plant products. Of note, spinach yielded a specific sterol signature composed of  $\alpha$ -spinasterol and 7-stigmastenol, of which  $\alpha$ -spinasterol dominates, shown in Figure 26 a and b. This unique set of sterols has been previously identified in modern food studies and stands spinach apart from other plant products such as brassica and barley for example (Rochester, Kjellbom and Larsson 1987). However,  $\alpha$ -spinasterol and 7-stigmastenol have not been previously identified in archaeological ceramics. The presence of these sterols in ceramics in this research would help to confirm the presence of spinach in medieval Sicily and its use in culinary practices. However, an important caveat, as noted in Chapter 3, is that sterols are subject to degradation and may not survive in the archaeological record.

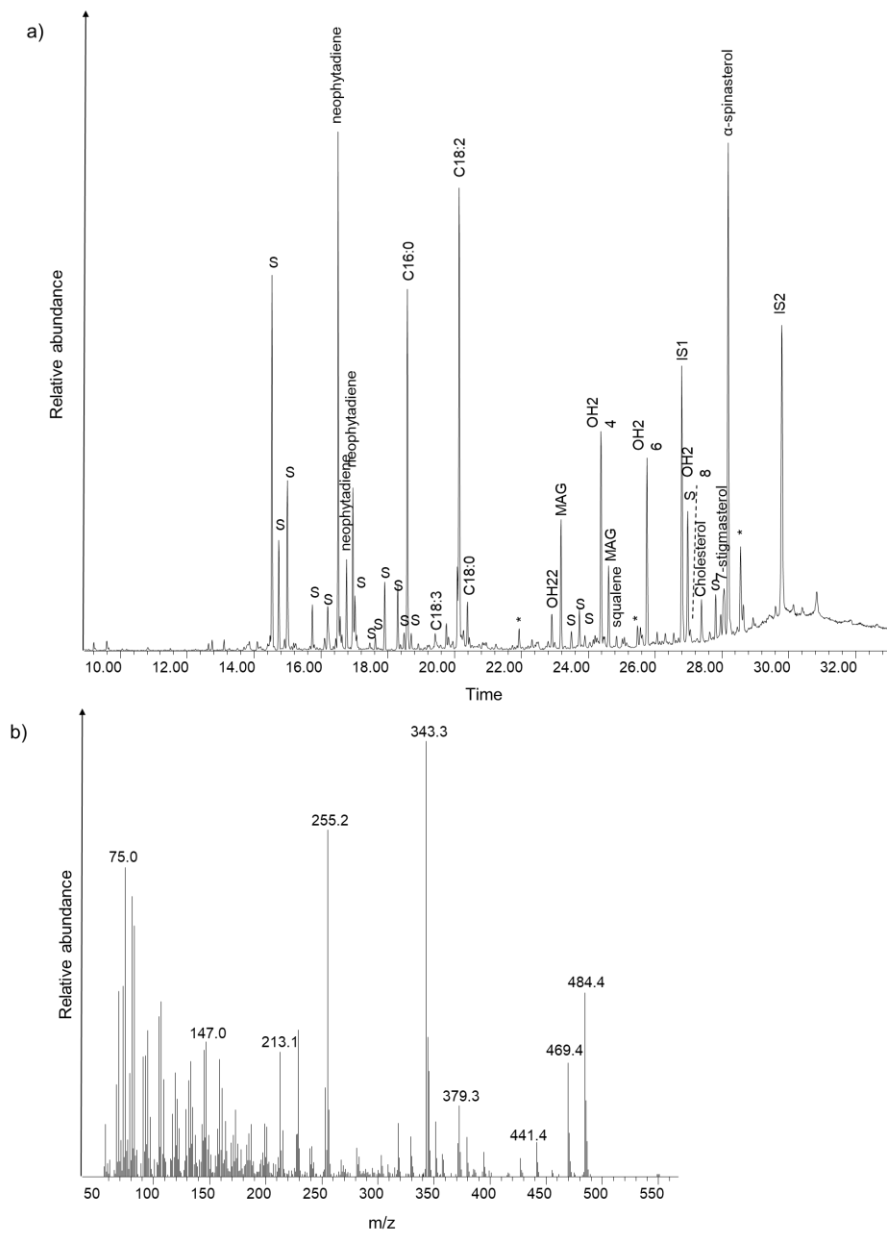


Figure 26: a) AE TIC chromatogram of sample CA\_MOD\_20 (spinach) showing the presence of fatty acids, alcohols (OH), unidentified sugars (S) and  $\alpha$ -spinasterol and 7-stigmasterol. b) Scan mode mass spectrum of  $\alpha$ -spinasterol.

Table 4: Summary of molecular information of all modern vegetables analysed

Sample name	Commodity	Origin	Part	Saturated even number fatty acids	Saturated off number fatty acids	Unsaturated fatty acids	Ketones	Alkanes	Alcohols (TMS)	Sterols	Other
CA_MOD_1	Turns (It: Girdi Campania)	Castronovo	Leaf	C16:0, C20:0, C22:0, C24:0, C26:0, C28:0 C30:0	C21:0	C21:1, C16:3, C18:3,	KT29(15)		OHC26, OHC28, OHC30	$\beta$ -sitosterol	sugars
CA_MOD_1B	Turns (It: Girdi Campania)	Castronovo	Stem	C16:0, C18:0, C20:0, C22:0, C24:0, C26:0, C28:0, C30:0	C21:0, C23:0, C25:0	C16:3, C18:2, C18:3, C20:2, C20:3, C21:1, C22:1				$\beta$ -sitosterol	sugars
CA_MOD_2	Cabbage (It: Cavoli)	Castronovo	Leaf	C16:0, C18:0, C20:0, C22:0, C24:0, C28:0, C30:0	C19:0, C21:0, C23:0	C16:3, C18:3, C20:2, C20:3, C21:1, C22:1	KT29(15)	ALK29, ALK31	OHC26, OHC28, OHC30, OH29(br)	$\beta$ -sitosterol	sugars
	Cabbage (It: Cavoli)	Castronovo	Stem	C16:0, C18:0, C20:0, C22:0	C19:0	C16:1, C16:3, C18:1, C18:2, C18:3	KT29(15)			$\beta$ -sitosterol	sugars
CA_MOD_5a	Carrots	Castronovo	Leaf	C16:0, C18:0, C20:0, C22:0, C24:0, C28:0, C30:0	C21:0, C25:0	C16:3, C18:2, C18:3			OHC26, OHC28	$\beta$ -sitosterol	sugars
CA_MOD_5b	Carrots	Castronovo	Carrot	C16:0, C18:0, C20:0, C22:0, C24:0	C23:0	C16:1, C18:2, C20:2				$\beta$ -sitosterol; stigmasterol	sugars
CA_MOD_7	Aubergines	Castronovo		C16:0, C18:0, C20:0, C22:0		C18:2, C18:2, C22:1				$\beta$ -sitosterol	sugars
CA_MOD_16A	Leeks	UK	Leaf	C16:0		C18:2, C18:2	KT31		OHC26, OHC28, OHC30	$\beta$ -sitosterol	sugars
CA_MOD_16B	Leeks	UK	Stem (white part)	C16:0, C18:0, C20:0, C22:0, C24:0, C26:0	C23:0	C16:1 C18:1, C18:3, C20:2, C20:2	KT31		OHC26, OHC28, OHC30	$\beta$ -sitosterol	sugars
CA_MOD_17	Onion	Castronovo		C16:0, C18:0, C22:0, C24:0, C26:0		C18:1, C18: 1, C20:1, C20:2				$\beta$ -sitosterol; cycloartenol	sugars
CA_MOD_18	Cabbage	UK	Leaf	C16:0, C18:0, C20:0 C22:0, C24:0, C26:0	C17:0, C19:0	C16:3, C18:2, C18:2, C18:3	KT29(15)	ALK29	OH29(br)	$\beta$ -sitosterol	sugars
CA_MOD_19	Kale	UK	Leaf	C16:0, C18:0	C21:0	C16:3, C18:2, C18:3, C21:1	KT29(15)	ALK29	OHC26, OHC27, OHC28, OH29(br)	$\beta$ -sitosterol	sugars
CA_MOD_20	Spinach	Italy	Leaf	C16:0, C20:0, C22:0, C24:0, C26:0		C16:1, C16:3, C18:1		ALK31	OHC22, OHC24, OHC26, OHC28	$\alpha$ -spinasterol; 7-stigmasterol	sugars
CA_MOD_27A	Lemon	Castronovo	Peel	C12:0, C14:0, C16:0, C18:0, C20:0, C22:0, C24:0, C26:0, C28:0, C30:0	C13:0, C17:0, C23:0, C25:0	C18:1, C18:2, C20:1, C22:1, C24:1,		ALK29	UI	$\beta$ -sitosterol; stigmasterol	sugars, citric acid
CA_MOD_27B	Lemon	Castronovo	Pulp	C16:0, C18:0, C20: C22:0, C24:0		C18:2, C18:3		ALK27	UI	$\beta$ -sitosterol; stigmasterol, campesterol	sugars, citric acid

#### 4.2.2.2 Alkanes, ketones and alcohols

Alongside sterols, a number of samples including Giri di Campania, leek, Cavoli, cabbage, carrot, kale and spinach yielded *n*-alkanes, *n*-alcohols and ketones. Leaf waxes may be distinguished in the archaeological record based on specific *n*-alkane, *n*-alkanol, and ketone distributions (Chapter 3). To identify specific profiles of these vegetables the relative distribution of these compounds were compared between the samples and are shown in Figure 27.

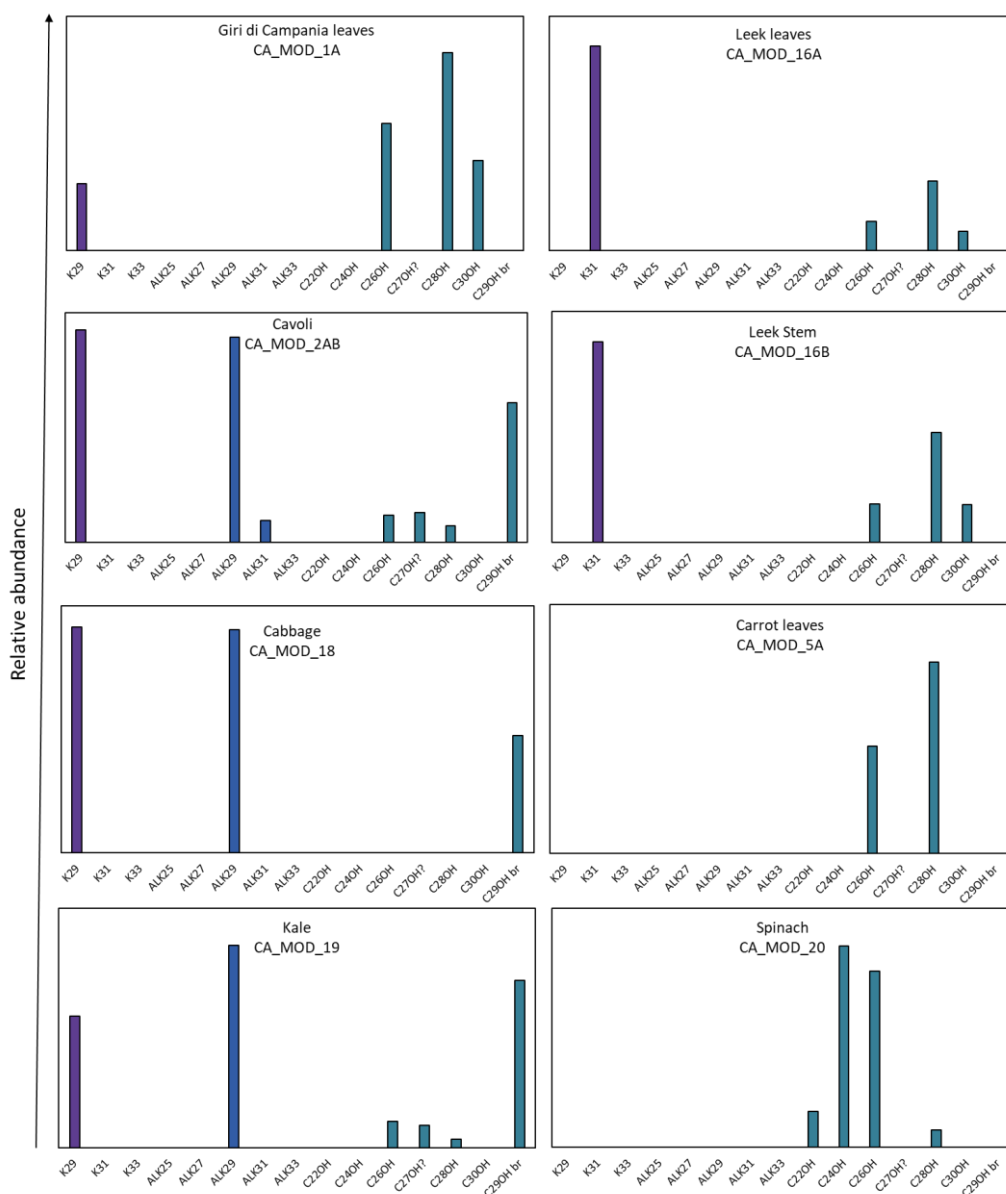


Figure 27: Long- chain *n*-alkanes, *n*-alcohols and ketone distributions of modern vegetable samples obtained from SE.

Here, cabbage, Cavoli and kale all revealed a molecular profile particular to the genus *Brassica*, where alkane C<sub>29</sub>, alcohol C<sub>29</sub> (nonacosane-15-ol) and ketone C<sub>29</sub> (nonacosane-15-one) were present (Charters et al. 1997). This specific profile has previously been used to identify brassicas in archaeological ceramics from medieval England (Evershed, Heron and Goad 1991; Dunne et al. 2019) and medieval Italy (Buonincontri et al. 2017; Giorgi, Salvini and Pecci 2010; Salvini, Pecci and Giorgi 2008). Whilst this profile can help to identify the processing of brassicas in archaeological ceramics, it is important to note that the genus *Brassica* covers a wide variety of vegetables. The results here further support that different species of the *Brassica* genus yield very similar lipid profiles. Therefore, while the identification of this profile gives confidence in the identification of brassicas in archaeological ceramics, it is not possible to go beyond genus level of identification.

Both parts of leek analysed (leaf and stem) yielded a profile containing ketone C<sub>31</sub> (hentriacontane-16-one) alkane C<sub>31</sub> and *n*-alcohols C<sub>22</sub>-C<sub>28</sub> where alcohol C<sub>28</sub> is dominant. This profile has already been observed and attributed to the presence of leek in several biomolecular archaeology papers studying medieval cooking pots (Evershed et al. 1992b, 1995; Raven et al. 1997; Rhee et al. 1998). However, it is uncertain how specific this profile is to leek and further analysis should analyse the profiles of other species from the *Allium* genus. Although it should be noted that in this experiment, onion, which is also of the genus *Allium*, did not deliver a similar profile to leek. Spinach also yielded a specific *n*-alkane C<sub>31</sub> and a *n*-alcohols C<sub>22</sub>-C<sub>26</sub> profile where alcohol C<sub>24</sub> is dominant. Although this profile cannot, with confidence, be used to identify spinach, it could be used alongside specific sterols.

#### **4.2.2.3 Degradation and burial**

As noted, the lipid profiles observed in archaeological ceramics are largely degraded and can be significantly different to that of the original fresh product. Furthermore, repeated processing of plant products are required to obtain a signal sufficient to be preserved in archaeological contexts, due to their lower lipid content in comparison to animal products. Once absorbed, the lipids undergo a number of transformation



processes through the use of the ceramic and in the burial environment. To assess how the lipids from the modern plant products absorb into the ceramic matrix and how they may be altered through heat and in the burial environment, the plant products with specific biomarkers (cabbage, spinach, and leek) were cooked in replica ceramic vessels and buried in Eze, South of France for one year. The vessels were wheel-thrown fired pottery made with 'Standard Red' clay and did not have any temper. These vessels were supplied by Mr Graham Taylor, Rothbury, UK. To cook the products, ~ 30g of fresh cabbage (30.0g), leek (31.66g) and spinach (29.4g) were placed in individual replica pottery vessels. The vessels were filled with 100ml of water, covered with aluminium foil and placed in a furnace oven for 2 hours at 150°C. The temperature inside the vessels, however, only reached a maximum of 88°C. Once cooled, as much of the fresh matter was removed as possible, and the samples were left to dry. The sample was split into two pieces; one was retained for analysis and the other half of the sample was buried in Eze the South of France for 1 year. Restrictions did not allow for burial of the samples in Sicily, but Eze was selected as a reasonably comparable burial environment to Sicily as the weather and pH levels are similar in both areas. Whilst a burial time of one year is a significantly shorter amount of time than archaeological samples remain in the burial environment, it has been proposed that 99% of lipids in ceramics are lost through microbial degradation and the majority of this degradation occurs in the first year after burial (Dudd, Regert and Evershed 1998a; Aillaud 2001). The samples before and after burial were sampled and lipids were extracted using the AE method and analysed using GC-MS techniques as described in Appendix A.

#### **4.2.3 Results after processing in the ceramic vessel and after burial**

The fatty acid profiles are not discussed here, but attention is paid to the specific profiles observed in the modern samples in terms of sterols, *n*-alkanes, *n*-alcohols and ketones. Sugars present in the modern vegetable samples were again identified in high quantities in the ceramic vessels after cooking, as they are soluble in water and therefore were easily absorbed into the matrix with the water, but then were lost in the burial environment. The loss of sugars after one year of burial further highlights that those sugars are unlikely to survive in the archaeological record. Here, no sterols

from either cabbage, leek or spinach were detected in the vessels after cooking the vegetable samples and subsequently they were not observed in the buried samples. This further highlights the issues of absorption of these compounds into the ceramic matrix as the samples were cooked at low temperature (<100 degrees) for a short period of time (<4 hours). Thus, it may be proposed that sterols observed in the archaeological record only occur when plant products have been processed in high quantities, for a long period of time in multiple cooking events in the same vessel, or result from contamination. Cabbage and leek did yield specific *n*-alkanes, *n*-alcohols, and ketones profiles, as shown in Figure 27, in the ceramic matrix after cooking. However, after burial, the profile for cabbage could not be detected, but the profile for leek could. These results, therefore, highlight that when the profile of brassicas are identified in archaeological ceramics it is because the ceramic was intensively used for their processing.

In conclusion, of the plants analysed in this experiment, only plants of the genus *Brassica*, leeks and spinach have sufficiently specific profiles to be identified in archaeological vessels. It is important here to refer to the commonly used saying 'absence of evidence is not evidence of absence' as, previously highlighted, our ability to identify plant products in the archaeological record through organic residue analysis is not only restricted by limited specific biomarkers, but also due to low fatty content meaning that: 1) to absorb into the ceramic matrix through processing, plant products have to be processed for a prolonged period or multiple events; 2) even when present these signatures are often masked by fattier products such as animal fats and may not be detected; and 3) these specific markers can be altered through cooking and lost to degradation in the burial environment.

### **4.3 Summary of chapter and consideration of experiments**

This chapter has outlined the results of two experiments. The first aimed to understand the impact of post-firing treatments on the interpretation of organic residues. It was found that post-firing treatments have variable impacts on the interpretation of organic residues. It was shown that a multi-faceted organic residue analysis approach

is essential in the study of organic residues from archaeological containers and provides rationale for the multiple extraction (acidified methanol, solvent and acid butylation extraction) and analytical techniques used in this thesis to interpret organic residues from archaeological containers. The second experiment extracted residues from modern plant products. Here, specific profiles were identified for spinach, brassicas, and leek. However, the cooking and burial experiments used were limited and more robust experiments should be carried out in the future to provide further insight into whether these profiles could be observed in the archaeological record. This chapter has highlighted a complexity of variables that may impact the absorption and survival of organic residues in ceramics which need to be considered when interpreting organic residues in archaeological contexts (e.g., cooking conditions including the length, temperature and how many times the product is processed, the burial conditions, type of clay, and parts of the vessel that is sampled). A consideration of these variables has enabled reflection and caution when interpreting the organic residue results of archaeological ceramics presented in the following chapters.

# Chapter 5 New insights into early medieval Islamic cuisine: Organic residue analysis of pottery from rural and urban Sicily

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## 1.3 Introduction

Sicily is the largest Island in the Mediterranean Sea and is centrally located, between mainland Italy, North Africa and the Eastern Mediterranean. For several reasons including political connections, environmental and arable conditions, and not least its geographic location, the island of Sicily has been at the epicentre of political interest and subsequent conquests throughout ancient and medieval history. Of profound impact to the island, was the transition from a Byzantine political control (6<sup>th</sup> – 9<sup>th</sup> century AD in western Sicily and 6<sup>th</sup> – 10<sup>th</sup> in the eastern part of the Island) to Islamic rule (9<sup>th</sup> to 11<sup>th</sup> century AD and 10<sup>th</sup> to 11<sup>th</sup>, in the west and east respectively) when Sicily became part of the *dār al-Islām*. In 827 AD the Aghlabid army landed in Mazara del Vallo, Trapani, located on the south-western coast of the island, following almost a century and a half of political unrest and regular raids from North Africa. In 831 AD

Palermo was captured and since became the political and economic capital of Sicily. In the next two centuries, under the control of first the Aghlabid emirate (827-909 AD), then the Fatimid caliphate, ruled from Ifrīqiya, and then from Egypt, and finally the Kalbid emirate, who ruled the Island on behalf of the Fatimids from 948 AD, Palermo thrived politically, economically and culturally. Despite this, exactly how these Islamic political regimes impacted the lifeways of people in Palermo and more broadly elsewhere in Sicily, particularly in lesser studied rural areas, at this time is not fully understood.

Agriculture was undoubtedly at the heart of the Islamic economy but the degree to which Sicily was transformed by an Islamic 'Green Revolution' requires careful evaluation. This term, originally coined by Watson (Watson 1974, 1983), refers to the association between the movement of Islamic communities and the introduction of new resources and agricultural innovations. Watson documents 17 plants that arrived in the Mediterranean from the Middle East with the Arabs, including spinach, cotton, durum wheat, sugar, citrus fruits and possibly broomcorn millet (Watson 1974, 1983). Some aspects of Watson's thesis have recently been revived, although not uncritically, both from historical research (Decker 2009; Squatriti 2014) and from the development of archaeobotanical research (Van der Veen 2010, 2011; Fuks, Amichay and Weiss 2020). Excavations at Mazara del Vallo (Trapani) have provided direct evidence of Watson's 'revolution plants', including citrus fruits in 10<sup>th</sup>-11<sup>th</sup> century contexts and watermelon, aubergine, cotton, durum wheat and spinach during the 11<sup>th</sup>-13<sup>th</sup> centuries (Primavera 2018a; Fiorentino, Lumaga and Zech-Matterne 2018). There is no doubt that an advanced knowledge of agricultural and irrigation systems was introduced into Sicily during this time. However, in the absence of data from preceding contexts, the extent and impact of these innovations remain unclear. While the introduction of new plants is not contested, it has since been suggested that some resources were already present in the region, but gained new importance at the arrival of the Arabs (Decker 2009). Furthermore, evidence from written sources and ceramic studies suggest a more complex dynamic with regards to the production of agricultural commodities in Palermo. It has been suggested that an evolution in farming methods and an integration of Sicily into an Islamic botanic-food horizon is more related to the

change of Palermo's role and to the investments of the new elites (Molinari 2015; Nef, Pezzini and Sacco 2017).

Alongside the agricultural economy, religion played an important and complex role in shaping the economy and consumption practices of Sicily during the early medieval period. Although under the Islamic political regime, Christian and Jewish communities remained in Sicily during the 9<sup>th</sup>-12<sup>th</sup> centuries, living alongside Muslims (Ardizzone and Pezzini 2014, 282). This complexity is often reduced to discussions of food taboos imposed by the Islamic religion. The consumption of pork and alcohol are prohibited in the hadiths but, to what extent these restrictions were observed in early medieval daily life is questionable, particularly in a pluralistic society as was Sicily at this time. For example, although generally found in low proportions, the discovery of pig remains found in Islamic contexts in Spain and Portugal, have been attributed to the presence of Christians or hunted wild boar (Salas-Salvadó et al. 2006; Grau-Sologestoa 2017). In terms of alcohol production and consumption, although prohibited by the hadith, wine may have been consumed by Christian and Jewish communities, or as Islamic medieval poems depict, by some members of the Muslim community also (Branca 2003; D'Alessandro 2010). The trade of wine to and from Islamic Sicily has also recently been observed by identifying organic biomarkers associated with grape products in amphorae (Drieu et al. 2021b).

Cuisine is a cultural phenomenon manifested in the way in which specific food resources are produced, procured, prepared, combined and consumed. By studying culinary practices in the archaeological record, it is possible to gain insight into the way that people both thought about and valued different foodstuffs and how food traditions and rituals were influenced by broader changes, such as in politics, economy, agricultural systems and religion (Belasco 1987, 2008; Rozin 1982). Recent archaeobotanical (Primavera 2018a; Fiorentino, Lumaga and Zech-Matterne 2018; Carver et al. 2019) and faunal analysis (Aniceti 2020; Arcoleo and Sineo 2014) from Islamic contexts in Sicily, have begun to address important questions regarding the resources that were available in Sicily under Islamic control. Written sources about Islamic cuisine have given insight into culinary practices across the Arabic-Islamic world (Zaouali 2009; Waines 2003; Perry 2005). However, distributed across a broad

chronological period and geographic area, these writings mostly describe practices of elite consumption. In contrast, here we present the first large scale study of culinary practices in the early medieval Islamic world focusing on the chemical analysis of domestic cooking wares from rural and urban settlements. Unlike written sources, such evidence is more likely to capture everyday utilitarian practice and allow a more nuanced understanding of how foodstuffs were utilized in Islamic cuisine. By understanding pottery use we can gain a unique perspective of how resources were prepared, consumed and combined, in turn helping to answer important questions regarding daily life under Islamic political control in Sicily.

We applied well-established methods in organic residue analysis (ORA) (Craig et al. 2013a; Correa-Ascencio and Evershed 2014; Garnier and Valamoti 2016; Craig et al. 2007; Dudd and Evershed 1998) (Appendix A) to identify organic residues from 134 cooking pots and other domestic containers from three sites in Palermo and from the rural settlement of Casale San Pietro dating to the 9<sup>th</sup>-12<sup>th</sup> centuries AD (Appendix E, S2 Data). The research benefits from recent analysis of ceramics from this period that has succeeded in refining the chrono-typologies and identifying production centres (Carver et al. 2019; Giarrusso and Mulone 2014; Arcifa and Bagnera 2014; Sacco 2014; Ardizzone, Pezzini and Sacco 2014; Messina et al. 2018). Only a small number of ceramics from medieval Sicily have previously been analysed using ORA (Lucejko et al. 2018). Until now, investigations have been mostly limited to typological and petrographic studies, and virtually nothing is known about the use of such pottery in Sicily nor more broadly in early medieval Islamic society. To aid this analysis, and to further understand the contents of these ceramics, ORA was performed on a selection of modern vegetables thought to be present in Sicily during the Islamic regime (Chapter 4). This study draws on evidence provided through archaeobotanical and faunal analysis, historical and contemporary accounts of Islamic-Arabic cuisine, and the identification of cooking wares.

With consideration of current ideas and evidence surrounding cuisine in early medieval Islamic societies, the focus of this research is two-fold: first, we ask, how does the contents of these domestic containers contribute to our knowledge of cuisine in

early medieval Islamic societies? Secondly, do the contents of these ceramics differ in urban and rural socio-economic settings?

## **5.1 Sites and ceramics**

### **5.1.1 Palermo (Urban)**

Pottery sherds ( $n= 83$ ) were obtained from three sites in urban capital of Palermo dating to the Islamic period; 24 from Castello San Pietro (CSP) (Arcifa and Bagnera 2014), 26 from Gancia church (GA) (Ardizzone, Pezzini and Sacco 2014) and 33 from Palazzo Bonagia (PB) (Sacco 2014) (Appendix E, Tables E.1, E.2 and E.3). Recent surveys at all three sites have enabled the identification of some of the earliest Islamic style pottery in Sicily. Strong typological links were made between tableware in the assemblage and those found in Aghlabid Ifrīqiya (located in Tunisia and part of Libya and Algeria), perhaps evidence of direct culinary links between Palermo and North Africa at this time (Messina et al. 2018). In contrast, the vast majority of domestic cooking wares were produced in Palermo and appear typologically dissimilar to those found in Ifrīqiya (Sacco, Chatti and Touihri 2018). As a major production site, Palermitan ceramics were distributed throughout the Island (Giarrusso and Mulone 2014).

Castello San Pietro (CSP) is a domestic site located adjacent to the harbour in Palermo, located in the urban core of the city (Di Stefano, 1998). The earliest context of this excavation revealed an Islamic cemetery, which was succeeded by a cluster of houses constructed with little hiatus after the cemetery's disuse (Arcifa and Bagnera 2014). One of the earliest closed contexts is US865, a well reused as a rubbish pit. The pottery from this context, after a re-examination of material culture from this site, has been dated between the 9<sup>th</sup> and the beginning of the 10<sup>th</sup> century based on their typology (Arcifa and Bagnera 2014) (Table E.1). In this study, ceramic samples from context US865, with assumed use by the populations that occupied the dwellings, were selected for analysis



The sequence seen at CSP is repeated at the site of the Gancia church (GA), where burials in the Islamic rite are superseded by settlement ('urbanisation') with walls and midden heaps associated with pottery assigned to the end of the 9<sup>th</sup> – beginning of the 10<sup>th</sup> century before the construction of the Fatimid citadel documented in 937 AD (Ardizzone, Pezzini and Sacco 2014, 197–200; Sacco 2017, 339). The same horizon (late 9<sup>th</sup>/early 11<sup>th</sup> century) has been noted at the excavations at Palazzo Bonagia (PB) (Sacco 2014, 226). The pottery at these sites, therefore, belongs to the early phase of Islamic Palermo (late 9<sup>th</sup>-early 10<sup>th</sup> at CSP, GA and PB), and the first phase of Islamic urbanisation (10<sup>th</sup>- 11<sup>th</sup> at GA and PB). The ceramic vessels selected for this study were again dated based on their typologies and expert comparison (see Chapter 2). As these were recovered as part of small-scale recovery excavations, the full stratigraphy and interpretations of these sites requires further evaluations. However, pottery associated with the Islamic phases have been associated with urban residence (Di Stefano 1998; Ardizzone, Pezzini and Sacco 2015; Sacco 2014; Sacco 2017).

It must be noted that on-going stable isotope analysis, ancient DNA analysis, and radiocarbon dating at these sites, as part of the wider Sicily in transition project, are helping to further aid our understanding of the people that lived at these sites.

### **5.1.2 Casale San Pietro (CLESP; rural)**

Pottery sherds ( $n= 51$ ) were obtained from the site of Casale San Pietro (CLESP), located on the plain outside of the town of Castronovo di Sicilia in the centre of Sicily within the province of Palermo (Figure 28) (Table E.4). The site of Casale San Pietro has been classed as rural as it lies on the rural hinterland in the Province of Palermo. Excavations at this site have been ongoing, as part of the Sicily in Transition project since 2014 and the current sequence and interpretation of the site are in progress (Carver et al. 2019, Carver et al. forthcoming). Nonetheless, excavations have revealed that the site was extensive and dedicated to agricultural production in the 3-5<sup>th</sup> centuries (a large *vicus* and a *statio*), continuing in a reduced form during the Byzantine period (6-9<sup>th</sup> centuries), flourishing again during the Islamic period (especially in the 10-11<sup>th</sup> centuries), and continuing but with a reduced economic profile in the 12/13<sup>th</sup>

centuries (Carver et al. 2019). During the 9<sup>th</sup>-12<sup>th</sup> centuries a series of walls were attributed to Islamic forms and have been interpreted as either as domestic houses for farmers and their families or as a station serving the road from Agrigento to Palermo. An impressive assemblage of ceramics has been identified from the 10<sup>th</sup>-12<sup>th</sup> centuries. Whereas, from the 9<sup>th</sup> century only a small number of cooking pots with unknown provenance have so far been identified. Analysis of the ceramic pastes of these ceramics has shown that the settlement relied heavily on pottery from Palermitan production at this time, depicting a strong link between this rural settlement and the capital of Palermo; likely one of economic reliance (Carver et al. 2019). This raises interesting questions regarding the use of these imported ceramics vessels for local culinary uses in the rural settlement. A collection of handmade/slow thrown cooking pots and wheel thrown calcite ware, of unknown provenance but likely imported from elsewhere, were also identified. In this study, pottery was selected from the waystation site (Int 5) from contexts associated with domestic occupation in walled rooms. An open cooking hearth associated with 10<sup>th</sup>-12<sup>th</sup> century contexts further supports domestication at the site (Carver et al., forthcoming; Meo forthcoming; Hummler forthcoming). The ceramic samples were dated based on their typologies and associated radiocarbon dates and coins in stratigraphic phases (Carver et al., forthcoming; Meo forthcoming; Hummler forthcoming). Samples of unknown date were not selected for analysis here.

### **5.1.3 Ceramic corpus**

Two main shapes of vessels suitable for use on fire have been distinguished, which correspond to at least two different technologies. Here we refer to '*olla*' as vessel shapes that are slightly closed and deeper than wide in capacity and often have handles and usually a curved base. We refer to 'cooking pots' here as vessels that are more wide than deep and generally have a flat base and straighter sides. In addition, some additional forms were analysed: pans, stone plates, so called 'braziers' and lids (Appendix E). Ceramics from CSP and PB were obtained from the Soprintendenza di Palermo Storage Rooms at Via Magione, 44, 90133 Palermo and ceramics from GA were obtained from Soprintendenza di Palermo Storage Rooms located in the church of

Santa Maria della Gancia, Via Alloro, 13, 90133 Palermo. Inventory numbers and sample numbers of the original studies of the ceramics from Palermo are referenced in (Tables E1-3). Samples from CLESP were obtained from the Comune di Castronovo storage rooms, located in Palazzo Giandalia, Via Fonte Regio, 49, 90030 Castronovo di Sicilia (PA) and analysed as part of this study. All necessary permits were obtained for the described study from the Soprintendenza dei Beni culturali e ambientali di Palermo, which complied with all relevant regulations.

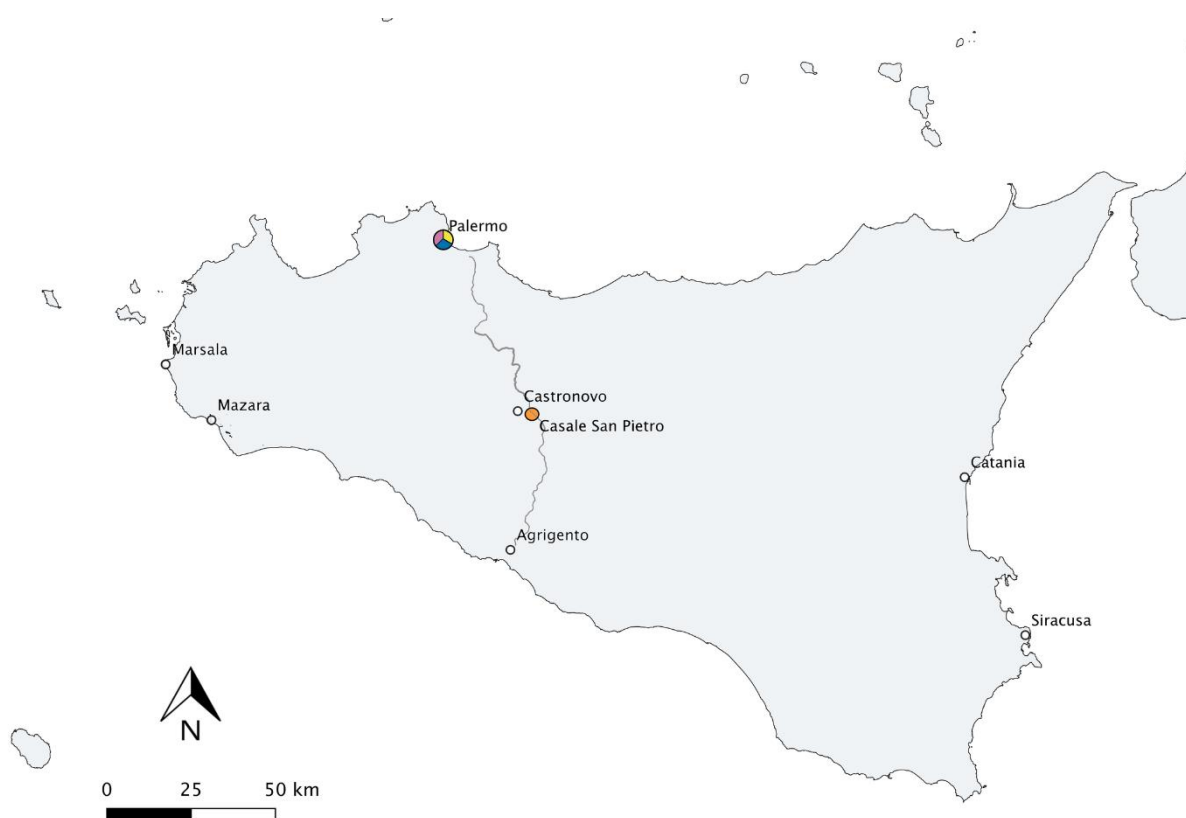


Figure 28: Map of Sicily showing the location of Palermo and Casale San Pietro. Colours represent the sites with ceramics used in this study where pink = CSP, yellow = GA, blue = PB and orange = CLESP. The main road between Palermo and Agrigento from North to South is marked on the map.

## 5.2 Results and discussion

Using an acid methanol extraction procedure (Correa-Ascencio and Evershed 2014) (Appendix A), over 90% (122/134) of the ceramic samples yielded lipid concentrations above  $5 \mu\text{g g}^{-1}$ , with a mean concentration of  $124.3 \mu\text{g g}^{-1}$  (CSP),  $52.7 \mu\text{g g}^{-1}$  (PB),  $96.7 \mu\text{g g}^{-1}$  (GA) and  $154.56 \mu\text{g g}^{-1}$  (CLESP) respectively. A value of  $5 \mu\text{g g}^{-1}$  has previously

been deemed the lowest lipid concentration that can be reliably attributed as endogenous and therefore interpretable (Evershed 2008b; Evershed et al. 1999).

### **5.2.1 Evidence of animal products**

Animal products undoubtedly played an important part in cuisine at this time. Meat was considered a staple in Arabic- Islamic cuisine where the regular dish typically contained meat (Lewicka 2011). Lamb and mutton, chicken and dairy products (milk, yogurts and cheeses) appear regularly in accounts of cuisine, where mutton was considered a delicacy consumed by the upper classes (Zaouali 2009; Waines 2003; Perry 2005). The consumption of pork is forbidden as part of the Islamic religion, which is reflected by its absence from culinary literary sources. However, the complete absence of pork in Sicily during this time cannot be assumed. For all of the four sites investigated, faunal remains of caprine (both sheep and goats), cattle and domestic fowl have been identified (Arcoleo and Sineo 2014; Arcoleo 2015; Aniceti 2020) (Appendix F). Faunal analysis at PB and GA have shown the near absence of suid remains compared to a higher abundance at the rural site of CLESP (Arcoleo and Sineo 2014; Arcoleo 2015; Aniceti 2020). In contrast to the other sites in Palermo, suid remains were identified at CSP (Arcoleo and Sineo 2014; Arcoleo 2015; Aniceti 2020) (Appendix F). The presence or absence of faunal remains in the archaeological record can, to some extent, inform us about how the meat or secondary products from these animals were processed through culling patterns, butchery and burning marks. Through organic residue analysis of ceramic cooking wares, we were interested in how animal products were incorporated into culinary practices at the site level.

Degraded animal fats were the most common lipid profiles encountered, characterised by an equal dominance of palmitic (C<sub>16:0</sub>) and stearic acids (C<sub>18:0</sub>) and the presence of cholesterol. Of these, a large proportion (90%) had branched and linear C<sub>15:0</sub> and C<sub>17:0</sub> fatty acids which are indicative of ruminant fats, formed through bacterial transformation of lipids in the rumen (Christie 1978). To further understand the origin of these lipids, 122 of the extracts were analysed by GC-c-IRMS to determine the stable carbon isotope value of the major fatty acids (C<sub>16:0</sub> and C<sub>18:0</sub>) (Appendix A and S2 Data).

This approach has shown to be useful for distinguishing ruminant adipose (i.e. carcass fats), non-ruminant adipose and ruminant dairy fats based on the difference  $\Delta^{13}\text{C}$  between the  $\delta^{13}\text{C}$  values of these fatty acids (Dudd and Evershed 1998; Copley et al. 2005c; Craig et al. 2007) as well as marine and freshwater resources based on their absolute values (Craig et al. 2013a) (Figure 29).

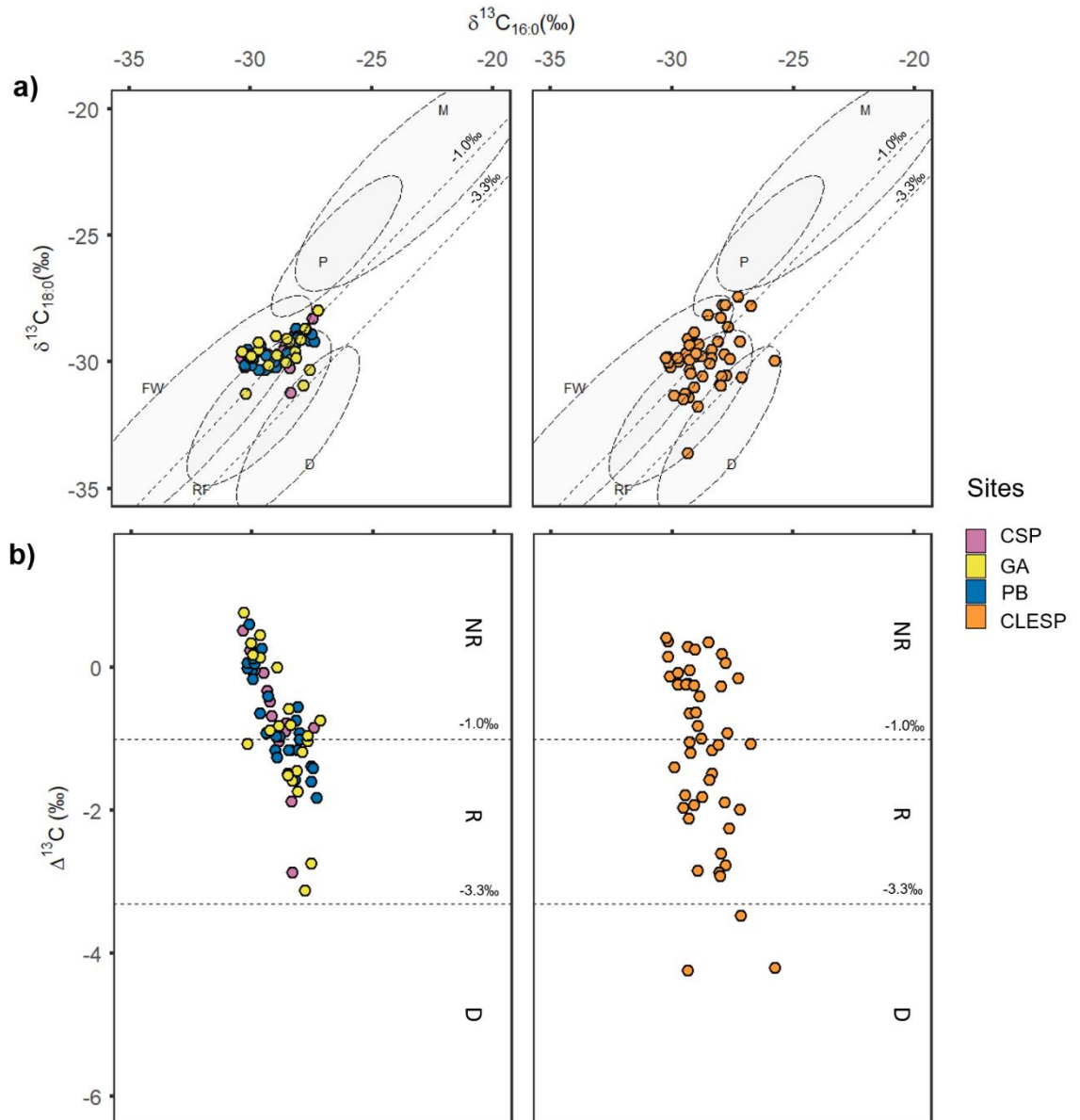


Figure 29: Plots of fatty acid stable isotope values obtained from individual vessels from Sicilian Islamic pottery. a) Plot of  $\delta^{13}\text{C}_{16:0}$  against  $\delta^{13}\text{C}_{18:0}$ . Ranges (68% confidence) of 269 modern authentic reference products are shown, D (Ruminant dairy), RF (Ruminant adipose), P (Porcine), M (Marine), and FW (Fresh water). These references are published elsewhere (Cubas et al. 2020) b) Plot of  $\Delta^{13}\text{C}$  against  $\delta^{13}\text{C}_{16:0}$ . values  $< -3.3\text{‰}$  are typically associated with D (Ruminant dairy), values between  $-3.3\text{‰}$  and  $-1.0\text{‰}$  are associated with R (Ruminant adipose) and above  $-1.0\text{‰}$  can be considered as NR (Non-ruminant) (Craig et al. 2012).

A high proportion of samples from all Palermo sites (64%) and CLESP (40%) fall within a relatively narrow range matching reference values of non-ruminant fats. However, no samples yielded fatty acid  $\delta^{13}\text{C}$  values that fall directly within the range of modern porcine fats which tend to have fatty acids more enriched in  $^{13}\text{C}$  than the values presented here (Figure 29a). At Palermo, these findings are in agreement with the near absence of suid remains at PB and GA (Arcolego and Sineo 2014; Arcolego 2015) (0.8% and 1.61% NISP respectively) (Appendix F). Although suid remains are in higher abundance at CSP (Aniceti 2020) (14.7% NISP) (Appendix F), it may be suggested that pork was not selected and processed in the ceramic containers. It is interesting to note that four samples from CLESP are clustered towards more enriched  $\delta^{13}\text{C}$  values and fall on the edge of the range of the reference porcine fats (Figure 29a). The processing of pork in the ceramic containers at CLESP would agree with the faunal evidence at this site whereby domestic suids are the second most dominant species in the faunal assemblage at CLESP (32% NISP) (Aniceti 2020) (Appendix F). However, the presence/absence of porcine fats based on these  $\delta^{13}\text{C}$  values must be treated with caution as the reference ellipses provided here for modern porcine from Northern Europe may not be representative of early medieval Sicilian porcine values due to the variability of  $\delta^{13}\text{C}_{16:0}$  values dependant on the animals diet (Mukherjee, Gibson and Evershed 2008).

Furthermore, the depleted  $\delta^{13}\text{C}_{16:0}$  values ( $\sim -30$  ‰) within the range of non-ruminant fats from all sites are difficult to interpret. These values could represent the processing of other non-ruminant animal fats such as domestic fowl (Colonese et al. 2017b) or hare (Drieu et al. 2021a) which are represented in the faunal assemblages of these sites (Arcolego and Sineo 2014; Arcolego 2015), but are present in very low proportions (<5% of the total NISP at all sites) (Appendix F). The contribution of  $\text{C}_3$  plant products such as vegetable oils and cereals etc., can impact  $\delta^{13}\text{C}$  values falling in the range of non-ruminant products. However, in most cases, the molecular profiles of these samples are dominated by  $\text{C}_{16:0}$  and  $\text{C}_{18:0}$  in equal proportions (Figure 31) and the presence of plant biomarkers is not consistent with samples that fall within the range of non-ruminant products. Thus, it is difficult to determine the source of these  $\delta^{13}\text{C}$  values and indeed mixtures of non-ruminant animal products, plant products and ruminant animals cannot be dismissed.

A number of fatty acids  $\delta^{13}\text{C}$  values obtained from the pottery from all sites fall within the range of ruminant adipose fat (CSP  $n=3$ ; GA  $n=8$ ; PB  $n=9$ ; CLESP  $n=20$ ) (Figure 29). The incorporation of ruminant animal products in these vessels is supported by the high dominance of ruminant species at these sites (cattle and caprines) (Aniceti 2020; Arcoleo 2015; Arcoleo and Sineo 2014) (Appendix F). It is important to note that the presence of ruminant fats may be underrepresented by the  $\delta^{13}\text{C}$  values, due to mixing of non-ruminant and ruminant fats. Of note, lipids from 3 samples from CLESP had  $\delta^{13}\text{C}$  values within the range of modern ruminant dairy products (Figure 29). This suggests that both primary and secondary ruminant products were processed in domestic containers at the rural site. Conversely, no evidence of dairy products was indicated by the fatty acid  $\delta^{13}\text{C}$  values obtained from the pottery from Palermo. Further use of dairy cannot be ruled out but here the signals are more difficult to interpret due to mixing between different types of animal fats and potentially plant oils, as shown by the density distribution of  $\Delta^{13}\text{C}$  values at this site (Figure 30c). Interestingly, the  $\Delta^{13}\text{C}$  values from all the Palermo pottery are not normally distributed around the mean perhaps indicating distinct uses, where vessels are dedicated for specific roles (Figure 30c). Whether these roles track specific food products or specific combinations of food products is more difficult to discern. In contrast, the  $\Delta^{13}\text{C}$  values from CLESP are much more widely dispersed compared to the samples from Palermo despite a similar sample size (Figure 30c), perhaps indicating more generalised uses of pottery vessels.

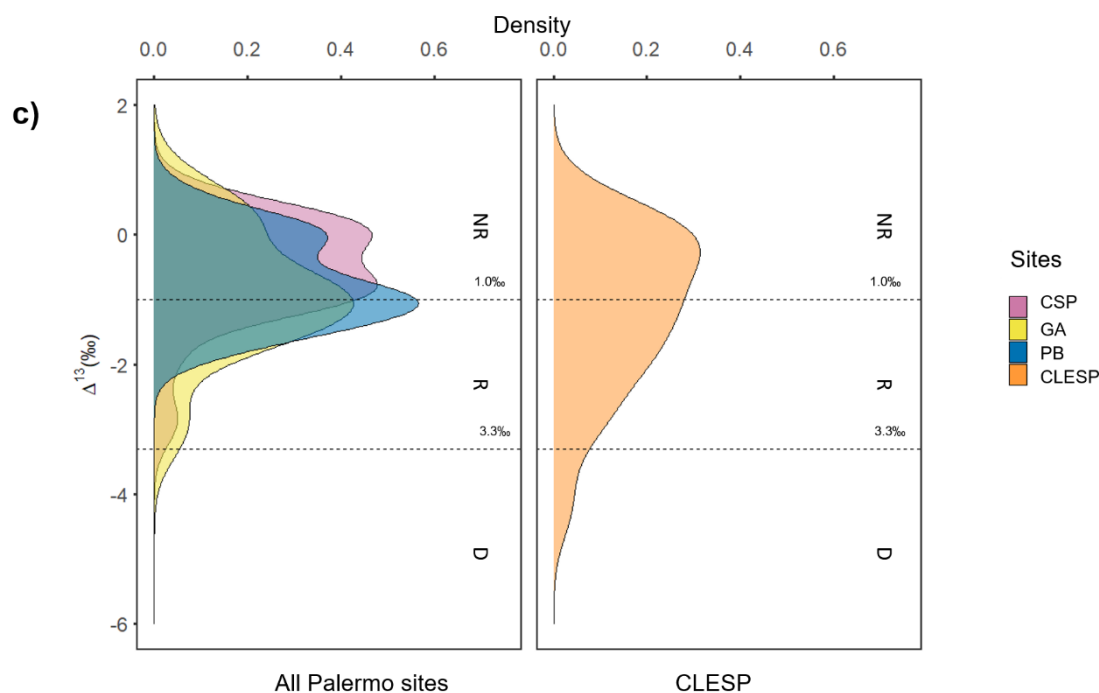


Figure 30: Kernel density estimate of  $\Delta^{13}\text{C}$  values. Bandwidth = 0.5.  $\Delta^{13}\text{C}$  values  $< -3.3\text{‰}$  are typically associated with D (Ruminant dairy), values between  $-3.3\text{‰}$  and  $-1.0\text{‰}$  are associated with R (Ruminant adipose) and above  $-1.0\text{‰}$  can be considered as NR (Non- ruminant) (Craig et al. 2012).

## 5.2.2 Further resolution with triacylglycerols (TAGs)

The distribution and relative abundance of different triacylglycerols (TAGs), can help to further understand the origin of animal fats (Dudd and Evershed 1998; Evershed et al. 1997b). Several samples extracted using solvent extraction (Appendix A), yielded intact TAGs (32/106 samples). In most cases, the TAG profiles were characteristic of ruminant adipose fats ( $T_{46}$  to  $T_{54}$ ) (Appendix G). These profiles are unlike non-ruminant adipose fats, where there is a clear predominance of  $T_{52}$  over  $T_{50}$  and  $T_{54}$  and the distribution for ruminant fats is centred on  $T_{52}$  (Dudd and Evershed 1998). No samples yielded TAG profiles indicative of non-ruminant adipose fats and of note, 3/4 samples from CLESP that fell within the  $\delta^{13}\text{C}$  values of modern porcine fats did not yield intact TAGs and 1 sample yielded a TAG profile indicative of ruminant adipose fats (Figure 31c). Thus, highlighting the clear evidence of mixing of products and the difficulty in interpreting these values. A broader range of TAGs ( $T_{42}$  to  $T_{54}$ ) is typical of dairy fats (Dudd and Evershed 1998) and was identified in 2 samples from CLESP (Figure 31d) (S2 Text)



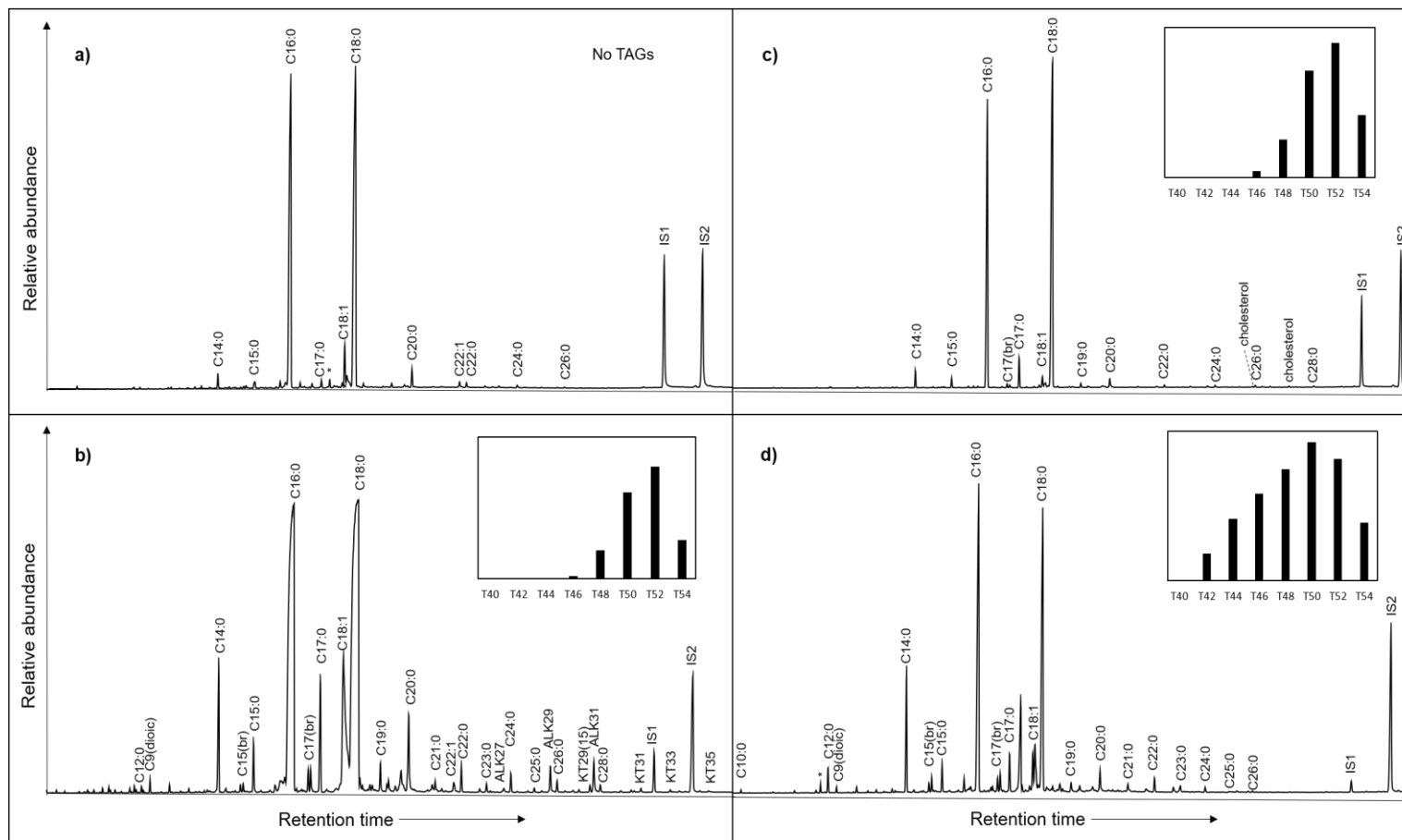


Figure 31: TIC chromatograms of pottery extracts. a) AE chromatogram of sample CSP\_25 that yielded  $\delta^{13}\text{C}$  values within the range of non-ruminant products, b) AE chromatogram of sample CLESP\_29 that yielded  $\delta^{13}\text{C}$  values within the range of non-ruminant products (porcine) and shows the TAG distribution profile associated with this sample after SE, c) AE chromatogram of sample GA\_34 that yielded  $\delta^{13}\text{C}$  values within the range of ruminant products adipose products and shows the TAG distribution profile associated with this sample after SE, d) AE chromatogram of sample CLESP\_26 that yielded  $\delta^{13}\text{C}$  values within the range of products and shows the TAG distribution profile associated with this sample after SE. Parentheses indicate the  $\delta^{13}\text{C}_{16:0}$  and  $\delta^{13}\text{C}_{18:0}$  fatty acid values.

