Wheat of the Americas? Evaluating the socio-political and economic significance of maize (*Zea mays*) in South America

a perspective from organic residue analysis of pottery from southern and northern Chile.

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Abstract

The “wheat of the Americas” is how some of the first Europeans arriving in the so-called “New World” referred to maize (*Zea mays*), a local Poaceae that was extensively cultivated and used in different culinary practices. Its identification in the archaeological record has supported many publications and studies, leading to a major focus on its culinary, economic and ideological importance for pre-colonial groups and the assumption of maize as the most outstanding cultivated crop in the Americas. Most assumptions have been extrapolated from historical records, while the analysis of stable isotopes on human remains and the archaeobotanical record has allowed researchers to infer its significance in diet. However, little has been done from the perspective of the culinary practices and foodways of pre-colonial groups. Pottery has largely been employed in the cuisine or foodways involving maize. Archaeobotanical remains have been found as well-preserved charred residues attached to pottery containers, indicating the direct use of ceramic vessels in maize processing since pre-colonial times; however, this evidence is limited to several cases and found in specific occations. Organic residue analysis allows to directly assess the molecular and carbon stable isotope composition of lipids trapped inside the porous matrix of ceramic vessels. Here I will present the use of organic residue analysis on ceramic containers from two different contexts from Chile: **