It has been shown that by plotting the average carbon number of the TAGs (M) and the dispersion factor (DF) ruminant adipose and dairy products can be broadly distinguished (Mirabaud, Rolando and Regert 2007). However, assigning specific products based on TAG profiles is undermined by preferential loss of lower molecular weight components (Dudd and Evershed 1998) and mixing of resources. Nevertheless, samples from CLESP have a wider distribution of TAGs compared to those from Palermo (Figure 32) indicating the occurrence of dairy fats in five samples with TAGs preserved i.e., those with a low average carbon number (48-49) and a higher dispersion factor (2.0-2.6). In other cases, the TAG distributions more closely resemble ruminant carcass fats (Mirabaud, Rolando and Regert 2007).

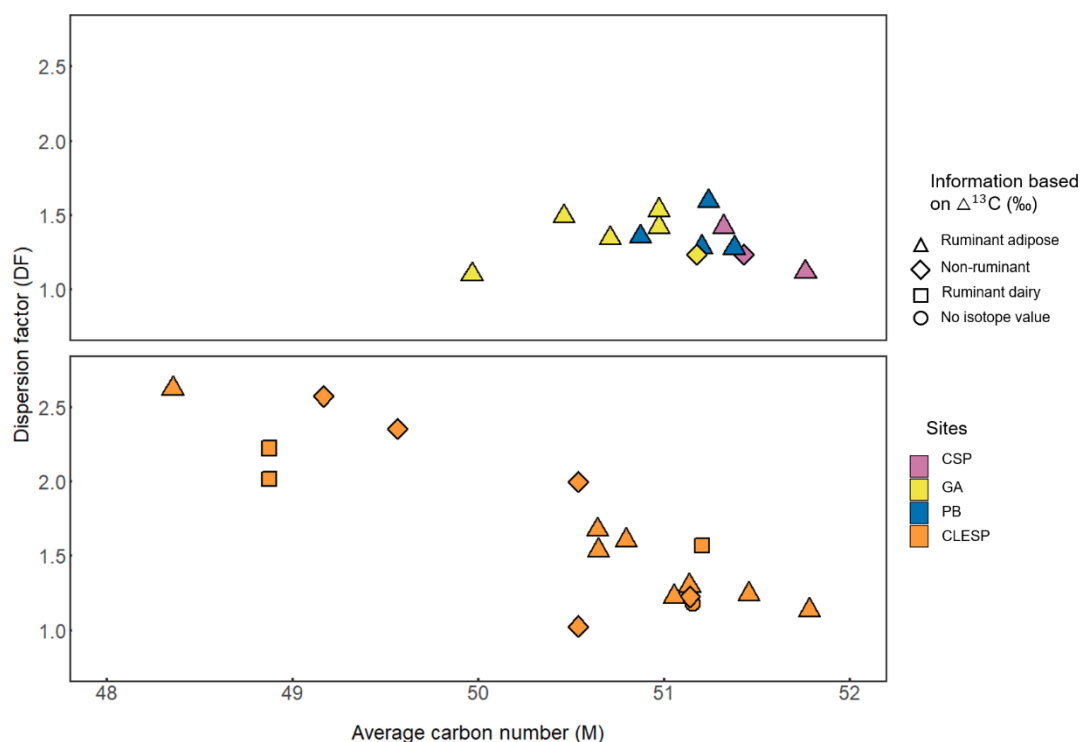


Figure 32: Plots of TAG information based on HT-GC data of individual pottery samples from Palermo sites (CSP, GA and PB) and Castronovo (CLESP) with interpretations based on fatty acid isotope values. The dispersion factor (DF) and average carbon number (M) were calculated using statistical equations outlined by Mirabaud et al. (Mirabaud, Rolando and Regert 2007). Shapes represent ruminant dairy (●) based on  $\Delta^{13}\text{C}$  values  $< -3.3\text{‰}$ , ruminant adipose (◆) based on  $\Delta^{13}\text{C}$  values between  $-3.3\text{‰}$  and  $-1.0\text{‰}$  and non-ruminant (■) based on  $\Delta^{13}\text{C}$  above  $-1.0\text{‰}$  (Craig et al. 2012).

The correspondence between vessels categorised by their fatty acid stable carbon isotope values (ruminant, non-ruminant, and dairy) and the distribution of TAGs is not straightforward to interpret for CLESP samples (Figure 32). Samples plotting in the ruminant dairy isotope range would be expected to have a lower M value which is not always the case. Similarly, two vessels from this site have non-ruminant isotope values but a low M and high DF values, which is more typical of dairy. This suggests substantial mixing of products within the pottery in most cases, with perhaps two vessels used more exclusively for dairy. At the Palermo sites, there is little evidence for dairy by considering either the TAG distribution or their stable isotope values, evidence of a contrasting pattern of resource use between the urban centre and rural settlement. Animal husbandry orientated towards meat production has been observed at urban centres in Al-Andalus compared to rural settlements where a more mixed economy prevails (Salas-Salvadó et al. 2006; Grau-Sologestoa 2017; García-García 2019).

### **5.2.3 Evidence of aquatic products**

The importance of fish and shellfish is not fully understood in Islamic Sicily. Fish is not generally considered in high regard in high-status Islamic cuisine and in Arabic recipes from the East fish is rare compared to meat, although fish recipes do appear marginally more frequently in Al-Andalus (Zaouali 2009, 191). When they are mentioned in recipes, both in Arab (Eastern) and Andalusian Medieval cookbooks, fish are baked or cooked in stews or sauces, rarely fried (García Sánchez 1996, 264–265). A small number of tuna remains were identified at PB, GA and CSP as well the inland site of CLESP (Aniceti 2020; Arcoleo and Sineo 2014; Arcoleo 2015) (Appendix F). A bias must be considered in the recovery of fish remains from archaeological excavations, due to sampling biases and preservation. Tuna fishing is thought to have been abundant in coastal sites of Trapani and Palermo during this time and tuna trapping techniques were spread through the island by the Arabs after they arrived on the island (Consolo et al. 2008; Longo and Clark 2012). It may be assumed that at major coastal towns, such as Palermo, fresh fish were consumed and perhaps preserved (salted or dried, for the

distribution of inland areas as observed in Roman sites inside and outside of the Mediterranean (Van Neer 1998; Van Neer, Ervynck and Monsieur 2010).

Freshwater or marine organisms could not be identified in any of the vessels analysed based on their fatty acid  $\delta^{13}\text{C}$  values (Figure 29a). Similarly, specific lipids derived from heating freshwater or marine animals (Craig et al. 2007; Cramp and Evershed 2014; Hansel et al. 2004), were absent in all the vessels analysed despite the use of very sensitive approaches for their detection (Appendix A). Isoprenoid fatty acids that are at high abundance in aquatic oils were identified in a number of samples in all four sites (S2 Data), including phytanic acids and 4,8,12-trimethyltridecanoic acid (TMTD), although these are present in some animal fats, albeit at lower concentration. It was not possible to distinguish the source of phytanic acid further based on their stereoisomer ratios (%SSR), as has been suggested (Lucquin et al. 2016c). The %SRR of many samples fell within the range of both aquatic oils and ruminant fats (S2 Data). The absence of fish oils in ceramics from the coastal sites of Palermo is somewhat surprising given the presumed availability of fresh fish. However, human isotope evidence for fish consumption in other medieval coastal sites in the Mediterranean do not indicate a high consumption of marine products (Toso et al. 2019). Although it is possible that fish were prepared and consumed in other ways (e.g. smoking, salting, cooked directly on fire, or processed as fish sauce) not detectable in the ceramics analysed here, these results likely reflect the lesser importance of fish in Arabic cuisine in contrast to other animal products (Zaouali 2009, 191).

#### **5.2.4 Evidence of vegetables, fruits and cereals**

In addition to meats and other animal products, vegetables, fruits and cereals likely played an important role in cuisine. With the Islamic green revolution, certain vegetables, fruits and cereals gained new importance and written sources of Islamic and complex mixtures of herbs, spices and vegetables are well documented in Arabic literature. Alongside spinach, aubergine and artichoke, other vegetables mentioned in historical sources include turnip, cabbage, cauliflower, onion, garlic and leek (Lewicka 2011; Riḍwān 1984, 132–137). Furthermore, dishes often reflect a sweet and sour/

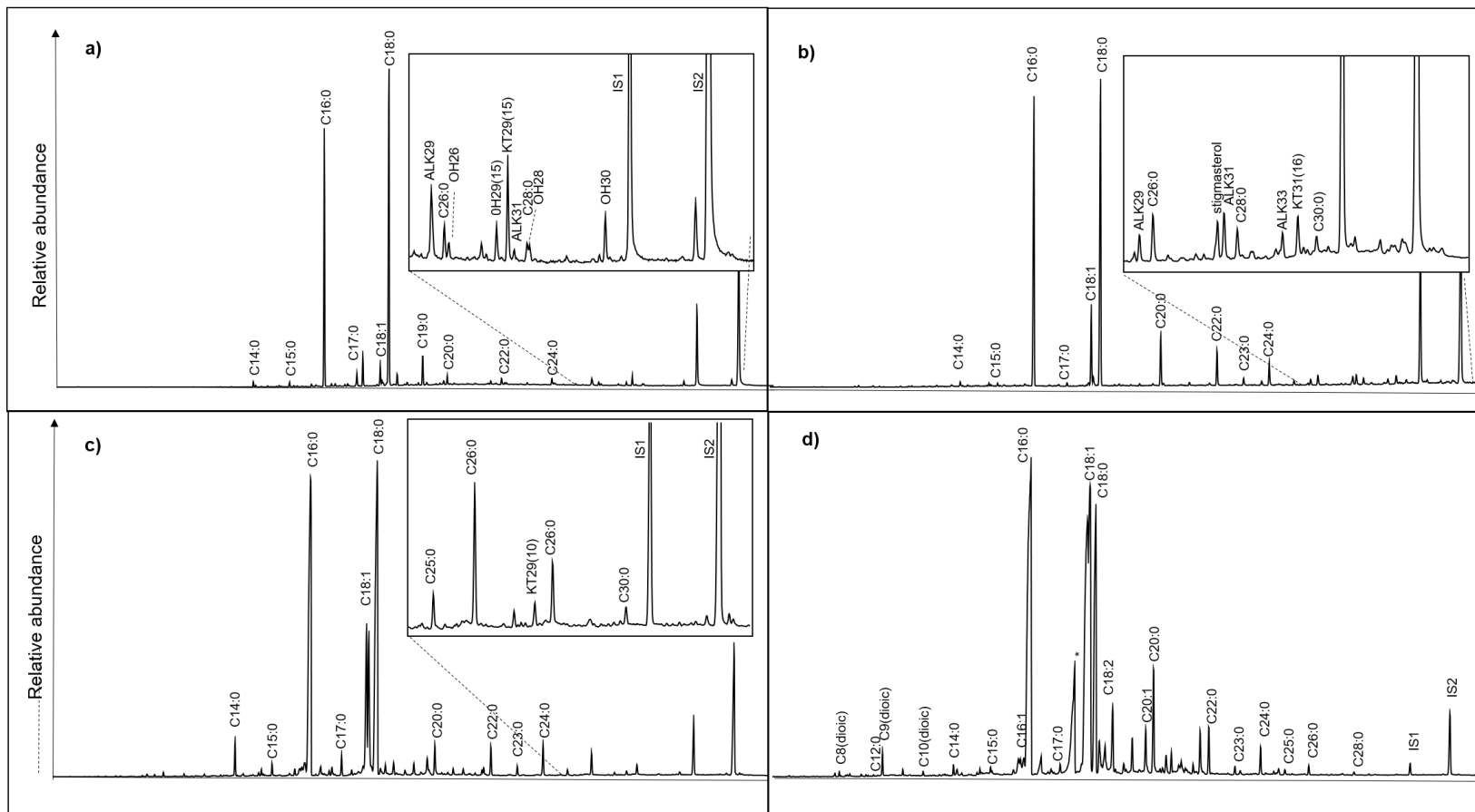
salty palate, where fruits and fruit juices were added to savoury meat dishes, for example citrus fruits (oranges and lemons), apples, pomegranates and grape products (Lewicka 2011; Peterson 1980). Recent archaeobotanical evidence has identified several species of vegetables, fruits and cereals in Islamic contexts from Sicily including several species of plums (*Prunus* spp.) at CLESP and citrus fruits, watermelon and aubergine at the site of Mazara del Vallo (Primavera 2018a; Fiorentino, Lumaga and Zech-Matterne 2018). However, in what way these resources were utilized in everyday cuisine is not fully understood.

In contrast to animal fats, the ability to assess the presence of plant products in archaeological ceramics is limited by their comparatively lower lipid yield (Evershed 2008a). Even when lipid profiles indicative of a plant source are encountered, they are rarely specific to a product/species. In order to maximise information, we have implemented an approach that involves the identification of leaf waxes, seed oils, phenolic lipids, terpenoids, fruit acids, cereal alkylresorcinols and miliacin. Lipid profiles with long-chain odd *n*-alkanes (with a predominance of C<sub>29</sub> or C<sub>31</sub>), *n*-alcohols and wax esters (W<sub>40</sub>-W<sub>48</sub>) indicate plant waxes (Eglinton and Hamilton 1967; Ribechini et al. 2008). When present, high oleic to stearic acid ratios (rare in archaeological samples) can indicate plant oils, alongside palmitic acid as a major constituent and sometimes unsaturated C<sub>18:2</sub> (Romanus et al. 2009; Copley et al. 2005a). Short chain carboxylic acids (fumaric, succinic, malic, and tartaric) can be used to indicate the presence of fruit products (Garnier and Valamoti 2016; Drieu et al. 2021b). The presence of alkylresorcinols can be used to identify cereals (wheat, barley and rye) (Hammann and Cramp 2018; Ross et al. 2003; Colonese et al. 2017a) and the presence broomcorn millet can be identified in archaeological ceramics by the presence miliacin (olean-18-en-3 $\beta$ -ol methyl ether) (Heron et al. 2016; Bossard et al. 2013).

Plant sterols (exclusively  $\beta$ -sitosterol) were identified in several samples across all four sites, often identified with other non-specific plant derived lipids. Spinach-specific sterols ( $\alpha$ -spinasterol and 7-stigmastenol (Chapter 4 and S2 Data) were not identified in any vessels, possibly because these compounds are easily degraded when cooked in pottery (Hammann et al. 2018). *n*-alkane, *n*-alkanol and ketone distributions indicated the presence of plant waxes in several samples. Additionally, C<sub>16:0</sub>, C<sub>18:0</sub>, C<sub>20:0</sub> and C<sub>22:0</sub>

wax esters were identified in a few samples, sometimes associated with odd-numbered *n*-alkanes, suggesting a plant wax or a mixture of plant wax and beeswax (Ribechini et al. 2008; Rageot et al. 2019c).

In some cases, it was possible to offer greater taxonomic resolution regarding the origin of the leaf waxes as they display a specific *n*-alkane, *n*-alkanol and ketone distributions. Ketone specific for *Brassica* (nonacosane-15-one) and alcohol nonacosane-15-ol (Charters et al. 1997) alongside other degraded leaf waxes were found in several samples ( $n=12$ ) (Figure 33a). Hentriacontane-16-one ( $C_{31}$  ketone) and *n*-hentriacontane ( $C_{31}$  alkane) in one sample from PB could be attributed to leek (Evershed et al. 1995; Raven et al. 1997; Rhee et al. 1998) (Figure 33b). Additionally, nonacosane-10-one, the major ketone found in the leaf/stem wax of broad-leaved sermountain (*Laserpitium latifolium*; (Huneck 1960)) and fennel (*Foeniculum vulgare* (Muckensturm et al. 1997)) was detected in pottery samples from CLESP ( $n=5$ ) (Figure 33c). Nonacosane-10-one may also be present in other apiaceous, for example *Apium* sp. However, the presence of nonacosane-10-one in archaeological ceramics has not previously been reported and no other biomarkers were detected to firmly identify any of these plants. Finally, one sample from CLESP indicated the presence of a plant oil, by a relatively high oleic to stearic acid ratio ( $C_{18:1}/C_{18:0} > 2$ ) in addition to a small amount of linoleic acid ( $C_{18:2}$ ) (Romanus et al. 2009; Copley et al. 2005a) (Figure 33d). Plant oils are likely underrepresented in these cooking vessels as oleic acid is susceptible to degradation in the burial environment and through prolonged cooking events (Dudd, Regert and Evershed 1998b) and mixtures with animal products is likely to mask their presence (Baeten et al. 2013).



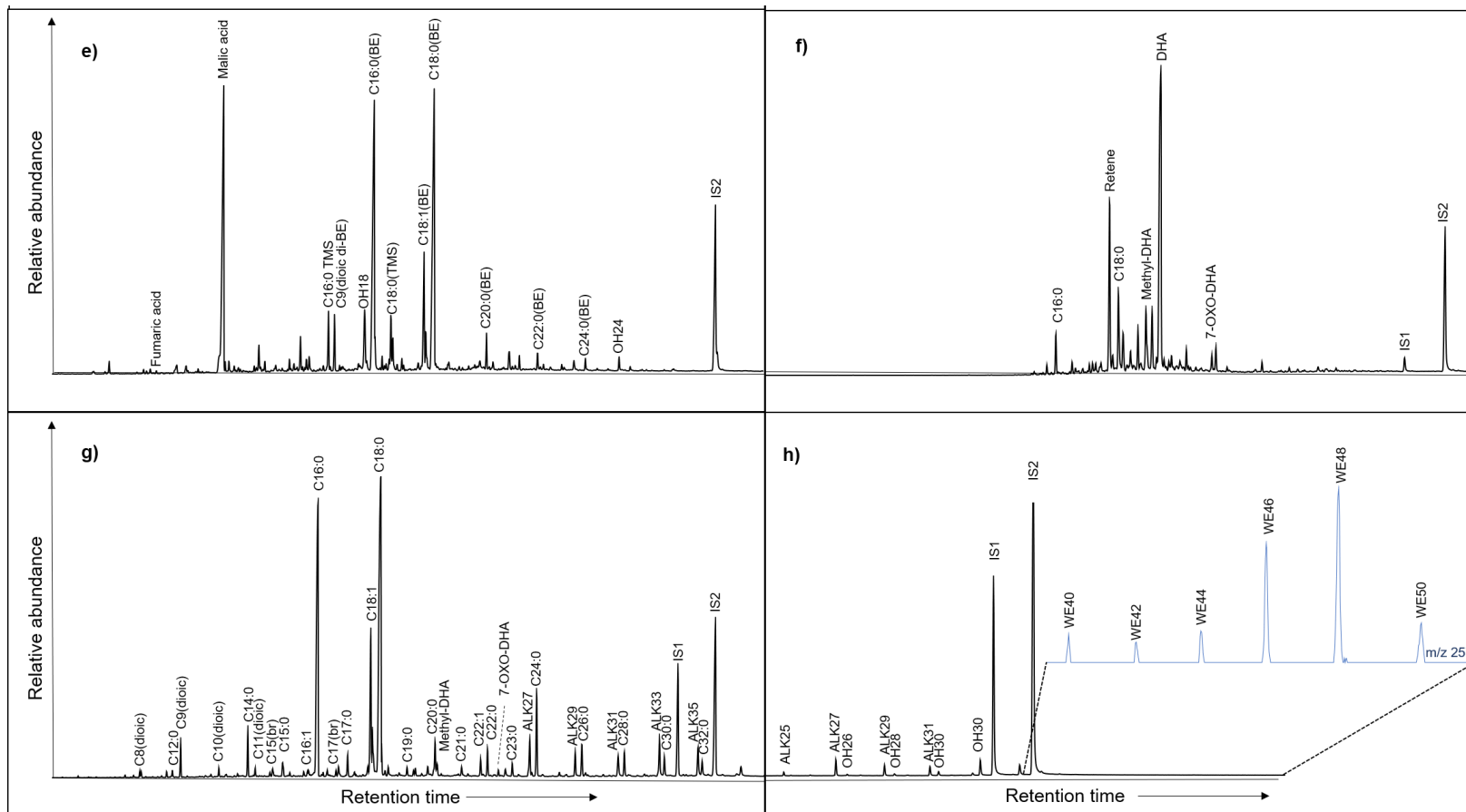




Figure 33: TIC chromatograms of extracts typical of a variety of products identified in these ceramics. a) AE chromatogram of sample GA\_10 showing the presence of C29(15) ketone that indicates the presumed presence of Brassica [9]. b) AE chromatogram of sample PB\_26 showing the presence of C31 ketone (hentriacontane-16-one) that indicates the presumed presence of leek (*Allium porrum*) in the ceramic samples [10–13]. c) AE chromatogram of CLESP\_58 showing the presence of C29(10) ketone (nonacosane-10-one) that indicates the presumed presence of (*Foeniculum vulgare*) (Fennel) [14]. d) AE chromatogram of CLESP\_12 indicating plant oil by a C18:1/C18:0 >2 and the presence of C18:2 [2, 15]. e) Chromatogram of sample CSP\_2 showing the presence of malic acid after acid butylation. f) SE chromatogram of sample CSP\_C5 indicating the presence of Pinaceae biomarkers: retene, methyl-dehydroabietic acid (Methyl-DHA), dehydroabietic acid (DHA), 7-oxo-dehydroabietic acid (7-oxo-DHA). g) AE chromatogram of sample CSP 4 typical of beeswax as well as animal fat and pine products. h) SE chromatogram of CSP 4 showing distribution of alkanes and alcohols typical of beeswax products alongside HT-GC of ion 257 showing the distribution of WE. Internal standards alkane C34 (IS1) and C36 (IS2) are shown.

Small organic acids, which are relatively insoluble in organic solvents were extracted from > 50% of the samples, using an acid butylation extraction developed by Garnier and Valamoti (2016) (Garnier and Valamoti 2016; Drieu et al. 2021b, 2020). Malic and tartaric acids were identified, in variable amounts, in 97% and 70% of them, respectively. Succinic acid, which occurs in a variety of food products and can form through the degradation of fatty acids, was also present in a number of samples (59%). Fumaric, maleic, malonic, and oxalic acids were detected less frequently, in 10%, 14 %, 8% and 3% of the samples respectively. Although tartaric acid is one of the main acids in grapes and wine, its mere presence is not sufficient to formally identify these products in CLESP and Palermo vessels, since it also exists in other plants (Drieu et al. 2020) and low amounts may be attributed to contamination from the burial environment (Drieu et al. 2020). Following the recommendations suggested by Drieu et al. (2020) and (2021), we performed a quantification of tartaric acid to consider only vessels with a significant amount of tartaric acid, which is unlikely to be contamination (Drieu et al. 2021b, 2020). Comparison of the proportions of malic and tartaric acids in the pots was used to distinguish between the presence of grapevine products and that of other plants (Figure 34) (Drieu et al. 2021b, 2020; Jaeggi, Wittmann and Garnier; Linger-Riquier et al. 2016; Cherel et al. 2018) (Appendix H). Four vessels from CLESP and one sample from CSP produced a ratio of tartaric acid to the sum of tartaric and malic acids (%TA) of greater than 35%, characteristic of ripe grapes and their products (wine, juice, vinegar), as well as tamarind and some pomegranate cultivars (Drieu et al. 2021b, 2020) (Figure 34b). Indeed, studies have suggested that cooking wares may have been used for the storage or heating of wine as well as being used as a flavouring for food in medieval Florence and Piombino (Salvini, Pecci and Giorgi 2008; Buonincontri et al. 2017). Interestingly, a long chain odd *n*-alkane distribution where

alkane C<sub>25</sub> is dominant, similar to the profile seen in the epicuticular wax of grape berries (Eglinton and Hamilton 1967), was detected in a vessel that had the highest absolute amount (3.2 µg g<sup>-1</sup>) and relative % of tartaric acid (93%). The addition of whole fresh grapes and raisins to medieval Islamic dishes is mentioned in the literature (Waines 2003, 275).

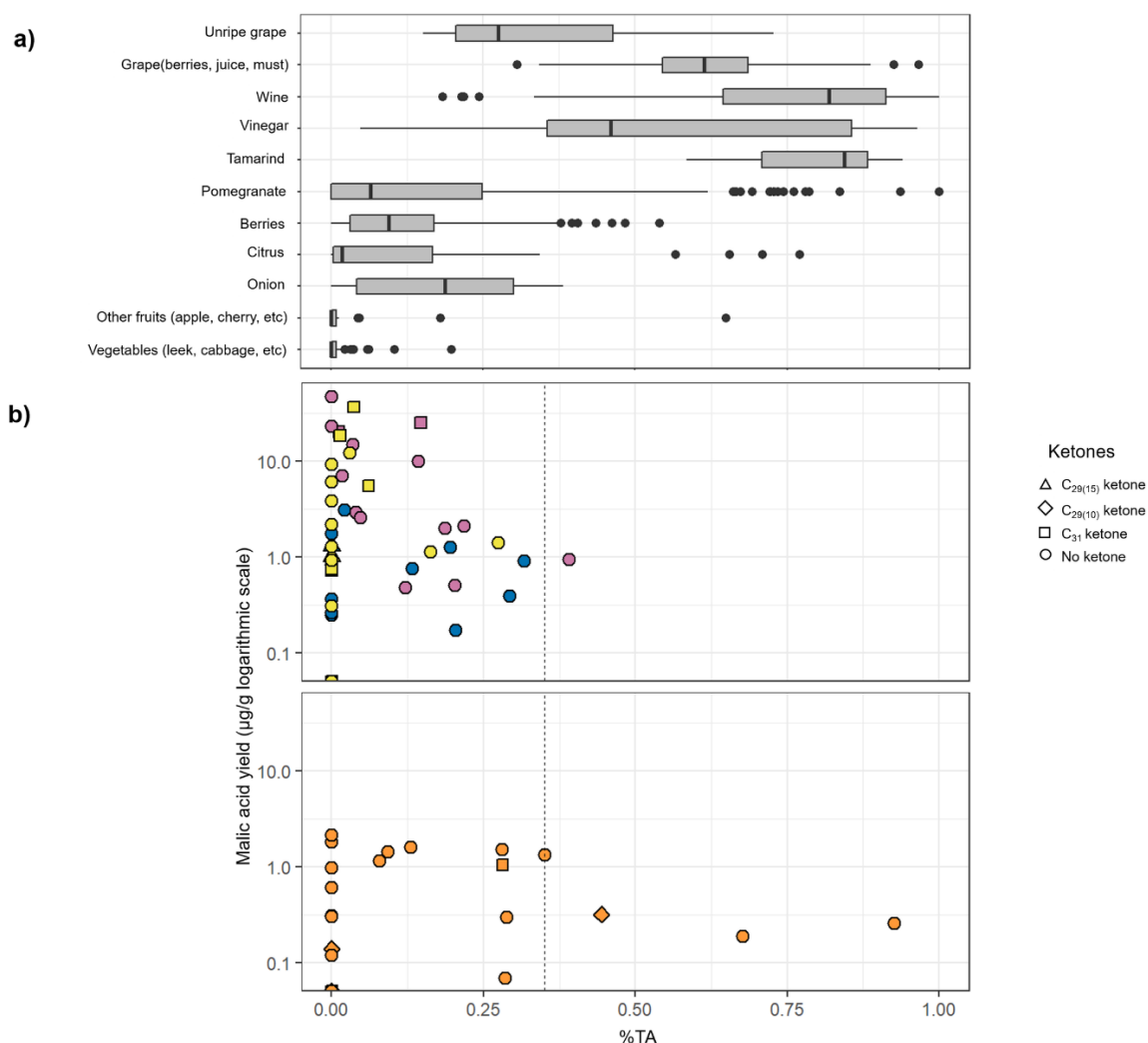


Figure 34: Malic acid yields and % tartaric acid (TA). a) Proportions of tartaric acid in various plants and plant products (Drieu et al. 2021b) (Appendix H); b) Proportions of tartaric acid in CLESP and Palermo cooking pots, plotted against the amount of malic acid extracted. % TA = tartaric acid/(tartaric + malic acid) (Drieu et al. 2021b). C<sub>29(10)</sub> ketone (nonacosane-10-one) indicates the presumed presence of broad-leaved sermountain (*Laserpitium latifolium*) (Huneck 1960) or (*Foeniculum vulgare*) (Fennel) (Muckensturm et al. 1997). C<sub>29(15)</sub> ketone indicates the presumed presence of *Brassica* (Charters et al. 1997). C<sub>31</sub> ketone (hentriacontane-16-one) indicates the presumed presence of leek (*Allium porrum*) in the ceramic samples (Evershed et al. 1992b; Raven et al. 1997; Rhee et al. 1998; Evershed et al. 1995).

Malic acid is one of the most common small acids in the plant kingdom and is present in large quantities in fruits (Walker and Famiani 2018). In many samples of CSP and GA (Figure 33e and 7b), we can suggest the presence of fruits characterised by low

proportions of tartaric acid compared to malic, such as apple, plum, cherry or peach (Figure 34a) (Drieu et al. 2021b; Jaeggi, Wittmann and Garnier; Linger-Riquier et al. 2016; Cherel et al. 2018). However, as malic acid is not restricted to fruit, it may have been derived from the other identified plant products, such as Brassicas or leeks (Ruhl and Herrmann 1985a).

Alkylresorcinols from cereals (wheat, barley or rye) were not found in any of the samples. However, as they are minor constituents and highly susceptible to degradation, their absence does not exclude cereals in these vessels (Ross et al. 2003; Hammann and Cramp 2018; Colonese et al. 2017a). Additionally, there was no evidence of broomcorn millet (*Panicum miliaceum*) in any of the vessels despite the fact that this product can be routinely identified through the presence of a specific biomarker (miliacin; olean-18-en-3 $\beta$ -ol methyl ether) (Heron et al. 2016; Bossard et al. 2013). This supports preceding evidence from stable isotope analysis that millet was mostly consumed in the north of Italy during the Medieval period but not in the south (Rolandsen, Arthur and Alexander 2019). Furthermore, no archaeobotanical evidence of millet has been identified in medieval Sicilian contexts.

## 5.2.5 Resins and Beeswax

A residue typical of Pinaceae resin, including diterpenoids (abietic, primaric and isoprimary acids) and their oxidation by-products (mainly dehydroabietic acid, 7-oxo-dehydroabietic and 15-hydroxy-dehydroabietic acid) were present in several of the Palermo vessels (5 CSP  $n=5$ ; GA  $n=1$ ; PB  $n=4$ ) (Colombini et al. 2005; Modugno and Ribechini 2009). There are a number of examples in the archaeological record where Pinaceae resins have been used as a waterproofing agent for ceramics, mainly in amphorae to store liquids (Dimitrakoudi et al. 2011; Izzo et al. 2013; Heron and Pollard 1988). Due to the low melting point of pine resin, when used to line cooking vessels the pine may impact the flavour of the contents, a taste that was favoured in ancient Greece and Rome, but also today (Reber and Hart 2008). Retene and methyl dehydroabietic acid were present in one *olla* sample from CSP (Figure 33f). The presence of methyl dehydroabietic acid indicates that the resin has been heated with wood, likely through

the production of pitch or tar (Mills and White 1989; Hjulström, Isaksson and Hennius 2006). Pitch or pine tar may have been stored and prepared in the cooking pots for use as a sealant, adhesive or to waterproof boats as observed in other medieval contexts (Pecci and Grassi-Munibe 2016; Salvini, Pecci and Giorgi 2008; Buonincontri et al. 2017). There are also accounts of dipping the stem of fruits in pitch (grapes and pears) to preserve them (Lewicka 2011; Mason 2013). Pears with red dipped stems are still present at Italian markets today (Mason 2013, 81).

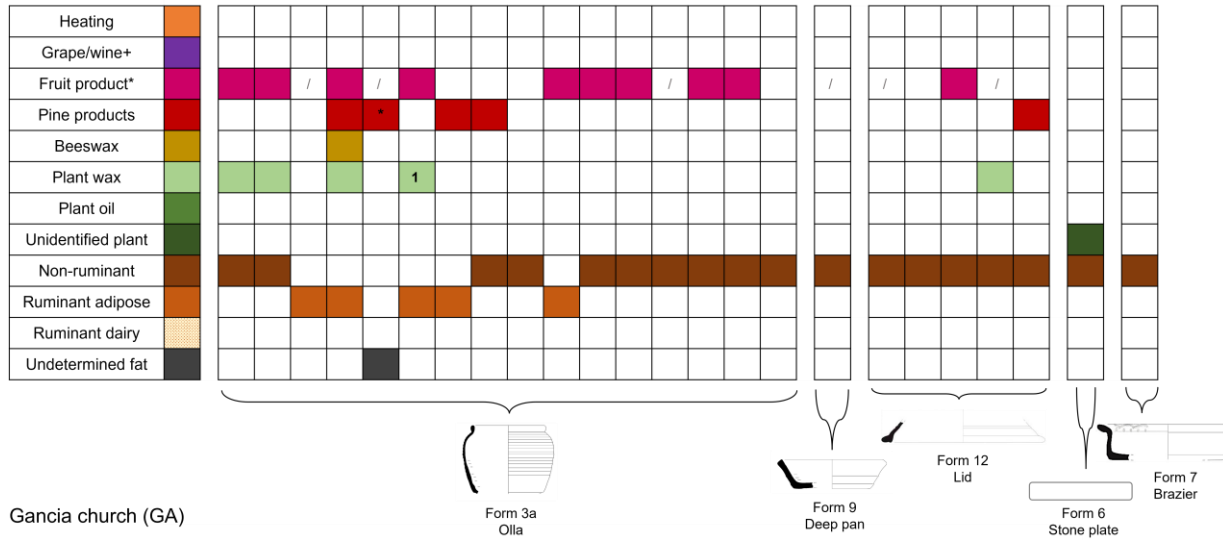
Alongside evidence of pine products, one sample yielded a distribution of long-chain odd *n*-alkanes (C<sub>25</sub>-C<sub>33</sub>, predominance of alkane C<sub>27</sub>), long chain even FAs (C<sub>20:0</sub>-C<sub>28:0</sub>, predominance of FA C<sub>24:0</sub>), and alcohols (C<sub>24</sub>-C<sub>32</sub>, predominance of C<sub>30</sub>) and palmitic acid wax esters (W<sub>40</sub>-W<sub>50</sub>) indicative of beeswax products (Roffet-Salque et al. 2015). Additionally, this sample displayed evidence of animal and plant products (Figure 33g and h). A mixture of beeswax and pine resin has been observed before in archaeological ceramics from Neolithic, Roman and Medieval contexts for example in England, Egypt, France and Greece (Charters et al. 1993a; Duce et al. 2015; Regert et al. 2001) as well as in Sicilian Bronze age ceramics (Mentesana, De Benedetto and Fiorentino 2018), with the suggestion that it provides an effective way to waterproof or repair vessels (Charters et al. 1993a; Duce et al. 2015; Regert et al. 2001). However, the presence of beeswax in pottery may not be due to technological uses. Beeswax products have various uses, such as cosmetic, medicinal or can come from the presence of honey (Regert et al. 2001; Abdulraouf et al. 2012). The co-occurrence of other plant and animal products may indicate the use of honey as a sweetener, contributing to the sweetness of Islamic Arabic cuisine.

### **5.3 Comparison of vessel forms and site variability**

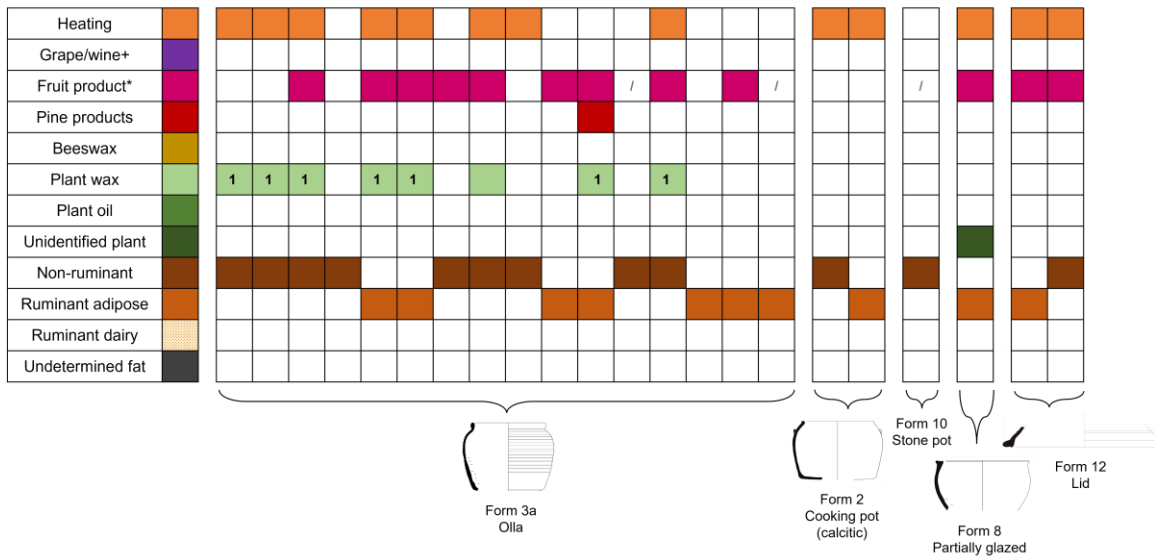
In Palermo generally, we found consistency in the use of cooking wares between sites with mixtures of animal fats, fruits and leafy vegetables without any clear distinction by vessel forms, such as between cooking pots and ollae (Figure 35a-c). Although not detected in samples from CSP, thermal alteration molecular products were frequent in GA and PB and overall, the residue evidence suggests that ollae and cooking pots were

general cooking wares, used to make stews or pottages. It is interesting to note that no clear difference in the use of *ollae* and cooking pots were observed in the sites of Palermo, despite differential techniques used to manufacture the two types of vessel (Pezzini, Sacco and Yenişehirlioğlu 2018). The high frequency of fruit products in these general cooking wares, reflects notions about Islamic- Arabic cuisine, where fruits products are regularly documented as important accompaniments to salty meat dishes (Zaouali 2009; Perry 2005; Lewicka 2011; Peterson 1980). Residues were also extracted from lids used to cover cooking vessels most likely during protracted boiling of the vessels. The presence of pine products, and in one sample beeswax products only found in *ollae* and lids, might suggest the importance of waterproofing these vessels to support more liquid dishes such as pottages and stews. Clear evidence of pitch identified in one *olla* sample from CSP may represent the reuse of these vessels for non-culinary uses such as the production or storage of pitch. It is interesting to note that pine products were not identified in samples from CLESP, either highlighting the differences in availability of conifer products or further supports the notion that pitch was being stored in these vessels and used to waterproof boats in the port of Palermo (Salvini, Pecci and Giorgi 2008; Buonincontri et al. 2017). Stone plates (form 6), braziers (form 7) and pans (form 1 and form 9) also contained animal products and plant products with little evidence of dedicated pottery use. Starchy cereals might also have been used but are difficult to detect using lipid residue analysis, despite a previous study, identifying starchy cereals in stone plates from Sicily (Lucejko et al. 2018).

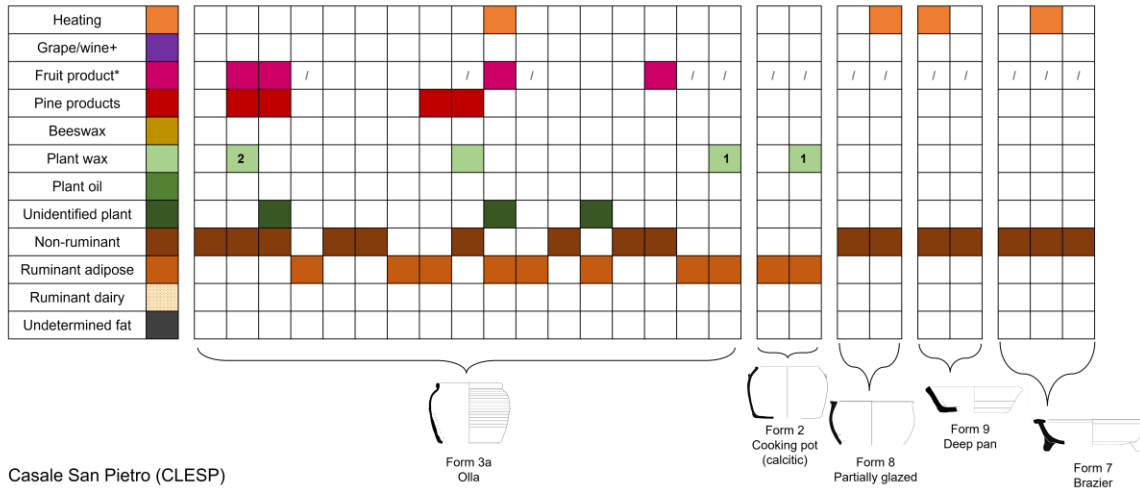
**a) Castello San Pietro (CSP)**



**b) Gancia church (GA)**



c) Palazzo Bonagia (PB)



d) Casale San Pietro (CLESP)

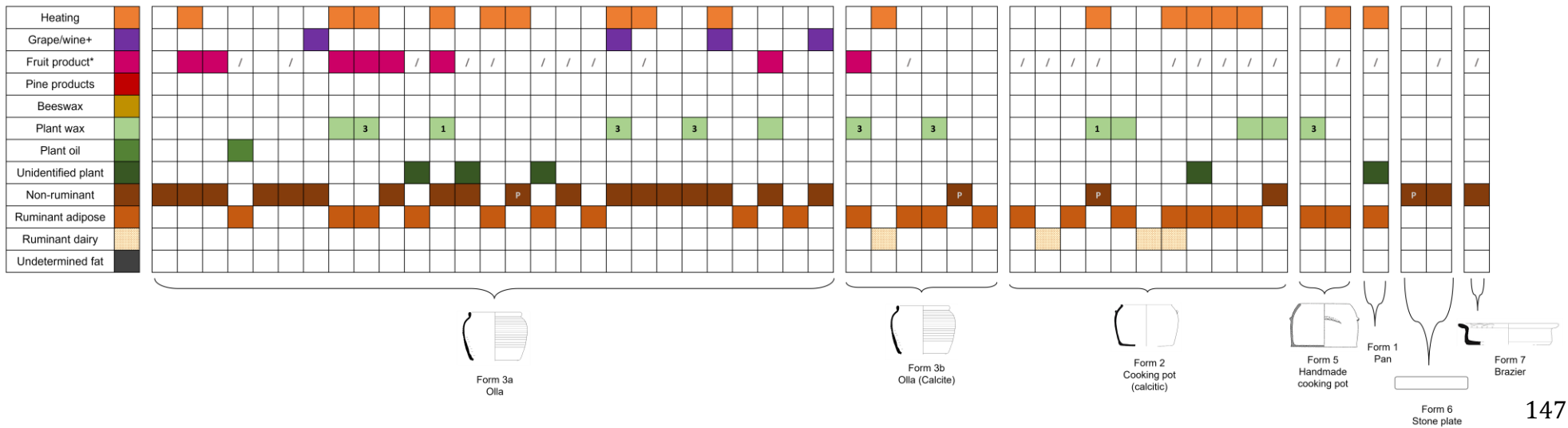


Figure 35: Summary figure of organic substances identified in pottery vessels from (a) CSP, (b) GA, (c) PB and (d) CLESP. Identification criteria of different commodities (ruminant dairy, ruminant adipose, non-ruminant, unidentified plant, plant oils, plant wax, beeswax, pine products, fruit products and grape products) are outlined in the text. Those not analysed for fruit acids are shown (/). Non-ruminant, ruminant and dairy were assigned based on  $\Delta^{13}\text{C}$  values. In one case both ruminant and dairy were identified based on clear dairy TAG distribution. Where P is noted in non-ruminant this refers to samples tentatively assigned to porcine. Specific taxonomy of plant waxes is indicated brassica (1), leek (2) and broadleaf sternum or fennel (3). In pine products pitch is indicated by (\*). Evidence of heating was presumed in the presence of ketones C31, C33 and C35 (Evershed et al. 1995) and/or APAAs (Shoda et al. 2018; Bondetti et al. 2021). Vessel drawings used as examples based on actual vessels from CSP and PB (Arcifa and Bagnera 2014; Sacco 2014).

In Casale San Pietro, we observed a similar consistency, with mixtures of animal fats, fruits and leafy vegetables present in all ceramic forms (Figure 35d). Of note, there was no evidence of waterproofing agents (pine products or beeswax) in samples from CLESP despite the fact ollae and cooking pots were seemingly used as general cooking wares for pottages and stews, as in Palermo. This may reflect a difference in the availability or need of these products between the urban centre and rural settlement. The presence of grape products/wine in four *ollae* samples from CLESP, alongside animal products and vegetable products suggests the integration of grape products such as wine or vinegar into cuisine at the rural site compared to limited evidence in samples from Palermo (1 sample from CSP).

Furthermore, evidence of porcine fats, tentatively identified in samples from CLESP, does not correlate with any particular vessel form (Figure 35d). The potential mixtures of pig fats, vegetables and fruits might suggest that their use was not tightly controlled, at least in a culinary sense. Conversely, in a number of samples from CLESP dairy is clearly separated from other products (i.e., animal adipose and plants). Additionally, in two samples where dairy products were present, thermal alteration markers were also identified. This suggests that vessels were dedicated for manipulation of dairy, perhaps in the production of ghee, yoghurts or cheeses. Of interest, evidence of dairy was only unambiguously identified in 'calcite wares' and not in other Palermo wares from CLESP or in samples from the Palermo sites. It cannot be assumed that dairy products were not being consumed at the Palermo sites and the lack of dairy traces in these vessels could be the result of the consumption of already processed dairy products (i.e., cheese) at these sites. However, we suggest this evidence constitutes not only a distinction between urban and rural resource use, and access to resources, but also sheds interesting light on specialised vessel use within the rural settlement. Although their provenance is still unknown, these wares were likely to have been imported,



raising questions regarding the production and use of 'calcite wares' at CLESP as they are seemingly representative of local culinary practices.

During the early 9<sup>th</sup> century, the inhabitants of Palermo were, at least to some degree, displaced by an incoming population and the city became increasingly urbanised. CLESP most likely benefited during the entire Islamic period being closely linked to the capital. The production of pottery at Palermo, and appearance of high-quality glazed wares at CLESP coincides with its rise in prosperity. However, it is likely that the capital also benefited from the rural site during this period as CLESP possibly supplied the capital with resources such as grains and processed dairy products. Overall, the archaeological material and the contents of the vessels analysed in this study shows that, in this period (9<sup>th</sup>-12<sup>th</sup> century), CLESP has an advantageous relationship with the capital, but there are differences in resource use at the site, most notably the production of dairy products in calcite ceramics, the integration of grape products in cuisine and possibly the consumption of pork. Future work on preceding periods should be undertaken to understand whether these differences reflect a differential impact of Islamic regimes at these sites.

## **5.4 Conclusions**

This study has provided the first direct evidence of cuisine from early medieval contexts in Sicily. We suggest that only by applying multifaceted organic residue analysis to many ceramic samples can complex cuisines begin to be untangled. The application of a range of analytical techniques and extraction methods enabled a wide variety of commodities and complex mixtures to be identified. For example, analytical procedures for the identification of small organic acids provided crucial evidence that fruits were incorporated in everyday culinary practices. A multifaceted analytical approach, including the identification of fruit products in domestic containers, has had its beginning in small corpus analysis of medieval ceramics (Salvini, Pecci and Giorgi 2008; Buonincontri et al. 2017; Pecci and Grassi-Munibe 2016; Notarstefano et al. 2011). This research has for the first time applied these methods systematically to a large corpus of samples. Due to the lack of food crusts on the surface of the ceramics,

an important caveat is that we are unable to distinguish whether foods were processed together to create complex 'dishes' or whether the residues build up over time (Miller et al. 2020) and therefore should be interpreted as a palimpsest reflecting multiple cooking events; indeed, both scenarios are likely.

Our main results can be summarised as follows:

1. Mixtures of commodities identified chemically, at all four sites across ceramic forms, are generally consistent with the colourful dishes noted in Arabic culinary literature, where meats, vegetables, and often fruits make up complex sweet, sour and salty recipes (Zaouali 2009; Perry 2005; Lewicka 2011; Peterson 1980). The organic residue data certainly does not contradict the uptake of these culinary practices in rural and urban settlements across North West Sicily, as has been suggested (Watson 1974, 1983).
2. Terrestrial animal products were widely processed in ceramics from all four sites but there was no evidence of marine or freshwater products. This study supports evidence that fish may not have been considered as important as meat products in medieval Islamic cuisine although fish may have been prepared using non- ceramic techniques.
3. Dairy products were unambiguously identified at CLESP but were only present in calcite wares. This find depicts a preference of these specific wares for the manipulation of dairy products. The absence of dairy products in ceramics from the Palermo sites and their absence in other Palermo production wares in CLESP suggests a distinction in the use of pottery of different productions and gives some insights into exogenous and local food practices associated with these vessels.
4. Dedicated uses of other forms (stone plates, braziers and pans etc.) could not be identified.
5. Porcine fats were tentatively identified in pottery from the rural site of Casale San Pietro. Alongside evidence from the faunal assemblage at this site, this may reflect less stringent food taboos applied in rural areas or the presence of Christian communities as suggested in other studies (Salas-Salvadó et al. 2006; Grau-Sologestoa 2017).

6. Brassicas, leeks and, possibly, fennel were identified providing an important complement to the archaeobotanical evidence. Fennel was exclusively found at the site of CLESP, where it grows prolifically today. However, specific biomarkers for other vegetable products (spinach, aubergine etc.) could not be identified.
7. The specific identification of grape products, more frequently in CLESP has opened questions regarding the use of these products and their integration into cuisine.
8. Non- culinary uses were identified in samples from Palermo sites in the form of pine products and beeswax products. This has provided insight not only into cuisine, but also pottery technologies during this period.

The evidence provided here should provide a useful baseline for further investigations aimed at examining continuity or change in pottery use as Sicily experienced profound social transformations during the Middle Ages, when under the control of different political powers. Furthermore, the analytical approach applied should provide a useful example to future studies of cuisine, particularly in contexts where complex mixtures of commodities might be expected.

## **5.5 Acknowledgments**

The authors of this article would like to thank Catalina Labra-Odde for her assistance in the extraction of modern plant products used in this study, Javier Montalvo Cabrera for his aid in the extraction of GA samples, Alice Di Muro for her help in preparing samples for extraction and Helen Talbot for her assistance in analysis and maintenance of the instruments. Finally, we are grateful to the two reviewers whose insightful comments contributed to the improvement of this article.

# Chapter 6 Cuisine in transition? Organic residue analysis of domestic containers from the 9<sup>th</sup>-14<sup>th</sup> Sicilian contexts

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## 6.2 Introduction

Under the rule of Islamic political regimes, notably the Aghlabid and Fatimid dynasties (9<sup>th</sup> -11<sup>th</sup> centuries), the introduction of new resources, agricultural systems and trade links generated considerable prosperity in Sicily (Watson 1974; Fiorentino, Lumaga and Zech-Matterne 2018; Carver, Fiorentino and Molinari 2019; Molinari 2021). However, the breakdown of the Kalbid dynasty, who ruled on behalf of the Fatimid dynasty from 948 AD, led to the end of Islamic rule in Sicily. The Normans, who had a longstanding interest in Sicily, took advantage of the political breakdown on the Island and began their conquest in 1061 AD. However, numerous defeats meant that the conquest took thirty years to complete (1061-1091 AD) and it was not until 1130 AD that a united Norman kingdom of Sicily was formed (Metcalf 2009, 87). The Norman regime under Roger II embraced the economic success of the Kalbid regime and the kingdom enjoyed its benefits for much of his reign and partly by his successor William II. The Norman period in Sicily held a unique character as several languages, cultures and religions existed in a single Kingdom with multiple layers and crossovers (Malette

1998; Abulafia 2005; Metcalfe 2002; Johns 2002; Metcalfe 2003; Lomax 1996). However, despite these ongoing relationships, the Norman invasion can also be seen as a turning point in Mediterranean history. Davis-Secord (2017,177) suggests, this invasion not only drew Sicily away from the Islamic world, but it also tipped the balance in power between Islam and Christianity, as Christianity began to push itself into the far east and the Levant. The Norman period is often defined as a time of coexistence between different groups, but these assumptions are often transposed onto the general Sicilian population from the viewpoint of the ruling elites and urban courts. It is important to move away from these assumptions, as the situation in Norman Sicily is much more complex, particularly for the non-elite populations (Molinari 2021). After the death of the Norman king William II in 1189, the unique multiculturalism that defined Norman Sicily disintegrated and in the 13<sup>th</sup> century social and cultural tensions were rife (Metcalfe 2009, 142, 2003; Lomax 1996) . In the rule of the Swabian king Frederick II, in 1194 AD, social and cultural tensions grew between different religious groups. By the 13<sup>th</sup> century Sicily became predominantly Latinized and Christianised as remaining Muslim and Jewish groups, who had increasingly become isolated, either left the Island or were expelled. Whilst it has been debated that the expulsion of remaining Jews and Muslims from Sicily under Fredrick's rule was a pragmatic choice, it undoubtedly changed the socio-cultural landscape of the Island (Booms and Higgs 2016). At the end of the 13<sup>th</sup> century, Sicily's political and economic dynamic shifted yet again, and by the 14<sup>th</sup> century Sicily had lost its independence and was ruled by the Crown of Aragon.

Although the period from the 9<sup>th</sup> -14<sup>th</sup> centuries saw changing socio-economic, political, and religious situations, each regime reflects an intricate tapestry where elements of the preceding regime have been continued, evolved, and in some cases abandoned. However, there is no doubt that Sicilian society by the 14<sup>th</sup> century was very different to the one experienced by the populations under the Islamic political regimes. The impact of these transitions in regime from the 9<sup>th</sup>-14<sup>th</sup> centuries on the population of Sicily is a complex subject that requires careful investigation and examination to be understood. The SICTRANSIT project was designed to investigate such changes, particularly as they affected farmers, merchants and their families (Carver, Fiorentino and Molinari 2019). As part of this project, new research was launched to give insight

into the impact of these changes in regime on the population of Sicily through the lens of cuisine over this 600-year period.

Cuisine- the way in which foods are combined, prepared, and consumed can yield important insight into everyday life. Culinary habits are often linked, but by no means limited to, socio-economic factors such as food availability and foodscapes, wealth, faith, and the technologies available for the procurement and processing of food. Indeed, it is hypothesised that the complexity of the transitions experienced in Sicily from the 9<sup>th</sup> to 14<sup>th</sup> centuries are likely reflected in the culinary habits of the populations that experienced them. As utilitarian artefacts, domestic cooking pots that are routinely found through archaeological investigation might be expected to capture a significant subset of foodstuffs processed daily. Organic residue analysis (ORA) of these domestic cooking wares can give direct chemical evidence of their contents and yield important insight into the types of foodstuffs that were prepared and combined by the communities that utilised them. A recent study of Islamic cooking pots and other domestic containers from 9<sup>th</sup>-12<sup>th</sup> century Sicily has shown that a wide range of animal products, fruits and vegetables were processed, and has yielded important insight into the use of resources in both urban and rural contexts under the same regimes (Lundy et al. 2021). Studies have aimed to assess culinary habits in Italian 12<sup>th</sup>- 13<sup>th</sup> century contexts through the analysis of ceramic containers (Giorgi,Salvini and Pecci 2010; Salvini,Pecci and Giorgi 2008; Buonincontri et al. 2017; Pecci and Grassi-Munibe 2016; Pecci et al. 2016) and a recent study also examined the impact of the Norman conquest in England through a multifaceted approach which included ORA of 10<sup>th</sup>-12<sup>th</sup> century cooking pots to examine their contents before and after the conquest, with particular focus on the dietary habits of lesser represented rural populations (Craig-Atkins et al. 2020b). However, to date no study has assessed the use of pottery vessels in post-Islamic contexts in Sicily.

To further understand the impact of these regime changes on culinary choices, 114 cooking wares were obtained from Islamic, Swabian and Aragonese contexts from the urban site of Mazara del Vallo and Norman and Swabian contexts from the rural settlement of Casale San Pietro. These results are compared to 134 previously published data from Islamic contexts from Palermo and Casale San Pietro (Chapter

5)(Lundy et al. 2021) to enable an in-depth comparison of pottery use in western Sicily over this significant period of transition and between different socio-economic settings. In order to gain an in-depth understanding of the commodities used in these pots, a multifaceted organic residue approach was used. Well-established methods for the extraction of residues from the ceramics (Craig et al. 2013a; Correa-Ascencio and Evershed 2014; Garnier and Valamoti 2016; Craig et al. 2007; Dudd and Evershed 1998) (Appendix A) were used and a range of gas chromatography and mass-spectrometry techniques were applied to identify these residues including; selected ion monitoring (SIM) modes to identify aquatic biomarkers and alkylresorcinols (Hansel et al. 2004; Hammann and Cramp 2018) and Gas Chromatography- combustion- Isotope Ratio Mass Spectrometry (GC-c-IRMS) to help to distinguish between animal fat origins (Copley et al. 2005c; Craig et al. 2011; Dudd and Evershed 1998) (Appendix A). We also employed a further extraction method to extract small organic acids to support identification of grapevine products and other fruit products (Drieu et al. 2020; Garnier and Valamoti 2016). The full methods and protocols used in this study are outlined in Appendices A and B. The application of these techniques to a large corpus of ceramics dating to the 9<sup>th</sup>-14<sup>th</sup> centuries in both rural and urban settings can enable a unique and in-depth insight into daily culinary habits during this transition in different socio-economic settings. Here we ask:

1. How do the contents of domestic containers from Norman, Swabian and Aragonese contexts compare to domestic containers from Islamic contexts?
2. How were culinary practices changed or maintained in both urban and rural settings as a result of these transitions?

## **6.3 Sites and Samples**

### **6.3.1 Mazara del Vallo (MZ)**

Mazara del Vallo (MZ) (Trapani) is located on the southwestern coast of Sicily (Figure 36). Archaeological investigations in the north-western part of the town, performed in 1997, have revealed streets with domestic residences and cess pits that serve them.

Excavations also reveal a limestone surface with buildings, silos, wells, and ditches that cut the ground, dated from the late 7<sup>th</sup>/ early 8<sup>th</sup> to the late 17<sup>th</sup>-18<sup>th</sup> centuries AD. Based on these finds, the first Islamic phase was dated to the late 10<sup>th</sup>-11<sup>th</sup> century, when the site was occupied, by pit latrines. Later, during the 11<sup>th</sup> century, after the demise of the major part of the latrines, a pottery workshop was built, with pits and a furnace. After the demolition of the pottery workshop, life continued at Mazara del Vallo until the modern period. The majority of 11<sup>th</sup> century ceramics analysed in this study were obtained from the usage and abandonment layers of a waste pit, that served the domestic residences of the town, (Latrine 5) from US (19, 27, 31, 45 and 55) (Meo forthcoming). The radiocarbon date from a chicken from US 19 dates between 925-1025 AD at 88.1% confidence (Carver et al., forthcoming). Two 11<sup>th</sup> century ceramic samples analysed in this study were obtained from the abandonment layers of the pottery workshop (US 78). Although Palermo was a major ceramic production site during the 9<sup>th</sup>-11<sup>th</sup> century, Mazara did not rely on kitchen wares produced in Palermo at this time, unlike other places, such as Casale San Pietro (Carver et al. 2019; Lundy et al. 2021). Instead, Mazara produced its own local ceramics, but because of its coastal location, it also received imports from elsewhere, particularly from North Africa. This depicts strong links between Mazara and North Africa at this time. The vessel types from this assemblage consist of cooking pots, pans, stone plates, braziers/tripods and some jugs with evidence of soot marks (Appendix I, Table I.1). The ceramic samples selected for this study have been dated based on their typology and associated radiocarbon dates in stratigraphic phases (Meo forthcoming)

The later pottery comes from the decommission of wells belonging to the mid-13<sup>th</sup> century (Well 1: US 7), the late 13<sup>th</sup> century (Well 3: US 52, 71), and the mid-14<sup>th</sup> century (Well 4: US 176). In these contexts, there is a shift in the types of containers. Of note, there is a rise in glazed cooking wares, particularly partially glazed wares from Messina, where the rim and internal base of the vessel was glazed (Meo and Orecchioni 2020; Orecchioni forthcoming). From the 13<sup>th</sup> -14<sup>th</sup> century contexts, a selection of partially glazed cooking pots, unglazed pots, bowls/pans, *Pentolino* (small cooking pots) and a jug were selected for analysis (Appendix I, Table I.1). Again, the samples selected for this study are from contexts associated with the use of domestic residences and have been dated based on their typology and associated radiocarbon dates



(Orecchioni forthcoming). The full sequence and investigation of Mazara del Vallo is being produced as part of the wider Sicily in Transition project (Meo et al. forthcoming).

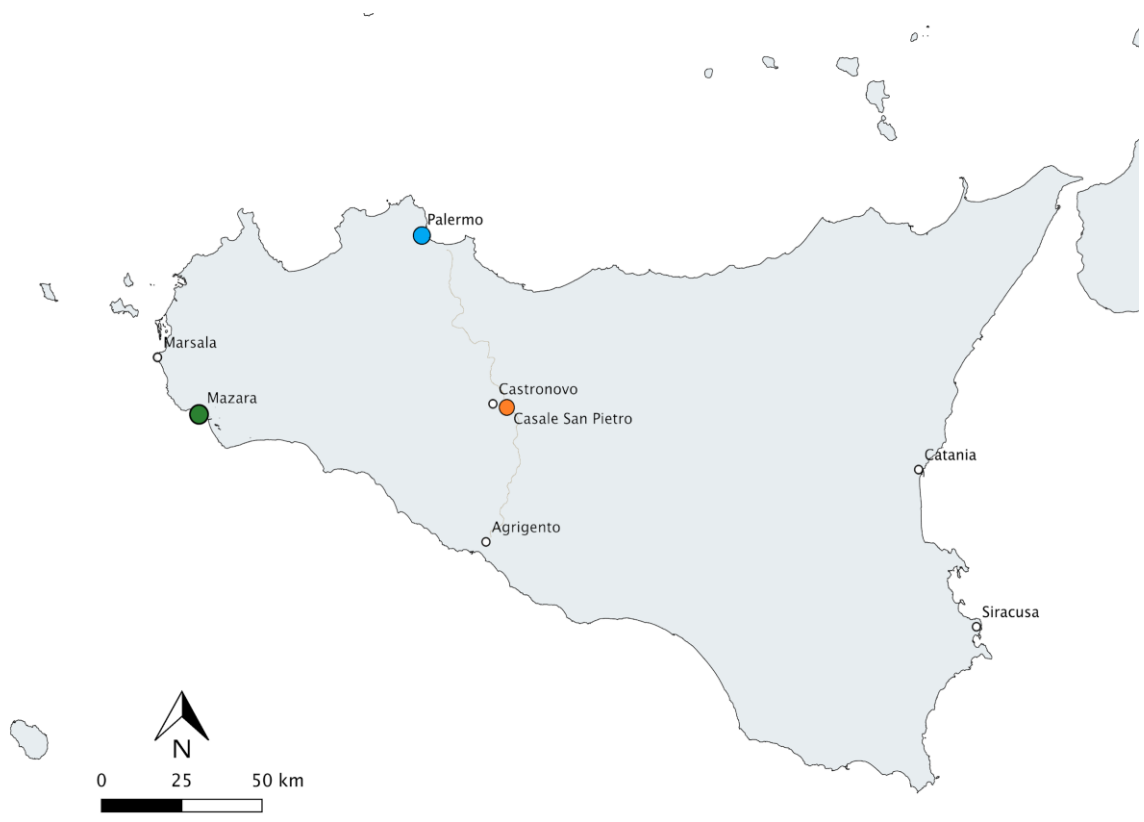


Figure 36: Map of Sicily showing the location of Mazara del Vallo, Casale San Pietro and Palermo. The main road between Palermo and Agrigento from North to South is marked on the map.

### 6.3.2 Casale San Pietro (CLESP)

Casale San Pietro (CLESP) is a site located on the plain outside of the town of Castronovo di Sicilia, in the rural hinterlands of the Palermo province. Excavations in 2016-2019 revealed that the site flourished during the 10<sup>th</sup>-11<sup>th</sup> century (Carver et al. 2017, 2019; Carver and Molinari 2016). An impressive assemblage of ceramics, faunal remains and other material culture was recovered from this period. A series of walls could be attributed to Islamic style architectural forms. In the 12<sup>th</sup> century the site was still occupied, either as a small agricultural settlement or a station on the main road between Agrigento and Palermo and evidence suggests at least in the early 12<sup>th</sup> century new buildings were built, utilizing the pre-existing structures of the Islamic and Roman

phases. Towards the later 12<sup>th</sup> century into the 13<sup>th</sup> century, it is likely that inhabitants predominantly occupied the nearby town of Castronovo and the Norman Castle of San Vitale on the hills overlooking Casale San Pietro. However, ceramics dated to the 12<sup>th</sup> and 13<sup>th</sup> century found at Casale San Pietro suggests that there was still some domestic occupation at the site in the Norman and Swabian periods, but with a reduced economic profile (Carver et al. 2017, 2019; Carver and Molinari 2016). The 10<sup>th</sup> -12<sup>th</sup> century ceramics previously studied by ORA consisted of *ollae*, cooking pots, pans and stone plates. In contrast to Mazara, these ceramics were predominantly imported from Palermo, with a small number of handmade pottery and calcite wares of unknown provenance (Appendix E)(Chapter 5)(Lundy et al. 2021). In the 12<sup>th</sup> century trade networks of ceramics into CLESP changed and new types were observed (Carver et al. 2017). In this assemblage we see the introduction of new glazed wares imported from Northeast Sicily, like those in Mazara. In the 13<sup>th</sup> century, unsurprisingly in comparison to the urban centre of Mazara, there is lesser variety in these cooking wares (Carver et al. 2017, 2019). However, a selection of cooking pots and pans, glazed, partially glazed and unglazed were analysed in this study (Appendix I, Table I.2). The ceramic samples selected for this study were taken from defined contexts dated with radiocarbon dates and associated finds (Carver et al. forthcoming). An effort was made to avoid samples that could not be assigned to chronological phases. However, the full interpretation of the ceramic assemblages and site sequence are forthcoming (Carver et al. forthcoming)

### **6.3.3 Palermo sites**

The organic residue analysis of these ceramics was compared to previously published ORA results from 9<sup>th</sup>-11<sup>th</sup> century ceramics from three sites in the urban capital of Palermo. The details of these sites and the ceramics studied are described in full elsewhere (Chapter 5) (Lundy et al. 2021), but in brief a total of 83 vessels were obtained from Castello San Pietro (CSP), La Gancia (GA) and Palazzo Bonagia (PB). These consisted of *ollae*, cooking pots, braziers, lids, pans and stone plates (Arcifa and Bagnera 2014; Ardizzone, Pezzini and Sacco 2014; Sacco 2014; Lundy et al. 2021). In the scope of this project, later Norman and Swabian domestic wares from Palermo were unavailable for study. For example, at the site of CSP, archaeological features from

Norman and Swabian have been uncovered, but their full interpretation is not yet known.

## 6.4 Results

Of the 114 samples analysed in this study a total of 97 samples (85%) yielded appreciable lipids (S3 Data) above the limit of reliable interpretation ( $5 \mu\text{g g}^{-1}$ ) (Evershed 2008b; Evershed et al. 1999). These results reflect the positive recovery of lipids from samples from Casale San Pietro and Palermo, where >90% of samples yielded appreciable concentrations of lipids (Chapter 5)(Lundy et al. 2021).

### 6.4.1 Animal products

In the majority of samples analysed, the lipid profiles were characteristic of degraded animal fats. These lipids were dominated by palmitic ( $\text{C}_{16:0}$ ) and stearic acids ( $\text{C}_{18:0}$ ) in equal proportions and, in many cases, branched and linear  $\text{C}_{15:0}$  and  $\text{C}_{17:0}$  fatty acids were present, which are formed through bacterial transformation in the rumen and therefore can be attributed to ruminant origin (Christie 1978)(S3 Data). Cholesterol, which is in high abundance in modern animal fats, was also observed in 34/114 samples. The presence of *n*-ketones  $\text{C}_{31}$ ,  $\text{C}_{33}$  and  $\text{C}_{35}$  in a number of samples indicated the heating of animal fats at temperatures above  $300 \text{ }^\circ\text{C}$  (Raven et al., 1997). Furthermore, 32 of the samples extracted using conventional solvent extraction methods yielded intact triacylglycerols (TAGs) (Appendix J). Figure 37 is a high temperature chromatogram showing a typical lipid profile of ruminant adipose fat. When observing the TAG profiles the majority of samples yielded profiles characteristic of ruminant adipose fats ( $\text{T}_{46}$  to  $\text{T}_{54}$ ). Unlike non-ruminant adipose fats, where there is a clear predominance of  $\text{T}_{52}$  over  $\text{T}_{50}$  and  $\text{T}_{54}$  and the distribution for ruminant fats is centred on  $\text{T}_{52}$  (Dudd and Evershed 1998). A broader range of TAGs ( $\text{T}_{42}$  to  $\text{T}_{54}$ ), distinctive of dairy fat origin, was observed in at least 2 samples from MZ, but no samples from CLESP showed this broad range (Dudd and Evershed 1998) (Appendix J).

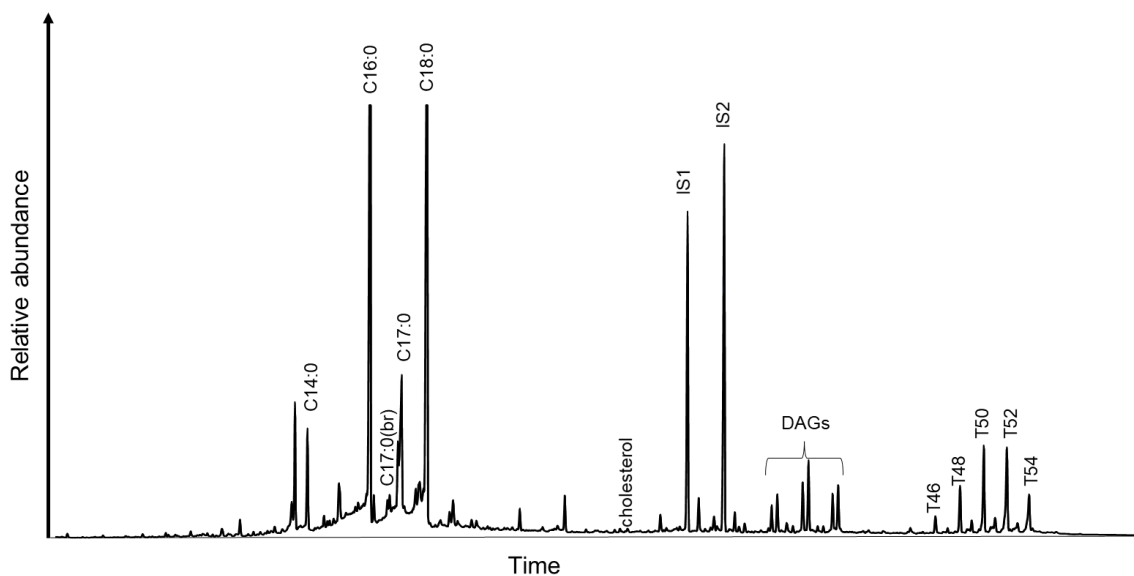


Figure 37: FID high temperature chromatogram of TLE extract MZ\_189 typical of ruminant adipose fat. Fatty acids C<sub>14:0</sub> C<sub>16:0</sub>, C<sub>18:0</sub> and branched (br) and unbranched C<sub>17</sub>, cholesterol, ketones (KT) monoacylglycerols (MAGs), diacylglycerols (DAGs) and TAGs (T44-54) are shown alongside the internal standards alkane C<sub>34</sub> (IS1) and C<sub>36</sub> (IS2).

## 6.4.2 Compound-specific isotopic values

Assigning resource contributions based on TAG profiles alone can be limited due to the preferential loss of lower molecular weight components and mixing of resources (Dudd and Evershed 1998). Therefore, to further understand the origin of these lipids, 100/114 of the samples were analysed by GC-c-IRMS to determine the stable carbon isotope value ( $\delta^{13}\text{C}$ ) of the major fatty acids (C<sub>16:0</sub> and C<sub>18:0</sub>). These values were compared to the 118 previously published fatty acid  $\delta^{13}\text{C}$  values from 9<sup>th</sup>-12<sup>th</sup> century ceramic samples from CLESP and Palermo sites (Chapter 5)(Lundy et al. 2021) (Figure 38) (S3 Data). This analysis helped to distinguish between ruminant adipose, ruminant dairy fats and non-ruminant fats (Copley et al. 2005c; Craig et al. 2011; Dudd and Evershed 1998). However, as previously noted it must be considered that the contribution of non- animal products, such as plant oils, waxes and nut oils in these ceramics may influence the  $\delta^{13}\text{C}$  values obtained here (Lundy et al. 2021).

Most of the fatty acid  $\delta^{13}\text{C}$  values from both Islamic vessels and Swabian vessels from MZ fall within the range of ruminant adipose (53% and 43%, respectively) and non-ruminant fats (45% and 48%, respectively) (Figure 38a). Two samples from Islamic contexts in MZ and one sample from Swabian contexts could be unambiguously attributed to dairy fats, as the  $\delta^{13}\text{C}$  values fall within the range of modern dairy fats ( $<-3.3$  ‰). The contribution of dairy fats is also reflected in the broad TAG distribution of two of these samples (MZ\_ 81 and MZ\_ 227) (Appendix J). The selection of ruminant adipose fats for processing in the ceramics is well reflected in the faunal assemblage at MZ, as in both Islamic and Swabian contexts caprines are the most dominant species of the main domesticates (caprines, cattle, suids and chicken) (Aniceti 2020, forthcoming)(Appendix K). Cattle, on the other hand, is barely represented at the site in both contexts and mainly consists of adult and elderly individuals, indicating beef was less likely processed and cattle were instead used for secondary products such as milk production, manure, and agricultural works (Aniceti 2020, forthcoming). The high occurrence of  $\delta^{13}\text{C}$  values that fall within the range of non-ruminant products may indicate the selection and processing of porcine. However, the faunal remains from MZ show that suid remains were scarce from Islamic contexts, supporting the assumption that pork was not consumed, as it was prohibited by Islamic hadiths (Aniceti 2020; Aniceti and Albarella 2022). Whilst the incidence of suid remains in the faunal assemblage from MZ increases in the following phase, it is not possible to unambiguously suggest presence of porcine fats in the ceramics based on these  $\delta^{13}\text{C}$  values alone, and no samples yielded TAG profiles indicative of porcine fats (Appendix J). Depleted  $\delta^{13}\text{C}_{16:0}$  values ( $\sim-30$  ‰) within the range of non-ruminant fats can indicate processing of other non-ruminant animal fats such as domestic fowl (Colonese et al. 2017b) or hare (Drieu et al. 2021a), albeit the latter is hardly represented in the Islamic and Swabian faunal assemblage at MZ (Aniceti 2020, forthcoming) (Appendix K). Alternatively, the contribution of  $\text{C}_3$  plant products such as vegetable oils, nuts and cereals can affect  $\delta^{13}\text{C}$  values falling in the range of non-ruminant products. Therefore, as discussed by Lundy et al. (2021), it is difficult to determine the source of these  $\delta^{13}\text{C}$  values and indeed mixtures of non-ruminant animal products, plant products and ruminant animals cannot be dismissed. Nonetheless, by comparing the two periods at

MZ, there is no statistical difference between the  $\Delta^{13}\text{C}$  values based on a Mann-Whitney U test ( $W = 633$ ,  $p\text{-value} = 0.6763$ ).

The  $\delta^{13}\text{C}$  values of samples from CLESP analysed in this study also predominantly fall within the range of both ruminant and non-ruminant adipose fats (Figure 38b). Previously published  $\delta^{13}\text{C}$  values from Islamic contexts from CLESP depict an equal distribution of samples falling within the range of ruminant and non-ruminant fats (46% and 48%, respectively) (Chapter 5) (Lundy et al. 2021). There is a slight increase in the percentage of samples that fall within the range of non-ruminant fats within the Norman period (34% ruminant compared to 50% non-ruminant values) and again in the Swabian period (41% compared to 58%). However, due to the variability in sample size across these periods there is no statistical difference between the distribution of  $\Delta^{13}\text{C}$  values across periods, as a Kruskal Wallis test revealed ( $\chi^2(2) = 3.74$ ,  $p\text{-value} = 0.15$ ). Furthermore, 3 Sicilian pots from the Norman period fell within the range of dairy fats, reflecting a continued processing of dairy products from the Islamic to Norman periods, whereas no samples from Swabian contexts could be unambiguously attributed to dairy fats, perhaps reflecting a disuse of ceramic vessels for the processing of dairy in this period. Faunal remains from Norman/Swabian periods at CLESP are currently under investigation as part of the wider Sicily in Transition project; further zooarchaeological results will provide more detailed insights on the statements discussed, which will enable comparisons to be made with organic residues in future works.

In comparison to the  $\delta^{13}\text{C}$  values of samples from Islamic vessels from sites in Palermo, both MZ and CLESP show a broader range of  $\Delta^{13}\text{C}$  values from all chronological periods (Figure 38c). In Palermo, no samples could be attributed to a dairy origin and it was previously reported that this may reflect a difference in the use of dairy products between Islamic urban and Islamic rural sites, due to the presence of dairy in ceramic samples at CLESP (Lundy et al. 2021). The differential use of dairy products between urban and rural sites has also been observed in medieval England. At the site of St Aldates, Oxford there was a near absence of dairy products in vessels at the urban site (Dunne et al. 2021) which contrasts to the rural site of West Cotton, Northamptonshire where dairy products were readily identified (Dudd and Evershed 1998; Dunne et al. 2019).

However, the presence of dairy in the vessels from both Islamic and Swabian Mazara, opens new questions regarding this trend.

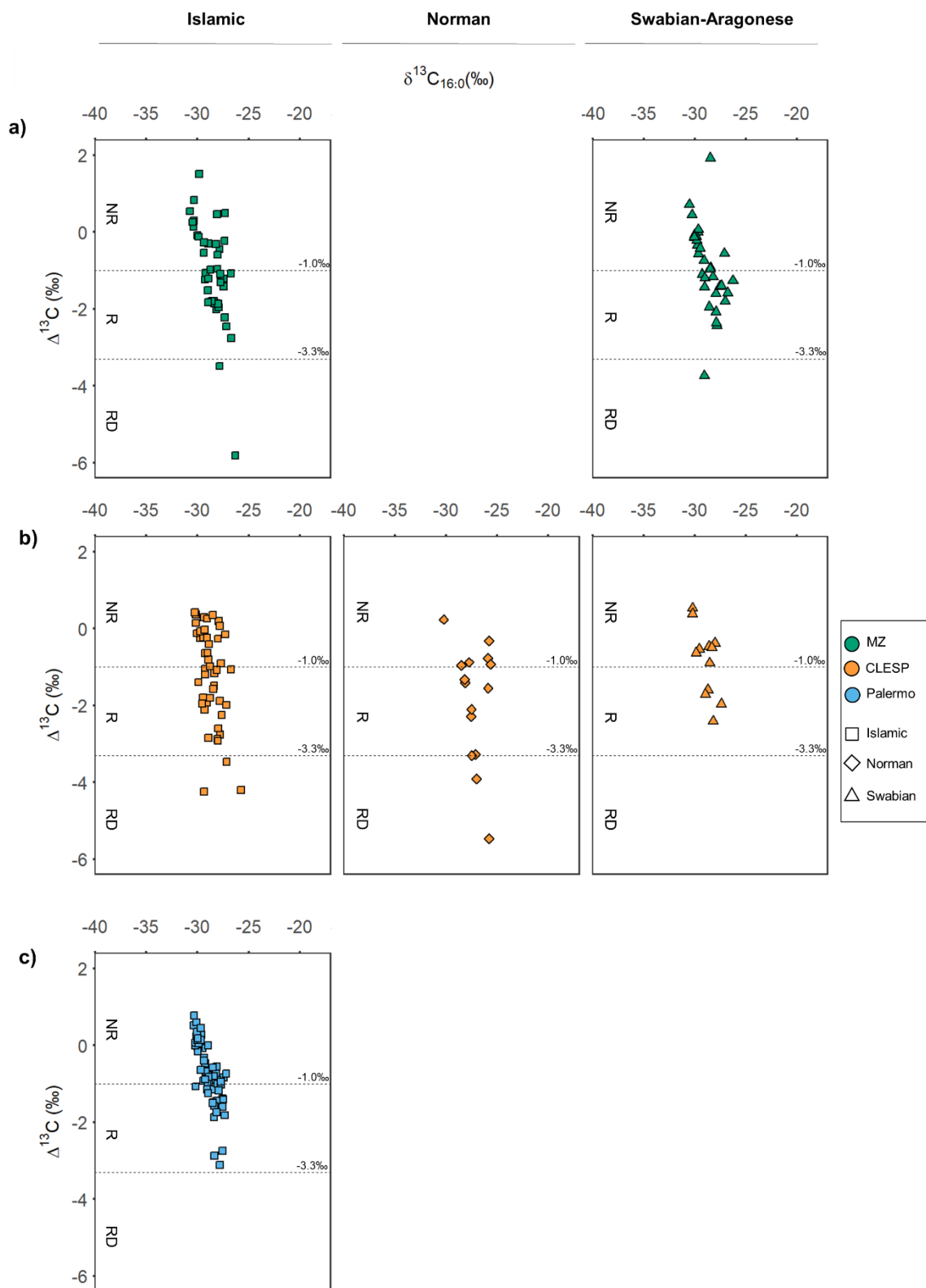


Figure 38: Plots of fatty acid stable isotope values obtained from individual vessels from Sicilian pottery. Plot of  $\Delta^{13}\text{C}$  against  $\delta^{13}\text{C}_{16:0}$ . values  $< -3.3\text{‰}$  are typically associated with RD (Ruminant dairy), values between  $-3.3\text{‰}$

and -1.0 ‰ are associated with R (Ruminant adipose) and above -1.0‰ can be considered as NR (Non- ruminant) (Craig et al. 2012).

### 6.4.3 Absence of aquatic products

Whilst a small number of samples were slightly enriched in  $\delta^{13}\text{C}$  ( $> -27\text{‰}$ ) which could indicate the presence of marine products in these ceramics, as discussed, the majority of samples yielded more depleted  $\delta^{13}\text{C}$  values than would be expected for marine products. Furthermore, despite the use of sensitive methods for their detection, no samples yielded a complete suite of aquatic biomarkers (Appendix A). Despite the presence of isoprenoid acids in several samples (S3 Data), these were either present in trace amounts or could not be distinguished from other sources such as ruminant products (e.g., when phytanic acid SRR diastereomers were calculated) (Lucquin et al., 2016). Furthermore, a complete absence of APAA C<sub>20</sub> and APAA C<sub>22</sub> in all samples analysed, formed through heating aquatic oils (Hansel et al., 2004; Evershed et al., 2008), meant that it was not possible to confirm the processing of aquatic sources in these vessels. Although the absence of APAA C<sub>20</sub> and APAA C<sub>22</sub> does not dismiss the presence of non-heated aquatic oils, potentially mixed with other animal products in these ceramic vessels (e.g., fish sauce), it has been shown that APAAs can be formed through relatively low intensity heating conditions (Bondetti et al. 2021).

### 6.4.4 Plants

A large majority of samples analysed from both MZ and CLESP revealed biomarkers and lipid profiles indicative of plant contribution ( $n=76$ ) (S3 Data). The presence of  $\beta$ -sitosterol and stigmasterol in a number of samples suggests the presence of plant products without offering a more precise identification because of their ubiquity in the plant kingdom. However, in several samples it was possible to identify the contribution of plant waxes through the specific distribution of *n*-alkanes, *n*-alkanols, and ketones.

Lipid profiles, indicative of leaf waxes of more specific taxon, were also identified in several samples from both sites and chronological periods. The presence of ketone



nonacosane-15-one and *n*-alcohol nonacosane-15-ol in conjunction with a dominance of *n*-alkane C<sub>29</sub> has been attributed to the presence of *Brassica* (Charters et al. 1997) and was identified in 20 samples from both Islamic and Swabian samples from MZ, but was not observed in Norman and Swabian samples from CLESP. Furthermore, hentriacontane-16-one (C<sub>31</sub> ketone) and *n*-hentriacontane (C<sub>31</sub> alkane) which has previously been attributed to the contribution of *Allium*, potentially leek (Evershed et al. 1992b, 1995; Raven et al. 1997; Rhee et al. 1998), was observed in the same vessels that yielded *Brassica* biomarkers from MZ (Figure 39a). However, again, this was not observed in any samples from CLESP. Additionally, nonacosane-10-one previously observed in 10<sup>th</sup>-12<sup>th</sup> century ceramics from CLESP and attributed to the possible contribution of fennel (*Foeniculum vulgare*) (Muckensturm et al. 1997; Lundy et al. 2021)(Chapter 5), was identified in 5 samples from Norman contexts from CLESP (Figure 39b). Clear evidence of plant oil could only be identified in one vessel from MZ, a jug known to be used on the fire as evidence of soot were identified on the side of the vessel. The presence of plant oil was indicated by the relatively high oleic to stearic acid ratio (C<sub>18:1</sub>/C<sub>18:0</sub> >2), in addition to a small amount of linoleic acid (C<sub>18:2</sub>)(Copley et al. 2005b; Romanus et al. 2009) (Figure 39c). A further 2 samples from CLESP also showed evidence of plant oil contribution in the 12<sup>th</sup> century using the same criteria (S3 Data).

As shown previously, plant biomarkers were often observed in samples that showed evidence of animal fat origins (Chapter 5)(Lundy et al. 2021). Although it is not possible to distinguish between mixing of commodities in a single cooking event and subsequent uses of different products (Miller et al. 2020), it is not unexpected that both plants and animal products were mixed in a single recipe.

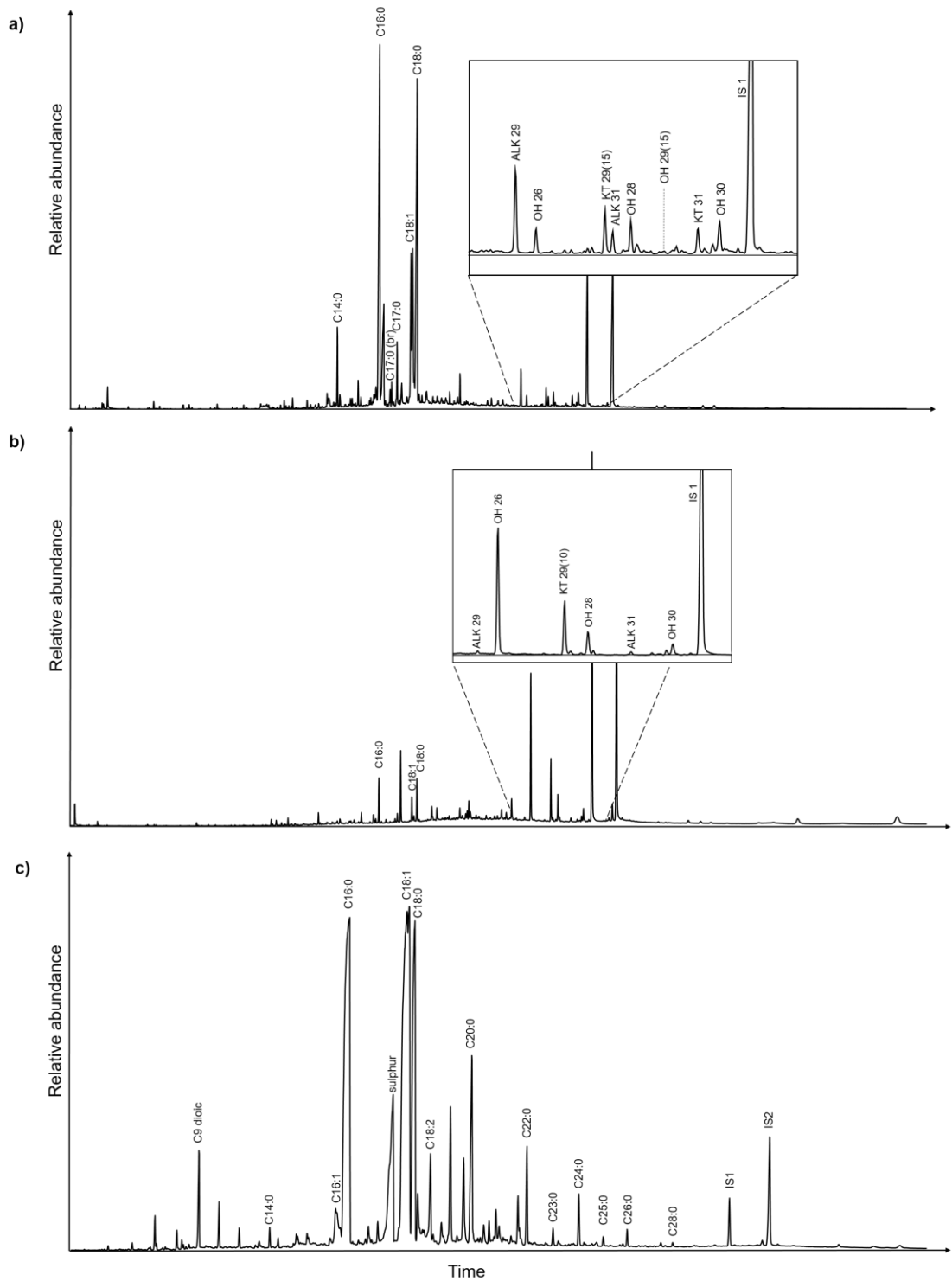


Figure 39: TIC chromatograms of extracts typical of plant contributions. a) TLE chromatogram of sample MZ\_14 showing the presence of C31 ketone (hentriacontane-16-one) that indicates the presumed presence of leek (*Allium porrum*) in the ceramic samples (Evershed et al. 1992b, 1995; Raven et al. 1997; Rhee et al. 1998) and C29(15) ketone that indicates the presumed presence of Brassica (Charters et al. 1997). b) TLE chromatogram of CLESP\_194 showing the presence of C29 (10) ketone (nonacosane-10-one) that indicates the presumed presence of (*Foeniculum vulgare*) (Fennel) (Muckensturm et al. 1997). c) AE chromatogram of CLESP\_69 indicating plant oil by a  $C_{18:1}/C_{18:0} > 2$  and the relatively small contribution of  $C_{18:2}$  (Copley et al. 2005b; Romanus et al. 2009).

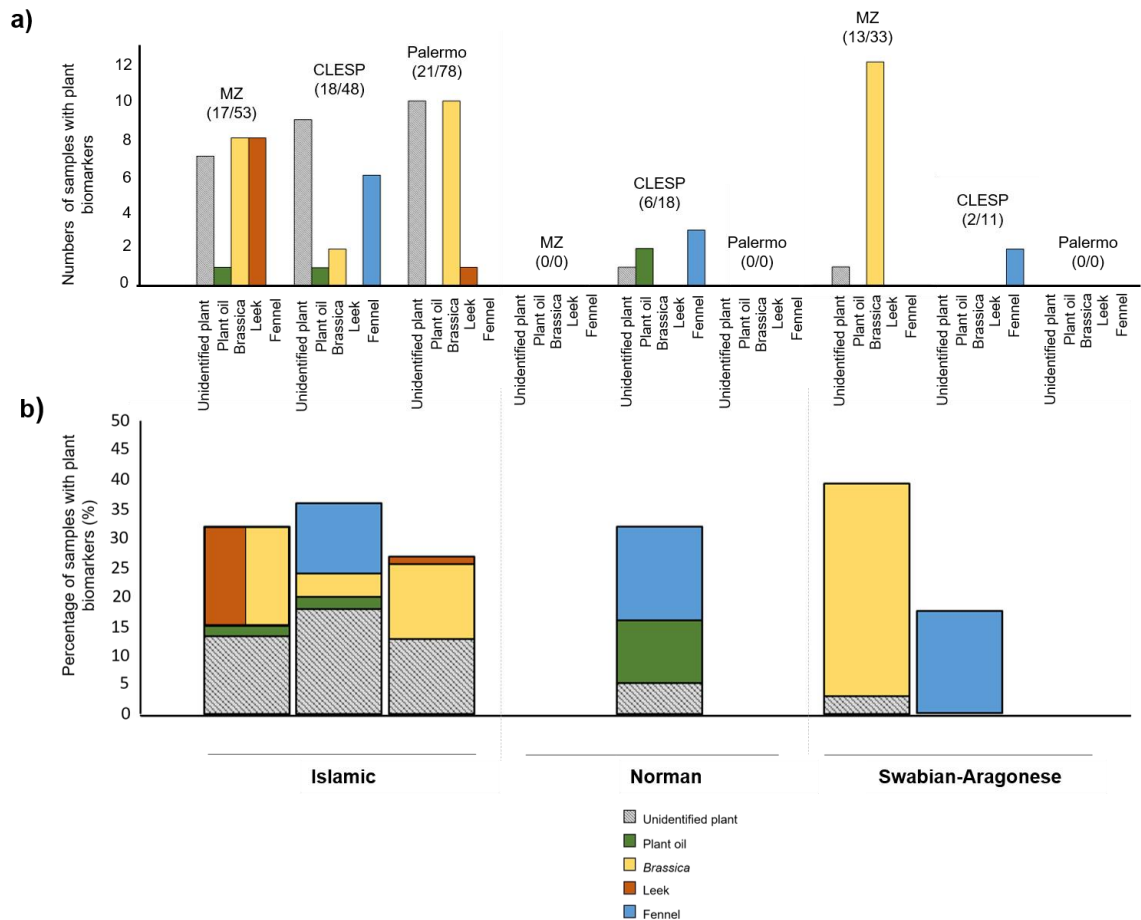


Figure 40: Molecular evidence of plant biomarkers from ceramic samples MZ, CLESP and Palermo. a) Number of samples with evidence of unidentified plant, plant oil, brassicas, leek and proposed fennel b) Percentage of samples with plant biomarkers whereby unidentified plants are assigned by the presence of sitosterol, stigmasterol and or/ specific n-alkane distributions. Plant oil is assigned by a  $C_{18:1}/C_{18:0} > 2$  and the relatively small contribution of  $C_{18:2}$  (Copley et al. 2005b; Romanus et al. 2009).  $C_{29}(10)$  ketone (nonacosane-10-one) indicates the presumed presence of (*Foeniculum vulgare*) (fennel) (Muckensturm et al. 1997).  $C_{29}(15)$  ketone indicates the presence of Brassica (Charters et al. 1997).  $C_{31}$  ketone (hentriacontane-16-one) indicates the presumed presence of leek (*Allium porrum*) in the ceramic samples (Evershed et al. 1992b, 1995; Raven et al. 1997; Rhee et al. 1998).

By comparing the presence of plant biomarkers from each of these sites and to previously published data from Islamic CLESP and Palermo, some observations can be made (Figure 40). A wide variety of plant products is observed in the ceramics from each site in the Islamic period, but with variations from one site to another. This diversity seems to persist at CLESP, at least during the Norman period, then disappear during the Swabian period. On the contrary, in Mazara, plant consumption seems to focus on *brassicas* during the Swabian period. This may indicate less diversity of vegetable products in ceramics as part of post-Islamic cuisines. However, this must be taken with caution, due to the difficulty in identifying plant products in archaeological

ceramics, which is hindered by non-specificity and lower yields compared to animal products (Evershed 2008a).

#### **6.4.5 Fruits and further evidence of plant products**

A number of samples from both MZ and CLESP were also extracted using the acid butylation extraction method to extract small organic acids ( $n=97$ ), as their recovery through conventional solvent extraction methods is poor because they are relatively insoluble in organic solvents (Drieu et al. 2020; Garnier and Valamoti 2016) (Appendix A). Small organic acids including fumaric, succinic, malic, and tartaric acid can indicate the presence of fruit products (Garnier and Valamoti 2016; Drieu et al. 2021b). Malic acid and tartaric acid were identified in a number of samples in varying quantities (S3 Data). However, because they are both present in different fruits and plant products (Drieu et al. 2020; Lundy et al. 2021; Drieu et al. 2021b) it was important to consider the proportions of the acids individually and in relation to each other to better understand their origin (Drieu et al. 2021b) (Figure 41).

Tartaric acid is the main acid in both grapes and wine, but previous research has shown that in order to assert the presence of grapevine products in archaeological ceramics it is important to compare the proportions of malic and tartaric acids (Drieu et al. 2021b, 2020; Jaeggi, Wittmann and Garnier; Linger-Riquier et al. 2016; Cherel et al. 2018). Drieu et al. (2021) suggest that the percentage contribution of tartaric acid (%TA) compared to malic acid should be  $>35\%$  in order to distinguish grapevine products from other fruit products (Figure 41a). Using this criterion, the presence of grapevine products was identified in two ceramic samples studied here. These included one cooking pot from CLESP and one cooking pot from MZ, both from Swabian contexts. This may indicate the use of these cooking pots to heat or store wine, or its use as a flavouring for foods as previously observed in medieval Italy (Salvini, Pecci and Giorgi 2008; Buonincontri et al. 2017). However, the presence of tartaric acid could also indicate the use of other grapevine products such as vinegar, juices, or syrups. The absence of grapevine markers in Islamic contexts from MZ mirrors the near absence of samples that yielded grapevine products from Islamic Palermo sites in comparison to

a higher occurrence from samples from Islamic contexts at the rural site of CLESP (Chapter 5) (Lundy et al. 2021).

A number of samples yielded  $> 1 \mu\text{g g}^{-1}$  of malic acid, which is one of the most abundant small acids present in a number of fruits such as plums, apples and peach (Walker and Famiani 2018), and therefore may indicate a fruit contribution, other than grapevine, in these vessels. However, it must be considered that malic acid is also present in a variety of other plants including brassicas and leeks and thus might simply strengthen our identification of these vegetables in the ceramics (Ruhl and Herrmann 1985a) (Appendix H). There is a difference in the yields of malic acid recovered from the urban sites of MZ and Palermo compared to the rural site of CLESP. The overall yield of malic acid from samples from MZ and Palermo are significantly higher than the yields recovered from CLESP (Figure 41a and b). This was supported by a Kruskal Wallis test which indicated a statistical difference between sites when comparing the yield of malic acid in Islamic samples ( $\chi^2(1) = 9.39, p - \text{value} = 0.002$ ), and when comparing MZ and Palermo to CLESP with consideration of all chronological periods (Islamic, Norman and Swabian) ( $\chi^2(1) = 10.63, p - \text{value} = 0.001$ ). These results could indicate suggest a more frequent incorporation of fruits in cuisine in urban areas in comparison to rural areas, as suggested in previous research (Chapter 5) (Lundy et al. 2021). However, the significance of these values is difficult to interpret due to the variability in potential contributions to these values (fruits vs brassicas or leeks) and factors that may impact the differential survival of malic acid that are not easily understood.

The presence of fruit products in Norman and Swabian samples from CLESP and MZ highlight the continued use of these products in post-Islamic periods. Although it is not possible to identify any variability in the species, these results reflect the archaeobotanical record where, at MZ at least, a continued variety of fruit remains have been identified in Swabian periods (Fiorentino et al. forthcoming; Primavera 2018a).

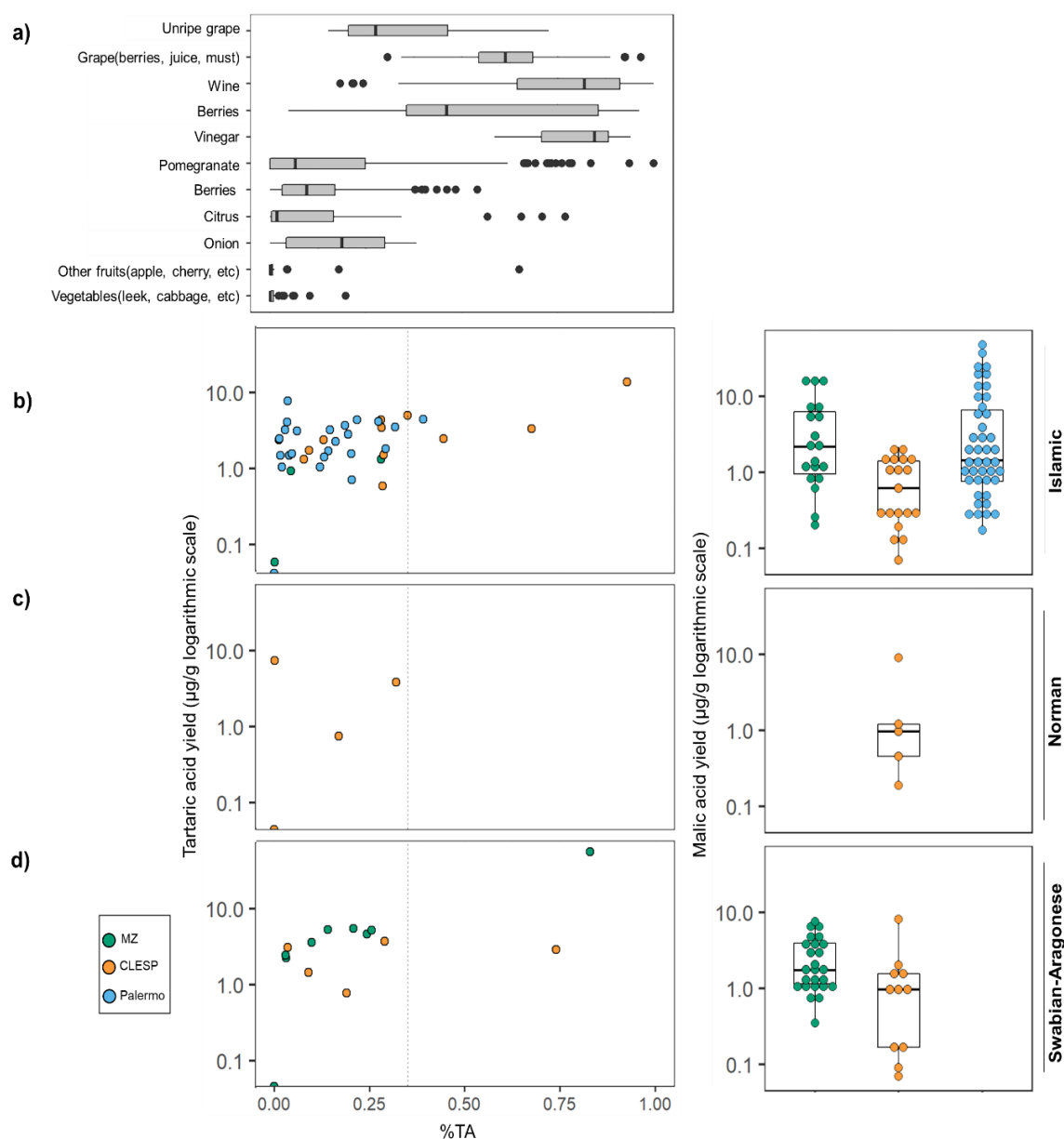


Figure 41: Tartaric yield plotted against %TA and malic acid yield of samples from MZ, CLESP and Palermo. a) Proportions of tartaric acid in various modern fruits and plant products (Lundy et al. 2021; Drieu et al. 2021b); b) % TA (tartaric acid/(tartaric + malic acid)) plotted against tartaric acid yield ( $\mu\text{g g}^{-1}$ ) for MZ, CLESP and Palermo across chronological periods and c) Malic acid yield ( $\mu\text{g g}^{-1}$ ) for samples from MZ, CLESP and Palermo across chronological periods.

## 6.4.6 Plant resins

One sample from MZ (MZ\_28) yielded a series of terpenoid compounds that may indicate the presence of birch-bark tar. The presence of pentacyclic terpenoid lupeol is characteristic of birch bark alongside the presence of betulin (O'Connell et al., 1988; Hayek et al., 1989; Hayek et al., 1990; Cole et al., 1991; Hua et al., 1991., Charters et al.,

1993 Aveling and Heron, 1998, Binder et al., 1990, Charters et al., 1993, Regert et al., 1998). Whilst the latter could not be identified, lup-2,20(29)-dien-28-ol and lup-2,20(29)-diene present in the sample indicate the heating of bark to produce tar, as these molecules result from the dehydration of betulin and lupeol respectively (Aveling and Heron, 1998, Regert, 2004). Other derivatives present included, naphthalene, lup-20(29)-en-3-one;  $\beta$ -amyrin, lupeol and lup-20(29)-en-3-ol (Regert, 2004). The archaeological record shows that birch-bark tar has been used as an adhesive since prehistory to the beginning of the Roman period, when it is believed there was a shift away from birch tar and pine products became the common source of resin (Pollard and Heron, 2008). However, in recent studies this has since been questioned, with the discovery of birch-bark tar in western Europe from the 1<sup>st</sup>-6<sup>th</sup> century A.D (Regert et al. 2019) and in early medieval contexts in England (Stacey et al. 2020). Although these findings only present evidence of birch-bark tar in one sample, this is the first evidence of birch-bark tar used in early medieval contexts outside of Britain. At present, the most recent attestation of birch pitch in Sicily dates back to the Bronze Age (Mentesana and Fragnoli 2020).

The presence of birch-bark tar so far south is surprising. It appears that over time, the use of this product, which was common in the Neolithic and Metal Ages in the Mediterranean (Regert et al. 2019; Rageot et al. 2019a), moved towards northern and eastern Europe in late Antiquity and the Middle Ages (Regert et al. 2019; Stacey et al. 2020). Birch-bark tar has been identified in archaeological ceramics for repairing as an adhesive sealing and as surface decoration in ceramics. Its presence here may also be attributed to the reuse of this cooking pot for the storage, production or transportation of birch-bark products (Urem-Kotsou et al. 2002; Charters et al. 1993a; Morandi, Porta and Ribechini 2018; Stacey et al. 2020; Rageot et al. 2019a; Robson et al. 2019). This sample also yielded evidence of ruminant adipose fats based on the  $\delta^{13}\text{C}$  values of palmitic and stearic acid and the presence of branched and linear chain  $\text{C}_{15:0}$  and  $\text{C}_{17:0}$  fatty acids (S3 data). Fatty products have been shown to modify the consistency or properties of birch-bark tar (e.g. fluidity, mechanical resistance of dry tar etc.) (Rageot et al. 2019a). Another consideration is that birch-bark biomarkers were introduced into the ceramic matrix from the fuel used to fire the vessel (Reber et al., 2019).

However, if this were the case, it is likely that evidence of birch-bark signals would exist in other ceramic vessels of the same production.

The presence of methyl dehydroabiatic acid identified in one 13<sup>th</sup> century sample from CLESP indicates the contribution of Pinaceae resin which has been heated with wood through the production of pitch resulting in methylation (Mills and White 1989; Hjulström, Isaksson and Hennius 2006). Pinaceae resin could have been used to seal the cooking pots to reduce porosity or was introduced into the pottery matrix from products stored in amphora, that may have been lined with resin (Pecci and Grassi-Munibe 2016; Buonincontri et al. 2017). However, due to the low concentration of methyl dehydroabiatic acid in this sample, it could have been introduced into the ceramic during firing and not from the deliberate use of pine (Reber et al. 2019b). Evidence of pine products in Islamic samples from Palermo were frequent (Chapter 5)(Lundy et al. 2021), but was not observed in any of the other samples studied here.

#### **6.4.7 Biomolecular evidence of heating and further resource identification**

Several samples from both MZ and CLESP yielded molecular thermal degradation markers. This included the presence of *n*-ketones C<sub>31</sub>, C<sub>33</sub> and C<sub>35</sub>, which can be formed through the heating process of animal fats at temperatures above 300 °C (Raven et al. 1997).  $\omega$ -(*o*-alkylphenyl) alkanolic acids (APAAs) were also detected in several samples. APAAs do not occur naturally but are formed through the heating of mono and polyunsaturated fatty acids (Hansel et al. 2004; Evershed, Copley and Dickson 2008; Bondetti et al. 2021). APAA C<sub>16</sub> and APAA C<sub>18</sub> can be formed through the heating of a number of different commodities: terrestrial animal fats, plant and starchy products and aquatic products. However, APAA C<sub>20</sub> and APAA C<sub>22</sub> are only formed through the heating of aquatic products and therefore are diagnostic for identifying aquatic products in ceramics. Only APAA C<sub>16</sub> and APAA C<sub>18</sub> were identified in samples from MZ and CLESP and no evidence of APAA C<sub>20</sub> and APAA C<sub>22</sub> was observed. Bondetti et al., (2021) suggest that it may be possible to distinguish between products by assessing the relative contribution of isomers in APAA C<sub>18</sub>, in particular the E and H



isomers (Bondetti et al. 2021). Figure 42 shows the APAA C<sub>18</sub> E/H isomer ratios of ceramic samples from Islamic contexts from MZ, CLESP and Palermo, Norman contexts from CLESP and Swabian contexts from MZ and CLESP. These are compared to APAA C<sub>18</sub> E/H isomers ratios of modern products as reported by Bondetti et al., (2021).

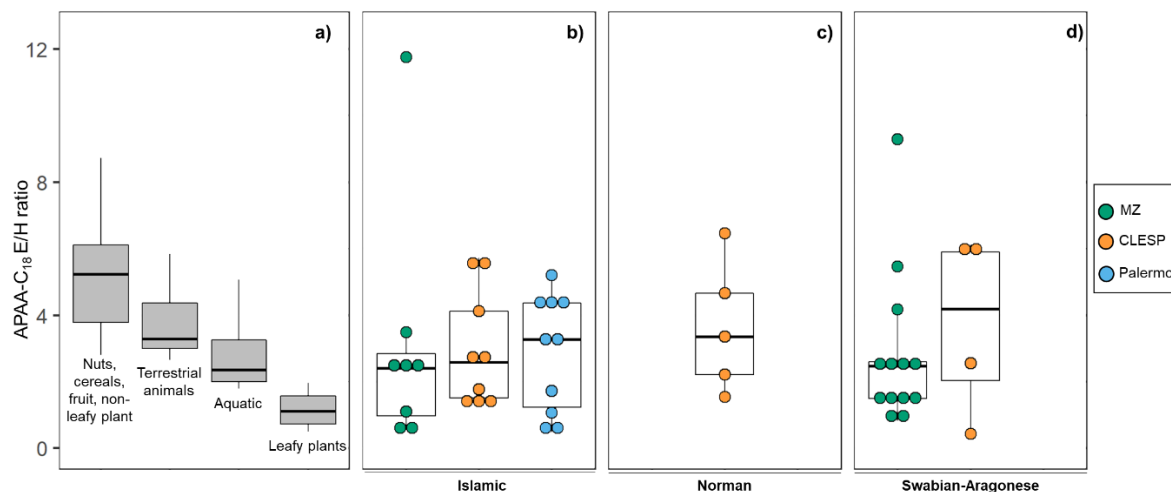


Figure 42: Box plot of APAA-18 E/H isomer ratios. a) APAA C<sub>18</sub> E/H isomers of modern products adapted from (Bondetti et al. 2021). b) APAA C<sub>18</sub> E/H isomers identified in domestic containers from Islamic contexts c) Norman contexts and d) Swabian-Aragonese contexts.

Overall, considering the mean APAA ratio of E/H APAA isomers, no statistical difference was found between sites or chronological periods. This was tested using the Kruskal test which gave a p-value of 0.7173 (Kruskal-Wallis chi-squared = 0.13112, df = 1, p-value = 0.7173). For the majority of samples, it was not possible to distinguish between terrestrial animal fats, plant and starchy products and aquatic products based on the APAA ratio of E/H APAA isomers, and no clear relationship was observed between the APPA E/H ratio and the lipid profiles of the extracts. It is important to note that the extracts obtained in this study show extensive mixtures and little is known about the effect of mixing different products on these distributions (Bondetti et al. 2021). Two specific outliers from MZ may be attributed to heating of nuts, cereals, fruits, or non-leafy plants as the E/H isomer ratio was >9 (9.28 and 11.75). Evidence of a fruit contribution was found in the former of the two samples and both samples yielded  $\delta^{13}\text{C}$  values that fall within the range of non-ruminant fats, which alongside non-ruminant adipose fats could also indicate plant or nut oils. However, the oleic to stearic acid ratio ( $C_{18:1}/C_{18:0}$ ) were close to 1 in both cases and no other clear plant biomarkers could be identified except malic acid in one sample (S3 Data). The majority of samples were run on the GC-MS using a SIM mode to look for fragment ions of

alkylresorcinols from cereals (wheat, barley or rye) (Appendix A), but no molecular evidence of cereals was found in any of the samples. However, as they are minor constituents and highly susceptible to degradation, the absence of alkylresorcinols cannot be used to exclude the use of cereals in these vessels (Ross et al. 2003; Hammann and Cramp 2018; Colonese et al. 2017a).

#### **6.4.8 Comparison of vessel use between sites and periods**

Due to the variation in vessel types between the two sites and between periods, they are difficult to directly compare. Furthermore, it is not possible to make definite conclusions about the uses of particular vessel types due to the small number of samples within each category. However, a number of observations could be made about the use of these vessels. In both MZ and CLESP, samples in all chronological periods, *ollae* and cooking pots of all fabrics and forms, frequently show evidence of mixing of animal products, plant products and fruits. Alongside molecular markers of heating it is possible to suggest these were general cooking wares perhaps used to make stews or pottages. This was previously observed in Islamic ceramics, reflecting Islamic-Arabic cuisines which often involve meat as a base, complemented by vegetables and fruits (Chapter 5)(Lundy et al. 2021). The evidence here suggests that these larger, closed vessels continued to have a similar use in the Norman and Swabian periods. It should be noted that in the absence of food crusts it is not possible to confirm whether these mixtures are processed together as part of a single recipe/cooking event or whether this represents the build-up of residues over time over multiple cooking events (Miller et al. 2020), although it is likely we are seeing both of these situations. Interestingly, despite the introduction of glazed wares in the Swabian periods, mostly imported from the east of the island from Messina, there does not appear to be a distinct difference in the ways the pots were used. This result perhaps lends to the idea that these were simply stylistic changes as opposed to functional changes.

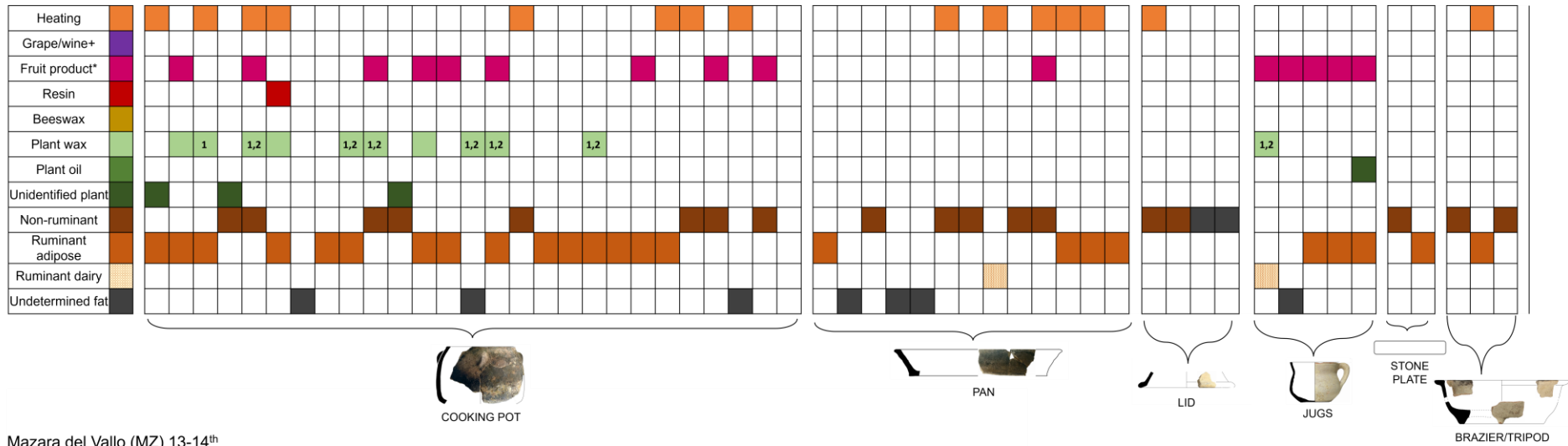
Flatter, more open vessel samples such as the stone plates and pans at both sites show lesser levels of mixing and seem to be predominantly used for processing animal products, as only a small number of these types yielded evidence of fruit or plant

products, perhaps used for frying meat on its own. Interestingly, one pan sample from Islamic contexts at MZ and one from Swabian contexts yielded evidence of dairy products. The use of dairy in pans contrasts to CLESP, where dairy products are solely processed in closed forms such as *ollae* and cooking pots. This highlights a differential use of dairy products between the two sites. The braziers sampled from MZ show evidence of animal fats, which likely reflects the dripping of fats and oils when meat was being prepared in a vessel placed above (Meo and Orecchioni, 2020). This is similarly observed in Islamic samples from CLESP and Palermo (Lundy et al. 2021). Of note, a selection of jugs from MZ showed evidence of animal fats, plants and fruit products together in the same vessel. This may suggest their use for small individual dishes or sauces being served, and perhaps were placed directly on the fire as evidence of soot was identified on the base and side of these samples (Meo and Orecchioni, 2020). One 13<sup>th</sup> century jug from MZ yielded clear evidence of dairy products alongside fruit and plant products, perhaps suggesting a dairy-based sauce or milk was prepared and flavoured with other products. These results show that these containers had a more complex use than the milk jug hypothesis formulated for the North African and Iberian Islamic contexts (Rosslter et al., 2012 p.259). A mixture of plant, fruit and potentially milk products has also been observed in 13<sup>th</sup> century jugs from Piombino, central Italy (Salvini, Pecci and Giorgi 2008).

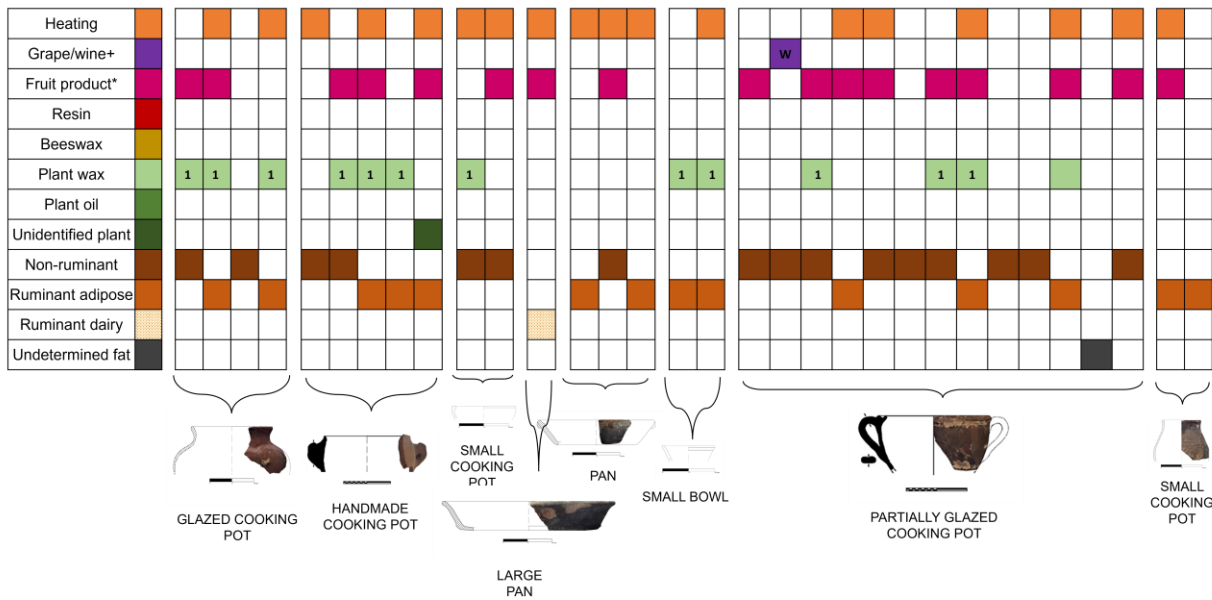
The use of dairy at these sites is interesting. In both Islamic and Norman ceramics from CLESP, the majority of pots that contain dairy, sometimes heated, show no evidence of other organic residues and as mentioned, are only found in *olla* and cooking pots. This suggests that dairy was likely being processed on its own, possibly into cheese or ghee in these ceramics as part of the rural economy, as previously observed in vessels from Islamic contexts at CLESP (Lundy et al. 2021). On the other hand, where dairy is identified in ceramics from MZ, evidence of other organic material is also found, such as plant products and fruit products. This may represent the multiple uses of these ceramic vessels for different products, but also lends to the idea that these ceramics were being used for recipes containing dairy, and not solely for the processing of dairy products, as observed at CLESP. Furthermore, dairy products at MZ were present in pans and a jug, but not in *olla* and cooking pots. This provides interesting insight into

the differential use of these products and the containers used to process/incorporate them within cuisines between the two sites.

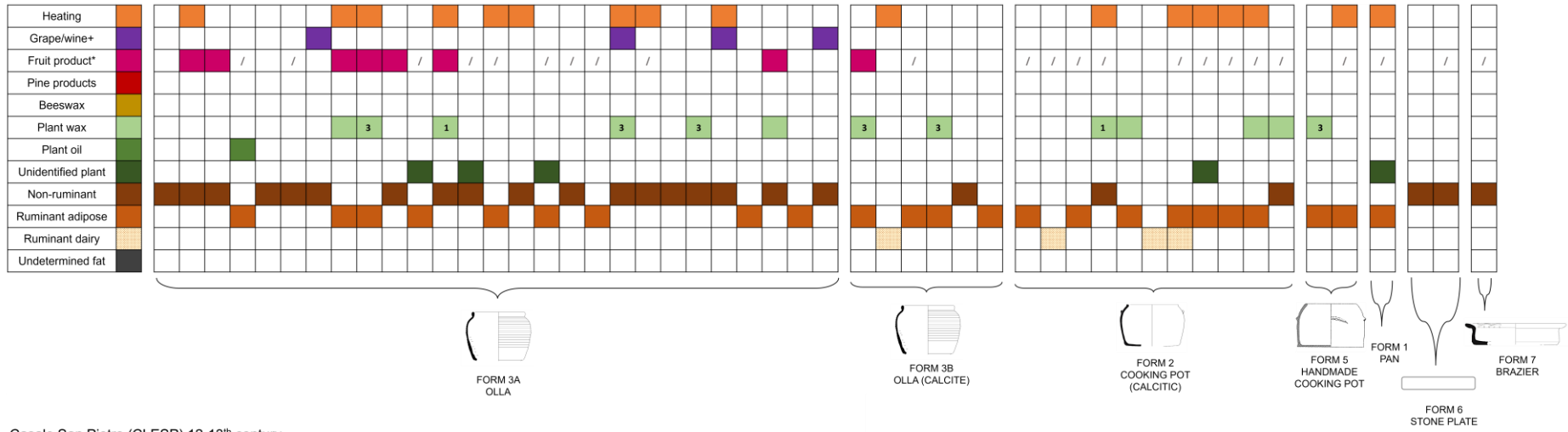
a) Mazara del Vallo (MZ) 10<sup>th</sup>-11<sup>th</sup>



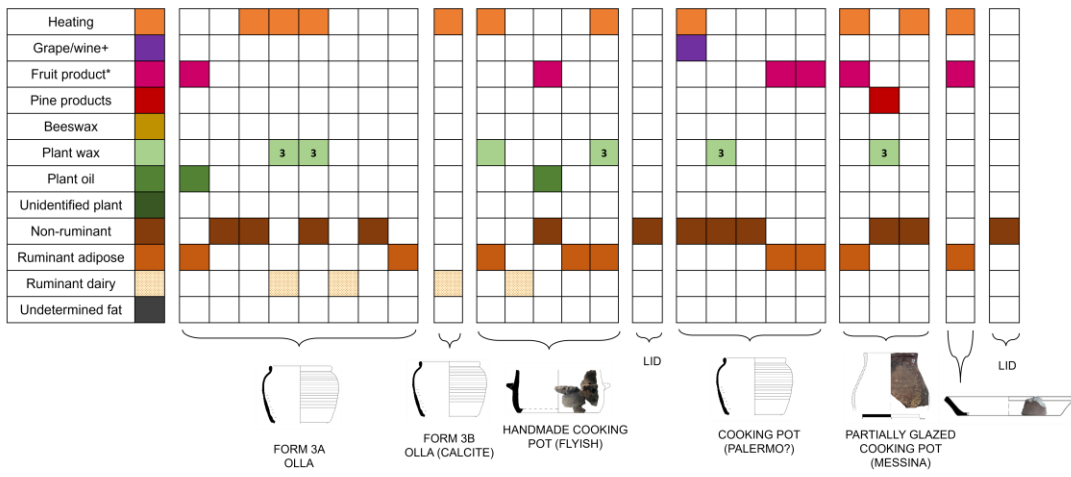
b) Mazara del Vallo (MZ) 13-14<sup>th</sup>



c) Casale San Pietro (CLESP) 10<sup>th</sup> – 11<sup>th</sup>



d) Casale San Pietro (CLESP) 12-13<sup>th</sup> century



12<sup>TH</sup> CENTURY

LATE 12<sup>TH</sup> – 13<sup>TH</sup> CENTURY

Figure 43: Summary figure of organic substances identified in pottery vessels. a) MZ 10<sup>th</sup> -11<sup>th</sup> century ceramics b) MZ 13<sup>th</sup> -14<sup>th</sup> century ceramics c) CLESP 10<sup>th</sup> -11<sup>th</sup> century d) CLESP 12<sup>th</sup>-13<sup>th</sup>. Identification criteria of different commodities (ruminant dairy, ruminant adipose, non-ruminant, unidentified plant, plant oils, plant wax, beeswax, pine products, fruit products and grape products) are outlined in the text. More specific taxonomy of plant waxes is indicated Brassica (1), leek (2) and fennel (3)

## 6.5 Conclusions

For the first time, this study has investigated cuisine across the transition from Islamic political control to post Islamic regimes of the Norman, Swabians and Aragonese periods in Sicily. Despite significant transformations in political power over the island, overall little change was observed in the commodities processed in pottery vessels through time periods. A substantial mixing of commodities such as animal fats, plant and fruit products was identified regardless of chronological period and geographical area, and although it is important to note that it is difficult to distinguish between a single cooking event and subsequent processing of different commodities, it is not unlikely that both scenarios would have existed.

This research shows a general continuation of this culinary tradition and recipes from the Islamic period through to the Aragonese period, as similar results were found in the analysis of cooking pots from Islamic Palermo and CLESP (Lundy et al. 2021). A continuation of culinary habits in post-Islamic Sicily may reflect the multi-cultural society that existed in Sicily throughout the 9<sup>th</sup> to at least the 12<sup>th</sup> century. This lends to the idea that the Normans flourished and profited from their predecessors, and it is without doubt that they benefited from the agricultural systems, resources and recipes introduced by the Arabs. However, some differences were observed in the general use of different wares such as greater mixing of products in open forms such as *olla* and cooking pots from all sites and chronological periods in comparison to more restrictive uses of pans and stone plates. Additionally, a decrease in the variety of plant products processed in ceramic vessels from later periods compared to those from Islamic contexts could indicate differences in the availability/selection of plant products between these periods. It is important to note, however, that organic residue analyses do not allow for very detailed taxonomic identification, and that changes may have occurred in the types of ruminants or fruits exploited, for example, without this being perceptible by molecular and isotopic analyses. The use of dairy in rural CLESP and the

urban sites of MZ and Palermo remain distinct and represent the use of ceramic vessels for the production of dairy products at the rural site, as opposed to their use as part of recipes in MZ and their absence from ceramics from Palermo. Furthermore, the presence of birch-bark tar in one cooking vessel from MZ has provided the first evidence of birch-bark products in medieval Sicily. These findings support new research that birch-bark tar had a continued use after the Roman period, despite the introduction of pine resins.

The application of a multifaceted organic residue approach to a large number of ceramics was essential here to identify a wide range of products and begin to unravel complex mixtures in the domestic cooking pots. Only by applying these methods to a large corpus of ceramics from multiple sites in medieval Sicily of both rural and urban setting has it been possible to gain insight into the culinary habits of these populations. Future studies investigating cuisine should use the approach here as a baseline for identifying a wide range of products and complex mixtures, particularly in medieval contexts.



# **Chapter 7 Discussion: reconciling organic residue analysis of cooking pots with other archaeological evidence.**

## **7.1 Introduction**

This thesis aimed to identify and understand cuisines and the impact of socio-cultural and political influences on the populations in Sicily during the 9<sup>th</sup>-14<sup>th</sup> centuries AD. This study used a multifaceted organic residue analysis approach to identify commodities in cooking vessels and other domestic containers to **1)** understand what foods were selected, combined and processed in ceramics vessels and **2)** how the contents of these ceramics compare in urban and rural socio-economic settings and between Islamic, Norman, Swabian and Aragonese contexts. Through a large-scale, multifaceted organic residue approach, a range of commodities were identified in the ceramic vessels including animal fats, vegetable products, fruit products, beeswax, and plant resins. In many cases a mixture of commodities were observed, reflecting either sequential cooking events and/or the complex mixtures of medieval recipes or likely a combination of the two (chapter 5 and 6). Both a spatial and temporal approach enabled insight into culinary habits of populations from different socio-economic environments (urban and rural) and diachronically (9<sup>th</sup>-14<sup>th</sup> centuries). Organic residue analysis has provided direct chemical evidence of the contents of the ceramic vessels and enabled insight into what foods were combined and prepared. However, foodways can be seen as complex systems which involve a number of different processes such as the production, procurement, processing, consumption and disposal of foods (Woolgar 2010; Twiss 2012; Hastorf 2016). Each of these processes are reflected differently in the archaeological record (Figure 44). Ceramics form only a small fraction of the material culture involved in cooking, but many other ways of processing foods exist (e.g., ovens, spit-roasting, open fires, barrels, skins and certain foods eaten raw (Lévi-Strauss 1966, 587). Therefore, it is important to consider other sources of evidence of foodways in the archaeological and historical record. By understanding what foods were available to the general population, it is possible to

gain deeper insight into what and how foods were specifically selected for processing in ceramic vessels.

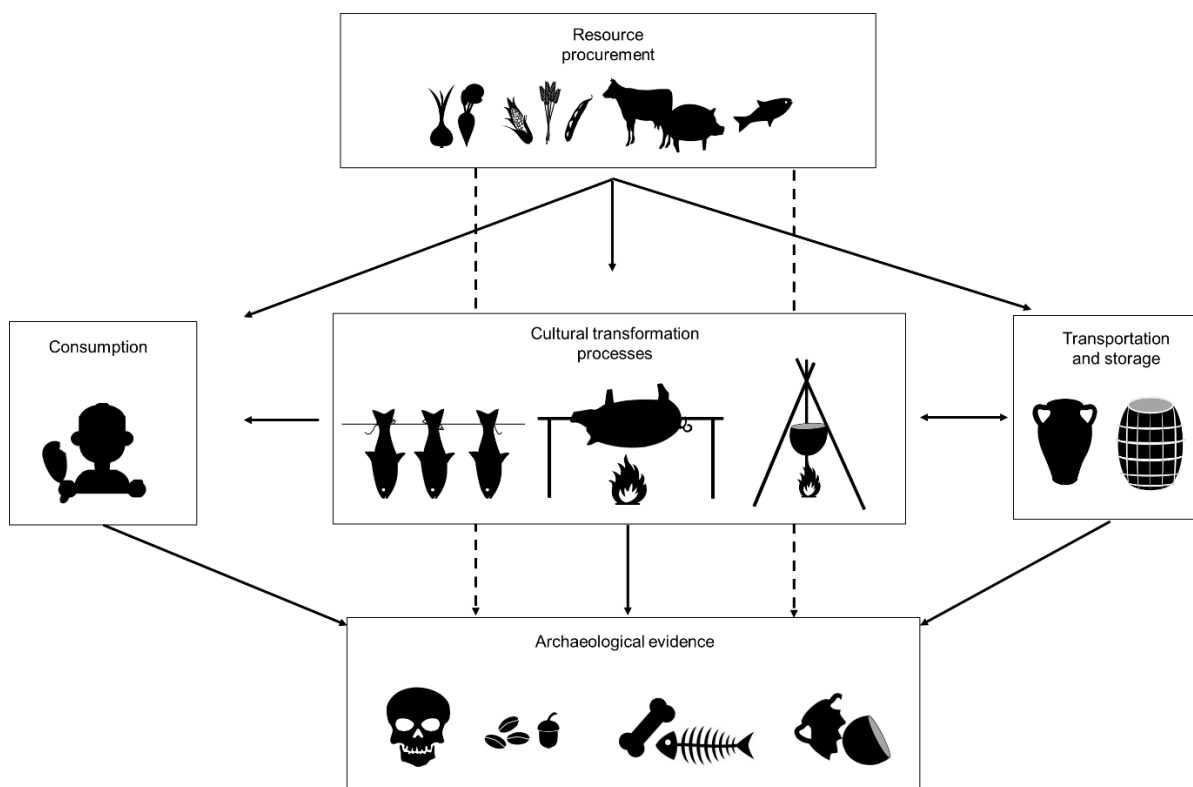


Figure 44: Schematic diagram representing the processes involved in food selection, preparation, consumption and storage and evidence represented in the archaeological record (from left to right: human remains for stable isotope analysis, archaeobotanical remains, faunal remains and ceramics). This figure was based on themes of foodways discussed by (Twiss 2012; Hastorf 2016; Woolgar 2010)

## 7.2 Sources of evidence overview

At the sites studied in this investigation, a number of other sources of food evidence were investigated within the scope of the SICTRANSIT project (faunal remains, archaeobotanical remains, human remains and transport and storage containers) (Figure 45). Although some of this evidence (faunal, archaeobotanical and textual sources) has been utilised to give context to the ORA results in previous chapters (chapters 5 and 6), in this chapter they are used in combination with the results of ORA to gain further insight into what foods were available, how they were selected and used in the ceramic containers and how they were consumed; thus, enabling a more robust

interpretation of the results presented in this thesis and to aid in our understanding of foodways as a whole in Sicily at this time. Here, each line of evidence and their contribution to food studies is outlined in the following section.

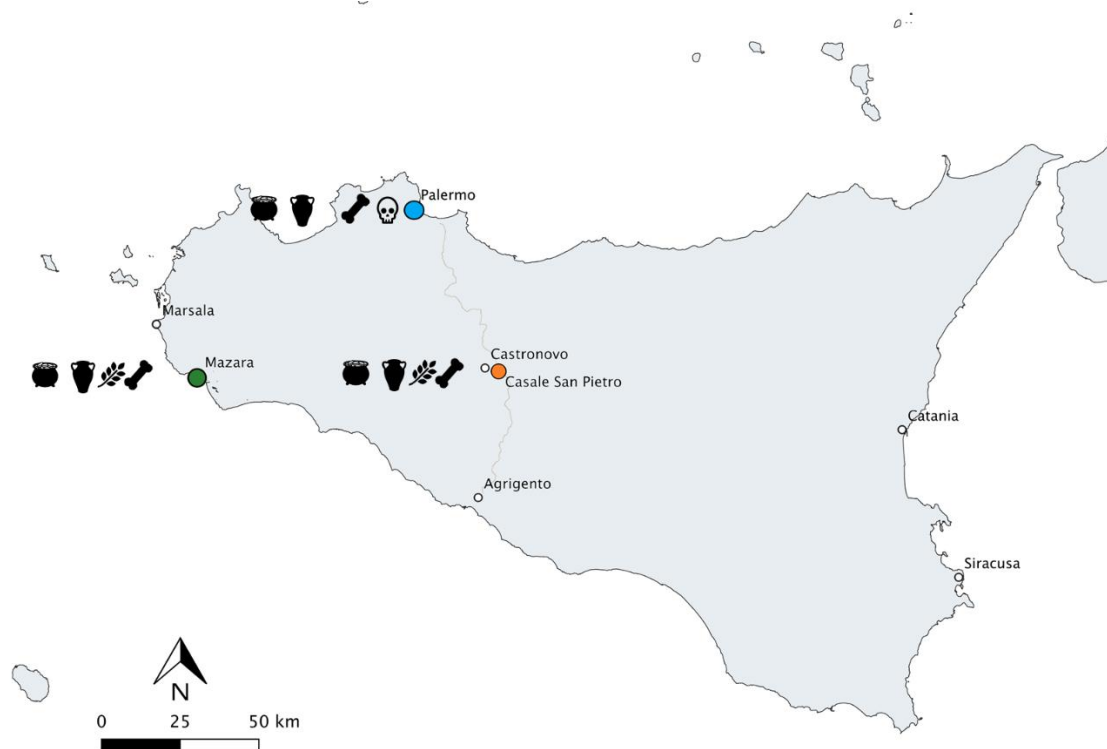


Figure 45: Map of Sicily showing the sites of Palermo (PB), Casale San Pietro (CLESP) and Mazara del Vallo (MZ) and the archaeological evidence of food found at these sites as part of the Sicily in Transition project (Carver, Fiorentino and Molinari 2019). Human remains (☠), faunal remains (🦴), archaeobotanical remains (🌿) and cooking pots (🍲) storage and transport vessels (🏺).

## 7.2.1 Faunal remains

The analysis of animal remains in the archaeological record provides important information about the presence and uses of animal species in the past (O'Connor 2018; Reitz and Wing 2008). Through the identification of taxon and taxonomic frequencies, age patterns, size biometrics, and processing marks (i.e. butchery, cut marks and charring) zooarchaeological studies can yield insight not only into what animals were present on site, but how they were managed and used, for both culinary (e.g. meat and milk) and non-culinary purposes (e.g. for traction, leather, wool or as pets) (O'Connor 2018; Reitz and Wing 2008; Davis 1997). In terms of assessing diet through animal remains, zooarchaeological analyses have investigated a variety of archaeological

questions such as use and preparation of animal products between religious (Morales-Muñiz et al. 2011; Valenzuela-Lamas et al. 2014; Pluskowski 2010) and socio-economic groups (Grau-Sologestoa 2017; Grau-Sologestoa, Albarella and Quirós Castillo 2016; Baker 1996). However, taphonomic processes and sampling biases can impede our interpretation of faunal remains (Lyman 1987; Payne 1972) and although in some cases faunal analysis can indicate the uses of animal products for food purposes, it is not a direct evidence of diet. For all of the five sites investigated in this thesis, a number of species were identified in the faunal record including the main domesticates caprine (both sheep and goats), cattle and domestic fowl. Fish remains and other animals such as dogs, horses, hare and deer have also been identified (Arcoleo and Sineo 2014; Arcoleo 2015; Aniceti 2020). The presence or absence of faunal remains in the archaeological record can, to some extent, inform us about how the meat or secondary products from these animals were processed through culling patterns, butchery, and charring marks. However, they do not provide direct evidence of diet and some limitations exist. Here an assessment of what species were available on site are compared to evidence of animal fats in the ceramics analysed to understand which animals and their products were selected and processed in these vessels.

### **7.2.2 Botanical remains**

When preserved, botanical remains can be identified to species and taxon specific levels, providing important information about the types and uses of plant products available (Bakels and Jacomet 2003; Cappers 2006; Ernst and Jacomet 2006; Hastorf 1999; Livarda 2011; Van der Veen 2018). Quite often, a large proportion of archaeobotanical assemblages contain the inedible and discarded parts of plants that have been disposed of, such as chaff, leaves, twigs and weeds etc. (Munson et al., 1971). Carbonised remains, which are more likely to survive in the archaeological record than unaltered products, can provide evidence for the processing of these food products, but 'non-dense' foods such as leafy greens and pulpy fruits that are boiled or eaten fresh, for example, are less likely to be preserved (Miksicek 1987). Additionally, distinguishing between plant products consumed by humans and those consumed by animals (animal fodder) can be difficult (Valamoti and Charles 2005). Therefore, it is

important to note that presence/ absence of archaeobotanical remains are not direct evidence of what was being consumed. Nonetheless, analysis of archaeobotanical remains can offer significant insights into the availability of products and offer a proximal view of human consumption and the dates at which new taxa became available in Sicily. As part of the Sicily in Transition project, archaeobotanical remains were collected and analysed at the site of Casale San Pietro and Mazara del Vallo (Fiorentino et al. forthcoming; Primavera 2018b; Fiorentino et al. forthcoming). The archaeobotanical remains at CLESP and MZ, both carbonised and mineralised, were well preserved and therefore provides an idiosyncratic opportunity to analyse plant remains, that otherwise may not have survived. A number of different commodities, both edible and non-edible were identified. In particular, the remains at Mazara present a unique case as samples were recovered from latrines in several phases of the city and therefore can be assumed to represent the waste of the populations that were living and consuming these foods (Primavera 2018b; Fiorentino et al. forthcoming). In combination with ORA of ceramics archaeobotanical evidence can aid our understanding of what resources were available and how they may have been selected, combined and prepared in ceramic vessels.

### **7.2.3 Carbon and nitrogen stable isotope analysis of human and animal remains**

Carbon ( $\delta^{13}\text{C}$ ) and nitrogen ( $\delta^{15}\text{N}$ ) stable isotope analysis of human remains can provide direct evidence of diet, as the stable isotope composition of bone collagen reflects the isotopic composition of the foodstuffs consumed over an individual's lifetime (dependant on the tissue used) (Schoeninger 2010; Hedges et al. 2007; Ambrose and Norr 1993). In brief,  $\delta^{13}\text{C}$  are used to identify the consumption of terrestrial resources and can distinguish between the intake of  $\text{C}_3$  and  $\text{C}_4$  plants in the diet, due to the differential fractionation of atmospheric  $\text{CO}_2$  between these plant types as outlined in Chapter 3 of this thesis (Ambrose and Norr 1993; Smith and Epstein 1971).  $\delta^{15}\text{N}$  isotopes are generally used to distinguish marine and terrestrial diets as the nitrogen isotope values increase with  $\delta^{15}\text{N}$  enrichment in an organism dependent on their position in the food chain (trophic level) (Deniro and Epstein 1981). Due to

differential sources of CO<sub>2</sub> in comparison to their terrestrial counterparts, marine foods also tend to have higher  $\delta^{13}\text{C}$  values (Schoeninger and DeNiro 1984). Stable isotope analysis of human remains can give a direct assessment of an individual's diet and comparison of individuals' diets in a population can yield important information regarding social structures within a community, including social organisation, faith and gender (Lee-Thorp 2008). Additionally, stable isotope analysis of animal remains not only provides an essential  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  local baseline needed to understand values obtained from humans, but animal isotopic values, through evidence of direct diet, can inform about animal husbandry practices, ecological and environmental differences (Casey and Post 2011; Goude and Fontugne 2016). However, it is important to emphasise that stable isotopes only provide a partial record of food consumption within a population and can be limited by the availability and selectivity of the burial record. The integration of human and animal stable isotope analysis and ORA of ceramics containers offer a complementary insight into the processing and consumption of foods in a population (Dunne et al. 2019; Craig-Atkins et al. 2020b; Buonincontri et al. 2017; Olsson and Isaksson 2008; Ganzarolli et al. 2018). There are few contexts within Sicily in Transition project where both ceramics and human bone were recovered. However, at sites in Palermo both ceramics and human remains have been analysed as part of on-going research undertaken by Alice Ughi as part of the Sicily in Transition project and therefore can be compared here (Ughi and Alexander forthcoming; Ughi unpublished).

#### **7.2.4 Storage and transport containers**

Often food products undergo some element of preparation or treatment before they are stored. The storage of foods is a stage that falls between the procurement, processing and utilisation of resources, either to be consumed or distributed (Curtis 2015, 174). The analysis of the contents of storage containers, therefore, can provide important information about the availability and treatment of food resources. The understanding of the contents of transport containers can reflect either processed or non-processed foods that have either been imported to the site for use or consumption by the local population or are being exported and represent economically significant

commodities. Evidence of the contents of these vessels sometimes exist as visible preserved contents (i.e., grain storage) or as inscriptions directly labelling the contents of the vessels. However, in the absence of visible contents or inscriptions, ORA residues can provide direct chemical evidence of the contents of these vessels, providing important information about commodities stored locally, imported or exported. As part of the Sicily in Transition project a number of transport amphorae were analysed using ORA from Palermo, Mazara and Casale San Pietro, alongside examples of amphorae probably reused for storage purposes at the site of Casale San Pietro (Drieu et al. 2021b; Drieu, Carver and Craig 2018; Drieu et al. forthcoming; Drieu forthcoming)

### **7.3 The use of animal products in medieval Sicilian cuisine**

In all sites and chronological periods, terrestrial animal products were the most abundant source of residues identified in the ceramics analysed in this study. Through the analysis of lipid profiles, animal fats were identified by the presence of fatty acids, whereby C<sub>16:0</sub> and C<sub>18:0</sub> were, in most cases, present in equal proportions. Triacylglycerol (TAG) profiles were consistent with animal fats, and in many cases, cholesterol was also identified in the samples, further confirming the contribution of animal products (Regert 2011)(Chapter 5 and 6). The  $\delta^{13}\text{C}$  values of fatty acids ( $\delta^{13}\text{C}_{\text{FA}}$  values) C<sub>16:0</sub> and C<sub>18:0</sub> were used to further determine the animal sources by distinguishing between ruminant adipose and ruminant dairy sources and non-ruminant sources including non-ruminant adipose fats as well as plant products, based on modern reference  $\delta^{13}\text{C}$  values (Copley et al., 2005; Craig et al., 2011; Dudd and Evershed, 1998; Cubas et al., 2020). There are several difficulties with identifying the origin of animal fat residues in the ceramics. Firstly, although ruminant and non-ruminant fats display different  $\delta^{13}\text{C}_{\text{FA}}$  values, it was not possible to further distinguish between different ruminant (e.g., cattle, sheep, and goat etc.) and non-ruminant species (e.g., between porcine, chicken, rabbit, and hare etc.). Secondly, plant products may make a significant contribution to the  $\delta^{13}\text{C}_{\text{FA}}$  values, such as plant oils, cereals, and nuts and in many cases the biomolecular profiles of the ceramic samples studied depicted a high occurrence of mixing of both animal products and plant products. These complex mixtures likely impacted the  $\delta^{13}\text{C}_{\text{FA}}$  values of the residues in the ceramics.

Furthermore, mixtures of different adipose fats (non-ruminant and ruminant products) will also impact the  $\delta^{13}\text{C}_{\text{FA}}$  values, making it difficult to interpret their origin or relative contribution.

To further understand the availability of animal products and how they were selected for processing in the ceramics analysed in this study, the assigned animal fat sources based on  $\delta^{13}\text{C}_{\text{FA}}$  values (non-ruminant, ruminant adipose and ruminant dairy) were compared to the evidence of the main domesticated species likely utilised for culinary purposes from the faunal assembles at these sites previously investigated in other works (Aniceti 2020; Arcoleo and Sineo 2014; Arcoleo 2015). Although the number of identified species (NISP) aids our understanding of the types of products available, this is not directly comparable to the lipid contents observed in the ceramic vessels. Therefore, NISP values of the main domesticates at each site (cattle, caprines, pigs and chicken) were converted to estimated meat weights by multiplying the total NISP of each species by assumed meat weight contributions of each species (kg.) Meat weight values (100kg for cattle, 15kg for caprines, 35kg for pigs and 0.7kg for chickens) were estimated based on medieval meat weight values reported in the literature (Boessneck et al. 1971; O'Connor 1991; Dyer 1989, 59) (Appendix K) (Figure 46). Estimated meat weight values assume that all animals present at the site were utilised for their meat and dismisses contributions of animal fats within the ceramics (e.g., bone marrow, dairy products etc.,) and other usable, but usually assumed to be inedible, tissues (e.g., for traction and leather) (Bowen 1992, 233–234). Thus, meat weight estimates provide a crude, but important assessment of the dietary contribution of animal species.



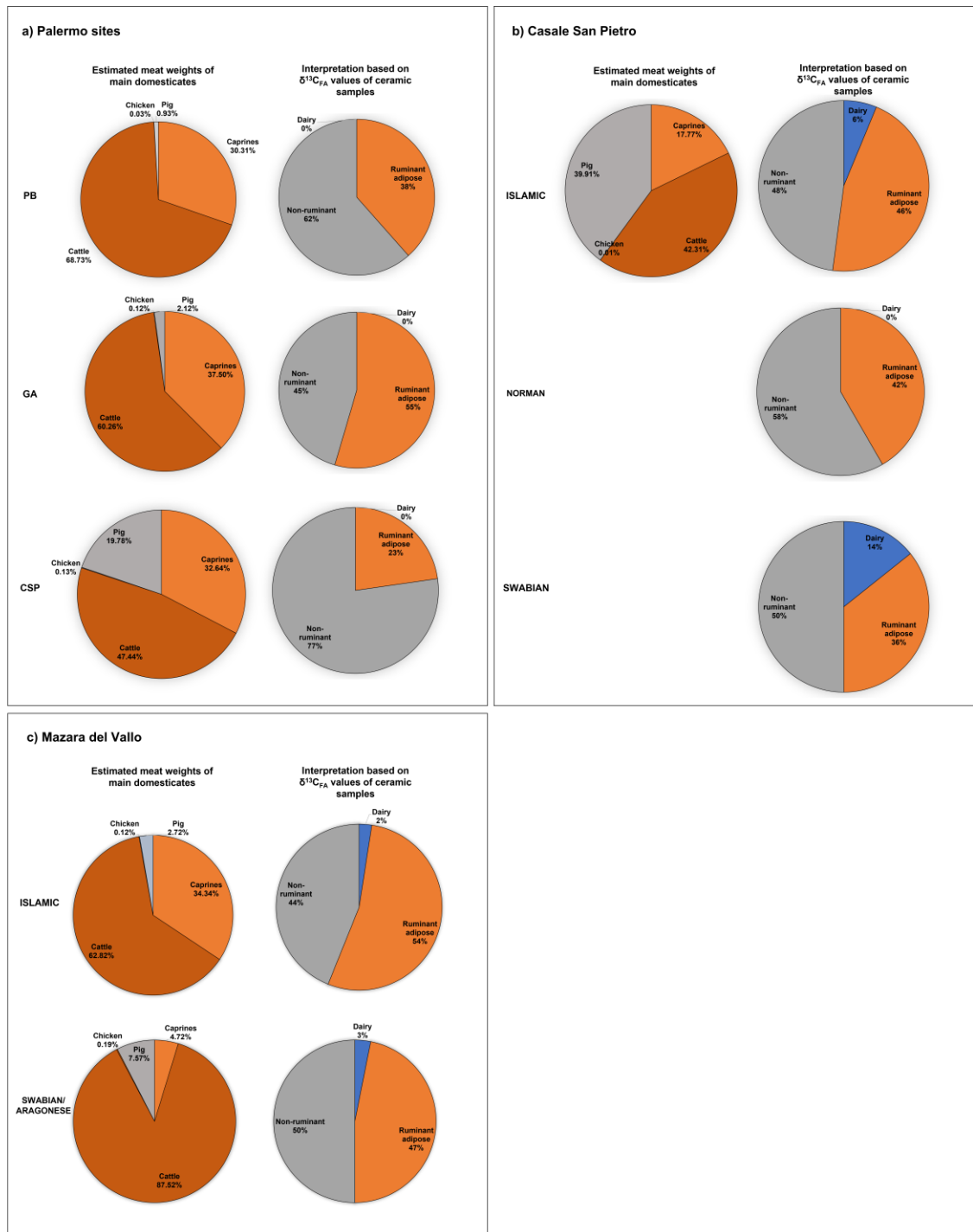


Figure 46: Pie graphs showing the estimated meat weight contribution of main domesticates (%) and non-ruminant (NR), ruminant (R) and dairy (D) interpretation based on  $\delta^{13}C_{FA}$  values of ceramic vessels from a) the three sites Palazzo Bonagia (PB), La Gancia (GA) and Castello San Pietro (CSP) in Palermo, Islamic; b) Norman and Swabian contexts in Casale San Pietro (CLESP) and c) Islamic and Swabian contexts in Mazara del Vallo (MZ).

Ruminant species, cattle and caprines, are both present in the faunal assemblages at all sites in high proportions (Appendix K). At all sites and chronological periods, caprines are the most dominant species based on NISP values. However, when crudely converted to meat weight, cattle appear to be the dominant species (Figure 46).

Together, the meat weight estimations of both cattle and caprines contribute >80% of the total meat weight in all Palermo sites and Mazara, and ~60% at Casale San Pietro. A number of  $\delta^{13}\text{C}_{\text{FA}}$  values obtained from ceramic samples from all sites and chronological periods fell within the range of modern ruminant adipose fat. At Palermo (38% of samples from PB, 55% of samples from GA, 23% of samples from CSP), Casale San Pietro (46%, 36% and 42% in Islamic, Norman contexts and Swabian contexts respectively) and at Mazara del Vallo (54% and 47% in Islamic and Swabian/Aragonese contexts, respectively) (Figure 46). Therefore, at all sites, ruminant adipose fats are underrepresented in the ceramic vessels compared to the assumed meat weight contributions of these species. The underrepresentation of ruminant adipose fats may suggest that only a proportion of ruminant carcasses were selected for processing in the ceramic vessels and other methods of cooking were used to process these products such as roasting, cooking directly on fire, or other cooking containers not analysed in this study. Evidence of charring identified on remains of cattle and caprines at Casale San Pietro and Mazara del Vallo for example, may support evidence of cooking carcasses directly on fire, although other indirect causes of charring on these remains cannot be dismissed (Aniceti 2020).

The use of caprines and cattle for secondary products can further support the disparity in meat weight contributions and  $\delta^{13}\text{C}_{\text{FA}}$  values. An assessment of kill off patterns and evidence of butchery and cut marks at these sites shows that both cattle and caprines were utilised for meat as well as secondary products (e.g., milk, wool, traction, and leather) (Aniceti 2020; Arcoleo and Sineo 2014; Arcoleo 2015). Interestingly, at the site of Mazara, although butchery marks observed on some of the faunal remains represent food waste, the high percentage of cattle that survived into later adulthood indicates that meat and milk were of secondary importance in the animal management strategies of cattle and that the majority of cattle were likely utilised for traction in Islamic contexts (Aniceti 2020). This suggests that the majority of ruminant adipose products processed in the pottery vessels came from caprines, in which the analysis of kill-off patterns depicted a much more generalised use of sheep and goat products both for meat, as well as milk and wool. Thus, in Islamic contexts, if caprines are assumed to be the predominant contribution of meat, alongside a smaller contribution of cattle (beef) in the ceramic containers, the meat weight contributions of % ruminant  $\delta^{13}\text{C}_{\text{FA}}$  values

are better correlated. This supports evidence from the literature, in which lamb and mutton appear to be preferred meats ahead of beef in Islamic-Arabic cuisines (Lewicka 2011; Zaouali 2009; Perry 2005). Conversely, in Swabian/ Aragonese contexts at Mazara del Vallo management practices of caprines were highly focused on the production of secondary products, represented in the very low meat weight contribution of caprines in this period <5% (Aniceti 2020). This may indicate a higher importance of beef in the later periods at these sites, but it is not possible to distinguish the contributions of cattle and caprines in the ceramic containers based on the  $\delta^{13}\text{C}_{\text{FA}}$  values alone. Regardless of species, however, the presence of ruminant adipose fats in the ceramic containers at all sites and chronological periods provides important insight into the processing of these products. The occurrence of ruminant fats in *ollae* and cooking pots suggest that ruminant fats were boiled, evidence of which cannot be identified in the faunal record (Aniceti forthcoming).

Evidence of dairy in some ceramic vessels from both MZ (2 samples in Islamic contexts and 1 in Swabian contexts) and CLESP (4 in Islamic contexts and 4 in Norman contexts) provides further support for the exploitation of ruminant animals for secondary products in these contexts. Based on the faunal evidence at MZ in both Islamic and Swabian periods, it is possible to suggest that caprines were the predominant source of dairy at this site in both periods (Aniceti forthcoming, 2020). However, the intensified management of caprines for secondary products in the Swabian period, as observed in the faunal investigations, cannot be seen in the use of dairy in the ceramic vessels here. The clear evidence of dairy in ceramic vessels from CLESP suggests that this site had a key role in the processing of dairy products (chapters 5 and 6), which has also been observed in the rural site of West Cotton, Northamptonshire (Dudd and Evershed 1998; Dunne et al. 2019). The culling ages of cattle in Islamic phases at CLESP suggest that although they may have been utilised for dairy products, it is more likely that these products come from caprines instead (Aniceti 2020).

Further evidence of dairy products in the ceramics from these sites may be underrepresented based on  $\delta^{13}\text{C}_{\text{FA}}$  values as a result of mixing of other products (ruminant and non-ruminant adipose or plant products) (Hendy et al. 2018). Processed products such as cheese may also be undetected in ceramic vessels as they are not as

easily absorbed into the ceramic matrix in comparison to milk, for example. It has been suggested that the use of such processed products in the ceramic vessels might explain the lack of dairy residues in vessels from Palermo (Lundy et al. 2021) (Chapter 5). This has also been used to explain the near absence of dairy products at the urban Jewish site of St Aldates (Dunne et al. 2021). Here, researchers suggest that the near absence of dairy products in the ceramic vessels analysed could be because dairy products were processed in non-ceramic containers such as wooden bowls or already processed products such as cheese and butter were more readily used, of which these signals are less likely to be observed in the ceramics (Dunne et al. 2021).

In contrast to ruminant fats, evidence of non-ruminant products are overrepresented in the ceramics based on the  $\delta^{13}\text{C}$  values in comparison to the observed meat weight values of non-ruminant animals (pigs and chickens) at the sites of PB, GA, CSP and MZ (Figure 46). The low meat weight contribution of pigs (<2.5%) in Islamic context from PB, GA and MZ support the notion that pork was of little culinary value at these sites, as a result of restrictions of the consumption of pork in the Islamic faith. In contrast, the meat contribution of pigs is higher at the site of CSP (~19%) and in Swabian/Aragonese contexts from Mazara (~7%). The presence of pig remains at CSP indicates that although the cemetery at CSP was Islamic, the settlement that superseded it in the later 9<sup>th</sup> century might not have been occupied only by practicing Muslims. Carver highlights that Christians were in the area at this time and continued to be buried at the site of via Guardienne throughout the 7<sup>th</sup>-12<sup>th</sup> centuries (Carver forthcoming). Furthermore, the presence of pig remains and presumed consumption of pork at Mazara during the 13<sup>th</sup>- 14<sup>th</sup> centuries can be attributed to the presence of Christians during the Swabian regime (Aniceti 2020; Aniceti and Albarella 2022). The higher meat weight values of pigs at CSP and later MZ contexts, therefore, could explain the higher percentage of samples within the non-ruminant values (Figure 46), but no direct evidence of the processing of porcine fats could be identified in the ceramic containers based on the  $\delta^{13}\text{C}_{\text{FA}}$  values or TAG profiles alone (chapters 5 and 6).

At CLESP the evidence of non-ruminant products based on  $\delta^{13}\text{C}_{\text{FA}}$  values (44%) in Islamic contexts are better correlated to the % meat weight contributions of non-ruminant species (39% for pigs). Only a small number of samples ( $n=4$ ) could be

tentatively assigned to porcine contribution based on slightly enriched  $\delta^{13}\text{C}_{\text{FA}}$  values (Lundy et al. 2021)(Chapter 5). However, based on the high percentage of pig remains at CLESP, it has been concluded that pork was likely consumed at this site (Aniceti 2020), and it may be assumed that pork was processed in the ceramic containers.

Furthermore, non-ruminant  $\delta^{13}\text{C}_{\text{FA}}$  values could be attributed to the processing of chicken meat in these ceramic containers. Cut marks indicate that to some extent chickens were processed for food purposes at these sites, rather than for egg or feather production (Aniceti 2020) and chicken meat appears regularly in recipes of this time (Waines 2003; Lewicka 2011). However, the percentage meat weights of chicken at all sites and chronological periods were very low (<0.2%). Thus, in comparison to the other domesticates, chicken meat unlikely accounts for the high number of  $\delta^{13}\text{C}_{\text{FA}}$  values that fall within the range of non-ruminant fats. However, it must be noted that the culinary use of eggs, also accounted for in medieval recipes (Pitchon 2020), cannot be detected in the archaeological ceramics and low meat weight contributions of chicken does not reflect their relative importance for the production of eggs. Sampling bias and biases in taphonomic processes must also be considered. The fact that larger, denser bones survive better in the archaeological record than smaller more porous bones from younger specimens and smaller species such as birds and fish, could result in domestic fowl being underrepresented in the faunal record. Furthermore, the importance of chicken meat or eggs as a food commodity may be underrepresented in the faunal record, as they could have been bred and their products prepared away from site and brought in for consumption. For example, chicken markets for both meat and eggs were an important feature of medieval Islamic towns (Pitchon 2020). Figure 47 shows the 10<sup>th</sup> century chicken market in Palermo documented in *The book of curiosities*, a series of Islamic Maps compiled by an unknown author in the late 11<sup>th</sup> century (as discussed by Johns and Savage-Smith 2003) (Figure 47). Aniceti (2020), suggests that, at the site of Mazara del Vallo from the Islamic periods to later periods, an increase in the size of domestic fowl indicates a shift to a more intensified breeding of chickens, which would not be correctly accounted for in the estimated meat weights (Aniceti, 2020). This could indicate the higher importance of these species as a food resource in later periods, as observed in Norman England where consumption of chicken is said to have increased at the arrival of the Normans (Craig-Atkins et al.

2020a). However, this is difficult to infer from  $\delta^{13}\text{C}_{\text{FA}}$  values alone, nor do the  $\delta^{13}\text{C}_{\text{FA}}$  values reflect a significant shift in differences in the kinds of non-ruminant adipose products that could have been processed in the ceramics from Islamic contexts to later Norman, Swabian and Aragonese.



Figure 47: A map of Sicily and Palermo from the Kitāb Gharā'ib al-funūn wa-mulāḥ al-'uyūn (وملح الفنون غرائب كتاب العيون) ("The Book of Curiosities") transcription of the 13<sup>th</sup> century copy (Unknown 1190–1210) Oxford, Bodleian Library, MS Arab. C. 90, f.32b-33a. The Map has been adapted by Nef et al., (2015) whereby 4 depicts the entrance of the chicken market. Figure taken from (Nef, Pezzini and Sacco 2015, 56).

The  $\delta^{13}\text{C}_{\text{FA}}$  values that fall within the range of non-ruminant might also reflect the contribution of rabbit or hare (Drieu et al. 2021a). Other studies have revealed rabbit remains at other sites in Palermo (Sarà 1997) and across Sicily, in Entella (Bedini 1999), Segesta (Di Martino 1997) and Calalthamet (Sarà and Cangialosi 2005) for example. A study of faunal remains in Termini Imerese depicts that hares were an important commodity during the 13<sup>th</sup> and 14<sup>th</sup> centuries in Sicily, representing over 40% of hunted fauna (Bresc 1980; Delort 1984). However, very few lagomorphs (inc. rabbit and hare) remains were identified in the faunal assemblages at the sites studied in this investigation, representing <1 % NISP, and the meat weight value would be expected to be almost negligible (Appendix K) (Aniceti 2020; Arcoleo and Sineo 2014; Arcoleo 2015). A supposed increase in hare as hunted fauna in the Norman, Swabian and Aragonese periods is also not observed in the archaeological record at least at

Mazara del Vallo (Aniceti 2020, forthcoming). Thus, whilst taphonomic processes and out-of-site processing of these animals may contribute to their misrepresentation in the faunal record, it seems unlikely, in comparison to the main domesticates (cattle, caprines, suids and even domestic fowl), that rabbit or hare were a staple source of daily diet amongst these populations.

It cannot be dismissed that non-ruminant animals (pigs, chicken, hare etc.) are underrepresented in the faunal record due to various factors and were still specifically selected for processing in these vessels. However, the over-representation of  $\delta^{13}\text{C}_{\text{FA}}$  values that fall within the range of non-ruminant products can be explained by other non-animal contributions. The isotopic value of  $\text{C}_3$  plants has been shown to cover a wide range in the non-ruminant region (which also covers the freshwater isotopic values) and some of the ruminant ranges (Chapter 3). Therefore, it is possible that mixtures with plant products are affecting the  $\delta^{13}\text{C}_{\text{FA}}$  values observed in these residues. Based on the faunal data it would appear that ruminant domestic species were the predominant contributor to foods and therefore mixtures of ruminant products with other products such as plants may have led to skewed  $\delta^{13}\text{C}_{\text{FA}}$  values. Indeed, many samples that fell within the range of non-ruminant products also displayed biomarkers of plant products, but plant products were also found alongside samples that fell within the range of ruminant adipose fats, and therefore it is difficult to deduce the true impact of plant products on the  $\delta^{13}\text{C}_{\text{FA}}$  values (see section 7.6).

Overall, therefore, both faunal remains and  $\delta^{13}\text{C}_{\text{FA}}$  values of ceramic samples show that at all sites and chronological periods, ruminant meats from cattle and caprines contributed significantly to cuisines and were selected for processing in ceramic vessels. Clear evidence for the processing of dairy products in ceramics from Casale San Pietro and Mazara supports the faunal evidence that ruminant animals were utilised for both meat and milk at these sites. An over-representation of  $\delta^{13}\text{C}_{\text{FA}}$  values that fell within the non-ruminant range may be explained by specific selection of non-ruminant adipose products such as pork or chicken, but also likely reflect the high levels of mixing of animal products and plant products in these vessels. In general, there is no clear difference of the processing of animal products in ceramic vessels between chronological periods based on the  $\delta^{13}\text{C}_{\text{FA}}$  values. However, faunal evidence

suggests that there may have been a shift in the importance of different animals such as chicken and pork in the later Norman and Swabian periods as well as changes in animal practices such as the more generalised use of caprines in Swabian periods at Mazara, that could not be observed in the organic residues of the ceramics (Aniceti 2020, forthcoming).

## **7.4 On the absence of aquatic resources in cooking pots**

In this study it was not possible to unambiguously identify a clear presence of aquatic resources in any of the ceramic samples studied, across all sites and chronological periods. Despite the presence of isoprenoid acids in several samples, these were either present in trace amounts or could not be distinguished from other sources such as ruminant products (e.g., when phytanic acid SRR diastereomers were calculated) (Lucquin et al., 2016) (Chapters 5 and 6). Importantly, although few samples were enriched in  $\delta^{13}\text{C}$  ( $> -27\text{‰}$ ), the majority of samples were more depleted in  $\delta^{13}\text{C}$  than would be expected for marine products and were therefore assigned to other non-ruminant sources, based on an incomplete aquatic biomarker suite. Furthermore, a complete absence of APAA C<sub>20</sub> and APAA C<sub>22</sub> in all samples analysed, formed through heating aquatic oils (Hansel et al., 2004; Evershed et al., 2008), meant that it was not possible to confirm the processing of aquatic sources in these vessels. Although the absence of APAA C<sub>20</sub> and APAA C<sub>22</sub> does not preclude the presence of non-heated aquatic oils contained in these ceramics (e.g. fish sauce), APAAs can be formed through relatively low intensity heating conditions (Bondetti et al. 2021) and evidence that other products (terrestrial fats and plant products) were heated in the ceramic vessels is clearly indicated in several samples through the presence of APAA C<sub>16</sub> and APAA C<sub>18</sub> and ketones.

The absence of evidence of aquatic products in the cooking vessels requires some reflection with regards to the consumption of fish amongst these populations. The absence of aquatics in the ceramic samples analysed does not necessarily mean that fish were not consumed by these populations. The large majority of ceramics analysed were of closed forms (*ollae* and cooking pots) and it is possible to assume that these



ceramic vessels were used for long boiling of meat carcasses and plants instead of fish. Although Eastern Arabic and Andalusian Medieval cookbooks do mention recipes whereby fish were cooking in stews or sauces, they may have been prepared and consumed in other ways (e.g. baked, smoked, salted, dried, or cooked directly on the fire) (García-Sánchez 1996, 264–265). Additionally, as shown in Chapter 4, the processing of non-fatty fish products in ceramic containers may limit the ability to identify these due to a low lipid content. However, it has been suggested that in places where fish was a staple (e.g., medieval sites in Maghreb) fatty fishes such as tuna and sardines were the most consumed species (Rosenberger 1999). Alternatively, fish may have been processed and incorporated into cuisines as fish sauce (garum, liquamen or salsamenta), but evidence of aquatics detected in organic residues in transport amphorae from these sites was also limited. For example, aquatic biomarkers were identified in only 2 samples from the 10<sup>th</sup>-11<sup>th</sup> century in Casale San Pietro. Drieu et al. (2021) suggests that these results indicate that marine products (sauces, garum, liquamen or salsamenta) were not a major commodity transported in these vessels unlike in the preceding periods This provides further support that non-heated aquatic products are minimally represented.

Some fish remains were identified in the faunal record, but fish remains at all sites studied made up only a small proportion of the assemblage (<3% of total NISP at all sites) (Aniceti 2020; Arcoleo and Sineo 2014; Arcoleo 2015)(Appendix K). Sampling bias (e.g., lack of sieving) and biases in taphonomic processes such as the preferential degradation of smaller, more porous bones, are likely to result in the underrepresentation of fish remains at these sites. Nonetheless, fish remains were identified at the sites of Palazzo Bonagia (NISP= 16) and La Gancia (NISP=88) and were assigned to tuna. Interestingly, traces of wear on these bones have been attributed to the trapping of these fish in traps, supporting sources that suggest this was an active trade in Trapani and Palermo (Consolo 2006; Longo and Clark 2012; Arcoleo and Sineo 2014). Additionally, a total of 13 countable fish remains were identified at the site of Castello San Pietro, of which 10 were assigned to tuna and 3 were unidentified due to a high degree of fragmentation and erosion (Aniceti 2020). At Casale San Pietro a total of 7 countable fish remains were identified and these were assigned to four different species (Tuna (*n*-4), European sea bass (*n*-1), Carangids (*n*-1) and Rajids (*n*-1). As

Casale San Pietro is an inland site, the presence of these remains suggest that fish products were introduced on site from elsewhere, an effort that reflects some importance of fish products. Furthermore, despite the low incidence of fish remains at Mazara del Vallo (NISP= 5 for Islamic contexts and 8 for Swabian context), clear evidence of cut marks identified on tuna remains indicates that fish were processed on-site (Aniceti, 2020; Aniceti, forthcoming).

Therefore, although incidences of fish remains were low at all sites, it is possible to suggest that fish products were utilised by the populations but not processed in the ceramic containers analysed here. However, the value of fish consumption by populations of Sicily at this time is difficult to discern from the faunal evidence and ORA analysis alone. In 10<sup>th</sup> century England it has been hypothesised that an increased consumption of fish was the result of growing urban centres and tighter restrictions of temporary meat fasting as part of the Christian religion (Barrett,Locker and Roberts 2004a; Sykes 2001). However, this phenomenon described as a 'Fish event horizon' has not been observed in Sicily (Barrett,Locker and Roberts 2004b; Barrett 2018). Furthermore, although meat fasting may have been implemented in post-Islamic Sicily, strict religious fasting is usually only seen clearly amongst monastic groups (Müldner et al. 2009; Sarkic et al. 2019) and such temporary fasting in the general populace is not easily identified in the archaeological record. Furthermore, amongst poorer populations fish was not necessarily used in replacement of terrestrial meats. More likely, accessible products such as cereals, legumes and vegetables were consumed without the contribution of meat or fish products, except in elite contexts (Adamson 2004, 189). The abstinence of meat products and replacement of either fish or plant products would not be identified in archaeological ceramics if processed in the same vessel, as it is not possible to distinguish between different cooking events without the presence of food crusts, as previously discussed (Miller et al. 2020).

Nonetheless, evidence from ceramics and the faunal evidence gives no indication that fish was more or less important between the Islamic and later Norman, Swabian and Aragonese periods. An understanding of the value of fish to cuisine in medieval Sicily requires further analysis and stable isotope analysis of human remains can aid our understanding by giving direct evidence of diet (see section 7.7).

## 7.5 Plant products and cuisine

As previously outlined (chapters 3, 4, 5 and 6), the potential to identify plant products (vegetables, plant oils, nuts, fruits, cereals and grains etc.) is somewhat limited compared to the identification of animal fats. This is predominantly due to their comparatively lower lipid yield and characteristic biomarkers that are more susceptible to degradation in the burial environment. Nonetheless, a wide variety of evidence for the use of plant products was identified in all sites and chronological periods studied here. In many cases, evidence of non-specific plant products were identified in the ceramic vessels, but plant oils, plant waxes including specific taxon biomarkers for *Brassica*, *Allium* and possible *Foeniculum vulgare* (fennel), plant resins (birch-bark and conifer) as well as evidence for grapevine and other fruit products were all identified. Biomolecular evidence of plant products in the ceramic samples has enabled important insight into the use of products and their incorporation into everyday cuisines. Despite this, the evidence of plant products is still lacking and the inability to identify more plant products at species level hinders further understanding of the use of certain resources, particularly those suggested as part of Watsons 'revolution plants' of which none were identified in the ceramic vessels analysed in this study (Watson 1974, 1983). Furthermore, cereals could not be identified in the ceramic vessels, but this does not necessarily exclude their use. Our ability to identify cereals is hindered by their low-lipid content and they are unlikely to survive in all but waterlogged or permafrost environments (Colonese et al. 2017a; Hammann and Cramp 2018; Ross et al. 2003). Due to these limitations and gaps in knowledge, further discussion is required here to enable a better insight into the availability and culinary uses of plant products in medieval Sicily.

### 7.5.1 Vegetables

Lipid profiles indicative of the epicuticular leaf wax of brassicas was identified in a several samples across all sites analysed in this study (in 12% of all samples from Palermo, in 15% and 36% of samples in Islamic and Swabian contexts from Mazara and 4% percent of samples from Islamic contexts at Casale San Pietro). Although these

profiles can distinguish *Brassica* from other plant genera, there are hundreds of cultivated varieties, which include kale, cabbage, broccoli and turnip leaves (*Brassica rapa*) and different regional varieties of these types (Evershed, Heron and Goad 1991). As shown in Chapter 4, it is not possible to differentiate further between these varieties, and therefore an understanding of what variety of *Brassica* may have been processed in these ceramic vessels requires other evidence of what was available. No archaeobotanical evidence of brassicas was found at the site of Mazara, but evidence of *Brassica* spp. was found at the site of Casale San Pietro in both Islamic and Norman contexts, albeit in lower concentrations in the latter (Fiorentino et al. forthcoming). However, these remains were unaltered and therefore it cannot be dismissed that the remains might be modern endogenous contaminants from the environment (Fiorentino et al. forthcoming). The identification of these products in the ceramic vessels, therefore, provides clear evidence that brassicas were available and selected for processing in ceramic containers. The concurrence of *Brassica* biomarkers and evidence of ruminant adipose fats processed in the same vessels from Mazara (4 samples in the Islamic contexts and 7 in Swabian contexts) and in Palermo (*n*-3) provides evidence that brassicas were routinely processed alongside meat products. Evidence for the processing of leafy vegetables and meat as part of pottages and stews has been previously shown in medieval England (Evershed, Heron and Goad 1991; Dunne et al. 2019) and medieval Italy (Buonincontri et al. 2017; Giorgi, Salvini and Pecci 2010; Salvini, Pecci and Giorgi 2008). Dishes that combine vegetables and meat products had high importance in medieval Arabic cuisines. This is evident in the names of meat dishes, named after the vegetable that they contain, for example the dish *Kurnubiyya* which means cabbage (Pitchon 2020). The identification of *Brassica* in Swabian cooking pots, also alongside ruminant adipose fats, shows that these dishes were still enjoyed by later populations.

Further evidence of the processing of leafy vegetables was demonstrated in this study through the identification of profiles characteristic of, but not necessarily specific to, *Allium porrum* (modern leek) in ceramic vessels from Palermo (*n*- 1) and from Islamic contexts at Mazara (*n*-7). It was stated in this research that without comparative profiles of other varieties within the *Allium* family, these cannot be dismissed, although the contribution of onion based on this profile was excluded in Chapter 4. In the

archaeobotanical record, no evidence of *Allium* was identified at the site of Casale San Pietro, in line with the concurrent absence of evidence in the ceramics at this site. Therefore, it can be suggested that these species of vegetable were not an important culinary commodity at this site (although evidence may not have preserved in the ceramic or archaeobotanical record). On the other hand, at the site of Mazara del Vallo, *Allium sp.* were identified in Swabian contexts and were attributed to wild garlic (Fiorentino et al. forthcoming; Primavera 2018b). Thus, the processing of wild garlic in the ceramic vessels cannot be dismissed but, without more information about these lipid profiles, it is difficult to be more conclusive. Additionally, lipid profiles similar to the leaf/stem wax of modern *Foeniculum vulgare* (fennel) were identified in a number of ceramic samples from Casale San Pietro. Although no evidence of fennel was observed in the archaeobotanical record from Sicily, native to the Mediterranean region, fennel was valued in the Roman period for its leaves and seeds. Furthermore evidence of fennel has been found in several Christian and Islamic sites of medieval Iberia (Peña-Chocarro et al. 2019; Peña-Chocarro and Pérez-Jordà 2019) and Roman-medieval contexts in Northern Europe (Livarda and van der Veen 2008). Fennel also grows prolifically at the site of Casale San Pietro today and has also been described in written sources for both medicinal and culinary purposes, as post- medieval botanist Parkinson describes:

*'The leaves, seede and rootes are both for meate and medicine; the Italians especially doe much delight in the use thereof, and therefore transplant and whiten it, to make it more tender to please the taste, which being sweete and somewhat hot helpeth to digest the crude qualitie of fish and other viscous meats. We use it to lay upon fish or to boyle it therewith and with divers other things, as also the seeds in bread and other things.'* (Parkinson 1640).

Therefore, this evidence could represent the first evidence of fennel in medieval Sicilian contexts from its detection in domestic cooking pots. However, future work should be done to strengthen the use of ketone nonacosan-10-one as a biomarker for fennel in archaeological contexts. Nonacosan-10-one is also present in the leaf wax of broadleaf sternum and its presence in other Apiaceae species is not yet known. *Apium* cfr. *Lectophyllum*, commonly known as marsh parsley was identified in Islamic contexts

at the site of Mazara del Vallo and could indicate the use of other Apiaceae species that need to be investigated molecularly (Fiorentino et al. forthcoming). Being non-dense products, as described by Miksicek (1987), fennel (or other Apiaceae species), brassicas and alliums may not be preserved in the archaeobotanical record unless in waterlogged environments, and they leave no trace of seeds or charred material when degraded or processed through boiling (Miksicek 1987). Therefore, evidence of the processing of these products in ceramic vessels has provided important insight into the processing of vegetables otherwise invisible in the archaeological record.

No other vegetable-specific evidence could be identified in the ceramic vessels, but a number of other vegetables varieties were identified at both Casale San Pietro and Mazara del Vallo in the archaeobotanical assemblages (Fiorentino et al. forthcoming; Primavera 2018a; Fiorentino et al. forthcoming). Of note, at the site of Mazara the archaeobotanical record showed evidence of vegetables forming part of Watson's 'revolution' plants including *Solanum melongena* (aubergine) in Islamic contexts and *Spinacia oleracea* (spinach) in Swabian contexts (Primavera 2018b; Fiorentino et al. forthcoming). Despite the identification of specific sterols present in modern spinach, these were not identified in any of the ceramic samples studied. However, the absence of these biomarkers does not exclude their presence in the ceramic vessels, as sterols are susceptible to degradation through anthropogenic and microbial processes as demonstrated in Chapter 4. Furthermore, aubergine does not have a distinct profile that would be observable in archaeological ceramics even if processed (Chapter 4). Additionally, plants from the *Cucurbitaceae* family were also identified in the Islamic phase at Mazara del Vallo and may include melon, cucumber, or watermelon (Primavera 2018b; Fiorentino et al. forthcoming). These products are all consumed fresh and do not require processing and thus would not be observed in the ceramic containers, regardless of specific biomarkers and preservation. Thus, although archaeobotanical remains can only be used as indirect evidence of food consumption, the identification of these remains tells us that these varieties were present and likely cultivated in vegetable gardens on the site, due to their perishable nature and unsuitability for long-distance transportation. This highlights the importance of archaeobotanical data in identifying products that are invisible through organic residue analysis, either because they have no specific biomarkers, would be degraded,

or are eaten fresh. Conversely, the identification of brassicas, alliums and potentially fennel in the ceramic containers, and which could not be unambiguously identified in the archaeobotanical record, indicates the advantage of ORA in identifying the processing of non-dense vegetable products.

Interestingly, in both MZ and CLESP there is a noticeable decrease in the variety of plant products observed in ceramics from the Islamic to the Swabian periods (Chapter 6). Despite differential identifications of products between the organic residue analysis and archaeobotanical evidence, this evidence compliments a similar decline in the variety vegetable products observed in the archaeobotanical record at both sites (Fiorentino et al. forthcoming, forthcominga); thus providing evidence that a reduced variability of vegetable products were available for culinary purposes during the Swabian period. However, at least at Mazara, a continued variety of fruit products was observed from Islamic contexts to Swabian contexts (Fiorentino et al. forthcoming).

### **7.5.2 Plant oils**

Plant oils could only be unambiguously identified in a small number of cooking pots and domesticated containers investigated in this study ( $n=2$  from Islamic and Swabian contexts in CLESP and  $n=1$  Swabian contexts MZ). Whilst a range of plant oils can theoretically be distinguished based on their specific molecular profiles, including brassica seed, castor, palm fruit and kernel oil (Colombini et al., 2005; Copley et al., 2001, 2005; Pecci et al., 2010; Steele et al., 2010), no such profiles were detected in the cooking vessels analysed here. The profiles indicative of plant oils detected in this study may indicate the presence of olive oil, but as the profile of olive oil is similar to that of most degraded plant oils it was not possible to be assertive here. Archaeobotanical evidence at CLESP and MZ show the presence of olive products (Fiorentino et al. forthcoming; Primavera 2018b; Fiorentino et al. forthcoming). However, it is difficult to know how these were used and incorporated into cuisine. An important caveat here is that plant oils are likely underrepresented in the cooking vessels analysed, as a result of mixing with animal products that mask the plant oil signal, degradation, and transformation through the cooking practice itself.

In contrast to cooking containers, transport or storage containers may offer a better way for identifying the presence/ absence of plant oils in the archaeological record. Although degradation and non-specificity is still very much a problem in ORA of these vessels, it can be expected that less mixing occurs in transport and storage vessels and generally they are used to carry one type of commodity (e.g., olive oil or wine). Furthermore, as these commodities are not processed and altered by heat, they might be more detectable. In research performed by Drieu, evidence of plant oils was detected in 6 amphorae samples from Casale San Pietro, the hypothesis that these profiles reflect the presence of olive oil cannot be ruled out, but for reasons as described above, may also represent other plant oils (Drieu et al. forthcoming). From Islamic contexts at Mazara, only two amphorae showed evidence of plant oils, one of which indicates the presence of another oil, possibly linseed or sesame (Drieu forthcoming; Copley et al. 2005a). Furthermore, only one amphora from 10<sup>th</sup>-11<sup>th</sup> century contexts at La Gancia indicated clear evidence of plant oils, but lipid degradation and mixing either through the single use of the vessel or successive uses limited further identification of these products in most cases (Drieu et al. 2021b; Drieu, Carver and Craig 2018). Therefore, whilst it may be assumed that plant oils, and particularly olive oil, were an important commodity in Sicily during the 9<sup>th</sup>-14<sup>th</sup> centuries, their use could not be clearly determined via organic residue analysis of cooking pots or transport vessels.

### **7.5.3 Fruit products**

Several samples yielded evidence of grapevine products using the criteria of identification as outlined by Drieu et al. (2020, 2021). The majority of these samples came from the site of Casale San Pietro ( $n=4$  from Islamic contexts and  $n= 1$  from Norman contexts), but evidence of grapevine products was also observed in 1 sample from Castello San Pietro (Palermo) and 1 sample from Swabian contexts at Mazara. Grapevine products may be representative of wine, but can also include other products such as grape juice and vinegar (Drieu et al. 2021b). Interestingly, despite the identification of grapevine products in the domestic ceramic vessels from CLESP, no evidence of grapevine products were identified in the archaeobotanical record in any



context (Fiorentino et al. forthcoming). Furthermore, in the analysis of transport amphora from CLESP there is little evidence of grapevine products from Islamic contexts, as only 3 amphorae dating to the end of the Islamic period (11-12<sup>th</sup>) met the criteria for grapevine products (Figure 48b). Drieu et al. (2021b) concluded that this represents a decline in wine trade between Palermo and Casale San Pietro in this period. Thus, the incorporation of grapevine in cooking vessels offers an important insight in the use and procurement of these products not previously evidenced at the site. Perhaps, grapevine products (whole fruits or processed) were transported to the site by other means (e.g., in wooden barrels), or remains of grapevine products did not survive in the archaeobotanical record due to degradation in the burial ground or anthropogenic processes. Furthermore, raisins are dried product that may not leave a chemical signal detectable through organic residue analysis if transported in amphorae.

Amphorae samples used for storage purposes in Norman contexts from Casale San Pietro did, however, yield evidence of grapevine products. This result depicts localised storage of grapevine products on the site, thus lending to the idea that grapevine was grown and processed locally (Drieu et al. 2021b). The concurrence of grapevine products and animal fats in storage amphorae may also depict the reuse of vessels for the transportation of wine for subsequent storage of animal products. As demonstrated in Chapter 4, the presence of both animal fats and grapevine products could be due to use of animal products in post-firing treatments to seal the vessel before containing liquid grape products. Additionally, the identification of grapevine products in the storage samples may indicate the use of vinegar used for pickling products to be stored.

Furthermore, grapevine products, most likely wine, were identified in a number of amphorae found in Palermo depicting local trade of wine within the city itself. Evidence of grapevine products found in amphorae samples transported from Palermo but found outside of Sicily (mainland Italy and Sardinia), show a clear persistence of wine trade during the Islamic period. This is despite uncertainty in the effect of the Islamic political regimes and associated hadiths that prohibit the consumption of alcohol in the Islamic faith (Drieu et al. 2021b). The near absence of grapevine products in the cooking pots from Palermo highlights that although these products were commercially

exported within the capital and outside of Sicily, they were not selected for incorporation in cuisine by these populations (Figure 48). These results could indicate that these populations did not utilise these products due to restrictions based on their faith (in terms of wine) or in comparison to the rural site of Casale San Pietro, preservation of foodstuffs was not needed through the use of vinegar, although vinegar and sour grape juice was widely used as a flavouring and for medicinal purposes also (Lewicka 2011; Zaouali 2009; Woolgar 2010; Waines 2003; Pitchon 2020).

On the other hand, the archaeobotanical record at Mazara shows that grapevine products dominated the fruit assemblage (Fiorentino et al. forthcoming) . The consumption of fresh or dried grapes were found and evidence of charred pips and a pedicel indicated that the grape products were being processed into wine, juices, syrups or other grape vine preparations (Drieu forthcoming). However, only one cooking pot from Swabian contexts and one transport amphorae from Islamic contexts yielded evidence of grapevine products (Figure 48a). This finding shows that these grapevine products, most likely wine, were not imported into Mazara via amphorae, and instead grapevine produced were processed and used locally instead. However, Drieu highlights this could represent a preservation issue at the site as overall lipid yield from the amphorae was low, but the high lipid yields obtained from cooking vessels from the same contexts suggests otherwise (Drieu forthcoming).

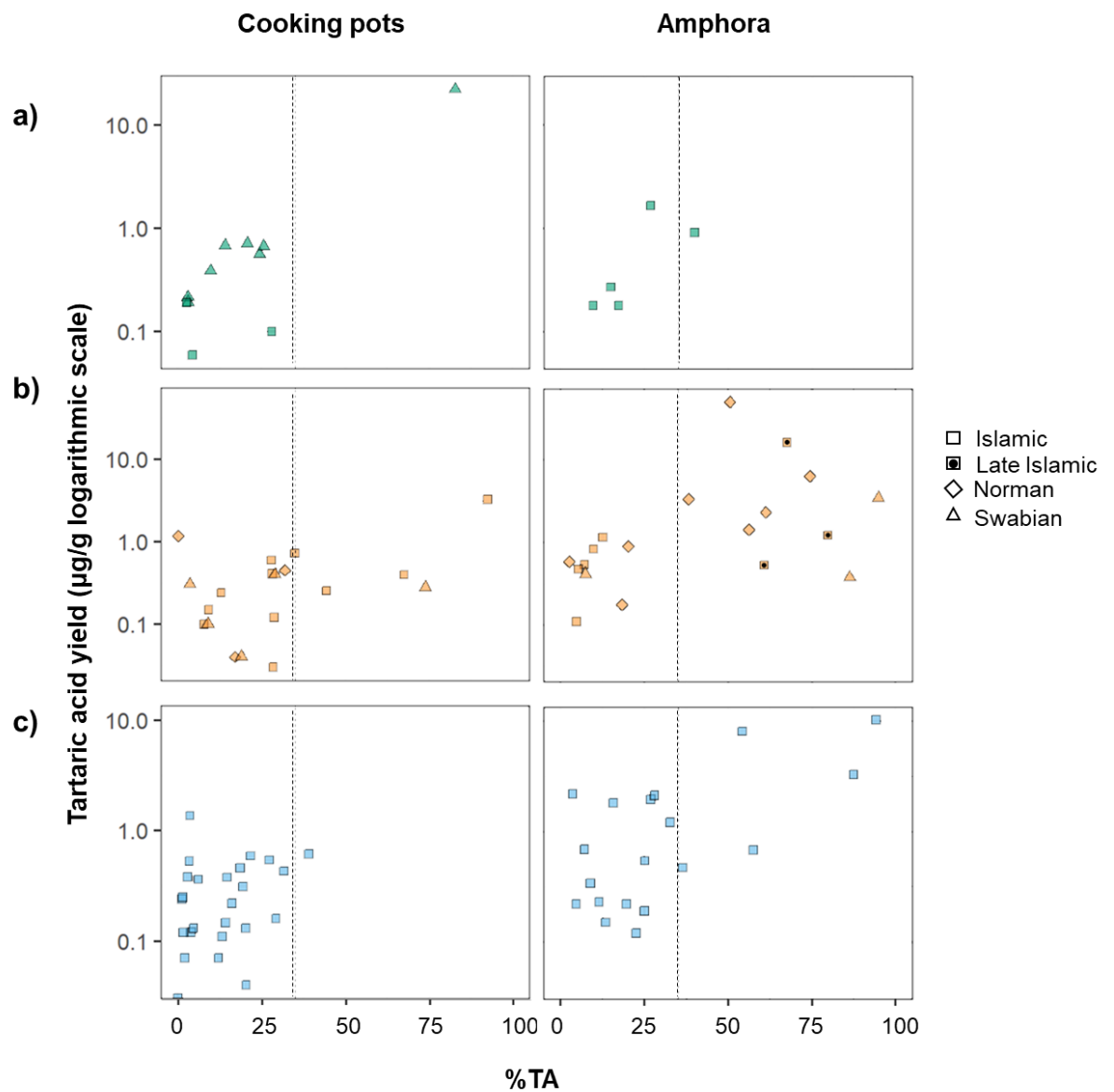


Figure 48: % TA (tartaric acid/ (tartaric + malic acid)) plotted against tartaric acid yield ( $\mu\text{g/g}$  logarithmic scale) for a) MZ b) CLESP and c) Palermo across chronological periods (Islamic, Late Islamic, Norman and Swabian for both cooking pots and amphora. The dashed line at %TA 35 shows the cut off point for the interpretation of grapevine products in these containers.

Evidence of other fruit products (based on the criteria set out in this investigation) was identified in several ceramic across all sites and chronological periods. Malic acid is the dominant acid present in fruits such as apple, plum, cherry, peach or apricot as it is ubiquitous throughout the plant kingdom and present in relatively high quantities in fruits (Walker and Famiani 2018). As noted however, malic acid is not restricted to fruit and may have been derived from the other identified plant products, such as brassicas or leeks which are present in many of the samples in this investigation some of which co-occur with evidence of fruit (Ruhl and Herrmann 1985a). Nonetheless,

Arabic-Islamic recipes widely refer to the incorporations of fruits into cuisines to add taste, often suggesting mixtures with meat to give a sweet and salty aesthetic (Al-Muḏaffar Ibn Naṣr Ibn, 2007). It is difficult to assert a fruit contribution in ceramics where both malic acid and markers of brassicas are found together. However, in samples where malic acid was identified without the presence of brassica markers a fruit contribution has been assumed. Using this criteria, evidence of fruit products and ruminant adipose products frequently occurred together in ceramic vessels from all sites and chronological periods showing a continuation of the addition of fruits to dishes in post Islamic contexts (see section 7.6).

A wide variety of fruit products other than grapes were identified in the archaeobotanical assemblages at both Casale San Pietro and Mazara. At the former, evidence of fig, apple, a variety of plums and peaches were identified in the Islamic contexts and with reduced variety in the Swabian periods where only plum and apple were identified (Fiorentino et al. forthcoming). At the site of Mazara, fruit products and their cultivation dominated the archaeobotanical record. Here citrus fruits, pomegranate and grape are the most represented in both Islamic and Swabian contexts, but evidence of plum, figs, peach, pear, apple, blackberries, and strawberries were also readily identified (Primavera 2018a; Fiorentino et al. forthcoming) . Thus, showing a wide variety of available fruit products at the site that continued from the Islamic to Swabian phase (Fiorentino et al. forthcoming). Although it is not possible to be more definitive about the types of fruit products present in the ceramic vessels or, in some cases, to be able to distinguish between fruits and vegetables, the high availability of these products at the sites studied here and our concepts of cuisine at the time, suggest that fruits were likely readily incorporated into dishes.

#### **7.5.4 Cereals and legumes**

Alkylresorcinols from cereals (wheat, barley or rye) were not found in any of the ceramic samples analysed in this study, despite the use of highly sensitive detection methods to identify their characteristic ions (Appendix A) (chapter 5 and 6). Whilst this could indicate that cereals were not processed in these vessels, the identification

of cereals in archaeological ceramics is somewhat an elusive task due to the high susceptibility of alkylresorcinols to degradation in contexts other than waterlogged sites. Therefore, their absence does not exclude the use of cereals in these vessels (Ross et al., 2003; Hammann and Cramp, 2018; Colonese et al., 2017). Indeed, a variety of cereals were identified in the archaeobotanical record at both Mazara and Casale San Pietro, where barley was most represented, followed by wheat species. At Mazara, in the Swabian and Aragonese period, there appears to have been a reduction in the presence of barley in comparison to earlier periods and an increase in the soft and durum wheat, the later presumed to be introduced to the island at the arrival of the Arabs as parts of Watsons 'green revolution' theory (Watson 1974, 1983; Fiorentino et al. forthcoming).

Whilst the archaeobotanical record provides evidence for the cultivation of cereals at the sites analysed here, it is possible to suggest that these may not have been a major part of cuisines prepared in closed forms such as *olla* and cooking pots. Instead, it is likely that barley and wheats were ground to flour and used for making breads and pastas instead of boiled or cooked in their raw form. Stone plates (*testelli*), analysed in this study, are thought to have been used to process flour based products such as bread (Corretti et al. 2016). No evidence of alkylresorcinols were identified in these ceramics, but as Hamman and Cramp (2018) demonstrate, for absorption of alkylresorcinols into the pottery matrix to occur, cereals need to be processed for a long time (Hammann and Cramp 2018). It is unlikely that plain contact with the ceramic would result in the absorption of these molecules.

Additionally, there was no evidence of broomcorn millet (*Panicum miliaceum*) in any of the vessels analysed in this study despite the fact that this product can be routinely identified through the presence of a specific biomarker (millacin; olean-18-en-3 $\beta$ -ol methyl ether) (Heron et al., 2016; Bossard et al., 2013). Studies have depicted the presence of millet in Northern and Central Italy through archaeobotanical, stable isotope studies and organic residue studies (Riccomi et al., 2020; Ganzarolli et al., 2018). However, no evidence of millet has been found in Southern Italy or Sicily. As part of the Sicily in Transition study, archaeobotanical evidence supports this notion as no evidence of millet was found. Whilst the absence of millet biomarkers in the

archaeological ceramics does not refute the use of millet for other culinary purposes, such as production into flour as opposed to boiling in ceramics, the results from ORA in combination with the archaeobotanical evidence does support previous evidence that millet was mostly consumed in the north of Italy during the medieval period but not in the south (Rolandsen, Arthur and Alexander, 2019).

Finally, it might be assumed that legumes would make up a significant part of the diet of populations in Sicily at this time. Legumes would have been an important source of carbohydrates and proteins, particularly in diets where meat contributions were low. Indeed, a variety of legumes are evident in the archaeobotanical assemblages from a number of sites from Sicily. For example, although rare and only occurring in the Islamic phases at the site, *Leguminosae* (fava bean, vetch and sweat pea) were identified at the site of Mazara del Vallo (Fiorentino et al. forthcoming). Furthermore, the fava bean was also identified in the archaeobotanical assemblages at Colmitella and Casale San Pietro (Carver et al. 2019). Additionally, legumes: the fava bean, chickpeas, chicking vetch, vetch and lentils, made up a significant proportion of the assemblages at the site of Contrada Castro, studied as part of other research (Castrorao Barba et al. 2021). However, it was not possible to identify the presence of legumes in the ceramics analysed through organic residue analysis despite the fact that legumes, such as the fava bean, were likely boiled in cooking vessels (Redon, Sabban and Serventi 2000). No specific biomarkers have thus far been identified for legumes and therefore their presence in archaeological ceramics is difficult to discern without the aid of charred food crusts or proteomic analysis.

## **7.6 Selectivity and mixing of products in ceramic vessels**

As previously noted, complex mixtures of different products (animal products, plants, fruits etc.) were frequently observed in the ceramic vessels analysed in this study. However, due to the lack of food crusts on the surface of the ceramics, an important caveat is that we are unable to distinguish whether foods were processed together to create complex 'dishes' or whether the residues build up over time (Miller et al. 2020). Whilst it is not possible to identify specific recipes in these vessels based solely on

evidence of mixing, it is possible to observe trends and patterns in the selectivity and combination of certain products. Figure 49 shows the number of times two different products were identified together in the same ceramic sample.

In comparison to open forms such as pans and stone plates from all sites and chronological periods, closed forms such as *ollae* and cooking pots show the highest amount of mixing. Whilst no clear pattern of the selectivity of certain resources could be observed, the mixtures of ruminant adipose, fruit products and plant products is highly indicative of recipes described in medieval culinary literature (Lewicka 2011; Waines 2003; Perry 2005). These mixtures likely reflect the uses of these closed vessels for the processing of stews or pottages. This pattern exists amongst both the Islamic forms and later Norman, Swabian and Aragonese periods, indicating the unchanged function of these vessels.

Conversely, a lesser mixing of products was identified in the open forms (e.g., pans and stone plates) in comparison to closed forms. Of note, non-ruminant products are more frequent in open forms compared to closed forms and identified without evidence of other products, suggesting a selective processing of these resources. Limited evidence of ruminant adipose fats processed in the open forms may indicate that ruminant meats were more likely boiled rather than fried. Pitchon (2020) suggests that in medieval Islamic cook books frying meat was relatively rare and, in any case, meats like lamb or kid were boiled prior to other cooking processes (Pitchon 2020). However, as previously discussed, based on the  $\delta^{13}\text{C}_{\text{FA}}$  values it was not possible to determine whether evidence of non-ruminant products come from non-ruminant adipose sources (pigs or chicken) or from  $\text{C}_3$  plant sources. In fact,  $\text{C}_3$  plant sources may have been processed in these types of vessels, but biomarkers are not visible due to the limited absorption of these products through frying alone. It has already been suggested that stone plates, for example, were used for cooking flour-based products such as bread, but our ability to identify these biomarkers are significantly limited based on the mode of cooking. Future investigations on a higher number of samples of this type will help to further deduce this pattern.

Furthermore, grapevine products only occur with non-ruminant products, mainly in closed forms potentially highlighting the selective mixing of grapevine and non-ruminant products. However, it is difficult to assert where these non-ruminant signals come from. Indeed, this evidence may suggest that non-ruminant adipose products were mixed with grapevine products such as wine, grape juice or vinegar for preservation and taste or could indicate the mixtures of these products with other plant products yielding a non-ruminant signal. It cannot be dismissed that the mixture of grapevine products with ruminant adipose fats either in the same cooking event or in sequential cooking could have impacted  $\delta^{13}\text{C}_{\text{FA}}$  values. However, due to the low concentrations of fatty acids in grapevine products, such an influence of  $\delta^{13}\text{C}_{\text{FA}}$  values seems unlikely and this signal likely comes from other resources (plant or animal).

The co-occurrence of both Pinaceae resins and beeswax products with ruminant adipose fats in some vessels might reflect the addition of ruminant fats to these products to modify their properties for better use as post-firing sealing products. However, as shown in Chapter 4 it is not possible to distinguish the presence of products used a sealing technology from the commodities processed in the vessels for culinary purposes. It is also possible that beeswax and pine products are associated with preserved ingredients added to the ceramic vessel. Pine resins: for example, are commonly used to waterproof amphorae vessels that contain liquid products. Thus, if products are first stored in other ceramic vessels, lined with waterproofing products such as pine or beeswax, their remnants may get into the cooking containers second hand.

Finally, as previously noted (chapters 5 and 6), dairy products in vessels from CLESP are, in most cases identified on their own, highlighting the selectivity of certain vessels for the processing of these products into either cheese, ghee, or yogurt. However, in samples from Mazara evidence of dairy co-exists with other products such as plant products and fruit products. This lends to the idea that these ceramics were being used for recipes containing dairy products and not solely for the processing of dairy products. Dairy products curdled or processed into yogurt have been mentioned as additions to stews in medieval Arabic sources (Pitchon 2020).



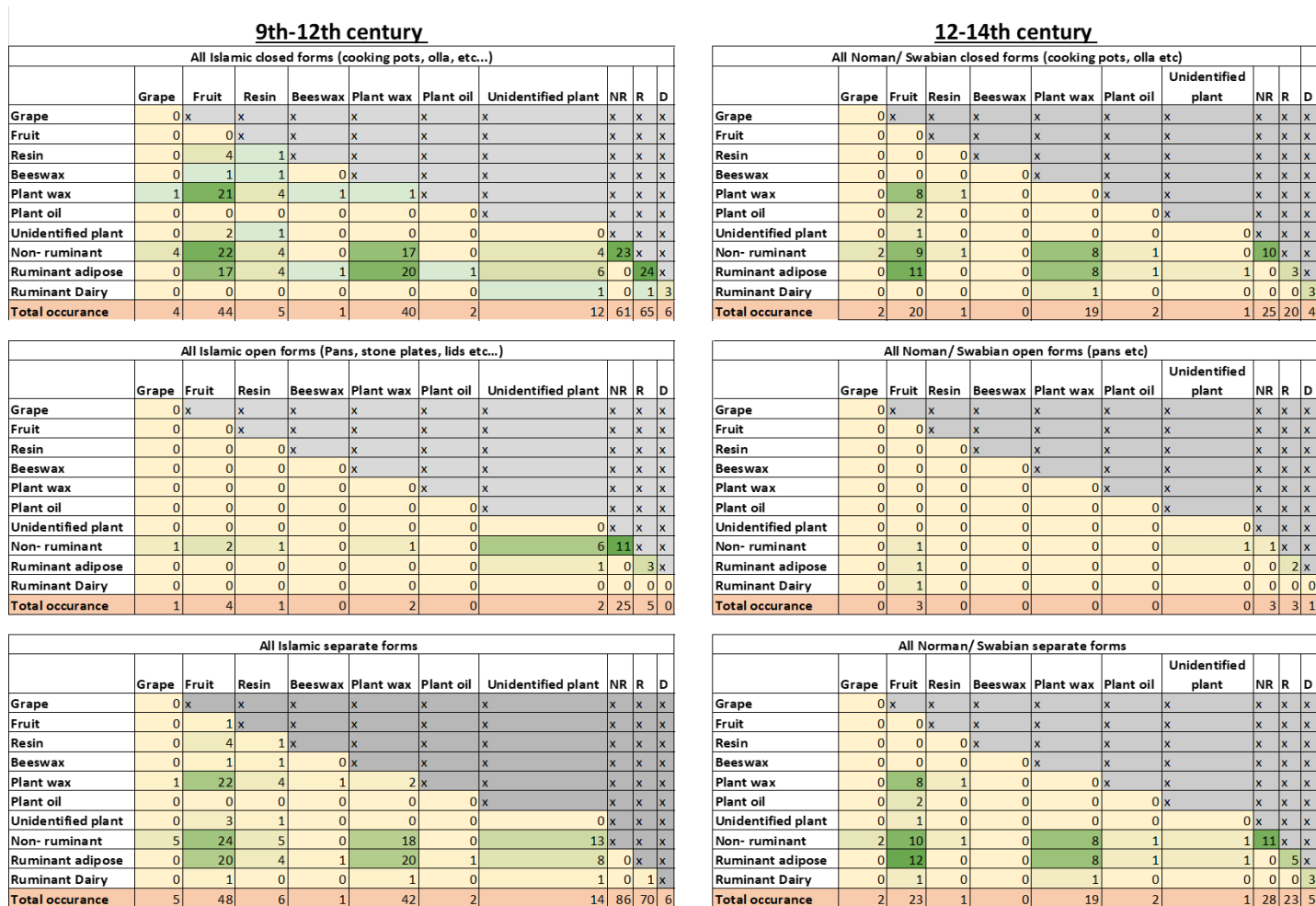


Figure 49: Mixing matrices of open and closed form vessels from 9<sup>th</sup>-12<sup>th</sup> century and 12<sup>th</sup>-13<sup>th</sup> century contexts from all sites. Each number represents the number of times two products are identified together and the total occurrence shows the total number of vessels that yielded evidence of that product.

Thus, whilst it is not possible through the use of organic residue analysis to determine specific recipes processed in cooking pots, it is possible through the analysis of trends of the processing of different products to gain insights into the functions of these ceramics and the selection of certain resources for processing. Future investigations on a wider range and larger sample size of different vessel forms may further aid our understanding of specific vessel and resource use.

## **7.7 Stable isotope analysis of human and animal bone collagen**

### **7.7.1 Direct evidence of animal and plant consumption through bulk stable isotope analysis of human remains from Islamic Palermo**

As shown, organic residue analysis of the ceramic vessels has yielded important evidence of the processing of animal and plant products. However, an important caveat with these datasets is that they only assume these products were consumed by the populations at these sites and products in ceramic vessels may not necessarily represent everyday cuisines. Furthermore, the identification of food sources in ceramic vessels only indicates one way of processing foods and does not account for other ways in which foods were prepared and consumed (e.g., roasting, smoking, pickling, drying or even just eaten raw). Alice Ughi as part of her forthcoming PhD research and as part of the wider Sicily in Transition project investigated direct diet of populations in medieval Sicily through carbon ( $\delta^{13}\text{C}$ ) and nitrogen ( $\delta^{15}\text{N}$ ) isotope data of human and faunal remains (Ughi and Alexander forthcoming; Ughi unpublished). Here, the carbon ( $\delta^{13}\text{C}$ ) and nitrogen ( $\delta^{15}\text{N}$ ) isotope data of human and faunal remains from sites in Palermo are compared to the ORA results presented in this thesis to better understand the consumption of animal products in Palermo as direct evidence of diet.

The human remains come from the sites of Castello San Pietro (CSP) and La Gancia Church (GA) and Oratorio dei Bianchi (OB). These remains have been radiocarbon dated between 7<sup>th</sup>-10<sup>th</sup> century at CSP, 8<sup>th</sup>-11<sup>th</sup> century at GA and 8<sup>th</sup>-10<sup>th</sup> at OB

(Appendix L). The  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  values of faunal bone collagen are used to establish isotopic base lines and are essential for understanding human consumption of these animal, but it also gives important insight into the consumption practices of these animals. The faunal remains are from two Islamic sites in Palermo (Corso dei Mille and Sant 'Antonino) (Appendix L). The  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  values of human and faunal bone collagen from Palermo sites are plotted in figure 50.

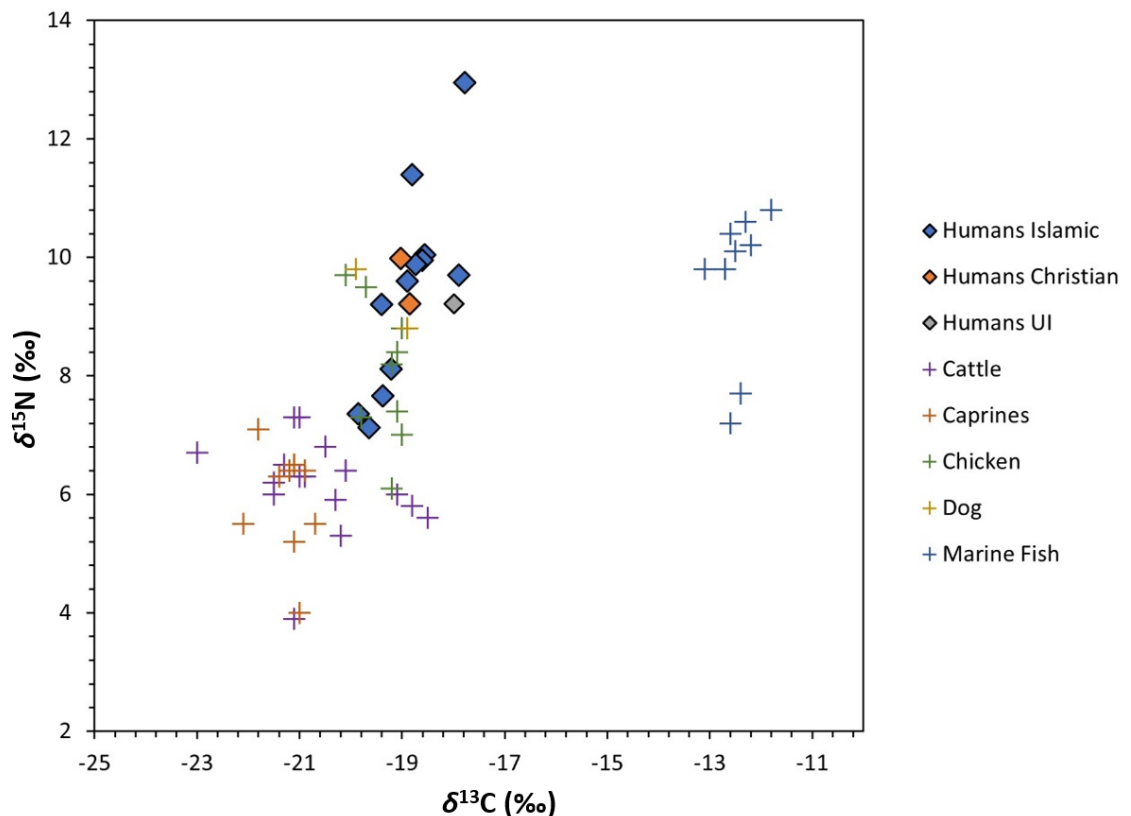


Figure 50: Scatterplot of bulk collagen  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  stable isotope values of faunal (dog ( $n=2$ ), cattle ( $n=16$ ), fish ( $n=10$ ), chicken( $n=10$ ), and caprines ( $n=13$ )) and human bone collagen from sites of Castello San Pietro (CSP) ( $n=4$ ), La Gancia (GA) ( $n=8$ ) and Oratorio dei Bianchi (OB) ( $n=3$ ), Palermo. The human values are separated by their faith, which was concluded from their burial position relating to either Islamic, non-Islamic/unidentified and Christian.

The  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  ratios of human remains at the sites of Palermo indicate that the individuals consumed predominantly  $\text{C}_3$  terrestrial diets (Figure 50). The  $\delta^{13}\text{C}$  values for humans were shown to remain relatively consistent amongst the population which signifies a predominantly  $\text{C}_3$  diet amongst all individuals regardless of site, sex or faith (Ughi unpublished). These results are consistent with the organic residues identified in the domestic containers at these sites, whereby both terrestrial animal products were identified. Some samples had relatively high nitrogen values perhaps indicating

more animal protein in the diet of some individuals at the site. The diverse range of  $\delta^{15}\text{N}$  values of chickens (6.1-9.5 ‰) supports the suggestion that, to some extent, chicken meat was being consumed by these populations and could have been a more significant part of the diet of those individuals with higher nitrogen values. However, it must be noted that a differentiation between meat and egg protein is not possible. On the other hand, at least four individuals have relatively lower  $\delta^{15}\text{N}$  values (median value of  $8.65 \text{ ‰} \pm 0.8$ ) indicating that  $\text{C}_3$  plant foods were a major staple for at least some of the population with either restricted access to, or a lesser preference for meat and fish. The large range of  $\delta^{15}\text{N}$  values reflects variable diets at Palermo, either due to differential access or selectivity of foods among these populations, which is not possible to identify through the contents of ceramic containers. However no statistical difference between  $\delta^{15}\text{N}$  values, as shown by a Kruskal Wallance test, was observed between sex (female, male and unidentified) ( $\chi^2(2) = 3.66, p - \text{value} = 0.16$ ) or burial rite (Islamic, Christian and unidentified) ( $\chi^2(1) = 0.75, p - \text{value} = 0.39$ ), suggesting that other factors may have influenced these food choices. Factors such as occupation, status, locality or individual choice could influence diet

Furthermore, lack of evidence of aquatic resources in ceramic samples, has led to the assumption that either fish was not held in high regard in Islamic cuisine or that fish products were being processed and consumed in other ways. However, the  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  ratios of human remains also shows that fish was not consumed at least in high proportions amongst individuals in Islamic Palermo (Figure 50) (Ughi unpublished). It is important to note that bulk  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  values may not detect more minor protein contributions in the diet, and therefore it is not precluded that aquatic products were incorporated in cuisines, but they still imply that fish did not make up a major part of the diet. For example, bulk  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  values of human collagen from individuals at the site of Segesta and Agrigento in Sicily were reflective of a purely terrestrial diet with no evidence of fish consumption. However, recent compound-specific amino acid analysis of the same individuals indicated that in a number of individuals, a significant percentage (~30%) of their protein intake came from marine sources (Ughi unpublished). This work highlights that caution needs to be taken when considering bulk isotopic values as they represent a long-term average of the major protein sources

in the diet and if the protein source is inadequate (<20% of overall contribution) these will likely be undetectable (Hedges 2004; Craig et al. 2013b).

Stable isotope values of collagen and  $\delta^{13}\text{C}$  fatty acids in pottery samples reflect differential consumptions of foods. Bulk stable isotope values of human collagen reflect habitual diets over an average of many years before death (though weighted toward adolescence), whereas evidence obtained from cooking pots provides more specific insights into consumption. The stable isotope analysis supports evidence from the cooking pots that both plant products and animal products were regularly consumed by these populations, whilst fish did not make up a considerable proportion of diet. Evidence of plant-based diets amongst the individuals at Palermo sites provides further evidence that the large amount of non-ruminant fats identified in the ceramic containers, and the discrepancy with the faunal record, could be assigned to  $\text{C}_3$  plant foods.

### **7.7.2 Bulk stable isotope analysis of faunal remains**

Furthermore, stable  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  isotope analysis of animal remains undertaken by Ughi, from all sites studied in this thesis (Palermo, MZ and CLESP), show that most of the animals have a  $\text{C}_3$  diet, which may include wheat, barley and grasses which is consistent with the  $\delta^{13}\text{C}_{\text{FA}}$  values  $\text{C}_3$  signals in the ceramic samples (Ughi unpublished). Interestingly, two cattle specimens from Islamic contexts at MZ yielded enriched  $\delta^{13}\text{C}$  values (14.8‰ and 14.6‰) indicative of the consumption of  $\text{C}_4$  plants (Ughi and Alexander forthcoming; Ughi unpublished). Although this may provide indirect evidence of the presence of millet, this assertion has been deemed unlikely due to the absence of evidence elsewhere and is in agreement with the absence of millet biomarkers in the ceramic containers (Ughi and Alexander forthcoming; Lundy et al. 2021). Furthermore, these results may indicate the consumption of the  $\text{C}_4$  plant sorghum, used as animal fodder, which is listed as one of the 'revolution plants' by Watson (1974, 1983) yet, as with millet, evidence of sorghum has only been attested in Northern Italy and has not yet been identified in medieval Sicily (Ughi and Alexander forthcoming). Therefore, the enriched  $\delta^{13}\text{C}$  values have been attributed to the

consumption of sugarcane leaves, suggesting the secondary use of sugarcane for animal fodder or the waste products of sugar cane which supports textual and material evidence for the production of sugar in Sicily at this time (Ughi and Alexander forthcoming; Canzonieri and Vassallo 2014; Ardizzone, Pezzini and Sacco 2018; Decker 2009). This does not directly provide insight into the use of this commodity in cuisines, and it is not possible to detect the presence of sugar in archaeological ceramics. A pilot study aimed to extract and analyse organic residues from 'sugar pots' found at Corso dei Mille, Palermo, but as expected no evidence of sugar could be identified (Carver, *Pers comms*). Nonetheless, other than these two examples from Mazara, consumption of C<sub>4</sub> amongst animals and human populations is very limited, which again supports the predominant processing of C<sub>3</sub> plant products and C<sub>3</sub>-fed animals in the ceramic vessels.

## 7.8 Summary

This chapter combined the results of ORA of cooking pots with other archaeological evidence of food consumption analysed as part of the Sicily in Transition project to gain deeper insight into the foodways of the populations that used these ceramic vessels. The combination of ORA of both domestic and transport vessels, zooarchaeology, archaeobotanical remains and carbon and nitrogen stable isotope analysis has shed important light not only on the food accessible to people in Sicily during the 9<sup>th</sup>-14<sup>th</sup> centuries, but how these products were selected, prepared, combined and consumed. The main conclusions are outlined as follows:

- Both faunal remains and  $\delta^{13}\text{C}_{\text{FA}}$  values of ceramic samples show that at all sites and chronological periods, ruminant meats contributed significantly to cuisines and were selected for processing in ceramic vessels. In Islamic contexts, at least, lamb and mutton were likely more highly regarded meats than beef. Evidence of ruminant adipose fats in the ceramic containers, particularly in closed forms such as *ollae* and cooking pots, provides important evidence that meat was boiled and likely combined with vegetables as stews.

- Clear evidence for the processing of dairy products in ceramics from Casale San Pietro and Mazara supports the faunal evidence that ruminant animals were utilised for both meat and milk at these sites.
- An over-representation of  $\delta^{13}\text{C}_{\text{FA}}$  values that fell within the non-ruminant range may be explained by specific selection of non-ruminant adipose products such as pork or chicken, but also likely reflect the high levels of mixing of animal products and plant products in these vessels.
- In general, there is no clear difference in the processing of animal products in ceramic vessels between chronological periods based on the  $\delta^{13}\text{C}_{\text{FA}}$  values alone. However, the faunal evidence suggests that there may have been a shift in the importance of different animals such as chicken and pork in the later Norman and Swabian periods and different animal management practices that could not be observed in the ceramics.
- Although the archaeobotanical remains and ORA of domestic cooking pots provide evidence of different products, the two lines of evidence are also complementary of each other. The identification of *Brassica*, *Allium* and potential fennel in the ceramic containers, which could not be unambiguously identified in the archaeobotanical record indicates the advantage of ORA in identifying the processing of non-dense vegetable products. In contrast, evidence of spinach and aubergine in the archaeobotanical record, provides important information about the availability of these resources that could not be identified using ORA alone. Together, both the ORA and archaeobotanical evidence suggests a diachronic decline in range of vegetable products available (archaeobotanical evidence) and used (ORA of ceramics) in the Norman and Swabian to Aragonese periods.
- Grapevine products identified in the ceramic containers provide important insight about their incorporation into cuisines. Particularly in cooking pots from Islamic contexts at Casale San Pietro where grapevine products were not identified in the archaeobotanical record or in transport amphorae.

Furthermore, whilst it is not possible to be specific about the fruit products identified in the ceramic containers, archaeobotanical evidence supports the fact that a wide range of fruits were available at these sites and processed in ceramic vessels as part of recipes in all chronological periods.

- It is not possible through the use of organic residue analysis to determine specific recipes through organic residue analysis of cooking pots, but through the analysis of trends of the processing of different products it was possible to gain insight into the functions of these ceramics and the selection of certain resources for processing. Of note, the regular mixing of animal, vegetable and fruit products supports the notion that *ollae* and cooking pots were used for processing stews and pottages. Whereas more open forms, for example stone plates and pans, appear to have been used for the processing of a more restricted range of products. These trends appear to continue from the Islamic periods to the later Norman, Swabian and Aragonese periods. This indicated that, despite some typological changes between these periods the general functions of the basic forms remains the same.
- Stable isotope analysis of human remains from Palermo provides further evidence for the importance of C<sub>3</sub> terrestrial plant and animal products in Islamic diets which complements the high occurrence of plant and animal fats identified in the ceramics from these sites. The diversity of diet amongst the populations is not clearly defined by faith, sex or site, lending to the idea that other influences may have had an impact on specific diets in these populations. The predominantly more plant-based diet observed in at least four individuals suggests that, whilst plant-products were undoubtedly a dietary staple for all individuals, for some they may have had a much more significant role. The limited evidence for fish consumption amongst these individuals supports the absence of aquatics found in the ceramic vessels. However, future investigations using compound specific amino analysis may help to better identify lesser contributions of fish products in the diet, not observable through bulk analysis.



- A predominantly C<sub>3</sub> plant diet observed at all sites in the stable isotope analysis of animals supports the C<sub>3</sub> signals observed in the ceramic containers. However, C<sub>4</sub> consumption of at least two cattle from Mazara provides important information about the availability of C<sub>4</sub> plants, such as waste sugar cane, not observed in the archaeobotanical record or ceramic vessels.

In the next chapter of this thesis the results of this research are summarised with respect to the main research questions set out at the beginning of this thesis. There then follows a further discussion about how future avenues can better aid our understanding of the use of ceramic vessels and foodways as a whole.

# Chapter 8 Conclusions

## 8.1 Overview of thesis rationale and aims

From the 9<sup>th</sup> the 14<sup>th</sup> centuries AD Sicily experienced a number of rapid and quite radical changes in regimes. During this time, Sicily was first under the control of the Aghlabid and Kalbid dynasties in the 9<sup>th</sup>-11<sup>th</sup> century, then ruled by the Normans in the 12<sup>th</sup> century and then the Swabians in the 13<sup>th</sup> and finally ruled by the crown of Aragon in the 14<sup>th</sup> century. The impact of these transitions in regime to the lifeways of the populations that experienced them is a complex subject, which required careful investigation and examination to be understood. This thesis used the concept of cuisine, the ways in which foods are prepared combined and consumed, as a proxy for understanding the lives of the general population of Sicily during these regimes. To do this, this research set out to understand the use of domestic containers, as utilitarian artifacts expected to capture a significant subset of daily foods processed throughout the 9<sup>th</sup>-14<sup>th</sup> centuries in both rural and urban environments.

Here, an overview of the findings of this research are outlined in relation to the five main research questions set out at the beginning of this thesis. These questions were:

- What impact do post firing treatments have on the identification of organic residues from ceramic containers?
- Is it possible to identify Watson's revolution plants in archaeological ceramics using organic residue analysis?
- Were the contents of domestic containers changed or maintained as a result of transitions in regimes from the 9<sup>th</sup>-14<sup>th</sup> century?
- How does the use of domestic containers compare between urban and rural settlements under the same regime and/or transition?
- What can we learn about foodways in medieval Sicily by integrating organic residue analysis with other archaeological evidence of food?

This chapter will then discuss the limitations of this research and prospects for future research before stating the final concluding remarks.

## **8.2 What impact do post-firing treatments have on the identification of organic residues from ceramics containers?**

As set out in Chapter 3 of this thesis, an inherent problem in organic residue analysis is the limited ability to distinguish mixed lipid signals in archaeological vessels. Without the presence of charred food remains, understanding whether mixtures identified in ceramics are due to multiple sequential cooking events of different products or intentional mixtures as part of a recipe, is complex. Further difficulties arise when post-firing treatments using organic products are used, such as sealants or polishes to reduce permeability before cooking or storing organic products in the vessel. To understand the impact of post-firing treatments on our ability to interpret organic residues from archaeological ceramics, experimental cooking pots that had been treated with post-firing treatments and supplied by the University of Innsbruck (Haas 2018), were analysed using a multi-faceted organic residue approach as part of this research.

The results of the organic residue analysis, presented in Chapter 4, showed that the use of post-firing treatments to seal or polish the ceramic before cooking or pouring commodities had a variable impact on the ability to interpret the organic residues. It was shown that products used for sealing the ceramic vessel have a mixed impact on the overall lipid yield and molecular profiles observed when different products are either cooked or poured into the ceramics. In some cases, the post-firing treatment completely masked the contents of the vessel, as was the case for beef tallow used as a sealant that completely masked the lipid profile of vegetable products that were cooked in the ceramics. This confirmed, in agreement with other studies that plant products yield very low concentrations of lipids in contrast to animal fats (Evershed 2008b, 2008a; Steele, Stern and Stott 2010; Hammann and Cramp 2018). However, in other cases the use of a post-firing treatment was not identified if other products were cooked or poured inside the vessels. For example, milk used as a sealant was only

identifiable when either no additional products or vegetable products were processed inside the vessel. The presence of lipid profiles relating to the commodities cooked or poured in the ceramic vessel, despite the use of a sealant, showed that only partial waterproofing of the vessel was achieved with the sealant as previously shown (Drieu, Lepère and Regert 2020). Furthermore, a higher concentration of tartaric acid extracted from the vessels treated with beef tallow and resin, in comparison to samples without a sealant, highlighted that the resin may play a role in protecting the tartaric acid inside the ceramic matrix. Of interest, thermal degradation markers (ketones and APAAs) were only identified in cases where a post-firing treatment was used, likely formed due to the higher temperatures reached when the organic is in contact with the ceramic surface. This led to the conclusion that, whilst it is not possible to use ketones or APAAs to distinguish between post-firing treatments and vessel contents, consideration should be given when these are observed in ceramic samples.

The main conclusion of these experiments was that post-firing treatments have varying impacts on the organic residues identified in ceramic containers and, in archaeological contexts, it would not be possible to distinguish between organic used as sealants and those used to process in the vessel. However, this investigation further highlighted the complexity of lipid residues identified in archaeological ceramics and that the commodities identified through lipid analysis may indicate the sealant rather than those processed in the vessels for culinary purposes or a mixture of both. Furthermore, these results shed some light on the complexities surrounding the differential absorption, survival and thus extraction of different products, which is influenced by various factors. Here, it was only possible to correctly identify contents of the ceramic by using a multi-faceted approach as the sealant had variable impact on: TAG distributions, fatty acid profiles and on the isotopic values and it was expected that the identification of products would be more complex in archaeological contexts because of degradation and alteration of lipid profile. This investigation therefore put forward the importance of combining different extraction methods and both isotopic and molecular data together in order to untangle complex mixtures in archaeological ceramics, as was the approach in this thesis. Finally, this investigation enabled reflection on the complex processes that lead to the organic residues that are observed in the archaeological record. This reflection was essential in the interpretation of the

contents of the archaeological ceramics studied in this thesis and ensured a rigorous and cautionary approach was taken before making definite conclusions whereby interpretations.

### **8.3 Is it possible to identify Watson's revolution plants in ceramic containers using organic residue analysis?**

A major consideration in understanding cuisine in Sicily from the 9<sup>th</sup> century onwards was the use of plant products, particularly the 'revolution plants' proposed by Watson to have arrived in the Mediterranean with the Arabs during the Islamic conquest (Watson 1974, 1983). Therefore, to aid the archaeological questions of this investigation organic residue analysis of aubergine, spinach and lemon (thought to have appeared/developed in Sicily as part of the 'Islamic green revolution' (Watson 1974, 1983) was performed to assess their lipid profiles and identify specific biomarkers that may be preserved and identified in archaeological ceramics. Additionally, other plant products; Giri di Campania, kale, leek, onion and carrot, were also analysed. The analysis revealed specific profiles for plants from the genus *Brassica* (cabbage, kale and Giri di Campania) and leek in agreement with published literature. Spinach revealed a specific sterol signature composed of  $\alpha$ -spinasterol and 7-stigmastenol, alongside *n*-alkane C<sub>31</sub> and *n*-alcohols C<sub>22</sub>-C<sub>26</sub>, where C<sub>26</sub> is dominant. No specific profile that would be observed in the archaeological record could be identified for any of the other plants analysed e.g., aubergine and lemon.

Cabbage, leek and spinach were processed in replica ceramic vessels and the ceramic sherds were buried in Eze, South of France for one year. However, the conditions of the experiments performed in this research were limited (e.g., low cooking temperatures and short cooking time, see Chapter 4). In these experiments, the specific sterols of Spinach were not absorbed into the ceramic vessel, which highlighted that plant products have to be processed for a prolonged period or over multiple cooking events to successfully absorb into the ceramic matrix due to their low lipid content. Cabbage and leek did yield specific *n*-alkanes, *n*-alcohols and ketones as shown in the ceramic matrix after cooking. However, after burial, whilst the specific profile of leek could be recovered, cabbage could not be detected. This highlighted that these molecules can

readily be lost to degradation in the burial environment even after one year. Again, this showed that when specific profiles of *Brassica* are identified in archaeological ceramics it reflects the intensive processing of these products. In this thesis, spinach could not be identified, despite targeting specific sterols in the analysis, in any of the archaeological ceramics investigated. However, profiles of brassicas and leek were readily identifiable. It is not possible to determine whether the absence of spinach biomarkers in the archaeological vessels is because it was not processed in the vessels or due to limitations in the absorption of sterols into the ceramic matrix, the latter, due to alteration through cooking or degradation in the burial environment.

This investigation highlighted the need for further experiments to understand the absorption and survival of sterols specific to spinach. Furthermore, it highlighted the inherent issues with identifying plant products in archaeological ceramics. In this thesis, it was important to consider that, despite lack of chemical signatures in the vessels analysed, products may have been processed in the ceramic vessels but were either 1) not absorbed, 2) altered through cooking, 3) did not survive in the burial environment and 4) could not be detected through limitations in the analytical methods used.

## **8.4 Were the contents of domestic containers changed or maintained as a result of transitions in regimes from the 9<sup>th</sup>-14<sup>th</sup> centuries?**

The period of the 9<sup>th</sup>-14<sup>th</sup> centuries AD represented a complex period for Sicily as the island saw rise to new religion, political and socio-economic ideas, and new people. It was hypothesised that the complexity of these regimes and transitions was likely reflected in the culinary practices of the people that experienced them. To assess how culinary practices may have been changed or maintained over these periods of transition 248 domestic containers from five sites dated to the 9<sup>th</sup>-14<sup>th</sup> centuries were analysed using a multi-faceted organic residue analysis approach in order to gain insight into how foods were combined and prepared.

The results reported in Chapter 6 showed that, despite significant transformations in political power over the island, little change was observed in the main commodities processed in pottery vessels through time. Substantial mixing of commodities such as, animal fats, vegetable and fruit products was identified, particularly in closed vessels such as *ollae* and cooking pots, regardless of chronological period and stylistic changes. It appears that throughout the 9<sup>th</sup>-14<sup>th</sup> centuries, closed forms maintained their function as containers for processing stews and pottages with combinations of meat, vegetables, and fruit products prepared in the same vessel. Although, as noted, it is difficult to distinguish between a single cooking event and subsequent processing of different commodities, it was concluded that due to the concurrent trends in mixtures observed in many vessels, both scenarios likely accounted for the mixed signal observed. In contrast, more open forms, in all periods, appear to have been used for the processing of a more restricted range of products, mainly unidentified non-ruminant products. Thus, despite some typological changes between these periods, the general functions of the basic forms remains the same.

In general, no significant difference of the processing of animal products in ceramic vessels between chronological periods were observed based on the  $\delta^{13}\text{C}_{\text{FA}}$  values alone, despite slight variations between sites as discussed in the next section. Of particular note, it was not possible to observe a preference for the selection and processing of pork products in the ceramic vessels between periods. This meant that it was not possible to assert whether a transition from Islamic politic rule to Christian rule (under the Normans, Swabians and Aragonese periods) had an influence on food preferences in these communities (at least in terms of the consumption of pork). However, other research has indicated that an assessment of the presence/absence of pork consumption amongst these populations is far from straight forward (Aniceti 2020; Castrorao Barba et al. 2021; García-García 2019; Aniceti and Albarella 2022). Furthermore, the identification of non-ruminant products based on  $\delta^{13}\text{C}_{\text{FA}}$  values was hindered due to a lack of region-specific modern reference values and mixed signals. It was concluded that the influence of C<sub>3</sub> plant products to these values cannot be dismissed, despite not being able to identify a clear link between  $\delta^{13}\text{C}_{\text{FA}}$  values and those samples that yielded biomarkers indicative of plant products.

Nonetheless, the continued use of meat products, vegetable products and fruit products reflects at least some continuation of culinary habits in Islamic-Aragonese Sicily. These results generally reflect the multi-cultural society that existed in Sicily and the continuation of agricultural and shared food practices throughout the 9<sup>th</sup> to at least the 12<sup>th</sup> century as described in the literature (Chapter 2). Indeed, the prevalence of basic pottery forms from the preceding Islamic contexts observed in Norman contexts has been attributed to the fact that pottery production in this period remained in the hands of the Muslim majority (Alfano and Sacco 2014). Thus, it is not surprising that these forms retained their culinary purpose from preceding periods. Furthermore, despite a clear shift in production of pottery types in the 13<sup>th</sup>-14<sup>th</sup> centuries, notably the widespread introduction of partially glazed wares from Messina and the abandonment of cooking wares with handles (Molinari 2012, 2010), the basic uses of these forms appear to remain the same.

However, a noticeable decrease in the variety of plant products was observed in the ceramic samples analysed through time, from both Mazara and Casale San Pietro. At the former site, evidence of *Brassica*, *Allium*, plant oil and unidentified products were identified in Islamic containers, but in Swabian contexts only *Brassica* and some unidentified plant products were observed. Of similar effect in Casale San Pietro, a biomarker that could indicate the presence of fennel, plant oil and unidentified plants were identified in Islamic domestic containers, but this was reduced and in Swabian contexts and only a biomarker indicative of fennel were observed in the domestic containers. This might represent a decrease in the availability of products in later post-Islamic periods or changes in the selectivity of plant products in recipes. A decrease in the availability of products may be due to a shift in agricultural practices from the start of the 13<sup>th</sup> century, whereby focus shifted back to the production of wheat, olive oil and wine (Booms and Higgs 2016). It is possible that these results begin to hint to an abandonment of well rooted cultural, social and culinary traditions at the start of the 13<sup>th</sup> century as proposed by (Molinari 2012, 2010). However, a larger sample size from the 13<sup>th</sup> to 14<sup>th</sup> centuries should be carried out in the future to further understand this trend.



An important caveat is that other factors such as degradation of identifiable plant biomarkers may have affected their identification. These plant products still existed in the culinary repertoire of later populations, but were processed and consumed in other ways not observable in the ceramic containers. Furthermore, it is important to note that due to the limitations of the absorption, survival and detailed taxonomic identification in organic residue analysis, more discrete changes could have occurred in the processing of different animal species, vegetable products or fruits exploited, for example, that are not visible by molecular and isotopic analyses. Culinary recipes are formed not just from the main products used, but their ascetics, texture and taste also. Spices and flavourings, for example, likely differentiated dishes cooked in these ceramic vessels despite the use of key unchanged ingredients (meat and vegetable bases), but their presence is difficult to detect via organic residue analysis.

Nonetheless, for the first time, this study has investigated cuisine across the transition from Islamic political control to post Islamic regimes of the Norman, Swabians and Aragonese periods in Sicily. Focusing on cooking wares from domestic contexts in Sicily has enabled insight into the culinary habits of the general population during these regimes. As part of this research, it was important to understand how cuisine may have been changed or maintained diachronically, but also to compare between different socio-economic settings under these regimes. The conclusions of these results are discussed in the next section.

## **8.5 How does the use of domestic containers compare between urban and rural sites under the same regime/transition?**

An assessment of the use of domestic containers in different socio-economic settings was an essential part of this thesis' research. As highlighted throughout, culinary habits are often linked to different socio-economic and cultural factors such as food availability and the foodscape, wealth, faith, and societal value. It has been proposed that the impact of the regimes and transitions in the 9<sup>th</sup>-14<sup>th</sup> centuries likely differed in

different socio-economic settings (Carver, Fiorentino and Molinari 2019; Molinari 2015). Therefore, to gain insight into the impact of these regimes and transitions in different socio-economic settings from a culinary perspective, the use of pottery was compared between both urban and rural settings (town and country). This question was first tackled in Chapter 5 of this thesis by comparing the contents of domestic containers from three 9<sup>th</sup>-11<sup>th</sup> century sites in the urban capital of Palermo and 10<sup>th</sup>-12<sup>th</sup> contexts in the rural site of Casale San Pietro. In Chapter 6, comparison was then made between Palermo, Casale San Pietro and Mazara del Vallo to widen the discussion of how culinary habits compared between sites during the period of Islamic political rule. Further comparison was made between Casale and Mazara in post-Islamic periods of Norman, Swabian and Aragonese rule.

Firstly, on comparison of the contents of 114 domestic containers from urban Palermo and rural Casale San Pietro, using a multifaceted organic residue approach, animal fat, plant products, fruit products and beeswax were identified, often with complex mixtures of various products at all sites. The role of *ollae* and cooking pots were fundamentally similar at these sites. The ORA showed that these vessels were used for processing a variety of products as part of stews or pottages, and mixtures of fruits, meats and vegetables, which generally reflects the sweet and salty dishes noted in Arabic culinary literature (Zaouali 2009; Perry 2005; Lewicka 2011; Peterson 1980). The similarity in the uses of *ollae*, at least those of Palermo type production, between Palermo and Casale San Pietro further indicates that the rural site had close connections with the capital at the time. These connections not only exist in the exchange of material culture but also likely in the transfer of ideas of the ruling regime reflected in the shared culinary habits of these populations. However, some distinct differences were observed between the two sites, which highlighted independent culinary practices between town and country. For example, the presence of dairy products in calcite wares from CLESP led to the conclusion that there was a preference of these specific wares for the manipulation of dairy products. In contrast, no dairy products were identified in Palermo production wares at the same site, as well as the absence of dairy identified in all sites from Palermo. These results do not preclude that dairy products were not consumed at the urban sites, but may have already been processed (i.e., into cheese) and are less visible in the ceramic containers as previously

suggested (Dunne et al. 2021). It was therefore possible to assume that dairy products were processed into cheese or ghee as part of the rural economy and supplied to the urban capital. This highlighted both a preference of specific vessels for the processing of dairy products, differential access to products in town and country, as well as the role of rural settlements within the wider economic landscape of Islamic Sicily.

Furthermore, differences were also observed in the use of plant products between these sites. For example, a biomarker indicative of fennel (but not specific to) was only observed at the rural site of Casale San Pietro, where it grows prolifically today. Additionally, grapevine products were more readily observed in ceramic samples at the rural site. The latter opened questions regarding the availability and use of grapevine products at the rural site. If used as wine, this may reflect a lesser restriction on the consumption of wine, as part of Islamic hadiths, in rural communities compared to in urban centres. However, these results may represent the use of other grapevine products such as vinegar for preserving or flavouring, or grape syrups and juices. It is possible to assume that vinegar as a preserving method would have been more necessary to store foods for a long period of time in the rural site where access to fresh foods were less readily available (Pitchon 2020). On the other hand, evidence of other fruit products (e.g., such as apple, plum, cherry or peach), were more readily identified in domestic wares from sites in Palermo which highlighted that there may have been a wider access to different fruit products at the urban sites in comparison to the rural settlement.

Additionally, the presence of pine products, only identified in samples from Palermo, indicated differences in technological treatments of ceramics. Pine resin might have been used to waterproof porous vessels, which would likely have acted also a flavouring given its low melting point. However, it was suggested that this could also indicate the reuse of ceramic vessels for the processing of pine products for other technological uses such as for waterproofing boats in the nearby ports, as suggested in other studies (Pecci and Grassi-Munibe 2016; Salvini, Pecci and Giorgi 2008; Buonincontri et al. 2017). Beeswax products were also identified in one sample from the Palermo site of Castello San Pietro with mixtures of plant products and ruminant fat in the vessel. However, it is not possible to discern if signals or beeswax indicate its

use for technological purposes such as waterproofing the vessel or if honey, used a sweetening product, was present in the vessel. However, regardless of the use of pine products and beeswax products, these results further indicated the differences in resource use between town and country, likely as a result of differential access to these products, but also requirements for their use (e.g., the necessity to process pitch to supply the port in Palermo). Additionally, if pine products and beeswax were used as waterproofing technologies, this raises questions of how the porous vessels in the rural settlement might have been sealed, if at all. It is reasonable to suggest that without access to pine resins or beeswax, animal fats may have instead been used. As demonstrated in Chapter 4, it is not possible to distinguish the use of animal fats as a sealant or culinary use. However, it is unlikely that animal fats would have been used solely for sealing domestic containers at this site and therefore a culinary use was concluded. Finally, signals of beeswax and pine resins in the cooking vessels could be associated vessel with preserved ingredients stored in other vessels. These results, therefore, could proximally reflect differences in the storage and/or treatments of products at these sites.

When the ORA results were compared to those from Islamic contexts at Mazara del Vallo, it was not appropriate to simplify these results based on a clear urban versus rural comparison. Whilst both Mazara and Palermo are urban sites, it had to be considered that these two centres could have had different roles within Islamic Sicily. This is certainly indicated in the distinct pottery assemblage at Mazara del Vallo whereby the production of domestic containers is largely independent from Palermo in all chronological periods, despite some similarity in shared forms (Meo and Orecchioni 2020). Nonetheless, some comparisons could be made. For example, the samples from Mazara also showed a more frequent occurrence of fruit products, other than grapevine, in the ceramic containers compared to Casale San Pietro and no samples from Islamic contexts in Mazara showed evidence of grapevine products in the vessels. This further supports differential access of fruit products between urban and rural sites and solidifies the distinct use of grapevine products at Casale San Pietro. However, in contrast to the result observed from Palermo samples, dairy products were identified in Islamic containers from Mazara. This meant that it could no longer be concluded that dairy products were not processed in ceramic vessels at urban

centres. However, the concurrence of dairy and other plant and fruit products and their appearance in pans and jugs, indicated differential uses of dairy products, as part of recipes in Mazara compared to the processing of milk into processed cheeses or ghee in *ollae* and cooking pots at Casale San Pietro. Thus, the use of dairy products between urban and rural sites remains distinct.

An assessment of pottery use between town and country was more difficult in the later Norman, Swabian and Aragonese periods due to the differences of sample size, the absence of 12<sup>th</sup> century ceramics at Mazara, and the absence of post Islamic samples from the sites in Palermo. Nonetheless, on comparison of the contents of ceramics from Swabian contexts at Mazara to those from Norman and Swabian contexts at Casale San Pietro, both the similarities and differences observed in the preceding periods were still present in later periods. For example, at both sites, dairy was still present in samples from later periods and CLESP continued to show a higher presence of grapevine products in later periods compared to Mazara. Furthermore, both sites showed the same pattern of decline in the variety of plant products observed as described previously. Thus, the impacts of the transitions on the use of products appear consistent between sites.

Therefore, the investigation of pottery use between town and country indicated some level of shared culinary practices across western Sicily in Islamic, Norman and Swabian to Aragonese contexts, but also highlighted some distinct differences between sites. These differences were not only separated between the urban and rural sites but also varied between Palermo and Mazara del Vallo.

## **8.6 What can we learn about foodways in medieval Sicily by integrating organic residue analysis with other archaeological evidence?**

In this thesis, the use of organic residue analysis provided a powerful tool in identifying the use of pottery and revealed a wide range of products processed in domestic containers. This has provided important insight into what resources were available to these populations and how these were prepared and combined as part of cuisines. However, to gain a deeper insight into the foodways of these populations it was essential to place the results from the ORA study into the wider context of food consumption at these sites. Throughout this thesis, the results of the organic residue analysis have been interpreted with reflection of both historical and archaeological evidence of food. Additional to this, a more in-depth discussion was provided by combining the results of this thesis with zooarchaeological, archaeobotanical evidence, organic residue analysis of amphorae and storage containers and stable isotope analysis of faunal and human remains as part of the wider Sicily in Transition project (Chapter 7).

As discussed in chapter 7, in many cases the products identified in the domestic containers complimented evidence of what was available at these sites and provided direct chemical evidence for how these products were selected, prepared, and combined. For example, both the faunal remains and  $\delta^{13}\text{C}_{\text{FA}}$  values of ceramic samples showed that, at all sites and chronological periods, ruminant meats contributed significantly to cuisines and were selected for processing in ceramic vessels. Furthermore, evidence for the processing of dairy products in ceramics from Casale San Pietro and Mazara supported the faunal evidence that ruminant animals were utilised for both meat and milk at these sites. The identification of vegetable products and other plant products in the ceramic containers complimented evidence for a wide variety of these products available at the sites studied. However, the combination of evidence also enabled a more nuanced assessment of availability, selection and processing of products that could not be detected through organic residue analysis alone. For example, no clear difference of the processing of animal products in ceramic

vessels between chronological periods could be observed in the  $\delta^{13}\text{C}_{\text{FA}}$  values alone. However, the faunal evidence indicates that there may have been a shift in the importance of different animals such as chicken and pork in the later Norman and Swabian periods that could not be observed in the ceramics. The identification of *Brassica*, *Allium* and potentially fennel in the ceramic containers, that could not be unambiguously identified in the archaeobotanical record, highlighted the advantage of ORA in identifying the processing of non-dense vegetable products. Whereas evidence of spinach and aubergine in the archaeobotanical record, provides important information about the availability of these resources that could not be identified using ORA alone.

Stable isotope analysis of human remains from Palermo indicated the importance of  $\text{C}_3$  terrestrial plant and animal products in Islamic diet in contrast to a lesser importance of marine products, which complements the high occurrence of plant and animal fats, but absence of aquatic products identified in the ceramics from these sites. However, evidence of a more plant-based diet obtained from stable isotope analysis reflected differential diets amongst these populations that could not be identified through organic residue analysis. Whether this is the result of restricted access to foods or personal choice is unclear, but further highlights the advantage of combining ORA and stable isotope analysis for a complete insight into diet and cuisine.

Overall, this research has highlighted the importance of combining multiple lines of evidence of diet together to enable a comprehensive insight into foodways of populations in the past.

## **8.7 Directions for future research**

In the next section, some considerations of the main methodological challenges faced in this study are addressed and avenues for future research are suggested. Furthermore, implications for future research to understand both cuisine and lifeways in Sicily are proposed with focus on future sampling strategies and contextual considerations.

## 8.8 Methodological improvements and considerations

### 8.8.1 Problems with mixtures and the potential of mixing models

As highlighted throughout this thesis, mixtures of different commodities can have significant impact on the interpretation of commodities identified in ceramics through organic residue analysis. The combination of extraction methods and suite of analytical techniques used to identify both molecular and isotopic signals of the residues significantly aided this study and allowed multiple products to be distinguished in a single vessel. However, there were still limitations caused in the identification of products due to unresolved mixtures of products. In particular, it was difficult to understand the origin of non-ruminant  $\delta^{13}\text{C}_{\text{FA}}$  values and it was proposed that these could be impacted by a contribution of  $\text{C}_3$  plants. Whelton et al., 2021 suggested recently that samples that yield evidence of plant biomarkers should be excluded from GC-c-IRMS analysis because of these problems (Whelton et al. 2021). However, this would have significantly reduced the number of samples that could be analysed using this technique and implemented a bias assumption that those that did not yield specific markers of plant products were exempt from this effect. Indeed, the large majority of samples yielded P/S ratios indicative of animal fat products, but this was not enough to assert that other contributions were not influencing the  $\delta^{13}\text{C}_{\text{FA}}$  values.

Attempts have been made to distinguish  $\delta^{13}\text{C}_{\text{FA}}$  values as a result of mixtures of two products, such as non-ruminant adipose fats with ruminant adipose fats, based upon the relative concentration of each fatty acid established from modern reference samples (Mukherjee, Gibson and Evershed 2008; Craig et al. 2011). However, resolving  $\delta^{13}\text{C}_{\text{FA}}$  values influenced by a mixture of different products, particularly plant products, remains very difficult. The use of FRUITS, a Bayesian mixing model, has been used to estimate the relative contributions of multiple different food stuffs to resolve mixed signals (Fernandes et al. 2018; Hendy et al. 2018). However, this technique relies on an assumption of which products were combined, and knowledge of their  $\delta^{13}\text{C}_{\text{FA}}$  end-values. Therefore, future research could use the specific identifications in this thesis to create a more informed mixing model approach. Before future attempts are made to produce mixing models, it is essential that more local specific modern references are



collected and analysed to ensure that environmental and dietary factors influencing the  $\delta^{13}\text{C}$  values are reduced.

## 8.8.2 Specificity of products

A variety of different products were identified in the ceramic containers analysed in the thesis and, in many cases, it was possible to identify species-specific products. In terms of plant products, specific markers for the genus *Brassica*, *Allium* and a potential biomarker for fennel were identified as well as pine and birch bark products. Furthermore, compound specific stable isotope analyses helped to distinguish between ruminant adipose, ruminant dairy and, although tentative, non-ruminant fats. However, the ability to be more assertive about specific cuisines is hindered by the lack of species-specific products that could be identified. For example, it was not possible to identify any of the 'revolution plants' proposed by Watson in the ceramic vessels, limiting our understanding of how these may have been incorporated into cuisines. Furthermore, it was not possible to distinguish between animal species used for their meat or milk in the ceramic vessels. Although, in most cases, a mixture of vegetable, fruit and animal products remained consistent between chronological phases and socio-economic settings, more nuanced differences in the products utilised by these populations may have existed but were unidentifiable by the molecular and isotopic signatures.

### 8.8.2.1 Experiments

This thesis aimed to identify specific biomarkers of vegetable products, including those proposed in Watson's thesis such as spinach and aubergine (Chapter 4). Although a specific molecular profile was identified for spinach, by extracting lipids from the fresh vegetable, further cooking and degradation experiments were inconclusive. Ultimately, the experiments were limited due to a short cooking time and low heating conditions. Furthermore, in this thesis, the identification of ketone nonacosan-10-one was proposed as a possible biomarker for fennel based on modern plant studies

(Muckensturm et al. 1997). However, caution must be taken when assigning new biomarkers to a specific source. As Whelton (2021) suggests, in order to validate biomarkers in archaeological studies, rigorous analysis should take place (Whelton et al. 2021). Therefore, future investigations should aim to assess the absorption of lipids from fennel, spinach, and other vegetable products into the ceramic matrix as well as the reaction of these lipids through cooking and degradation after burial. This should be done through more rigorous, controlled, and well-defined experiments such as those previously performed to validate biomarkers indicative of *Brassica* (Evershed et al. 1995; Raven et al. 1997) .

### **8.8.2.2 Analytical techniques**

As shown in this thesis, the distribution of TAGs identified in archaeological ceramics can help to distinguish between animal fat sources such as non-ruminant and ruminant adipose fats and dairy products (Evershed et al. 2002; Regert 2011). However, GC-MS techniques are limited in their ability to identify low concentrations of TAGs in archaeological samples and is unable to target structural information of individual TAGs. Detailed information regarding TAG structures is highly beneficial as it has been shown to improve species identification of animals (Mirabaud, Rolando and Regert 2007) and plants (Garnier et al. 2002). Targeted analysis of TAG structures can be achieved through the application of Matrix-Assisted Laser Desorption (MALDI), high performance liquid chromatography (HPLC) or nano electrospray ionization MS (Nano ESI MS) (Mirabaud, Rolando and Regert 2007; Garnier et al. 2002). Therefore, future investigations using these techniques may help to better identify plant products in these ceramics and could help to distinguish between animal products processed in these vessels. The later would aid our understanding of which animals were preferred for meat and which were preferred for dairy exploitation for example.

### **8.8.2.3 The potential of proteomics**

Proteins are essential components of living organisms and, although more susceptible to degradation than lipids, can provide important information when found in

archaeological contexts. Proteomic analysis has begun to make significant contributions to the study of archaeological material. If present, proteins can provide taxonomic, and tissue specific identifications and studies of proteins extracted from archaeological ceramics have enabled the identification of specific plant products, aquatic animals, and terrestrial animals (Hendy et al. 2018; Solazzo et al. 2008; Chowdhury, Campbell and Buckley 2021). Applied to archaeological ceramics in Sicily proteomic analysis could greatly enhance our understanding of the contents of these ceramics and may provide better species-specific identifications. This could help identify more nuanced culinary uses not identified through lipid analysis. Furthermore, used in conjunction with lipid analysis, proteomics could help to validate certain lipid biomarkers, such as those used to identify fennel in this study and therefore improve future ORA studies. However, proteins are more susceptible to leaching in the burial environment than lipids and, important to this study, are impacted by the effects of heating through the cooking process (Evershed 1996). Proteomic studies of pottery from the Neolithic site of Çatalhöyük, Turkey (Hendy et al. 2018) and 2<sup>nd</sup> Millennium BCE site of Tell Khaiber, southern Iraq (Chowdhury, Campbell and Buckley 2021) has provided promising results demonstrating the survival of proteins in relatively arid conditions for thousands of years. However, considering the number of factors that can affect their survival, future work should investigate the possibility of protein preservation in medieval Sicilian cooking pots.

## **8.9 Samples, contexts, and implications for wider research**

With the aim to perform an in-depth, multifaceted study of pottery use in Sicily during the 9<sup>th</sup>-14<sup>th</sup> century, this thesis focused on five main sites in Western Sicily. A focus on lesser sites and a more restrictive time range ensured that no less than 20 ceramic sherds per geographical and chronological context were analysed using a number of different extraction methods and analytical techniques. By focusing on a select number of sites and chronological periods several commodities it was possible to identify several commodities using a multifaceted approach, whilst ensuring that a significant number of samples from each context were analysed. This enabled an in-depth interpretation and comparison of the uses of pottery vessels on a chronological,

geographical, and inter-site level. However, whilst this study has for the first time given important insight into culinary habits in Sicily through a large-scale multifaceted approach, there is still more to be learned about culinary habits in medieval Sicily. Future research should focus on increasing the range of chronological and geographical contexts to further understand the use of pottery across the Middle Ages and Sicily. Some suggestions follow.

The transition from the Byzantine Empire to the Islamic regimes was a significant, but complex period of transition. Although this thesis has provided important insight into culinary habits in Islamic ruled Sicily at multiple sites in both rural and urban settings, without information about culinary habits in preceding Byzantine contexts it was not possible to assess whether culinary habits were changed or maintained because of this transition. Future research should focus on organic residue analysis of pre-Islamic contexts and contexts of the transition period to assess the evolution and use of pottery in Sicily. The ability to do this is somewhat limited at present due to the current lack of identified domestic cooking wares from 8<sup>th</sup>-9<sup>th</sup> century contexts. However, an increase archaeological investigations and recovery of material culture from these contexts is promising for the recovery and identification of these ceramic types and formation of more refined chrono-typologies. Of note, radiocarbon dating as part of the Sicily in Transition project is aiding to untangle chronological sequences at sites across Sicily (Carver et al. forthcoming). This is particularly useful for ceramic vessels of common forms, such as the *olla*, which are difficult to assign to a chronological period based on their typology alone.

Furthermore, this research has yielded important insight into culinary habits at rural sites in comparison to better studied urban contexts and enabled interpretation of the relationship of this site with the capital of Palermo. However, it would be of great advantage to our overall understanding of populations in medieval rural settings to analyse samples from more rural sites across Sicily, particularly as they are generally underrepresented in historical and archaeological studies. Several rural sites have been excavated in recent years such as Contrada Castro, central western Sicily (Castrorao Barba et al. 2021; Montana et al. 2022) and Colmitella, province of Agrigento (Rizzo, 2014). ORA analysis of ceramics at these sites will further aid our

understanding of culinary habits of these populations and how they were impacted as a result of these regimes. Additionally, this thesis focused only on sites in western Sicily. This enabled significant comparison between chronological periods and sites under the same regime. However, future work should aim assess the use of pottery in the lesser-studied central and eastern Sicily. As discussed in Chapter 1, the impact and timing of regimes differed significantly between the East and West. Although lesser archaeological attention has limited the number of ceramic samples recovered from important central and eastern Sicilian sites, recent research has enabled the characterisation of ceramics from sites in eastern Sicily such as Rochicella and the site of Paterno (Catania) (Messina et al. 2018). An analysis of the use of pottery from these sites will further broaden our understanding of the life ways of people Sicily as a whole and enable comparison of culinary habits between the east and west.

Additionally, whilst this thesis has shed some light on the use of different pottery forms including *ollae*, cooking pots, braziers, stone plates, pans etc., there was a significant bias in the number of samples from each vessel category analysed. From each site, the ceramic assemblage was heavily dominated by closed form vessels such as *ollae* and cooking pots. Conversely, open form vessels such as pans, stone plate and braziers were not well represented. Future research should aim to analysis a larger sample set of specific vessel types to add to our understanding of their use. Furthermore, future research should aim to assess more subtle difference in pottery types and their uses. For example, there are a wide variety of ceramic pastes, sizes, and stylistic differences within each vessel category such as *ollae*, which come in a variety of sizes, can be handled or unhandled and have flat or round bases. By using the results presented in this research and insights from forthcoming ceramic studies, there is great potential to understand these subtleties in ceramic types and how they might be related to their contents.

Throughout this thesis it was essential to consider the results of ORA alongside other evidence of food consumption in Sicily, either already from published literature or evidence obtained as part of the Sicily in Transition project. This was important to help guide the design of this ORA study. Hypotheses regarding the types of products that might have been contained in these ceramic containers aided the selection of samples

size, extraction methods and analytical techniques that were applied. Furthermore, as explained previously, limits of ORA in terms of degradation, specificity and mixing, can be complimented by other forms of evidence such as archaeobotanical and zooarchaeological remains. This approach enabled robust interpretation of the results presented in this thesis and enabled further insight into the food ways of populations in Sicily during these transitions. However, the research in this thesis and that of the wider Sicily in Transition project was undertaken concurrently and much of this research is still forthcoming. Thus, it is important to continue to reconcile these results as new research unfold. Future excavations and studies should use this research as an example of the importance of combining different lines of evidence in understanding food ways as a whole.

Finally, it is important to note that, regardless of methodological improvements there will always remain impediment in what we can identify, not only in ORA, but archaeological studies as a whole. This is important because there is always a level of bias in our interpretations steered by what we can find. It is often assumed that what we find in the archaeological record, including organic residues from archaeological ceramics, reflect the main or most important resources utilised by the population. However, as noted throughout this thesis, the fact that certain products cannot be identified, does not equate to their absence or non-use. Furthermore, there are elements of personal choice, for example, that one simply cannot understand through residue analyse or the material culture left behind. Whilst, assessing trends in the use of pottery can help to inform about different socio-economic factors that influence the selectivity of products, it is important to ensure that 'utilisation fallacy' is avoided. Future studies of past cuisines should carefully consider the limitations of study and subsequently the archaeological questions that we can answer. As excavations increase, interpretations produced and methodologies improved, the limitations of study will undoubtedly change and these archaeological questions can thus be adapted, but should always be considered.

## 8.10 Concluding remarks

This thesis has, for the first time, provided direct insights into the cuisines of populations in Sicily during the 9<sup>th</sup>-14<sup>th</sup> century in both urban and rural locations and has made a significant contribution to our understanding of the lifeways of people in Sicily were impacted during this significant period of political, cultural, and socio-economic change. The multi-faceted organic residue approach enabled a variety of different products to be identified in domestic containers, which has provided important information about the types of products used, and how they were prepared and combined for consumption by the general populations that utilised these vessels. This thesis has highlighted the complexity of identifying products in archaeological ceramics particularly when complex mixtures of products exist in the same vessel and shows that only by applying a multi-faceted organic residue analysis approach can complex cuisines begin to be untangled. This thesis has, for the first time, analysed a large corpus (>200 ceramics) from medieval Mediterranean contexts and was essential for interpreting pottery use both diachronically and between sites. Finally, it was essential to combine the results of organic residue analysis with other historical and archaeological evidence of food consumption to gain a more in-depth assessment of foodways in Sicily as a whole and placed the results of this thesis within the wider research scope of the Sicily in Transition project. The approach taken in this thesis should provide a useful example to future studies of cuisine, particularly in contexts where complex mixtures of commodities might be expected. Furthermore, there is still more to be learned about the culinary habits of populations in Sicily during the Middle Ages and future work is essential to further our understanding of how people in Sicily lived during this time.





## **Appendix A Organic residue analysis methods**

### **A.1 Sampling of the ceramic**

The ceramic sample was first 'cleaned' by removing the outer layer of the ceramic surface (approx. 1 mm deep) using a low speed Derma drill with an abrasive metal drill bit. The first millimetre of samples was discarded to avoid exogenous contamination. Approximately 2g of ceramic powder was obtained overall for analysis by drilling into the pot and collecting the powder.

### **A.2 Extractions**

#### **A.2.1 A.2.1 Acidified methanol extraction**

Acidified methanol extraction method (Craig et al. 2013a) is now the most commonly used method in archaeological samples that favour both high extraction yield and direct methylation for stable carbon isotopes measurements. The details of this extraction are recorded elsewhere (Craig et al. 2013a) but in brief, 4 ml of methanol and 10 µl of alkane C<sub>34:0</sub> were added to 1g of powdered ceramic sample and ultrasonicated. Sulphuric acid (H<sub>2</sub>SO<sub>4</sub>) was added to each sample and heated at 70°C for 4 hours. After centrifuging, the supernatant was extracted and transferred to a clean labelled hatch tube. The samples were dried to completion under a gentle stream of nitrogen. The sample was then re-suspended in hexane and 10 µl of alkane C<sub>36:0</sub> was added as an internal standard before further analysis by gas chromatography techniques (Correa-Ascencio and Evershed 2014).

#### **A.2.2 A.Solvent (SE) / total lipid extraction (TLE)**

Solvent extraction (SE) method (Charters et al. 1993b) ensures the preservation of the most-informative molecules in fatty and waxy products (triacylglycerols and wax esters). It is worth noting that these are not always preserved in archaeological

samples. The preparation for these samples was the same as with acid extraction and 1g of sample powder was used. After the addition of 10  $\mu\text{L}$  of *n*-alkane  $\text{C}_{34:0}$ , 5ml of DCM: MeOH (2:1, v/v) was added to each ceramic sample including the blank and standard. The samples were then sonicated for 15 minutes. They were then centrifuged for 10 mins. The supernatant was then transferred to a clean labelled hatch tube. These steps were performed 3 times. The solution was then reduced to 1-2 ml under a gentle stream of nitrogen and transferred to a large hydrolysis vial. This was then dried to completion under a gentle stream of nitrogen.

### **A.2.3 Acid butylation extraction**

Small organic acids were extracted following the acid butylation extraction protocol developed by Garnier and Valamoti (2016) (Garnier and Valamoti 2016). In brief a BF 3 -butanol/hexane mixture (1:2, v/v) was added to the remaining powder previously extracted using the SE methods described above. The mixture was then heated for 2 hours at 80°C. After centrifuging, the supernatant was transferred to a clean vial and neutralised using a saturated sodium carbonate solution. The samples were extracted in DCM 3 times, and washed twice with distilled water. The extracts were then dried to completion under a gentle stream of nitrogen.

### **A.3 Trimethylsilylation (TMS)**

The dried extracts from solvent, acid butylation extraction and some select acidified methanol extracts were derivatized using *N, O*-bis(trimethylsilyl) trifluoroacetamide (BSTFA) with 1% trimethyl-chlorosilane and heated for 1 hour at 70°C. The solution and excess BSTFA was dried to completion under a gentle stream of nitrogen. 90  $\mu\text{l}$  of *n*-hexane was added to re-dissolve the extract and vortexed before transferring to an auto sampling vial which contained 10  $\mu\text{l}$  of the second internal standard  $\text{C}_{36:0}$ .

## **A.4 Instrumentation**

Gas chromatography - Flame ionisation detector (GC-FID) was used to screen for fatty acids and for basic quantification (methyl esters and TMS derivatives). Gas chromatography-Mass spectrometry (GC-MS) was used to identify complex molecules according to their mass and their fragmentation pattern.

### **A.4.1 Gas chromatography - Flame ionisation detector (GC-FID)**

Acidified methanol extracts were screened by using an Agilent 7890A Series gas chromatograph (Agilent Technologies Cheadle, Cheshire, UK) connected to a Flame Ion Detector. The column used was a 100% Dimethylpolysiloxane DB1-High temperature (HT) column (15 m × 0.32 mm × 0.1 µm; J&W Scientific, Folsom, CA, USA). 1 µl of sample was injected via a splitless injector maintained at a temperature of 300°C. The temperature of the column was kept at 100 °C for 2 minutes and then increased by 20 °C every minute until a final temperature of 325 °C was reached. A temperature of 325 °C was then held for 2 mins. Helium was used as the carrier gas at a constant flow rate of 2 ml min<sup>-1</sup>. The detector was kept at 300 °C with hydrogen flow of 30 ml min<sup>-1</sup>. This was a short temperature programme for 20 minutes.

Derivatised solvent extracts were screened with the same GC-FID instrument, fitted with the same DB1-HT column. The temperature programme was adapted to identify higher molecular weight molecules such as monoacylglycerols (MAGs), diacylglycerols (DAGs) and triacylglycerols (TAGs). The temperature of the column was kept at 50 °C for 2 minutes and then increased by 10 °C every minute until a final temperature of 375 °C was reached. A temperature of 375 °C was then held for 10 mins. This was a longer temperature programme for 44.5 minutes.

#### **A.4.2 Gas Chromatography - Mass Spectrometry (GC-MS)**

All extracts were analysed using gas chromatography mass spectrometry to identify biomarkers. The GC component was an Agilent 7890A series chromatography attached to an MS Agilent 5975 Inert XL mass selective detector with a quadrupole mass analyser (Agilent technologies, Cheadle Cheshire, UK). A DB-5MS (5%-phenyl)-methylpolysiloxane column (30 m x 0.250 mm x 0.25 µm; J&W Scientific, Folsom, CA, USA) was used. The GC column was inserted directly into the ion source of the mass spectrometer. 1 µl of sample was injected via a splitless injector maintained at a temperature of 300 °C. Helium was used as the carrier gas at a constant flow rate of 3 mL min<sup>-1</sup>. The ionisation energy of the spectrometer was 70eV and spectra were obtained by scanning between *m/z* 50 and 800. The temperature of the column was kept at 50 °C for 2 minutes and then increased by 10 °C every minute until a final temperature of 325 °C was reached. 325 °C was then held for 15 mins. Thus, the total run time was 44.5 minutes. Compounds were identified using Agilent ChemStation software with the NIST 14.0 mass spectral library.

To target ions specific to alkylresorcinols extracts were run using the same chromatographic conditions with the mass spectrometer in selected ion monitoring (SIM) mode. The ions *m/z* 73, 268, 464, 492, 520, 548, 576, 604, 632, corresponding to alkylresorcinols with cyclic carbon chain lengths C17 to C25, were monitored.

#### **A.4.3 Gas chromatography - Mass spectrometry (GC-MS) for aquatic biomarkers (DB23 AQUA SIM)**

Extracts were also analysed using an Agilent 7890A series chromatography attached to an MS Agilent 5975 Inert XL mass selective detector with a quadrupole mass analyser (Agilent technologies, Cheadle Cheshire, UK) equipped with a DB-23 (50%-Cyanopropyl)-methylpolysiloxane column (60 m x 0.250 mm x 0.25 µm; J&W Scientific, Folsom, CA, USA). The temperature of the column was kept at 50 °C for 2 minutes and then increased by 10 °C every minute until 100 °C. The temperature increased then until 140 °C by 4 °C every minute, then until 160 °C by 0.5 °C every minute and finally

until 250 °C by 20 °C every minute. Helium was used as the carrier gas at a flow rate of 1.5 mL/min. The Selective Ion Monitoring mode (SIM) was used to target different groups of ions corresponding to  $\omega$ -(*o*-alkylphenyl) alkanolic acids (APAAs) and other aquatic markers such as 4,8,12-trimethyl tridecanoic acid (TMTD), pristanic acid and phytanic acid. These groups were: *m/z* 74, 105, 262, 290, 318, 346 for the detection of  $\omega$ -(*o*-alkylphenyl)alkanoic acids of carbon lengths C<sub>16</sub> to C<sub>22</sub> (APAA<sub>16-22</sub>), *m/z* 74, 87, 213, 270 for TMTD, *m/z* 74, 88, 101, 312 for pristanic acid, *m/z* 74, 101, 171, 326 for phytanic acid and *m/z* 74, 105, 262, 290, 318, 346 for the detection of  $\omega$ -(*o*-alkylphenyl)alkanoic acids of carbon lengths C<sub>16</sub> to C<sub>22</sub> (APAA<sub>16-22</sub>). The relative abundance of the two diastereomers of phytanic acid was obtained by integrating ion *m/z* 101. These were analysed using ChemStation software or Mass Hunter.

#### **A.4.4 High temperature HT-GCMS**

TMS solvent extracts were analysed using a high temperature column and programme which was adapted to detect the presence of TAGs and wax esters. An Agilent 7890A Series gas chromatograph was used fitted with a DB5-HT column (30 m × 0.25 mm × 0.1  $\mu$ m) instead of a DB-5MS and the temperature of the column was kept at 50 degrees for 2 minutes and then increased by 10 degrees every minute until a final temperature of 375°C was reached. The maximum temperature of 375°C was then held for 10 mins. The total run time was 44.5mins.

#### **A.4.5 Gas Chromatography-Combustion-Isotope Ratio Mass Spectrometry (GC-c-IRMS)**

Stable carbon isotope ( $\delta^{13}\text{C}$ ) values of the major saturated fatty acids (FA; C<sub>16:0</sub> and C<sub>18:0</sub>) were obtained by GC-c-IRMS. A Delta V Advantage isotope ratio mass spectrometer (Thermo Fisher, Bremen, Germany) linked to a Trace Ultra gas chromatograph (Thermo Fisher) with a GC Isolink II interface was used. Alternatively, an Isoprime 100 (Isoprime, Cheadle, UK) with a Hewlett Packard 7890B series GC (Agilent Technologies, Santa Clara, CA, USA) and an Isoprime GC5 interface (Isoprime Cheadle, UK) was used. Both instruments were equipped with a DB-5MS ultra- inert

fused silica column (US, 60m x 0.25mm x 0.25 µm) and 1 µl of the samples diluted in hexane were injected via a splitless injector maintained at a temperature of 300 °C. The temperature was set at 50 °C for 0.5 min and raised by 25 °C every minute to 175 °C and then raised by 8 °C every minute to 325 °C where it was held for 20 minutes. Ultra-high-grade helium with a flow rate of 3 mL/min was used as the carrier gas. A parallel acquisition of the molecular data was achieved by splitting the gas flow into two streams. One was directed to an ISQ inert mass spectrometer (MSD) or an Agilent 5975C MSD for the Thermo and Isoprime respectively. The other half of the gas eluting from the column was directed to the creature tube to oxidise carbon species in CO<sub>2</sub>. The ionisation energy of the mass spectrometer was 70 eV and ion intensities of *m/z* 44, 45 and 46 were recorded for computing the <sup>13</sup>C/<sup>12</sup>C ratio of the peaks of each of the extracts.

Isodat (Thermo Fisher Scientific, Bremen, Germany) or Ion Vantage and IonOS software Isoprime, Cheadle, UK) software were used to compute the <sup>13</sup>C/<sup>12</sup>C ratio of the peaks in the extracts. The <sup>13</sup>C/<sup>12</sup>C ratio was calculated in comparison with a standard reference gas (CO<sub>2</sub>) of known isotopic composition that was repeatedly measured. All samples were injected twice and only regarded if the mean standard deviation of both C<sub>16:0</sub> and C<sub>18:0</sub> was <0.3 ‰. The δ<sup>13</sup>C values were expressed as per mill (‰) relative to the internal standard V-PDB (Vienna Pee Dee Belemnite) (Rieley 1994; Woodbury et al. 1995). This expression is defined by the following equation (equation 1) where R =:  $\frac{\delta^{12}C}{\delta^{13}C}$

$$\delta^{13}C_{\text{‰}} = \frac{R_{\text{sample}} - R_{\text{standard}}}{R_{\text{standard}}} \times 1000 \quad (\text{Equation 1})$$

An *n*-alkanoic acid ester standard of known isotopic composition (Indiana standard F8-3) was used to determine the precision and accuracy of the instrument which needed to remain <0.5 ‰ and <0.3 ‰ respectively. Values needed to be corrected to account for subsequent methylation of carboxyl groups during the acidified methanol extraction. These values were corrected in relation to the method standard (STD

METHYL-n) a mixture of C<sub>16:0</sub> and C<sub>18:0</sub> FAs of known isotopic composition. This is shown by the following balance equation (equation 2):

$$\delta^{13}C_{FA} = \frac{(C_{FAME} \times \delta^{13}C_{FAME}) - \delta^{13}C_{MeOH}}{C_{FA}} \quad (\text{Equation 2})$$

## Appendix B Protocols

### B.1 Lipid extraction with acidified methanol

#### LOCATION:

BioArch, University of York

#### COSHH REF:

Refer to the COSHH risk assessment for the GC process

#### PRINCIPAL:

Acid extraction of lipid residues from pottery sherds.

#### SAMPLE TYPE:

Ceramic powder from pottery sherds

#### CAUTION:

Sulfuric 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), Sulfuric 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).

#### PROCEDURE:

All persons following this protocol must ensure that the procedures detailed in the SOP are followed when carrying out acid extraction of organic residues from pottery sherds.

#### 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 C16/C18 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: note 2 g if doing both AE and SE**

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  $-20^{\circ}\text{C}$ .

4.2 for standard Using syringe add 100 $\mu\text{l}$  of isotopically measured 1 $\mu\text{g}/\text{ul}$  C16/C18 fatty acid standard to one clean labelled Hach tube and evaporate under nitrogen to dryness.

Add C34 10 $\mu\text{l}$  1 $\mu\text{g}/\text{ul}$  to all samples and standards and blanks

4.3 Using Pasteur pipette add approximately 4ml of MEOH to pottery samples + C16/C18 standard + method blank.

4.4 Sonicate for 15 minutes.

4.4 Using Pasteur pipette add 800 $\mu\text{l}$  of pure sulphuric acid (under fume hood, wearing eye protection).

4.5 Heat at  $70^{\circ}\text{C}$  for 4 hours on the heating block. Foil under cap

4.6 Prepare a Pasteur pipette by sample packing glass wool enough to plug the pipette and adding cleaned DCM cleaned (3 rinse) 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. Centrifuge 1min if needed
- 4.10 Repeat steps 4.8 and 4.9 twice more, combining the extracts.
- 4.11 Add finally 1ml of hexane through the pipette.
- 4.12 Evaporate to dryness under a very gentle stream of nitrogen with gentle warmth. Acid methanol no dry
- 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.
- 4.15 Store extracts in a freezer at  $-20^{\circ}\text{C}$  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 10 $\mu\text{l}$  of the C36 alkane standard ( $1\mu\text{g}\cdot\mu\text{l}^{-1}$ ) to a clean, labelled auto-sampling vial with 0.1ml conical insert.
- 4.18 Transfer the 90 $\mu\text{l}$  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  $4^{\circ}\text{C}$  (short-term) or in a freezer at  $-20^{\circ}\text{C}$  (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.

## **B.2 Lipid extraction with solvent method**

### **LOCATION:**

BioArch, University of York

### **COSHH REF:**

Refer to the COSHH risk assessment for the GC process

### **PRINCIPAL:**

Acid extraction of lipid residues from pottery sherds.

### **SAMPLE TYPE:**

Ceramic powder from pottery sherds

### **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, C34 alkane standard, Dichloromethane (HPLC grade), Hexane (HPLC grade), Methanol (HPLC grade), Pasteur pipettes, sterile glass wear.

### **PROCEDURE:**

All persons following this protocol must ensure that the procedures detailed in the SOP are followed when carrying out solvent extraction of organic residues from pottery sherds.

#### **1.0 PREPARATION PROCEDURES:**

1.1 Make sure all glassware and tools are solvent rinsed (3x rinsing in DCM).

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/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 SOLVENT 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  $-20^{\circ}\text{C}$ . Add 10-100ul 1ul/g of the C34 alkane standard to the powder.

4.2 Transfer powder into a hatch tub

4.3 Add C34 10ul 1ug/ul to all samples and standards and blanks

4.4 Add 2ml of DCM: MEOH 2/1 v/v.

4.5 Sonicate for 15 minutes at  $25^{\circ}\text{C}$ .

4.6 Centrifuge at 3000rpm for 10minutes.

4.7 Carefully pipette off the liquid extract into a clean, labelled scintillation vial.

4.8 Repeat steps 4.4 to 4.7 twice more, combining the extracts.

4.9 Reduce volume of extracts to about 2ml under a stream of nitrogen with gentle heat.

4.10 Transfer to a clean, labelled, small vial and continue to evaporate to dryness.

4.11 Store in a refrigerator at  $4^{\circ}\text{C}$  (short-term) or in a freezer at  $-20^{\circ}\text{C}$  (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 above procedure.

### **B.3 Acid butylation extraction of small organic acids**

#### **LOCATION:**

BioArch, University of York

#### **PRINCIPAL:**

Acid butylation extraction of small organic acids

#### **SAMPLE TYPE:**

Pottery sherds, ceramics.

#### **CAUTION:**

DCM and  $\text{BF}_3$  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, C34 alkane standard, Dichloromethane (HPLC grade), Hexane (HPLC grade), Boron trifluoride - 1-butanol solution ~10% in 1-butanol (~1.3 M), for GC derivatization ( $\text{BF}_3$  - Butanol solution), distilled water, saturated sodium carbonate solution, Pasteur pipettes, sterile glass wear.

#### **PROCEDURE:**

All persons following this protocol must ensure that the procedures detailed in the SOP are followed when carrying out solvent extraction of organic residues from pottery sherds.

#### **1.0 PREPARATION PROCEDURES:**

1.1 Make sure all glassware and tools are solvent rinsed (3x rinsing in DCM).

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 This extraction is performed on the pottery powder after solvent extraction

3.2 Ensure that the sample is dry before starting. If not, dry powder under a gentle stream of nitrogen to ensure that the solvent has evaporated

## **4.0 ACID BUTYLATION EXTRACTION:**

4.1 Add 4 pipettes of hexane to the powder

4.2 Add 2 pipettes of BF<sub>3</sub> - Butanol solution to the powder with hexane

4.3 Sonicate for 15 minutes at 25°C.

4.4 Put on heat for 2 hours at 80°C after 1 hour vortex or shake the hatch tube to make sure that everything is mixed and return to heat

4.5 After 2 hours remove tubes check the pH of the mixture.

4.6 Centrifuge at 3000rpm for 10 minutes.

4.7 Carefully pipette off the supernatant into a clean, labelled hatch tube.

4.8 Reduce volume of extracts to about 2ml under a stream of nitrogen with gentle heat.

4.9 Add 2ml of DCM to the powder, vortex and centrifuge and transfer supernatant to the same hatch tube

4.10 To the solution add 1 ml of saturated sodium carbonate (this neutralises the acid) and add 1 ml of distilled water

4.11 Vortex and centrifuge for a few minutes to facilitate phase separation

4.12 Carefully pipette off the organic layer and transfer to a new clean labelled hatch tube

4.13 Then add 4 ml of DCM to the initial mixture and vortex and centrifuge

4.14 Again carefully pipette off the organic layer and transfer to the same hatch tube

- 4.15 Repeat steps 4.13 and 4.12 with 2ml of DCM
- 4.16 To the organic mixture add 2ml of distilled water and then fill the rest of the hatch tube with DCM so that it is filled to the top
- 4.17 Cap the test tube and shake vigorously to mix
- 4.18 Pipette off water later and discard in a beaker
- 4.19 Repeat steps 4.16, 4.17 and 4.18 once more
- 4.20 Reduce volume of extracts to about 2ml under a stream of nitrogen with gentle heat.
- 4.21 Transfer to a clean, labelled, small vial and continue to evaporate to dryness.
- 4.22 Store in a refrigerator at 4°C (short-term) or in a freezer at -20°C (long term).

## **B.4 Silylation of organic residues for GC/GC-MS**

### **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. 399

### **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 70°C 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 C Plant $\delta^{13}\text{C}$ values

Table C.1:  $\delta^{13}\text{C}$  values of modern  $\text{C}_3$  plants and  $\text{C}_4$  plant products used to create figure 22 (Chapter 3) with reference to original study

Common name	Class	Provenance	$\delta^{13}\text{C}_{16:0}$ (‰)	$\delta^{13}\text{C}_{18:0}$ (‰)	$\Delta^{13}\text{C} (\text{C}_{18:0}-\text{C}_{16:0})$	reference of study
Olive	C3	Slovenia	-32.95	-31.85	-1.10	(Spangenberg and Ogrinc 2001)
Olive	C3	Slovenia	-29.83	-29.73	-0.10	(Spangenberg and Ogrinc 2001)
Olive	C3	Slovenia	-30.63	-30.73	0.10	(Spangenberg and Ogrinc 2001)
Olive	C3	Slovenia	-31.33	-30.73	-0.60	(Spangenberg and Ogrinc 2001)
Olive	C3	Slovenia	-30.93	-31.33	0.40	(Spangenberg and Ogrinc 2001)
Olive	C3	Croatia	-31.43	-30.93	-0.50	(Spangenberg and Ogrinc 2001)
Olive	C3	Croatia	-30.33	-30.53	0.20	(Spangenberg and Ogrinc 2001)
Olive	C3	Croatia	-29.03	-29.93	0.90	(Spangenberg and Ogrinc 2001)
Olive	C3	Croatia	-31.93	-31.23	-0.70	(Spangenberg and Ogrinc 2001)
Olive	C3	Croatia	-30.93	-29.63	-1.30	(Spangenberg and Ogrinc 2001)
Pumpkin	C3	Slovenia	-29.63	-31.13	1.50	(Spangenberg and Ogrinc 2001)
Pumpkin	C3	Slovenia	-29.63	-27.23	-2.40	(Spangenberg and Ogrinc 2001)
Sunflower	C3	Slovenia	-31.93	-31.13	-0.80	(Spangenberg and Ogrinc 2001)
Sunflower	C3	Slovenia	-30.73	-29.53	-1.20	(Spangenberg and Ogrinc 2001)
Soybean	C3		-32.33	-32.43	0.10	(Spangenberg and Ogrinc 2001)
Sesame	C3		-28.03	-27.83	-0.20	(Spangenberg and Ogrinc 2001)
Acorn	C3	Japan	-35.08	-35.95	0.87	(Lucquin et al. 2016b)
Acorn	C3	Japan	-33.12	-34.63	1.51	(Lucquin et al. 2016b)
Acorn	C3	Japan	-34.01	-34.45	0.44	(Lucquin et al. 2016b)
Acorn	C3	Japan	-32.12	-34.02	1.90	(Lucquin et al. 2016b)
Acorn	C3	Japan	-32.69	-33.64	0.95	(Lucquin et al. 2016b)
Walnut	C3	Japan	-31.87	-33.07	1.20	(Horiuchi et al. 2015)
Chestnut	C3	Japan	-35.47	-33.07	-2.40	(Horiuchi et al., 2015)

<b>Corn (Pisingallo)</b>	C4	Andes	-17.20	-19.20	2.00	(Lantos et al. 2015)
<b>Corn (Chullpi)</b>	C4	Andes	-19.20	-20.90	1.70	(Lantos et al. 2015)
<b>Corn (Dentado blanco)</b>	C4	Andes	-18.10	-21.10	3.00	(Lantos et al. 2015)
<b>Corn (Capia blanco)</b>	C4	Andes	-18.70	-21.00	2.30	(Lantos et al. 2015)
<b>Corn (Chullpi)</b>	C4	Andes	-18.50	-18.50	0.00	(Lantos et al. 2015)
<b>Corn (Pisingallo from Punta Colorada)</b>	C4	Andes	-15.10	-17.80	2.70	(Lantos et al. 2015)
<b>Maize</b>	C4		-13.30	-14.20	-2.00	(Guo et al. 2010)
<b>Antephora</b>	C4		-20.00	-19.40	-1.70	(Ballentine et al. 1998)
<b>Cenchrus</b>	C4		-20.80	-19.60	-1.70	(Ballentine et al. 1998)
<b>Sugar Cane</b>	C4		-20.70	-19.30	-1.70	(Ballentine et al. 1998)
<b>Sorghum</b>	C4		-20.50	-20.90	-1.60	(Chikaraiashi et al. 2004)
<b>Saccharum officinarum</b>	C4		-21.00	-20.50	-1.60	(Chikaraiashi et al. 2004)
<b>Miscanthus sinensis</b>	C4		-19.50	-19.30	-1.70	(Chikaraiashi et al. 2004)
<b>Zea mays</b>	C4		-22.60	-23.70	-1.70	(Chikaraiashi et al. 2004)

## Appendix D Summary post-firing treatment cooking experiments

Here, a summary of the experiments performed of post-firing treatments on replica vessels prior to organic residue analysis performed in chapter 4.2 of this thesis are outlined in Table D.I.1. These experiments were not performed by the author of this thesis. These experiments were performed as part of the Monte Iato experimental project funded by the Nachwuchsförderung der Universität Innsbruck by Dr. Birgit Öhlinger (Principal Investigator) and Julia Haas. The full details of these experiments are outlined in (Haas 2018) available online at <https://www.uibk.ac.at/projects/monte-iato/ex-inhalt/ora>

Table D.1: Summary of replica pottery vessels treated with post firing treatments (polishing or sealing) before different products were cooked or poured into the vessel. All samples analysed using ORA in this thesis are reported and the commodities used are summarised alongside the sealing and polishing procedure and cooking/pouring procedure.

Sample	Sealing	Content	Use	Field fire temperature used for sealing °C	Summary of sealing/Polishing procedure	Summary of cooking/ pouring procedure
1	milk	-		460	Unpasteurized milk was used from a Tyrolean farmer. After firing the ceramic for 13-15 mins reaching temperatures between 300-500°C the vessel was dipped in milk 3 times.	
2	milk	RAF	Cooking	494	Unpasteurized milk was used from a Tyrolean farmer. After firing the ceramic for 13-15 mins reaching temperatures between 300-500°C the vessel was dipped in milk 3 times.	2 cooking events over 2 days were performed on the same vessels. The vessels were placed over a charcoal oven. On the first day ~155g of cattle belly fat was filled inside the vessel and cooked for 4 hours. The temperature inside the vessel reached 80-100°C. The same was repeated on day 2 with 100g of leg meat.
3	milk	NRAF	Cooking	300	Unpasteurized milk was used from a Tyrolean farmer. After firing the ceramic for 13-15 mins reaching temperatures between 300-500°C the vessel was dipped in milk 3 times.	2 cooking events over 2 days were performed on the same vessels. The vessels were placed over a charcoal oven. On the first day ~230g of porcine belly fat was filled inside the vessel and cooked for 4 hours. The temperature inside the vessel reached 80-

						100°C. The same was repeated on day 2.
4	milk	Fish	Cooking	444	Unpasteurized milk was used from a Tyrolean farmer. After firing the ceramic for 13-15 mins reaching temperatures between 300-500°C the vessel was dipped in milk 3 times.	2 cooking events over 2 days were performed on the same vessels. The vessels were placed over a charcoal oven. On day 1 70g of mixed fish (Redfish, seas bass, coalfish, and Salmon) vessel and cooked for 4 hours. The temperature inside the vessel reached 80-100°C. On day 2 a further 70g of the same fish was added with the addition of 15g of pasta di acciughe.
5	milk	Veg	Cooking	477	Unpasteurized milk was used from a Tyrolean farmer. After firing the ceramic for 13-15 mins reaching temperatures between 300-500°C the vessel was dipped in milk 3 times.	
6	milk	Wine	Pouring	484	Unpasteurized milk was used from a Tyrolean farmer. After firing the ceramic for 13-15 mins reaching temperatures between 300-500°C the vessel was dipped in milk 3 times.	
8	Beef tallow	-		350	Beef tallow was brought from Tyrol and made from cattle sued. After heating the ceramic in fire for 12-15 minutes reaching temperatures between 200-490 °C. The beef tallow was then placed in the vessel and moved around the cover the internal surface.	
9	Beef tallow	Milk	Cooking	200	Beef tallow was brought from Tyrol and made from cattle sued. After heating the ceramic in fire for 12-15 minutes reaching temperatures between 200-490 °C. The beef tallow was then placed in the vessel and moved around the cover the internal surface.	2 cooking events over 2 days were performed on the same vessels. The vessels were placed over a charcoal oven. On day both days ~300ml of milk was filled inside the vessel and cooked for 4 hours. The temperature inside the vessel reached 80-100°C.
10	Beef tallow	NRAF	Cooking	400	Beef tallow was brought from Tyrol and made from cattle sued. After heating the ceramic in fire for 12-15 minutes reaching temperatures between 200-490 °C. The beef tallow was then placed in the vessel and moved	2 cooking events over 2 days were performed on the same vessels. The vessels were placed over a charcoal oven. On the first day ~230g of porcine belly fat was filled inside the vessel and cooked for 4 hours. The temperature inside the vessel reached 80-

					around the cover the internal surface.	100°C. The same was repeated on day 2.
11	Beef tallow	Fish	Cooking	300	Beef tallow was brought from Tyrol and made from cattle sued. After heating the ceramic in fire for 12-15 minutes reaching temperatures between 200-490 °C. The beef tallow was then placed in the vessel and moved around the cover the internal surface.	2 cooking events over 2 days were performed on the same vessels. The vessels were placed over a charcoal oven. On day 1 ~70 g of mixed fish (redfish, sea bass, coalfish, salmon) were added to the vessel and cooked for 4 hours. The temperature inside the vessel reached 80-100°C. The same was repeated on day 2 with a further ~15g of the same mixed fish was added with the addition of pasta di acciughe (fish paste)
12	Beef tallow	Veg	Cooking	490	Beef tallow was brought from Tyrol and made from cattle sued. After heating the ceramic in fire for 12-15 minutes reaching temperatures between 200-490 °C. The beef tallow was then placed in the vessel and moved around the cover the internal surface.	
13	Beef tallow	Wine	Pouring	270	Beef tallow was brought from Tyrol and made from cattle sued. After heating the ceramic in fire for 12-15 minutes reaching temperatures between 200-490 °C. The beef tallow was then placed in the vessel and moved around the cover the internal surface.	
15	Beef tallow	Oil	Pouring	360	Beef tallow was brought from Tyrol and made from cattle sued. After heating the ceramic in fire for 12-15 minutes reaching temperatures between 200-490 °C. The beef tallow was then placed in the vessel and moved around the cover the internal surface.	
16	Resin	-		250	Pine resin was collected in the woods of Tyrol. 50ml of clean resin was enriched with some beef tallow. After firing the ceramic to temperatures between 200-300 °C. the mixture was applied to the interior surface of the ceramic vessel	
18	Resin	Milk	Cooking	250	Pine resin was collected in the woods of Tyrol. 50ml	2 cooking events over 2 days were performed on the same

					of clean resin was enriched with some beef tallow. After firing the ceramic to temperatures between 200-300 °C. the mixture was applied to the interior surface of the ceramic vessel	vessels. The vessels were placed over a charcoal oven. On day both days ~300ml of milk was filled inside the vessel and cooked for 4 hours. The temperature inside the vessel reached 80-100°C.
19	Resin	NRFA	Cooking	290	Pine resin was collected in the woods of Tyrol. 50ml of clean resin was enriched with some beef tallow. After firing the ceramic to temperatures between 200-300 °C. the mixture was applied to the interior surface of the ceramic vessel	2 cooking events over 2 days were performed on the same vessels. The vessels were placed over a charcoal oven. On the first day ~230g of porcine belly fat was filled inside the vessel and cooked for 4 hours. The temperature inside the vessel reached 80-100°C. The same was repeated on day 2.
20	Resin	Fish	Cooking	290	Pine resin was collected in the woods of Tyrol. 50ml of clean resin was enriched with some beef tallow. After firing the ceramic to temperatures between 200-300 °C. the mixture was applied to the interior surface of the ceramic vessel	2 cooking events over 2 days were performed on the same vessels. The vessels were placed over a charcoal oven. On day 1 ~70 g of mixed fish (redfish, sea bass, coalfish, salmon) were added to the vessel and cooked for 4 hours. The temperature inside the vessel reached 80-100°C. The same was repeated on day 2 with a further ~15g of the same mixed fish was added with the addition of pasta di acciughe (fish paste)
21	Resin	Wine	Pouring	220	Pine resin was collected in the woods of Tyrol. 50ml of clean resin was enriched with some beef tallow. After firing the ceramic to temperatures between 200-300 °C. the mixture was applied to the interior surface of the ceramic vessel	The vessel was filled with red wine a placed in an oven at 70 °C. These were warmed for 3 weeks and filled when needed
23	Resin	Oil	Pouring	330	Pine resin was collected in the woods of Tyrol. 50ml of clean resin was enriched with some beef tallow. After firing the ceramic to temperatures between 200-300 °C. the mixture was applied to the interior surface of the ceramic vessel	The vessel was filled with olive oil bought and produced in Sicily a placed in an oven at 70 °C. These were warmed for 3 weeks and filled when needed
31	Beef tallow		Polished		The interior of the ceramic vessel was polished with beef tallow using a rose quartz stone for several hours	
32	Oil		Polished		The interior of the ceramic vessel was polished with olive oil	

					bought and produced in Sicily using a rose quartz stone for several hours	
33	None	Wine	Pouring			The vessel was filled with red wine a placed in an oven at 70 °C. These were warmed for 3 weeks and filled when needed.
35	None	Milk	Cooking			2 cooking events over 2 days were performed on the same vessels. The vessels were placed over a charcoal oven. On day both days ~300ml of milk was filled inside the vessel and cooked for 4 hours. The temperature inside the vessel reached 80-100°C.
36	None	RAF	Cooking			2 cooking events over 2 days were performed on the same vessels. The vessels were placed over a charcoal oven. On the first day ~155g of cattle belly fat was filled inside the vessel and cooked for 4 hours. The temperature inside the vessel reached 80-100°C. The same was repeated on day 2 with 100g of leg meat.
37	None	NRAF	Cooking			2 cooking events over 2 days were performed on the same vessels. The vessels were placed over a charcoal oven. On the first day ~230g of porcine belly fat was filled inside the vessel and cooked for 4 hours. The temperature inside the vessel reached 80-100°C. The same was repeated on day 2.
38	None	Fish	Cooking			2 cooking events over 2 days were performed on the same vessels. The vessels were placed over a charcoal oven. On day 1 ~70 g of mixed fish (redfish, sea bass, coalfish, salmon) were added to the vessel and cooked for 4 hours. The temperature inside the vessel reached 80-100°C. The same was repeated on day 2 with a further ~15g of the same mixed fish was added with the addition of pasta di acciughe (fish paste)
39	None	Veg	Cooking			2 cooking events over 2 days were performed on the same vessels. The vessels were placed over a charcoal oven. On day 1 ~300 g of mixed vegetables (chard, cabbage, parsley, yellow carrot) were added to the vessel and cooked for 4 hours. The



						temperature inside the vessel reached 80-100°C. The same was repeated on day 2 with a further ~300g of the same mixed vegetables.
40	None	Wine	Pouring			The vessel was filled with red wine a placed in an oven at 70 °C. These were warmed for 3 weeks and filled when needed.
42	None	Oil	Pouring			The vessel was filled with olive oil bought and produced in Sicily a placed in an oven at 70 °C. These were warmed for 3 weeks and filled when needed.
43	None	Milk	Pouring			The vessel was filled with milk a placed in an oven at 70 °C. These were warmed for 3 weeks and filled when needed.

## **Appendix E Ceramic samples from Palermo sites (CSP, GA and PB) and 10<sup>th</sup>-12<sup>th</sup> century CLESP and summary of substance identification of organic residue analysis**

Summary tables of all ceramic samples and their relative information are presented alongside a summary of substance identification from the organic residue analysis relating to chapter 5 of this thesis. All samples analysed from Castello San Pietro (CSP) are presented in Table E.1. Samples from La Gancia are presented in Table E.2. Samples from Palazzo Bonagia are presented in Table E.3. Samples from 10<sup>th</sup>-12<sup>th</sup> century contexts from Casale San Pietro are presented in table E.4.

The full results of organic residue analysis for each of these sites are presented in the supporting information of this thesis: **S2 data. Ceramic samples and organic residue analysis results from 9<sup>th</sup>-12<sup>th</sup> century Palermo and Casale San Pietro (chapter 5).**

All raw data files relating to these samples have been deposited in an online depository available from Dryad: <https://doi.org/10.5061/dryad.9ghx3ffhd>.

Table E.1: Summary of ceramic samples from Castello San Pietro (CSP) and summary substance identification of organic residue analysis. Here the reference to the original ceramic study is given and the Inventory number of each sample reported in the original ceramic study

Site name	Site code	Sample code	Chronology	Form	Technology	Provenance	Inventory number/ original form code	Reference for ceramic study	Form Id	Part of ceramic sampled	Yield (µg/g)	Summary substance identification
Castello San Pietro (PA)	CSP	CSP_C1	Late 9th-early 10th century	Olla	Wheel thrown	Palermo	B/2	(Arcifa and Bagnera 2014)	Form 3a	Body	39.72	Non-ruminant + plant wax + fruit product
Castello San Pietro (PA)	CSP	CSP_C2	Late 9th-early 10th century	Olla	Wheel thrown	Palermo	B/80	(Arcifa and Bagnera 2014)	Form 3a	Body	83.75	Non-ruminant + plant wax +fruit product
Castello San	CSP	CSP_C3	Late 9th-early 10th century	Olla	Wheel thrown	Palermo	B/6	(Arcifa and Bagnera 2014)	Form 3a	Body	285.83	Ruminant adipose

<b>Pietro (PA)</b>												
<b>Castello San Pietro (PA)</b>	CSP	CSP_C4	Late 9th-early 10th century	Olla	Wheel thrown	Palermo	B/81	(Arcifa and Bagnera 2014)	Form 3a	Body	91.52	Ruminant adipose +plant wax +fruit product +beeswax + pine products
<b>Castello San Pietro (PA)</b>	CSP	CSP_C5	Late 9th-early 10th century	Olla	Wheel thrown	Palermo	B/10	(Arcifa and Bagnera 2014)	Form 3a	Base	1121.51	Pine products (pitch)+ unidentified fat
<b>Castello San Pietro (PA)</b>	CSP	CSP_C6	Late 9th-early 10th century	Olla	Wheel thrown	Palermo	B/5	(Arcifa and Bagnera 2014)	Form 3a	Body	142.85	Ruminant adipose +plant wax(brassica)+ fruit products
<b>Castello San Pietro (PA)</b>	CSP	CSP_C7	Late 9th-early 10th century	Lid	Wheel thrown	Palermo	B/13	(Arcifa and Bagnera 2014)	Form 12	Body	28.55	Non-ruminant
<b>Castello San</b>	CSP	CSP_C8	Late 9th-early 10th century	Lid	Wheel thrown	Palermo	B/13	(Arcifa and Bagnera 2014)	Form 12	Body	25.70	Non-ruminant + fruit product + grape product

<b>Pietro (PA)</b>												
<b>Castello San Pietro (PA)</b>	CSP	CSP_C9	Late 9th-early 10th century	Olla	Wheel thrown	Palermo	B/8	(Arcifa and Bagnera 2014)	Form 3a	Body	35.11	Non-ruminant +pine products
<b>Castello San Pietro (PA)</b>	CSP	CSP_C10	Late 9th-early 10th century	Olla	Wheel thrown	Palermo	B/11	(Arcifa and Bagnera 2014)	Form 3a	Base	133.51	Ruminant +pine products
<b>Castello San Pietro (PA)</b>	CSP	CSP_C11	Late 9th-early 10th century	Olla	Wheel thrown	Palermo	B/11	(Arcifa and Bagnera 2014)	Form 3a	Base	56.50	Non-ruminant
<b>Castello San Pietro (PA)</b>	CSP	CSP_C13	Late 9th-early 10th century	Lid	Wheel thrown	Palermo	B/16	(Arcifa and Bagnera 2014)	Form 12	Body	61.75	Non-ruminant
<b>Castello San</b>	CSP	CSP_C14	Late 9th-early 10th century	Lid	Wheel thrown	Palermo	B/17	(Arcifa and Bagnera 2014)	Form 12	Rim	51.21	Non-ruminant + plant wax

<b>Pietro (PA)</b>												
<b>Castello San Pietro (PA)</b>	CSP	CSP_C15	Late 9th-early 10th century	Lid	Wheel thrown	Palermo	B/19	(Arcifa and Bagnera 2014)	Form 12	Rim	74.91	Non-ruminant + pine products
<b>Castello San Pietro (PA)</b>	CSP	CSP_C16	Late 9th-early 10th century	Brazier	Wheel thrown	Palermo	B/12	(Arcifa and Bagnera 2014)	Form 7	Rim and body	68.97	Non ruminant fat
<b>Castello San Pietro (PA)</b>	CSP	CSP_C18	First half of 10th	Pan	Wheel thrown	Palermo	B/24	(Arcifa and Bagnera 2014)	Form 9	Body	31.00	Non-ruminant
<b>Castello San Pietro (PA)</b>	CSP	CSP_C19	First half of 10th	Olla	Wheel thrown	Palermo	B/9	(Arcifa and Bagnera 2014)	Form 3a	Body	162.72	Ruminant + fruit products
<b>Castello San</b>	CSP	CSP_C20	First half of 10th	Olla	Wheel thrown	Palermo	B/29	(Arcifa and Bagnera 2014)	Form 3a	Body	101.37	Non-ruminant + fruit products

<b>Pietro (PA)</b>												
<b>Castello San Pietro (PA)</b>	CSP	CSP_C21	First half of 10th	Olla	Wheel thrown	Palermo	B/5	(Arcifa and Bagnera 2014)	Form 3a	Body	78.83	Non-ruminant + fruit products
<b>Castello San Pietro (PA)</b>	CSP	CSP_C22	First half of 10th	Olla	Wheel thrown	Palermo	B/10	(Arcifa and Bagnera 2014)	Form 3a	Base	74.52	Non- ruminant
<b>Castello San Pietro (PA)</b>	CSP	CSP_C23	First half of 10th	Olla	Wheel thrown	Palermo	B/28	(Arcifa and Bagnera 2014)	Form 3a	Body	101.39	Non-ruminant + fruit products
<b>Castello San Pietro (PA)</b>	CSP	CSP_C24	First half of 10th	Olla	Wheel thrown	Palermo	B/10	(Arcifa and Bagnera 2014)	Form 3a	Base	31.00	Non-ruminant + fruit products
<b>Castello San</b>	CSP	CSP_C25	First half of 10th	Olla	Wheel thrown	Palermo	B/10	(Arcifa and Bagnera 2014)	Form 3a	Base	68.55	Non- ruminant

<b>Pietro (PA)</b>												
<b>Castello San Pietro (PA)</b>	CSP	CSP_C33b	First half of 10th	Stone plate	Wheel thrown	Palermo		(Arcifa and Bagnera 2014)	Form 6	Body/Base	37.73	Ruminant adipose + unidentified plant



Table E.2: Summary of ceramic samples from La Gancia (GA) and summary substance identification of organic residue analysis. Here the reference to the original ceramic study is given and the Inventory number of each sample reported in the original ceramic study

Site name	Site code	Sample code	Chronology	Form	Technology	Provenance	Inventory number/ original form code	Reference for ceramic study	Part of ceramic sampled	yield (µg/g)	Form Id	Summary substance identification
La Gancia (PA)	GA	GA_C9	Late 10th - early 11th century	Olla	Wheel thrown	Palermo	GA2201	(Ardizzone, Pezzini and Sacco 2014)	Body	121.68	Form 3a	Non-ruminant + plant waxes (Brassica) +heating
La Gancia (PA)	GA	GA_C10	Late 10th - early 11th century	Olla	Wheel thrown	Palermo	GA2203	(Ardizzone, Pezzini and Sacco 2014)	Body	50.56	Form 3a	Non-ruminant + plant waxes (Brassica) +heating
La Gancia (PA)	GA	GA_C11	Late 10th - early 11th century	Olla	Wheel thrown	Palermo	GA2202	(Ardizzone, Pezzini and Sacco 2014)	Body	185.95	Form 3a	Non-ruminant + plant waxes (Brassica) + fruit +heating
La Gancia (PA)	GA	GA_C12b	Late 10th - early 11th century	Olla	Wheel thrown	Palermo	GA549	(Ardizzone, Pezzini and Sacco 2014)	Body	42.60	Form 3a	Non-ruminant
La Gancia (PA)	GA	GA_C13	Late 10th - early 11th century	Olla	Wheel thrown	Palermo	GA545	(Ardizzone, Pezzini and Sacco 2014)	Body	122.96	Form 3a	Ruminant + plant waxes (Brassica) + fruit +heating

<b>La Gancia (PA)</b>	GA	GA_C14	Late 10th - early 11th century	Olla	Wheel thrown	Palermo	GA546	(Ardizzone,Pezzini and Sacco 2014)	Body	161.43	Form 3a	Ruminant adipose +plant waxes (Brassica) + fruit +heating
<b>La Gancia (PA)</b>	GA	GA_C15	Late 10th - early 11th century	Olla	Wheel thrown	Palermo	GA539	(Ardizzone,Pezzini and Sacco 2014)	Body	13.02	Form 3a	Non-ruminant + fruit
<b>La Gancia (PA)</b>	GA	GA_C16	Late 10th - early 11th century	pan	Wheel thrown	Palermo	GA552	(Ardizzone,Pezzini and Sacco 2014)	Base	14.57	Form 9	UI Below limit of interpretation
<b>La Gancia (PA)</b>	GA	GA_C17	Late 10th - early 11th century	pan	Wheel thrown	Palermo	GA551	(Ardizzone,Pezzini and Sacco 2014)	Base	23.21	Form 9	UI Below limit of interpretation
<b>La Gancia (PA)</b>	GA	GA_C18	Late 9th- early 10th century	Olla	Wheel thrown	Palermo	GA17	(Ardizzone,Pezzini and Sacco 2014)	Body	41.59	Form 3a	Non-ruminant + plant waxes + fruit +heating
<b>La Gancia (PA)</b>	GA	GA_C19	Late 9th- early 10th century	Olla	Wheel thrown	Palermo	GA67	(Ardizzone,Pezzini and Sacco 2014)	Body	22.13	Form 3a	Non-ruminant + heating
<b>La Gancia (PA)</b>	GA	GA_C20b	Late 9th- early 10th century	Olla	Wheel thrown	Palermo	GA7	(Ardizzone,Pezzini and Sacco 2014)	Body	372.03	Form 8	Ruminant + unidentified plant + fruit + heating
<b>La Gancia (PA)</b>	GA	GA_C21	Late 9th- early 10th century	Olla	Wheel thrown	Palermo	GA40	(Ardizzone,Pezzini and Sacco 2014)	Body	333.72	Form 3a	Ruminant + fruit
<b>La Gancia (PA)</b>	GA	GA_C22	Late 9th- early 10th century	Olla	Wheel thrown	Palermo	GA18	(Ardizzone,Pezzini and Sacco 2014)	Body	35.94	Form 3a	Ruminant adipose +plant waxes (Brassica) + pine products+ fruit

<b>La Gancia (PA)</b>	GA	GA_C23	Late 9th- early 10th century	Olla	Wheel thrown	Palermo	GA19	(Ardizzone,Pezzini and Sacco 2014)	Body	26.65	Form 3a	Non-ruminant
<b>La Gancia (PA)</b>	GA	GA_C24	Late 9th- early 10th century	Stone pot	Wheel thrown	?		(Ardizzone,Pezzini and Sacco 2014)	Body	29.77	Form 10	Non-ruminant
<b>La Gancia (PA)</b>	GA	GA_C25	Late 9th- early 10th century	Lid	Wheel thrown	Palermo		(Ardizzone,Pezzini and Sacco 2014)	Body	286.17	Form 12	Ruminant adipose + fruit+ heating
<b>La Gancia (PA)</b>	GA	GA_C26	Late 9th- early 10th century	Cooking pot	Wheel thrown	Calcitic*		(Ardizzone,Pezzini and Sacco 2014)	Body	51.98	Form 2	Non-ruminant + heating
<b>La Gancia (PA)</b>	GA	GA_C27	Late 9th- early 10th century	Olla	Wheel thrown	Palermo	GA31	(Ardizzone,Pezzini and Sacco 2014)	Body	111.42	Form 3a	Non-ruminant + plant wax (Brassica)+ fruit + heating
<b>La Gancia (PA)</b>	GA	GA_C28	Late 10th century	Olla	Wheel thrown	Palermo	GA485	(Ardizzone,Pezzini and Sacco 2014)	Body	52.98	Form 3a	Ruminant adipose
<b>La Gancia (PA)</b>	GA	GA_C29	Late 10th century	Olla	Wheel thrown	Palermo	GA483	(Ardizzone,Pezzini and Sacco 2014)	Body	121.39	Form 3a	Ruminant adipose + fruit
<b>La Gancia (PA)</b>	GA	GA_C30	Late 10th century	Cooking pot	Wheel thrown	Calcitic*		(Ardizzone,Pezzini and Sacco 2014)	Base	34.21	Form 2	Ruminant adipose + heating
<b>La Gancia (PA)</b>	GA	GA_C31	Late 10th century	Lid	Wheel thrown	Palermo	GA483	(Ardizzone,Pezzini and Sacco 2014)	Body	113.24	Form 12	Non- ruminant +fruit
<b>La Gancia (PA)</b>	GA	GA_C32	Late 10th century	Pan	Wheel thrown	Palermo	GA484	(Ardizzone,Pezzini and Sacco 2014)	Body	9.07	Form 9	UI Below limit of interpretation
<b>La Gancia (PA)</b>	GA	GA_C33	Late 10th century	Stone plate	Wheel thrown	?	GA484	(Ardizzone,Pezzini and Sacco 2014)	Base	9.78	Form 6	UI Below limit of interpretation

<b>La Gancia (PA)</b>	GA	GA_C34	Late century	10th	Olla	Wheel thrown	Palermo	GA480	(Ardizzone, Pezzini and Sacco 2014)	Body	167.10	Form 3a	Ruminant adipose
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Table E.3: Summary of ceramic samples from Palazzo Bonagia (PB) and summary substance identification of organic residue analysis. Here the reference to the original ceramic study is given and the Inventory number of each sample reported in the original ceramic study

Site name	Site code	Sample code	Chronology	Form	Technology	Provenance	Inventory number/ original form code	Reference for ceramic study	Part of ceramic sampled	yield (µg/g)	Form Id	Summary substance identification
Palazzo Bonagia (PA)	PB	PB_C25	late 10th-early 11th century	Olla	Wheel thrown	Palermo	PB468	(Sacco 2014)	Body	144.08	Form 3a	Non-ruminant
Palazzo Bonagia (PA)	PB	PB_C26	Second half of 10th century	Olla	Wheel thrown	Palermo	PB519	(Sacco 2014)	Body	44.14	Form 3a	Non-ruminant+ plant waxes (leek)+ pine products + fruit
Palazzo Bonagia (PA)	PB	PB_C27	10th -11th century	Olla	Wheel thrown	Palermo	PB609	(Sacco 2014)	Body	70.87	Form 3a	Non-ruminant + unidentified plant + pine products + fruit
Palazzo Bonagia (PA)	PB	PB_C28	Late 9th - early 10th century	Olla	Wheel thrown	Palermo	PB155	(Sacco 2014)	Body	0.12	Form 3a	UI Below limit of interpretation
Palazzo Bonagia (PA)	PB	PB_C29	Late 9th - early 10th century	Olla	Wheel thrown	Palermo	PB160	(Sacco 2014)	Body	24.47	Form 3a	Ruminant adipose

<b>Palazzo Bonagia (PA)</b>	PB	PB_C30	Late 9th - early 10th century	Olla	Wheel thrown	Palermo	PB151	(Sacco 2014)	Body	41.36	Form 3a	Non-ruminant
<b>Palazzo Bonagia (PA)</b>	PB	PB_C31	late 10th-early 11th century	Olla	Wheel thrown	Palermo	PB108	(Sacco 2014)	Body	22.05	Form 3a	Non-ruminant
<b>Palazzo Bonagia (PA)</b>	PB	PB_C32	Second half of 10th century	Olla	Wheel thrown	Palermo	PB891	(Sacco 2014)	Body	1.12	Form 3a	UI Below limit of interpretation
<b>Palazzo Bonagia (PA)</b>	PB	PB_C33	Second half of 10th century	Olla	Wheel thrown	Palermo	PB25	(Sacco 2014)	Body	2.32	Form 3a	UI Below limit of interpretation
<b>Palazzo Bonagia (PA)</b>	PB	PB_C34	Second half of 10th century	Olla	Wheel thrown	Palermo	PB16	(Sacco 2014)	Body	51.49	Form 3a	Ruminant adipose
<b>Palazzo Bonagia (PA)</b>	PB	PB_C35	Second half of 10th century	Olla	Wheel thrown	Palermo	PB518	(Sacco 2014)	Body	1.27	Form 3a	UI Below limit of interpretation

<b>Palazzo Bonagia (PA)</b>	PB	PB_C36	11th century	Olla	Wheel thrown	Palermo	PB948	(Sacco 2014)	Body	17.03	Form 3a	Ruminant adipose + pine products
<b>Palazzo Bonagia (PA)</b>	PB	PB_C37	Late 10th-early 11th century	Olla	Wheel thrown	Palermo	PB464	(Sacco 2014)	Body	2.71	Form 3a	UI Below limit of interpretation
<b>Palazzo Bonagia (PA)</b>	PB	PB_C38	11th century	Olla	Wheel thrown	Palermo	PB949	(Sacco 2014)	Body	9.02	Form 3a	Non-ruminant + unidentified plant wax + pine products
<b>Palazzo Bonagia (PA)</b>	PB	PB_C39	10th -11th century	Olla	Wheel thrown	Sicilia occidentale	PB559	(Sacco 2014)	Body	70.11	Form 3a	Ruminant adipose + unidentified plant + fruit + heating
<b>Palazzo Bonagia (PA)</b>	PB	PB_C40	Second half of 10th century	Olla	Wheel thrown	Palermo	PB23	(Sacco 2014)	Body	79.84	Form 3a	Ruminant adipose
<b>Palazzo Bonagia (PA)</b>	PB	PB_C41	Late 10th-early 11th century	Olla	Wheel thrown	Palermo	PB223	(Sacco 2014)	Body	71.40	Form 3a	Non-ruminant
<b>Palazzo Bonagia (PA)</b>	PB	PB_C42	Second half of 10th century	Olla	Wheel thrown	Palermo	PB895	(Sacco 2014)	Body	170.78	Form 3a	Ruminant adipose + plant

<b>Palazzo Bonagia (PA)</b>	PB	PB_C44	Second half of 10th century	Olla	Wheel thrown	Palermo	PB984	(Sacco 2014)	Body	127.40	Form 3a	Non-ruminant
<b>Palazzo Bonagia (PA)</b>	PB	PB_C45	Second half of 10th century	Olla	Wheel thrown	Palermo	PB42	(Sacco 2014)	Body	140.67	Form 3a	Non-ruminant + fruit
<b>Palazzo Bonagia (PA)</b>	PB	PB_C46	Early Islamic	Olla	Wheel thrown	Palermo	PB44	(Sacco 2014)	Body	58.54	Form 3a	Ruminant adipose
<b>Palazzo Bonagia (PA)</b>	PB	PB_C47	Late 9th - early 19th century	Olla	Wheel thrown	Palermo	PB175	(Sacco 2014)	Body	70.41	Form 3a	Ruminant adipose + plant wax (Brassica)
<b>Palazzo Bonagia (PA)</b>	PB	PB_C48	Late 10th-early 11th century	Cooking pot	Slow thrown	Calcitic	PB148	(Sacco 2014)	Body	116.00	Form 2	Ruminant
<b>Palazzo Bonagia (PA)</b>	PB	PB_C49	Late 10th-early 11th century	Cooking pot	Slow thrown	Calcitic	PB483	(Sacco 2014)	Body	52.33	Form 2	Ruminant adipose + plant wax (Brassica)



<b>Palazzo Bonagia (PA)</b>	PB	PB_C50	Late early 10th-11th century	Partially glazed cooking pot	Wheel thrown	East Sicily	PB482	(Sacco 2014)	Body	25.40	Form 8	Non-ruminant
<b>Palazzo Bonagia (PA)</b>	PB	PB_C51	Late early 10th-11th century	Partially glazed cooking pot	Wheel thrown	East Sicily	PB988	(Sacco 2014)	Body	46.81	Form 8	Non-ruminant +heating
<b>Palazzo Bonagia (PA)</b>	PB	PB_C52	Second half of 10th century	Pan	Wheel thrown	Palermo	PB989	(Sacco 2014)	Body	35.05	Form 9	Non-ruminant +heating
<b>Palazzo Bonagia (PA)</b>	PB	PB_C53	Late early 10th-11th century	Pan	Wheel thrown	Palermo	PB913	(Sacco 2014)	Body	1.89	Form 9	UI Below limit of interpretation
<b>Palazzo Bonagia (PA)</b>	PB	PB_C54	Second half of 10th century	Pan	Wheel thrown	Palermo	PB856	(Sacco 2014)	Body	29.71	Form 9	Non-ruminant
<b>Palazzo Bonagia (PA)</b>	PB	PB_C55	Late early 10th-11th century	Brazier	Wheel thrown	Palermo	PB909	(Sacco 2014)	Body	39.72	Form 7	Non-ruminant

<b>Palazzo Bonagia (PA)</b>	PB	PB_C55(BIS)	Second half of 10th century	Brazier	Wheel thrown	Palermo	PB478	(Sacco 2014)	Body	138.43	Form 7	Non-ruminant +heating
<b>Palazzo Bonagia (PA)</b>	PB	PB_C56	Early Islamic	Brazier	Wheel thrown	Palermo	PB739	(Sacco 2014)	Body	81.68	Form 7	Non-ruminant
<b>Palazzo Bonagia (PA)</b>	PB	PB_C57	Early Islamic	Stone plate	Limestone		PB738	(Sacco 2014)	Base	0.00	Form 6	UI Below limit of interpretation

Table E.4: Summary of ceramic samples from Casale San Pietro (CLESP) and summary substance identification of organic residue analysis. Here the reference to the original ceramic study is given and the Inventory number of each sample reported in the original ceramic study

Site name	Site code	Sample code	Chronology	Form	Technology	Provenance	Form Id	Part of ceramic sampled	yield (µg/g)	Summary substance identification	
Casale Pietro	San	CLESP	CLESP_1	10th-11th century	Stone plate	Limestone	?	Form 6	Base	11.2	Non-ruminant
Casale Pietro	San	CLESP	CLESP_2	10th-11th century	Olla	Wheel thrown	Palermo	Form 3a	Body	406.7	Non-ruminant
Casale Pietro	San	CLESP	CLESP_3	10th-11th century	Olla	Wheel thrown	Palermo	Form 3a	Body	153.2	Non-ruminant + fruit +heating
Casale Pietro	San	CLESP	CLESP_4	10th-11th century	Cooking pot	Handmade	Local?	Form 5	Body	1,213.6	Ruminant + plant wax (Fennel)
Casale Pietro	San	CLESP	CLESP_5	10th-11th century	Olla	Wheel thrown?	Calcitic	Form 3b	Body	13.4	Ruminant + plant wax (Fennel)+ fruit
Casale Pietro	San	CLESP	CLESP_11	10th-11th century	Olla	Wheel thrown	Palermo	Form 3a	Body	318.2	Non-ruminant+ fruit
Casale Pietro	San	CLESP	CLESP_12	10th-11th century	Olla	Wheel thrown	Palermo	Form 3a	Body	535.7	Ruminant adipose + Plant oil
Casale Pietro	San	CLESP	CLESP_14	10th-11th century	Olla	Wheel thrown	Palermo	Form 3a	Body	52.8	Non-ruminant
Casale Pietro	San	CLESP	CLESP_15	10th-11th century	Olla	Wheel thrown	Palermo	Form 3a	Body	67.6	Non-ruminant

<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_16	10th-century	11th	Olla	Wheel thrown	Palermo	Form 3a	Base and body	94.4	Non-ruminant +grape product
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_18	10th-century	11th	Olla	Wheel thrown	Palermo	Form 3a	Body	382.4	Ruminant adipose + Unidentified wax + fruit + heating
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_19	10th-century	11th	Olla	Wheel thrown	Palermo	Form 3a	Rim	118.9	Ruminant adipose + plant wax (Fennel) fruit +heating
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_20	10th-century	11th	Olla	Wheel thrown	Palermo	Form 3a	Body	131.3	Non-ruminant + fruit
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_21	10th-century	11th	Cooking pot	Handmade	Local?	Form 5	Body	471.2	Ruminant adipose + heating
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_22	10th-century	11th	Cooking pot	Slow thrown	Calcitic	Form 2	Body	26.0	Non-ruminant
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_23	10th-century	11th	Olla	Wheel thrown	Palermo	Form 3a	Rim	109.4	Ruminant + unidentified plant
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_24	10th-century	11th	Olla	Wheel thrown	Palermo	Form 3a	Body	143.0	Non-ruminant + plant wax (Brassica)+fruit product heating
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_25	10th-century	11th	Olla	Wheel thrown	Palermo	Form 3a	Body	21.1	Non-ruminant + unidentified plant
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_26	10th-century	11th	Cooking pot	Slow thrown	Calcitic	Form 2	Body	64.6	Dairy

<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_27	10th-century	11th	Cooking pot	Slow thrown	Calcitic	Form 2	Rim	76.9	Ruminant adipose
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_28	10th-century	11th	Olla	Wheel thrown	Palermo	Form 3a	Body and base	16.1	Ruminant + heating
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_29	10th-century	11th	Olla	Wheel thrown	Palermo	Form 3a	Body	491.5	Non-ruminant (porcine?) + heating
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_30	10th-century	11th	Olla	Wheel thrown	Palermo	Form 3a	Body	22.7	Ruminant adipose + unidentified plant
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_31	10th-century	11th	Pan?	Slow thrown	Calcitic	Form 1	Base and body	369.9	Ruminant adipose + unidentified plant + heating
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_33	10th-century	11th	Cooking pot	Slow thrown	Calcitic	Form 2	Body	267.1	Non-ruminant (porcine?) + plant wax (brassica)+ heating
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_35	10th-century	11th	Cooking pot	Slow thrown	Calcitic	Form 2	Rim	105.7	Ruminant adipose + Unidentified plant wax
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_37	10th-century	11th	Cooking pot	Slow thrown	Calcitic	Form 2	Rim	26.9	Dairy
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_38	10th-century	11th	Olla	Wheel thrown	Calcitic	Form 3b	Body	9.4	Ruminant adipose
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_39	10th-century	11th	Cooking pot	Slow thrown	Calcitic	Form 2	Body	90.8	Ruminant adipose + dairy + heating

<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_40	10th- century	11th	Cooking pot	Slow thrown	Calcitic	Form 2	Body	72.1	Ruminant adipose + unidentified plant + heating
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_41	10th- century	11th	Olla	Wheel thrown	Calcitic	Form 3b	Body and base	166.5	Dairy + heating
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_42	10th- century	11th	Olla	Wheel thrown	Palermo	Form 3a	Body	86.3	Non-ruminant
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_43	10th- century	11th	Olla	Wheel thrown	Palermo	Form 3a	Body	166.3	Ruminant adipose
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_46	10th- century	11th	Olla	Wheel thrown	Palermo	Form 3a	Body	176.0	Non-ruminant +plant wax (Fennel)+ grape product +heating
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_47	10th- century	11th	Brazier	Handmade	Local?	Form 7	Body	48.7	Non-ruminant
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_49	11th-12th century		Olla	Wheel thrown	Palermo	Form 3a	Body	449.6	Non-ruminant +heating
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_51	11th-12th century		Cooking pot	Slow thrown	Calcitic	Form 2	Body	99.3	Ruminant adipose + heating
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_54	10th- century	11th	Olla	Wheel thrown	Calcitic	Form 3b	Body	29.9	Ruminant adipose
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_55	10th- century	11th	Cooking pot	Slow thrown	Calcitic	Form 2	Body	97.1	Ruminant adipose + unidentified plant wax + heating

<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_56	10th-11th century	Olla	Wheel thrown	Palermo	Form 3a	Body	59.1	Non-ruminant
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_57	10th-11th century	Cooking pot	Slow thrown	Flyish	n/a	Body	4.8	UI
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_58	11th-12th century	Olla	Wheel thrown	Palermo	Form 3a	Rim	79.0	Non-ruminant +plant wax (Fennel)
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_59	11th-12th century	Stone plate	Limestone		Form 6	Base	8.8	Non-ruminant (porcine?)
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_61	10th-11th century	Olla	Wheel thrown	Palermo	Form 3a	Body	112.4	Non-ruminant + grape product +heating
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_73	11th-12th century	Cooking pot	Slow thrown	Calcitic	Form 2	Body	108.1	Non-ruminant +unidentified plant wax
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_C107A	10th-11th century	Olla	Wheel thrown	Palermo	Form 3a	Rim	245.0	Ruminant adipose
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_111	9th-10th century	Olla	Wheel thrown	Palermo	Form 3a	Body	136.9	Non-ruminant + unidentified plant wax + fruit
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_121	11th century	Olla	Wheel thrown	Calcitic	Form 3b	Body	88.3	Ruminant adipose + plant wax (Fennel)
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_122	11th century	Olla	Wheel thrown	Calcitic	Form 3b	Body	39.7	Non-ruminant (porcine?)
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_123	11th century	Olla	Wheel thrown	Palermo	Form 3a	Rim	183.6	Ruminant adipose

<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_130	10th-11th century	Olla	Wheel thrown	Palermo	Form 3a	Rim	17.1	Non-ruminant + grape product
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## **Appendix F Summary of faunal remains from Palermo sites (CSP, GA and PB) and CLESP**

A summary of faunal remains identified at the sites studied in this research (Palazzo Bonagia (Arcoleo 2015; Arcoleo and Sineo 2014); the Gancia Church (Arcoleo 2015; Arcoleo and Sineo 2014); Castello San Pietro (Aniceti 2020) and Casale San Pietro (Aniceti 2020) . These are presented in terms of number of identified species (NISP) and percentage of number of identified species (%NISP) and reference to the original studies that analysed these samples is cited.

Table F.1: Summary of faunal remains at Palazzo Bonagia (PB) reported as number of identified species (NISP) and % NISP

<b>Site</b>	<b>Species</b>	<b>NISP</b>	<b>%NISP</b>	<b>Reference to original study</b>
Palazzo Bonagia (PB)	Caprines	652	65.26	(Arcoleo 2015; Arcoleo and Sineo 2014)
Palazzo Bonagia (PB)	Cattle	207	20.74	
Palazzo Bonagia (PB)	Pig	8	0.8	
Palazzo Bonagia (PB)	Chicken	15	1.5	
Palazzo Bonagia (PB)	Horse	14	1.4	
Palazzo Bonagia (PB)	Deer	7	0.7	
Palazzo Bonagia (PB)	Tuna	16	1.6	
Palazzo Bonagia (PB)	Dog	21	2.1	
Palazzo Bonagia (PB)	Cat	3	0.3	
Palazzo Bonagia (PB)	Rat	2	0.2	
Palazzo Bonagia (PB)	Total	998		

Table F.2: Summary of faunal remains at the Gancia Church (GA) reported as number of identified species (NISP) and % NISP

Site	Species	NISP	%NISP	Reference to original study
La Gancia (GA)	Caprines	2258	71.21	(Arcoleo 2015; Arcoleo and Sineo 2014)
La Gancia (GA)	Cattle	404	12.74	
La Gancia (GA)	Pig	51	1.61	
La Gancia (GA)	Chicken	145	4.57	
La Gancia (GA)	Horse	46	1.45	
La Gancia (GA)	Deer	14	0.44	
La Gancia (GA)	Tuna	88	2.77	
La Gancia (GA)	Other birds	50	1.59	
La Gancia (GA)	Dog	17	0.54	
La Gancia (GA)	Cat	15	0.47	
La Gancia (GA)	Fox	3	0.09	
	Total	3171		

Table F.3: Summary of faunal remains at Castello San Pietro (CSP) reported as number of identified species (NISP) and % NISP

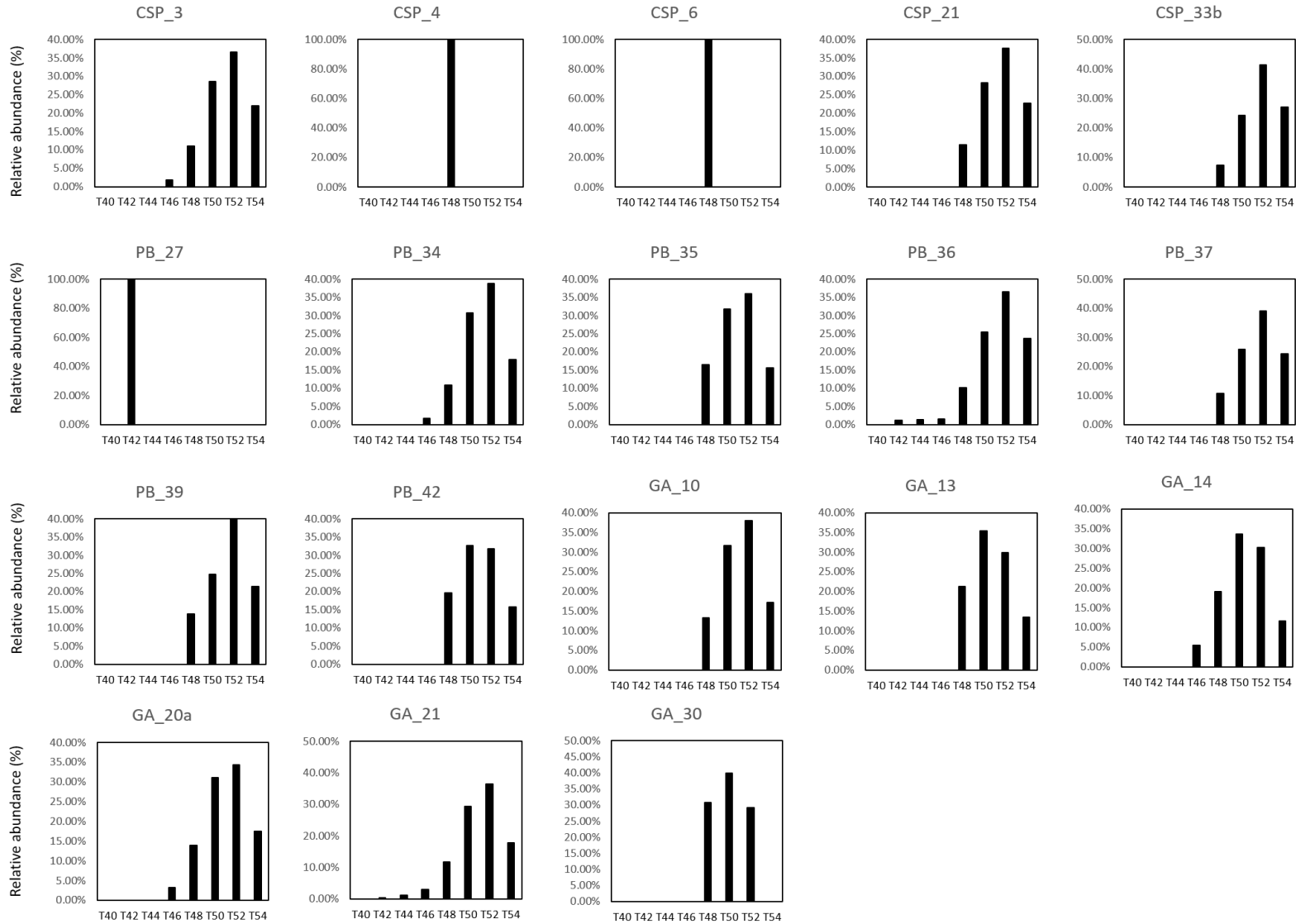
Site	Species	NISP	%NISP	Reference to original study
Castello San Pietro (CSP)	Caprines	231	60.4712	(Aniceti 2020)
Castello San Pietro (CSP)	Cattle	47	12.30366	
Castello San Pietro (CSP)	Pig	56	14.65969	
Castello San Pietro (CSP)	Chicken	18	4.712042	
Castello San Pietro (CSP)	Horse	1	0.26178	
Castello San Pietro (CSP)	Tuna	10	2.617801	
Castello San Pietro (CSP)	Dog	2	0.52356	
Castello San Pietro (CSP)	Cat	3	0.78534	
Castello San Pietro (CSP)	Total	382		

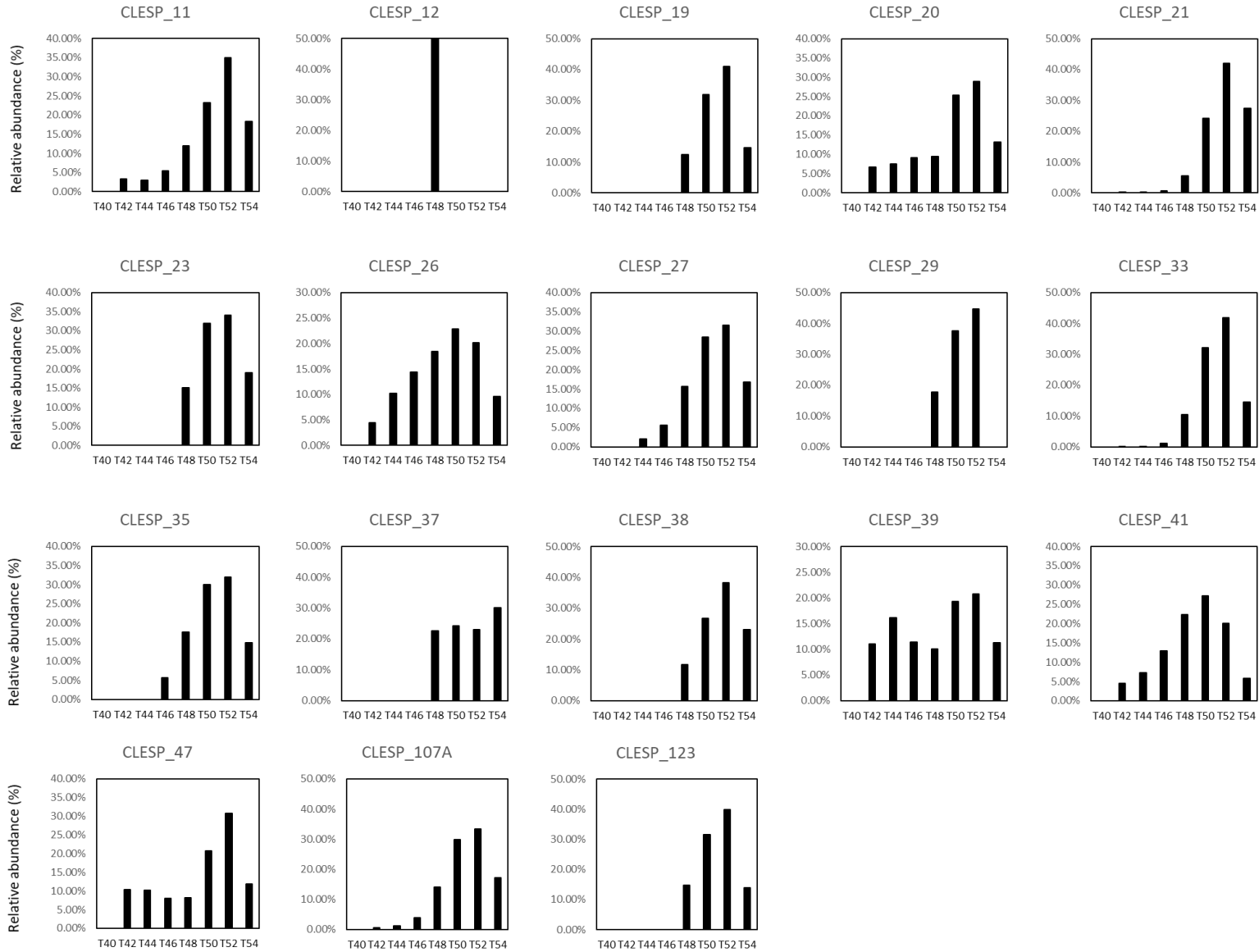
Table F.4: Summary of faunal remains at Casale San Pietro (CSP) reported as number of identified species (NISP) and % NISP

Site	Species	NISP	%NISP	Reference to original study
Casale San Pietro (CLESP)	Caprines	69	35.9375	(Aniceti 2020)
Casale San Pietro (CLESP)	Cattle	23	11.97917	
Casale San Pietro (CLESP)	Pig	62	32.29167	
Casale San Pietro (CLESP)	Chicken	1	0.520833	
Casale San Pietro (CLESP)	Horse	14	7.291667	
Casale San Pietro (CLESP)	Deer	8	4.166667	
Casale San Pietro (CLESP)	Tuna	4	2.083333	
Casale San Pietro (CLESP)	Dog	21	10.9375	
Casale San Pietro (CLESP)	Total	192		

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## **Appendix G TAG distribution profiles of Palermo sites and 10<sup>th</sup>-12<sup>th</sup> century CLESP**





## Appendix H Malic acid and tartaric acid quantities in vegetables

Here the malic acid and tartaric acid quantities identified in a variety of vegetables from previously published studies are presented. These values, alongside values for different fruits published in (Drieu et al. 2021b), were used to produce Figure 34a and Figure 48a in this thesis.

Table H.1. Malic acid and tartaric acid quantities in vegetables. Values taken from previously published studies as shown in table. %TA = tartaric acid/ (tartaric + malic acid)

Category	Product	Reference	Yield tartaric acid (TA) (µg/g)	Yield malic acid (MA) (µg/g)	TA/MA	%TA
Vegetables	Spinach	(Askar,El-Samahy and Abd El-Fadeel 1982)	0.00	13.00	0.00	0.00
Vegetables	Eddoe	(Askar,El-Samahy and Abd El-Fadeel 1982)	18.30	73.90	0.25	0.20
Vegetables	Carrot	(Mabesa,Baldwin and Garner 1979)	0.00	93.38	0.00	0.00
Vegetables	Peas	(Mabesa,Baldwin and Garner 1979)	0.00	15.56	0.00	0.00
Vegetables	Carrot	(Ruhl and Herrmann 1985b)	1.80	236.00	0.01	0.01
Vegetables	Carrot	(Ruhl and Herrmann 1985b)	2.00	524.00	0.00	0.00
Vegetables	Carrot	(Ruhl and Herrmann 1985b)	2.70	502.00	0.01	0.01
Vegetables	Carrot	(Ruhl and Herrmann 1985b)	5.50	327.00	0.02	0.02
Vegetables	Carrot	(Ruhl and Herrmann 1985b)	1.90	305.00	0.01	0.01
Vegetables	Carrot	(Ruhl and Herrmann 1985b)	1.80	275.00	0.01	0.01
Vegetables	Carrot	(Morvai,Molnár-Perl and Knausz 1991)	0.01	0.59	0.01	0.01
Vegetables	Carrot	(Morvai,Molnár-Perl and Knausz 1991)	0.01	0.59	0.01	0.01
Vegetables	Carrot	(Morvai,Molnár-Perl and Knausz 1991)	0.01	0.58	0.01	0.01
Vegetables	Carrot	(Morvai,Molnár-Perl and Knausz 1991)	0.01	0.58	0.01	0.01
Vegetables	Cucumber	(Morvai,Molnár-Perl and Knausz 1991)	0.01	0.17	0.06	0.06
Vegetables	Leek	(Ruhl and Herrmann 1985b)	0.00	192.00	0.00	0.00
Vegetables	Leek	(Ruhl and Herrmann 1985b)	0.00	175.00	0.00	0.00
Vegetables	Leek	(Ruhl and Herrmann 1985b)	0.00	155.00	0.00	0.00
Vegetables	Chives	(Ruhl and Herrmann 1985b)	0.00	240.00	0.00	0.00



Vegetables	Chinese cabbage	(Ruhl and Herrmann 1985b)	0.00	96.00	0.00	0.00
Vegetables	Chinese cabbage	(Ruhl and Herrmann 1985b)	0.00	126.00	0.00	0.00
Vegetables	Chinese cabbage	(Ruhl and Herrmann 1985b)	0.00	108.00	0.00	0.00
Vegetables	Cauliflower	(Ruhl and Herrmann 1985b)	0.00	126.00	0.00	0.00
Vegetables	Cauliflower	(Ruhl and Herrmann 1985b)	0.00	276.00	0.00	0.00
Vegetables	Cauliflower	(Ruhl and Herrmann 1985b)	0.00	239.00	0.00	0.00
Vegetables	Kale	(Ruhl and Herrmann 1985b)	0.00	174.00	0.00	0.00
Vegetables	Kale	(Ruhl and Herrmann 1985b)	0.00	158.00	0.00	0.00
Vegetables	Brussels sprouts	(Ruhl and Herrmann 1985b)	0.00	189.00	0.00	0.00
Vegetables	Brussels sprouts	(Ruhl and Herrmann 1985b)	0.00	262.00	0.00	0.00
Vegetables	Brussels sprouts	(Ruhl and Herrmann 1985b)	0.00	410.00	0.00	0.00
Vegetables	Red cabbage	(Ruhl and Herrmann 1985b)	0.00	75.00	0.00	0.00
Vegetables	Red cabbage	(Ruhl and Herrmann 1985b)	0.00	63.00	0.00	0.00
Vegetables	Red cabbage	(Ruhl and Herrmann 1985b)	0.00	71.00	0.00	0.00
Vegetables	White cabbage	(Ruhl and Herrmann 1985b)	0.00	107.00	0.00	0.00
Vegetables	White cabbage	(Ruhl and Herrmann 1985b)	0.00	96.00	0.00	0.00
Vegetables	White cabbage	(Ruhl and Herrmann 1985b)	0.00	84.00	0.00	0.00
Vegetables	Savoy cabbage	(Ruhl and Herrmann 1985b)	0.00	93.00	0.00	0.00
Vegetables	Savoy cabbage	(Ruhl and Herrmann 1985b)	0.00	105.00	0.00	0.00

Vegetables	Lettuce	(Ruhl and Herrmann 1985b)	5.60	168.00	0.03	0.03
Vegetables	Lettuce	(Ruhl and Herrmann 1985b)	8.10	243.00	0.03	0.03
Vegetables	Lettuce	(Ruhl and Herrmann 1985b)	10.70	92.00	0.12	0.10
Vegetables	Endive	(Ruhl and Herrmann 1985b)	7.00	179.00	0.04	0.04
Vegetables	Endive	(Ruhl and Herrmann 1985b)	6.00	155.00	0.04	0.04
Vegetables	Chicory	(Ruhl and Herrmann 1985b)	6.10	258.00	0.02	0.02
Vegetables	Chicory	(Ruhl and Herrmann 1985b)	12.50	185.00	0.07	0.06
Vegetables	Celery	(Ruhl and Herrmann 1985b)	0.00	495.00	0.00	0.00
Vegetables	Celery	(Ruhl and Herrmann 1985b)	0.00	474.00	0.00	0.00
Vegetables	Celery	(Ruhl and Herrmann 1985b)	0.60	447.00	0.00	0.00
Vegetables	Celery	(Ruhl and Herrmann 1985b)	2.30	226.00	0.01	0.01
Vegetables	Celery	(Ruhl and Herrmann 1985b)	0.00	397.00	0.00	0.00
Vegetables	Beets	(Ruhl and Herrmann 1985b)	0.00	22.00	0.00	0.00
Vegetables	Beets	(Ruhl and Herrmann 1985b)	0.00	18.00	0.00	0.00
Vegetables	Beets	(Ruhl and Herrmann 1985b)	0.00	17.00	0.00	0.00
Vegetables	Spinach	(Ruhl and Herrmann 1985b)	0.00	37.00	0.00	0.00
Vegetables	Spinach	(Ruhl and Herrmann 1985b)	0.00	47.00	0.00	0.00
Vegetables	Spinach	(Ruhl and Herrmann 1985b)	0.00	36.00	0.00	0.00
Vegetables	Spinach	(Ruhl and Herrmann 1985b)	0.00	64.00	0.00	0.00
Onion	Onion	(Liguori et al. 2017)	13.93	60.43	0.23	0.19
Onion	Onion	(Liguori et al. 2017)	6.10	66.33	0.09	0.08
Onion	Onion	(Liguori et al. 2017)	16.15	61.85	0.26	0.21
Onion	Onion	(Liguori et al. 2017)	25.88	57.61	0.45	0.31
Onion	Onion	(Liguori et al. 2017)	12.02	78.94	0.15	0.13
Onion	Onion	(Ruhl and Herrmann 1985b)	0.00	194.00	0.00	0.00
Onion	Onion	(Ruhl and Herrmann 1985b)	0.00	196.00	0.00	0.00
Onion	Onion	(Ruhl and Herrmann 1985b)	0.00	155.00	0.00	0.00
Onion	Onion	(Ruhl and Herrmann 1985b)	0.00	184.00	0.00	0.00
Onion	Onion	(Rodriguez Galdon et al. 2008)	8.90	47.80	0.19	0.16
Onion	Onion	(Rodriguez Galdon et al. 2008)	23.30	49.30	0.47	0.32
Onion	Onion	(Rodriguez Galdon et al. 2008)	19.20	31.10	0.62	0.38
Onion	Onion	(Rodriguez Galdon et al. 2008)	15.50	50.30	0.31	0.24
Onion	Onion	(Rodriguez Galdon et al. 2008)	14.00	34.30	0.41	0.29
Onion	Onion	(Rodriguez Galdon et al. 2008)	25.20	44.70	0.56	0.36

## **Appendix I Ceramic samples from Mazara del Vallo (MZ) and 12<sup>th</sup>-13<sup>th</sup> century CLESP and summary of substance identification of organic residue analysis**

Summary tables of all ceramic samples and their relative information are presented alongside a summary of substance identification from the organic residue analysis relating to chapter 6 of this thesis. All samples analysed from Mazara del Vallo (MZ) are presented in Table I.1. Samples from 12<sup>th</sup>-13<sup>th</sup> century Casale San Pietro are presented in Table I.2.

The full results of organic residue analysis for each of these sites are presented in the supporting information of this thesis: **S3 data. Ceramic samples and organic residue analysis results from 11<sup>th</sup>-14<sup>th</sup> century Mazara del Vallo and Casale San Pietro (chapter 6).**

Table I.1: Summary of ceramic samples from Mazara del Vallo (MZ) and summary substance identification of organic residue analysis. Here the reference to the original ceramic study is given and the Inventory number of each sample reported in the original ceramic study

Site name	Site code	Sample code	Chronology	Form	Technology	Provenance	Reference for ceramic study	Inventory number/ original form code			Summary substance identification
Mazara del Vallo	MZ	MZ_C2	11th century	Cooking pot	Handmade/slow-thrown cooking pot	Mazara	XIII.2.2	Meo, forthcoming	Body	257.71	Ruminant adipose + unidentified plant + heating
Mazara del Vallo	MZ	MZ_C7	11th century	Cooking pot	Handmade/slow-thrown cooking pot	Mazara	XIII.1.2a	Meo, forthcoming	Body	160.27	Ruminant adipose + plant wax? + fruit
Mazara del Vallo	MZ	MZ_C10	11th century	Cooking pot	Handmade/slow-thrown cooking pot	Mazara	XIII.1.1b	Meo, forthcoming	Body	1997.6	Ruminant adipose + plant wax (brassica)+ plant wax (leek) + heating
Mazara del Vallo	MZ	MZ_C12	11th century	Colander?		Mazara	XIII.4.1	Meo, forthcoming	Base	3.75	Under limit of detection
Mazara del Vallo	MZ	MZ_C13	11th century	Cooking pot	Handmade/slow-thrown cooking pot	Mazara	XIII.1.1b	Meo, forthcoming	Body	78.98	Non ruminant + unidentified plant

<b>Mazara del Vallo</b>	MZ	MZ_C14	11th century	Cooking pot	Handmade/slow-thrown cooking pot	Imported	XXXIXb.2.1	Meo, forthcoming	Rim	79.04	Non ruminant + plant wax (brassica+ leek) fruit + heating
<b>Mazara del Vallo</b>	MZ	MZ_C15	11th century	Pan		Mazara	XIII.4.1	Meo, forthcoming	Rim	12.07	Ruminant adipose
<b>Mazara del Vallo</b>	MZ	MZ_C18	11th century	Pan		Mazara	XII.2.5b	Meo, forthcoming	Body and base	6.54	Unidentified animal fat?
<b>Mazara del Vallo</b>	MZ	MZ_C20	11th century	Pan		Mazara	XIII.4.2b	Meo, forthcoming	Rim	1	Under limit of detection
<b>Mazara del Vallo</b>	MZ	MZ_C23	11th century	Pan		Mazara	XIII.5.1	Meo, forthcoming	Rim	24.04	Non ruminant
<b>Mazara del Vallo</b>	MZ	MZ_C25	11th century	Cooking pot	Handmade/slow-thrown cooking pot	West Sicily	XV.1.1	Meo, forthcoming	Body	9.87	Non-ruminant + heating
<b>Mazara del Vallo</b>	MZ	MZ_C26	11th century	Stone plate		West Sicily	n/a	Meo, forthcoming	Base	43.22	Non ruminant

<b>Mazara del Vallo</b>	MZ	MZ_C28	11th century	Cooking pot	Handmade/slow-thrown cooking pot	Mazara	XII.1.6	Meo, forthcoming	Body	149.44	Ruminant + birch bark tar + heating
<b>Mazara del Vallo</b>	MZ	MZ_C34	11th century	Pan		Mazara	XIII.4.2b	Meo, forthcoming	Body and rim	20.31	Undetermined fat
<b>Mazara del Vallo</b>	MZ	MZ_C37	11th century	Lid		Mazara	XIII.6.1	Meo, forthcoming	Rim	37.56	Undetermined fat
<b>Mazara del Vallo</b>	MZ	MZ_C38	11th century	Lid		Mazara	XII.3.1	Meo, forthcoming	Rim	96.77	Undetermined fat
<b>Mazara del Vallo</b>	MZ	MZ_C40	11th century	Cooking pot	Handmade/slow-thrown cooking pot	Mazara	XIII.2.1	Meo, forthcoming	Rim	186.92	Ruminant
<b>Mazara del Vallo</b>	MZ	MZ_C42	11th century	Cooking pot	cooking ware	Mazara	XIV.1.1b	Meo, forthcoming	Body	149.06	Ruminant + plant wax (brassica+ leek)
<b>Mazara del Vallo</b>	MZ	MZ_C44	11th century	Cooking pot	Handmade/slow-thrown cooking pot	Mazara	XXII.1.4a	Meo, forthcoming	Rim	912.3	Non ruminant + plant wax (brassica +leek) + fruit

<b>Mazara del Vallo</b>	MZ	MZ_C45	11th century	Pan		Mazara	XIII.4.2a	Meo, forthcoming	Base	250.41	Non ruminant + heating
<b>Mazara del Vallo</b>	MZ	MZ_C51	11th century	Pan		Mazara	XIII.3.2	Meo, forthcoming	Body	45.01	Non ruminant
<b>Mazara del Vallo</b>	MZ	MZ_C52	11th century	Pan		Mazara	XIII.4.2a	Meo, forthcoming	Base and body	2.53	below limit of detection
<b>Mazara del Vallo</b>	MZ	MZ_C53	11th century	Cooking pot	Handmade/slow-thrown cooking pot	Mazara	XII.1.5a	Meo, forthcoming	Body	139.87	Non ruminant + unidentified plant
<b>Mazara del Vallo</b>	MZ	MZ_C55	11th century	Cooking pot	Handmade/slow-thrown cooking pot	Mazara	XII.1.6	Meo, forthcoming	Body	299.25	Ruminant adipose + unidentified plant wax + fruit
<b>Mazara del Vallo</b>	MZ	MZ_C57	11th century	Cooking pot	Handmade/slow-thrown cooking pot	Mazara	XXII.1.4a	Meo, forthcoming	Body	1135.69	Ruminant adipose + fruit
<b>Mazara del Vallo</b>	MZ	MZ_59	11th century	Cooking pot	Handmade/slow-thrown cooking pot	West Sicily	XV.1.1	Meo, forthcoming	Rim	645.58	Ruminant adipose + plant wax (Brassica+leek)

<b>Mazara del Vallo</b>	MZ	MZ_C60	11th century	Cooking pot	cooking ware	Mazara	XIV.1.1b	Meo, forthcoming	Rim	275.54	Ruminant adipose + plant wax (brassica+leek) + fruit
<b>Mazara del Vallo</b>	MZ	MZ_C61	11th century	Cooking pot	cooking ware	Mazara	n/a	Meo, forthcoming	Rim	18.02	Non ruminant +heating
<b>Mazara del Vallo</b>	MZ	MZ_C62	11th century	Cooking pot	cooking ware	Mazara	XIV.1.1c	Meo, forthcoming	Rim	2000.69	Ruminant adipose
<b>Mazara del Vallo</b>	MZ	MZ_C66	11th century	Cooking pot	Handmade/slow-thrown cooking pot	Mazara	XII.1.5a	Meo, forthcoming	Rim	352.37	Ruminant adipose
<b>Mazara del Vallo</b>	MZ	MZ_C68	11th century	Brazier		Mazara	XX.19	Meo, forthcoming	Body	10.25	Non ruminant
<b>Mazara del Vallo</b>	MZ	MZ_C77	11th century	Cooking pot	Handmade/slow-thrown cooking pot	Mazara	XIII.2.2	Meo, forthcoming	Body	164.42	Ruminant adipose +plant wax (brassica +leek)
<b>Mazara del Vallo</b>	MZ	MZ_C78	11th century	Cooking pot	Handmade/slow-thrown cooking pot	Mazara	XIII.2.2	Meo, forthcoming	Body	92.68	Ruminant adipose + fruit
<b>Mazara del Vallo</b>	MZ	MZ_C79	11th century	Cooking pot	Handmade/slow-thrown cooking pot	Mazara	XIII.2.1	Meo, forthcoming	Body and base	1020.26	Ruminant adipose + heating
<b>Mazara del Vallo</b>	MZ	MZ_C81	11th century	Pan		Mazara	XIII.3.1	Meo, forthcoming	Body	194.72	Dairy + heating



<b>Mazara del Vallo</b>	MZ	MZ_C82	11th century	Cooking pot	Handmade/slow-thrown cooking pot	Mazara	XIV.1.2	Meo, forthcoming	Body	63.61	Non- ruminant + heating
<b>Mazara del Vallo</b>	MZ	MZ_C83	11th century	Pan		Egypt	XVIII.1.1	Meo, forthcoming	Rim	30.04	Non- ruminant
<b>Mazara del Vallo</b>	MZ	MZ__84	11th century	Cooking pot	Handmade/slow-thrown cooking pot	West Mediterranean	XVII.1.1	Meo, forthcoming	Rim	13.73	Non- ruminant
<b>Mazara del Vallo</b>	MZ	MZ_C85	11th century	Lid		West Sicily	XV.3.1	Meo, forthcoming	Body	89.94	Non ruminant + heating
<b>Mazara del Vallo</b>	MZ	MZ_C86B	11th century	Cooking pot		Mazara	XIII.1.1	Meo, forthcoming	Body	227.54	Ruminant adipose + heating
<b>Mazara del Vallo</b>	MZ	MZ_C88	11th century	Cooking pot		Mazara	XIV.1.1a	Meo, forthcoming	Rim	10.87	Non ruminant +fruit
<b>Mazara del Vallo</b>	MZ	MZ_C128	Late 13th-early 14th	Small cooking pot	Partially glazed cooking ware	Messina	XXXVII.2.4	Orecchioni, forthcoming	Rim	34.69	Non- ruminant + fruit
<b>Mazara del Vallo</b>	MZ	MZ_C129	13th century	Bowl	Unglazed cooking ware	Mazara	XII.2.3	Orecchioni, forthcoming	Body	100.77	Dairy + fruit+ heating
<b>Mazara del Vallo</b>	MZ	MZ_C130	13th century	Small cooking pot	Partially glazed cooking ware	Messina	XXXVII.2.3	Orecchioni, forthcoming	Rim	16.85	Non ruminant + wine (Grape product)
<b>Mazara del Vallo</b>	MZ	MZ_C131	13th century	Small bowl	Glazed cooking ware	Mazara	XXXVIII.1.1	Orecchioni, forthcoming	Body	251.47	Ruminant+ plant wax (brassica)
<b>Mazara del Vallo</b>	MZ	MZ_C132	13th century	Cooking pot	partially glazed cooking ware	Mazara?		Orecchioni, forthcoming	Rim	263.92	Non ruminant + plant wax (brassica)+ fruit
<b>Mazara del Vallo</b>	MZ	MZ_C133	13th century	Cooking pot	Partially glazed cooking ware	Messina	XXXVII.3.1	Orecchioni, forthcoming	Rim	73.27	Non ruminant + plant wax (brassica)+ fruit

<b>Mazara del Vallo</b>	MZ	MZ_C134	13th century	Small cooking pot	Partially glazed cooking ware	Messina	XXXVII.2.3	Orecchioni, forthcoming	Base	114.82	Ruminant + fruit + heating
<b>Mazara del Vallo</b>	MZ	MZ_C135	13th century	Small bowl	Glazed cooking ware	Mazara	XXXVIII.2.1	Orecchioni, forthcoming	Rim	5845.75	Ruminant + plant wax (brassica)+heating
<b>Mazara del Vallo</b>	MZ	MZ_C136	13th century	Small cooking pot	Glazed cooking ware	Mazara	XXXVIII.4.1	Orecchioni, forthcoming	Body	223.86	Non ruminant +plant wax (brassica)+ heating
<b>Mazara del Vallo</b>	MZ	MZ_C137	13th century	Cooking pot	Partially glazed cooking ware	Messina	XXXVII.3.3	Orecchioni, forthcoming	Rim	37.56	Non ruminant + fruit + heating
<b>Mazara del Vallo</b>	MZ	MZ_C138	13th century	Cooking pot	Partially glazed cooking ware	Messina	XXXVII.3.3	Orecchioni, forthcoming	Base	96.77	Non ruminant
<b>Mazara del Vallo</b>	MZ	MZ_C139	13th century	Cooking pot	Handmade	Mazara	XXXVI.4.7	Orecchioni, forthcoming	Body	113.53	Non-ruminant + plant wax (brassica)+ fruit
<b>Mazara del Vallo</b>	MZ	MZ_C140	13th century	Small cooking pot	Unglazed cooking ware	Mazara	XL15.5	Orecchioni, forthcoming	Rim	43.93	Non- ruminant + fruit +heating
<b>Mazara del Vallo</b>	MZ	MZ_C141	14th century	Cooking pot	Unglazed cooking ware	Catalonia	XXXIV.2.2b	Orecchioni, forthcoming	Body	143.77	Non ruminant + heating
<b>Mazara del Vallo</b>	MZ	MZ_C143	13th century	Cooking pot	Handmade	Mazara	n/a	Orecchioni, forthcoming	Body	58.99	Ruminant+ plant wax (brassica)
<b>Mazara del Vallo</b>	MZ	MZ_C144	13th century	Cooking pot	Handmade	Mazara	XXXVI.4.10	Orecchioni, forthcoming	Base	31.19	Ruminant + plant wax (brassica) +fruit + heating
<b>Mazara del Vallo</b>	MZ	MZ_C145	13th century	Cooking pot	Partially glazed cooking ware	Messina	XXXVII.3.1	Orecchioni, forthcoming	Rim	132.19	Non - ruminant + plant wax (brassica)+ fruit
<b>Mazara del Vallo</b>	MZ	MZ_C160	13th century	Cooking pot	Partially glazed cooking ware	Messina	I.2a1/a	Molinari, Cassi 2006	Rim	417.86	Ruminant + plant wax (brassica)+ fruit + heating

<b>Mazara del Vallo</b>	MZ	MZ_C162	13th century	Small cooking pot	Partially glazed cooking ware	Messina	XXXVII.2.2	Molinari, Cassi 2006	Body	145.28	Ruminant + fruit + heating
<b>Mazara del Vallo</b>	MZ	MZ_C163	13th century	Small cooking pot	Partially glazed cooking ware	Messina	XXXVII.2.4	Molinari, Cassi 2006	Rim	69.95	Ruminant
<b>Mazara del Vallo</b>	MZ	MZ_C165	13th century	Cooking pot	Partially glazed cooking ware	Messina	I.2.4	Molinari, Cassi 2006	Base	44.95	Non- ruminant
<b>Mazara del Vallo</b>	MZ	MZ_C166	13th century	Cooking pot	Partially glazed cooking ware	Messina	I.2.4	Molinari, Cassi 2006	Base	33.37	Non- ruminant
<b>Mazara del Vallo</b>	MZ	MZ_C170	13th century	Small cooking pot	Partially glazed cooking ware	Messina	I.2.3	Molinari, Cassi 2006	Base and body	732.76	Ruminant + plant wax + fruit+ heating
<b>Mazara del Vallo</b>	MZ	MZ_C176	13th century	Small cooking pot	Partially glazed cooking ware	Messina	I.2.3	Molinari, Cassi 2006	Handle and Body	<1	below limit of detection
<b>Mazara del Vallo</b>	MZ	MC_C178	13th century	Cooking pot	Partially glazed cooking ware	Messina	I.2.4	Molinari, Cassi 2006	Rim	129.62	Non- ruminant + fruit +heating
<b>Mazara del Vallo</b>	MZ	MZ_C179	14th century	Cooking pot	Glazed ware cooking ware	Catalonia	XXXIV.2.2a	Orecchioni, forthcoming	Body	1357.54	Ruminant + plant wax (brassica) + fruit+ heating
<b>Mazara del Vallo</b>	MZ	MZ_C180	14th century	Bowl/pan	Glazed ware cooking ware	Catalonia	XXXIX.1.2	Orecchioni, forthcoming	Base	515.36	Ruminant + heating
<b>Mazara del Vallo</b>	MZ	MZ_C181	14th century	Bowl/pan	Glazed ware cooking ware	Catalonia	XXXIX.1.1	Orecchioni, forthcoming	Body	232.36	Non- ruminant + fruit + heating
<b>Mazara del Vallo</b>	MZ	MZ_C182	14th century	Bowl/pan	Glazed ware cooking ware	Catalonia	XXXIX.1.3	Orecchioni, forthcoming	Rim	106.21	Ruminant + heating
<b>Mazara del Vallo</b>	MZ	MZ_C183	late 13th-early 14th	Cooking pot	Glazed ware cooking ware	Mazara	XXXVII.5.1	Orecchioni, forthcoming	Rim	47.87	Non- ruminant

<b>Mazara del Vallo</b>	MZ	MZ_C184	14th century	Cooking pot	Glazed cooking ware	Catalonia	XXXIV.2.2a	Orecchioni, forthcoming	Body	117.42	Ruminant + plant wax (brassica) + heating +fruit
<b>Mazara del Vallo</b>	MZ	MZ_C189b	13th century	Cooking pot	Handmade	Mazara	I.1.1/a	Molinari, Cassi 2006	Body and base	6032.09	Ruminant adipose + unidentified plant + fruit
<b>Mazara del Vallo</b>	MZ	MZ_C227	11th century	Jug	Fine ware	Mazara	XX.18.2a	Meo, forthcoming	Body	474.2	Dairy +fruit + plant wax (brassica +leek)
<b>Mazara del Vallo</b>	MZ	MZ_C232	11th century	Jug	Fine ware	Mazara	XX.18.2a	Meo, forthcoming	Body	99.05	Non- ruminant + fruit
<b>Mazara del Vallo</b>	MZ	MZ_C236	11th century	Jug	Fine ware	Mazara	XX.18.2a	Meo, forthcoming	Body	21.93	Ruminant + fruit
<b>Mazara del Vallo</b>	MZ	MZ_C240	11th century	Jug	Fine ware	Mazara	XX.12.1	Meo, forthcoming	Body	9.65	Ruminant + fruit
<b>Mazara del Vallo</b>	MZ	MZ_C241	11th century	Bowl/lid		Mazara	XX.6.1	Meo, forthcoming	Body	28.81	Non ruminant
<b>Mazara del Vallo</b>	MZ	MZ_C242	11th century	Jug	Fine ware	Mazara		Meo, forthcoming	Body	453.74	Ruminant + Plant oil + fruit
<b>Mazara del Vallo</b>	MZ	MZ_C265	13th century	Jug	Fine ware	Mazara	XX.18.2a	Orecchioni, forthcoming	Body	3.5	below limit of detection
<b>Mazara del Vallo</b>	MZ	MZ_387	11th century	Cooking pot		Mazara	n/a	Meo, forthcoming	Body	4	below limit of detection
<b>Mazara del Vallo</b>	MZ	MZ_C396	11th century	Stone plate		West Sicily	n/a	Meo, forthcoming	Body	5.05	Unidentified fat
<b>Mazara del Vallo</b>	MZ	MZ_C400	11th century	Tripod		Mazara	XX.19.3	Meo, forthcoming	Body	218.65	Ruminant + heating
<b>Mazara del Vallo</b>	MZ	MZ_C401	11th century	Tripod		Mazara	XX.19.3	Meo, forthcoming	Body	8.59	Non- ruminant

Mazara del Vallo	MZ	MZ_C244	11th century	Tripod		Mazara	XX.19.3	Meo, forthcoming	Body	<1	below limit of detection
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Table I.2: Summary of ceramic samples from 12<sup>th</sup>-13<sup>th</sup> century Casale San Pietro (CLESP) and summary substance identification of organic residue analysis. Here the reference to the original ceramic study is given and the Inventory number of each sample reported in the original ceramic study

Site name	Site code	Sample code	Chronology	Form	Technology	Provenance	Reference for ceramic study	Summary substance identification
Casale Pietro	San	CLESP	CLESP_69	12th Century	olla	wheel thrown C. ware	Palermo	Ruminant adipose +plant oil +fruit
Casale Pietro	San	CLESP	CLESP_70	12th Century	cooking pot	handmade C. ware	Fly	Ruminant adipose +plant wax (fennel) + heating
Casale Pietro	San	CLESP	CLESP_72	12th Century	cooking pot	handmade C. ware	Fly	Dairy + unidentified plant
Casale Pietro	San	CLESP	CLESP_88	12th Century	olla	wheel thrown C. ware	Palermo	Non- ruminant
Casale Pietro	San	CLESP	CLESP_89	12th Century	cooking pot	handmade C. ware	Fly	Non- ruminant + plant oil + fruit

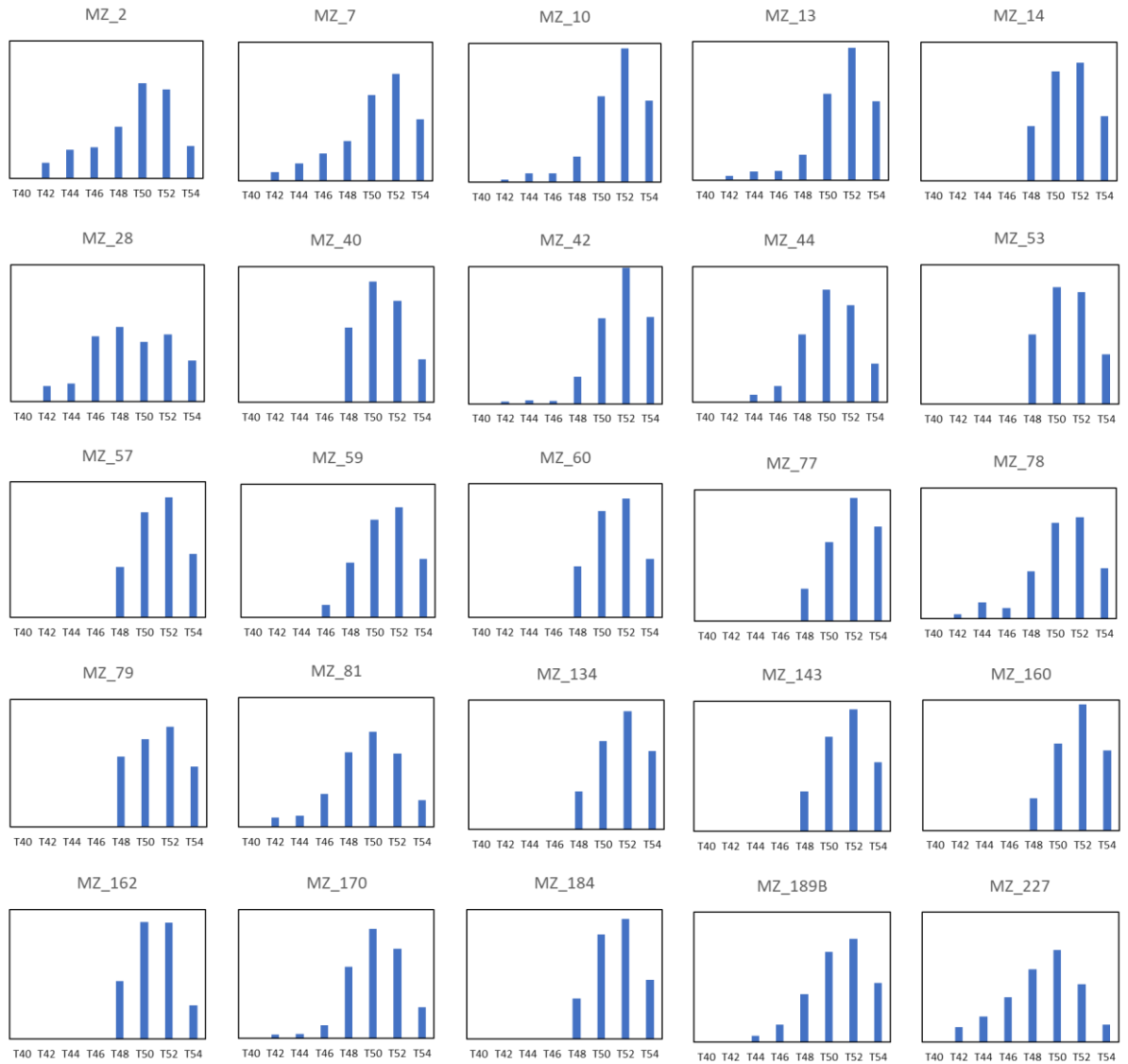
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_93	12th Century	olla	wheel thrown C. ware	Palermo		Non ruminant + heating
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_96A	12th Century	cooking pot	handmade C. ware	Fly		Ruminant adipose
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_97	12th Century	cooking pot	handmade C. ware	Fly		Below limit of detection
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_98	12th Century	olla	wheel thrown C. ware	Palermo		Dairy + plant wax (fennel) + heating
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_99	12th Century	olla	wheel thrown C. ware	Ca		Dairy + heating
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_100	12th Century	olla	wheel thrown C. ware	Palermo		Non ruminant + heating
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_104	12th Century	cooking pot	handmade C. ware	Fly		Ruminant + plant wax (fennel)
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_105	12th Century	olla	wheel thrown C. ware	Palermo		Dairy
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_115	12th Century	olla	wheel thrown C. ware	Palermo		Non - ruminant
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_116	12th Century	olla	wheel thrown C. ware	Palermo		Ruminant adipose

<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_117	12th Century	cooking pot	handmade C. ware	Fly		Below limit of detection
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_129	12th Century	lid	wheel thrown C. ware	Palermo		Non- ruminant
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_C178b	12th Century		Handmade cooking ware			Below limit of detection
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_C187	Late 12th - early 13th Century	cooking pot	partially glazed cooking ware	Messina		Ruminant adipose + fruit + heating
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_C188	Late 12th - early 13th Century	cooking pot	partially glazed cooking ware	Messina		Non ruminant + plant wax (fennel) + pine resin
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_C189	Late 12th - early 13th Century	cooking pot	partially glazed cooking ware	Messina		Non- ruminant + heating
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_C190	late 12th Century	pan/bowl	Handmade cooking ware	local?		Ruminant + fruit + heating
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_C191	late 12th Century	lid	cooking ware	Palermo?		Non ruminant
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_C192	13th century	cooking pot	cooking ware	Palermo?		Non- ruminant

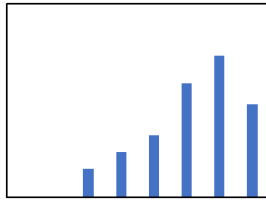
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_C193	late 12th century	cooking pot	cooking ware	Palermo?		Non-ruminant + grape product +heating
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_C194	13th century	cooking pot	cooking ware	Palermo?		Non-ruminant + plant wax (fennel)
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_C195	late 12th Century	cooking pot	cooking ware	Palermo?		Non-ruminant
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_C196	13th century	cooking pot	cooking ware	Palermo?		Ruminant + fruit
<b>Casale Pietro</b>	<b>San</b>	CLESP	CLESP_C197	13th century	cooking pot with handle	cooking ware	Palermo?		Ruminant + fruit



## Appendix J TAG distribution profile of ceramic samples from Mazara del Vallo (MZ) and 12<sup>th</sup>-13<sup>th</sup> century Casale San Pietro (CLESP)

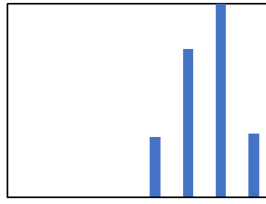


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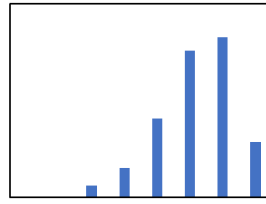
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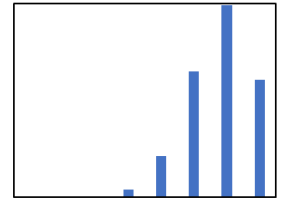
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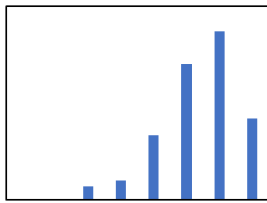
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CLESP\_187



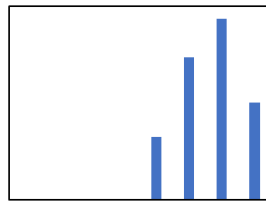
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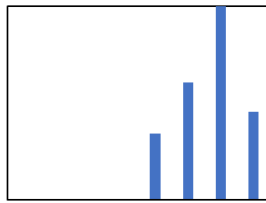
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CLESP\_191



T40 T42 T44 T46 T48 T50 T52 T54

CLESP\_194



T40 T42 T44 T46 T48 T50 T52 T54

## Appendix K Summary of faunal assemblages from all sites and estimated meat weights of main domesticates.

NISP values of the main domesticates at each site (cattle, caprines, pigs and chicken) were converted to estimated meat weights by multiplying the total NISP of each species by assumed meat weight contributions of each species (kg.) Meat weight values (100kg for cattle, 15kg for caprines, 35kg for pigs and 0.7kg for chickens) were estimated based on medieval meat weight values reported in the literature (Boessneck et al. 1971; O'Connor 1991; Dyer 1989, 59)

Table K.1: Summary of faunal assemblages and estimated meat weights of main domesticates from Palazzo Bonagia (PB)

Site name	Site code	Chronology	Species (en)	Species (la)	NISP	%NISP	MNI
Palazzo Bonagia	PB	10th-11th	Caprines	<i>Caprovini</i>	652	65.26	46
Palazzo Bonagia	PB	10th-11th	Sheep	<i>Ovis aries</i>	46	4.6	
Palazzo Bonagia	PB	10th-11th	Goat	<i>Capra hircus</i>	7	0.7	
Palazzo Bonagia	PB	10th-11th	Cattle	<i>Bos taurus</i>	207	20.74	9
Palazzo Bonagia	PB	10th-11th	Dog	<i>Canis familiaris</i>	21	2.1	2
Palazzo Bonagia	PB	10th-11th	Tuna	<i>Thunnus thynnus</i>	16	1.6	2
Palazzo Bonagia	PB	10th-11th	Chicken	<i>Gallus domesticus</i>	15	1.5	3
Palazzo Bonagia	PB	10th-11th	Horse	<i>Equus caballus</i>	14	1.4	1
Palazzo Bonagia	PB	10th-11th	Pig	<i>Sus domesticus</i>	8	0.8	1
Palazzo Bonagia	PB	10th-11th	Deer	<i>Cervus elaphus</i>	7	0.7	1
Palazzo Bonagia	PB	10th-11th	Cat	<i>Felis catus</i>	3	0.3	1
Palazzo Bonagia	PB	10th-11th	Rat	<i>Rattus rattus</i>	2	0.2	1
Palazzo Bonagia	PB		Total		998		

Species	NISP	Estimated meat weight based on literature (kg)	Meat weight contribution of species (Estimated meat weight x NISP (kg))	Percentage meat weight contribution of species (%)
Caprines	625	14	9128	30.30
Cattle	207	100	20700	68.78
Chicken	15	0.7	10.5	0.03
Pig	8	35	280	0.9

Table K.2: Summary of faunal assemblages and estimated meat weights of main domesticates from La Gancia (GA)

Site name	Site code	Chronology	Species (en)	Species (la)	NISP	%NISP	MNI
La Gancia	GA	10th-11th	Caprines	<i>Caprovini</i>	2258	71.21	69
La Gancia	GA	10th-11th	Sheep	<i>Ovis aries</i>	30	0.95	
La Gancia	GA	10th-11th	Goat	<i>Capra hircus</i>	43	1.36	
La Gancia	GA	10th-11th	Cattle	<i>Bos taurus</i>	404	12.74	7
La Gancia	GA	10th-11th	chicken	<i>Gallus domesticus</i>	145	4.57	17
La Gancia	GA	10th-11th	Horse	<i>Equus caballus</i>	46	1.45	1
La Gancia	GA	10th-11th	Pig	<i>Sus domesticus</i>	51	1.61	4
La Gancia	GA	10th-11th	Dog	<i>Canis familiaris</i>	17	0.54	2
La Gancia	GA	10th-11th	Cat	<i>Felis catus</i>	15	0.47	1
La Gancia	GA	10th-11th	Deer	<i>Cervus elaphus</i>	14	0.44	2
La Gancia	GA	10th-11th	Fox	<i>Vulpes vulpes</i>	3	0.09	1
La Gancia	GA	10th-11th	Rabbit	<i>Oryctolagus cuniculus</i>	7	0.22	1
La Gancia	GA	10th-11th	Mallard	<i>Anas platyrhynchos domesticus</i>	5	0.16	1
La Gancia	GA	10th-11th	Goose	<i>Anser anser</i>	10	0.32	1
La Gancia	GA	10th-11th	Pheasant	<i>Fasianidi</i>	10	0.32	
La Gancia	GA	10th-11th	Duck	<i>Antatidi</i>	25	0.79	
La Gancia	GA	10th-11th	Tuna	<i>Thunnus thynnus</i>	88	2.77	
La Gancia	GA	10th-11th	total		3171	100	107

Species	NISP	Estimated meat weight based on literature (kg)	Meat weight contribution of species (Estimated meat weight x NISP (kg))	Percentage meat weight contribution of species (%)
Caprines	2258	14	999.9	42.78
Cattle	404	100	1274.0	54.67
Chicken	145	0.7	3.20	0.14
Pig	51	35	56.35	2.42

Table K.3: Summary of faunal assemblages and estimated meat weights of main domesticates from Castello San Pietro (CSP)

Site name	Site code	Chronology	Species (en)	Species (la)	NISP	%NISP
Castello San Pietro	CSP	9th	Caprines	<i>Caprovini</i>	231	60.47120419
Castello San Pietro	CSP	9th	Sheep	<i>Ovis aries</i>		
Castello San Pietro	CSP	9th	Goat	<i>Capra hircus</i>		
Castello San Pietro	CSP	9th	Cattle	<i>Bos taurus</i>	47	12.30366492
Castello San Pietro	CSP	9th	Dog	<i>Canis familiaris</i>	2	0.523560209
Castello San Pietro	CSP	9th	Tuna	<i>Thunnus thynnus</i>	10	2.617801047
Castello San Pietro	CSP	9th	Chicken	<i>Gallus domesticus</i>	18	4.712041885
Castello San Pietro	CSP	9th	Horse	<i>Equus caballus</i>	1	0.261780105
Castello San Pietro	CSP	9th	Pig	<i>Sus domesticus</i>	56	14.65968586
Castello San Pietro	CSP	9th	Deer	<i>Cervus elaphus</i>		0
Castello San Pietro	CSP	9th	Cat	<i>Felis catus</i>	3	0.785340314
Castello San Pietro	CSP	9th	Rat	<i>Rattus rattus</i>		0
Castello San Pietro	CSP		Total		382	

Species	NISP	Estimated meat weight based on literature (kg)	Meat weight contribution of species (Estimated meat weight x NISP (kg))	Percentage meat weight contribution of species (%)
Caprines	2258	14	3234	32.64
Cattle	404	100	4700	47.44
Chicken	145	0.7	12.6	0.12
Pig	51	35	1960	19.78

Table K.4: Summary of faunal assemblages and estimated meat weights of main domesticates from Casale San Pietro (CLSEP)

Site name	Site code	Chronology	Species (en)	Species (la)	NISP	%NISP
Casale San Pietro	CLESP	10th-11th	Caprines	<i>Caprovini</i>	69	35.9375
Casale San Pietro	CLESP	10th-11th	Sheep	<i>Ovis aries</i>	69	35.9375
Casale San Pietro	CLESP	10th-11th	Goat	<i>Capra hircus</i>	0	0
Casale San Pietro	CLESP	10th-11th	Cattle	<i>Bos taurus</i>	23	11.97916667
Casale San Pietro	CLESP	10th-11th	Dog	<i>Canis familiaris</i>	21	10.9375
Casale San Pietro	CLESP	10th-11th	Tuna	<i>Thunnus thynnus</i>	4	2.083333333
Casale San Pietro	CLESP	10th-11th	Chicken	<i>Gallus domesticus</i>	1	0.5208333333
Casale San Pietro	CLESP	10th-11th	Horse	<i>Equus caballus</i>	14	7.291666667
Casale San Pietro	CLESP	10th-11th	Pig	<i>Sus domesticus</i>	62	32.29166667
Casale San Pietro	CLESP	10th-11th	Deer	<i>Cervus elaphus</i>	8	4.166666667
Casale San Pietro	CLESP	10th-11th	Cat	<i>Felis catus</i>		0
Casale San Pietro	CLESP	10th-11th	Rat	<i>Rattus rattus</i>		0
Casale San Pietro	CLESP		Total		192	

Species	NISP	Estimated meat weight based on literature (kg)	Meat weight contribution of species (Estimated meat weight x NISP (kg))	Percentage meat weight contribution of species (%)
Caprines	2258	14	966	17.77
Cattle	404	100	2300	42.31
Chicken	145	0.7	0.7	0.013
Pig	51	35	2170	39.91

## Appendix L Carbon and nitrogen stable isotopes

Presented here are the bulk carbon ( $\delta^{13}\text{C}$ ) and nitrogen ( $\delta^{15}\text{N}$ ) isotope values of human and faunal remains used to make Figure 50 of this thesis. These values were obtained, with permission, from Alice Ughi who as part of her forthcoming PhD research and as part of the wider SICTRANSIT project investigated direct diet of populations in medieval Sicily through carbon ( $\delta^{13}\text{C}$ ) and nitrogen ( $\delta^{15}\text{N}$ ) isotope data of human and faunal remains (Ughi and Alexander forthcoming; Ughi unpublished). The human remains come from the sites of Castello San Pietro (CSP) and La Gancia Church (GA) and Oratorio dei Bianchi (OB). The faunal remains are from two Islamic sites in Palermo (Corso dei Mille and Sant 'Antonino).

Table L.1: Carbon ( $\delta^{13}\text{C}$ ) and nitrogen ( $\delta^{15}\text{N}$ ) stable isotopes of human and faunal remains from sites in Palermo (Ughi and Alexander forthcoming; Ughi unpublished)

Sample	Region	Site	Site name	Species	Chronological period	Historical period	Faith	Age	Age category	Sex (ost.)	Radiocarbon date@95.4% bar	$\delta^{13}\text{C}$ (‰)	$\delta^{15}\text{N}$ (‰)
CSP_BN 01	Palermo	CSP	Castello San Pietro	Human	late 7th-8th	Byz-Isl	Islamic	Adult		M	665-770	-18.8	11.4
CSP_BN 02	Palermo	CSP	Castello San Pietro	Human	late 7th-8th	Byz-Isl	Islamic	Adult		M	685-869	-17.9	9.7
CSP_BN 03	Palermo	CSP	Castello San Pietro	Human	late 7th-8th	Byz-Isl	Islamic	Adult		F	687-870	-19.4	9.2
CSP_BN 04	Palermo	CSP	Castello San Pietro	Human	late 7th-8th	Byz-Isl	Islamic	Adult		F	671-770	-18.9	9.6
GA_BN 01	Palermo	GA	La Gancia	Human	8-11th	Isl	Christian	Adult		M	901-1025	-19.03	9.98
GA_BN 02	Palermo	GA	La Gancia	Human	8-11th	Isl	Islamic	Adult		M	771-941	-18.56	10.04

<b>GA_BNO 3</b>	Palermo	GA	La Gancia	Human	8-11th	Isl		Non adult		N/A	721-889	-17.99	13.3
<b>GA_BNO 4</b>	Palermo	GA	La Gancia	Human	8-11th	Isl	Christian	Adult		M		-18.85	9.22
<b>GA_BNO 5</b>	Palermo	GA	La Gancia	Human	8-11th	Isl	Islamic	Adult		M	666-770	-17.78	12.95
<b>GA_BNO 7</b>	Palermo	GA	La Gancia	Human	8-11th	Isl	Islamic	Adult		F	977-1035	-19.21	8.11
<b>GA_BNO 8</b>	Palermo	GA	La Gancia	Human	8-11th	Isl	Islamic	Adult		M		-18.61	9.95
<b>GA_BNO 9</b>	Palermo	GA	La Gancia	Human	8-11th	Isl	Islamic	Adult		N/A	775-968	-19.38	7.66
<b>OB_BNO 1</b>	Palermo	OB	Oratorio dei Bianchi	Human	9-10th	Isl	Islamic	Adult		M	777-973	-19.86	7.36
<b>OB_BNO 2</b>	Palermo	OB	Oratorio dei Bianchi	Human	9-10th	Isl	Islamic	Adult		F	770-889	-19.65	7.13
<b>OB_BNO 3</b>	Palermo	OB	Oratorio dei Bianchi	Human	9-10th	Isl	Islamic	Adult		M	776-969	-18.74	9.88
<b>CDM_B 1</b>	Palermo	CDM	Corso dei Mille	Cattle	7-8th						1060-1153	-23	6.7
<b>CDM_B 2</b>	Palermo	CDM	Corso dei Mille	Cattle	7-8th							-20.9	6.3
<b>CDM_B 3</b>	Palermo	CDM	Corso dei Mille	Cattle	7-8th							-21.5	6
<b>CDM_B 4</b>	Palermo	CDM	Corso dei Mille	Sheep	7-8th						1292-1407	-21	6.3



<b>CDM_B5</b>	Palermo	CDM	Corso dei Mille	Cattle	7-8th														-18.8	5.8	
<b>CDM_B6</b>	Palermo	CDM	Corso dei Mille	Sheep	7-8th														-20.2	5.3	
<b>CDM_B7</b>	Palermo	CDM	Corso dei Mille	Cattle	7-8th														-20.3	5.9	
<b>CDM_B8</b>	Palermo	CDM	Corso dei Mille	Cattle	7-8th							1214-1389							-20.1	6.4	
<b>SAN_B1</b>	Palermo	SAN	Sant 'Antonino	Bos taurus	8-10th														-20.1	6.4	
<b>SAN_B2</b>	Palermo	SAN	Sant 'Antonino	Bos taurus	8-10th							777-974							-19.1	6	
<b>SAN_B3</b>	Palermo	SAN	Sant 'Antonino	Bos taurus	8-10th														-21.5	6.2	
<b>SAN_B4</b>	Palermo	SAN	Sant 'Antonino	Bos taurus	8-10th														-20.5	6.8	
<b>SAN_B5</b>	Palermo	SAN	Sant 'Antonino	Bos taurus	8-10th							776-968							-21	7.3	
<b>SAN_B6</b>	Palermo	SAN	Sant 'Antonino	Bos taurus	8-10th														-21.3	6.5	
<b>SAN_B7</b>	Palermo	SAN	Sant 'Antonino	Bos taurus	8-10th														-21.1	3.9	
<b>SAN_B8</b>	Palermo	SAN	Sant 'Antonino	Bos taurus	8-10th																
<b>SAN_B9</b>	Palermo	SAN	Sant 'Antonino	Bos taurus	8-10th															-18.5	5.6

<b>SAN_B1 0</b>	Palermo	SAN	Sant 'Antonino	Bos taurus	8-10th						771-950	-21.1	7.3
<b>SAN_B1 1</b>	Palermo	SAN	Sant 'Antonino	Ovis aries	8-10th							-21.1	5.2
<b>SAN_B1 2</b>	Palermo	SAN	Sant 'Antonino	Ovis aries	8-10th						776-969	-20.9	6.4
<b>SAN_B1 3</b>	Palermo	SAN	Sant 'Antonino	Ovis aries	8-10th						773-961	-21.8	7.1
<b>SAN_B1 4</b>	Palermo	SAN	Sant 'Antonino	Ovis aries	8-10th						694-882	-22.1	5.5
<b>SAN_B1 5</b>	Palermo	SAN	Sant 'Antonino	Ovis aries	8-10th							-21.1	6.5
<b>SAN_B1 6</b>	Palermo	SAN	Sant 'Antonino	Ovis aries	8-10th						726-891	-21.4	6.3
<b>SAN_B1 7</b>	Palermo	SAN	Sant 'Antonino	Ovis aries	8-10th							-20.7	5.5
<b>SAN_B1 8</b>	Palermo	SAN	Sant 'Antonino	Ovis aries	8-10th						774-962	-21.2	6.4
<b>SAN_B1 9</b>	Palermo	SAN	Sant 'Antonino	Ovis aries	8-10th						667-775	-21	4
<b>SAN_B2 0</b>	Palermo	SAN	Sant 'Antonino	Ovis aries	8-10th						726-891	-21.1	6.5
<b>SAN_B2 1</b>	Palermo	SAN	Sant 'Antonino	Ovis aries	8-10th								

<b>SAN_B2 2</b>	Paler mo	SAN	Sant 'Antonino	Gallus gallus domesti cus	8-10th									-20.1	9.7
<b>SAN_B2 3</b>	Paler mo	SAN	Sant 'Antonino	Gallus gallus domesti cus	8-10th									-19.1	7.4
<b>SAN_B2 4</b>	Paler mo	SAN	Sant 'Antonino	Gallus gallus domesti cus	8-10th									-19	7
<b>SAN_B2 5</b>	Paler mo	SAN	Sant 'Antonino	Gallus gallus domesti cus	8-10th									-19	8.8
<b>SAN_B2 6</b>	Paler mo	SAN	Sant 'Antonino	Gallus gallus domesti cus	8-10th									-19.2	6.1
<b>SAN_B2 7</b>	Paler mo	SAN	Sant 'Antonino	Gallus gallus domesti cus	8-10th									-19.2	8.2

<b>SAN_B2 8</b>	Palermo	SAN	Sant 'Antonino	Gallus gallus domesti cus	8-10th								
<b>SAN_B2 9</b>	Palermo	SAN	Sant 'Antonino	Gallus gallus domesti cus	8-10th							-19.7	9.5
<b>SAN_B3 0</b>	Palermo	SAN	Sant 'Antonino	Gallus gallus domesti cus	8-10th							-19.1	8.4
<b>SAN_B3 1</b>	Palermo	SAN	Sant 'Antonino	Gallus gallus domesti cus	8-10th					657-768		-19.8	7.3
<b>SAN_B3 2</b>	Palermo	SAN	Sant 'Antonino	canis familiari s	8-10th					775-964		-19.9	9.8
<b>SAN_B3 3</b>	Palermo	SAN	Sant 'Antonino	canis familiari s	8-10th							-18.9	8.8



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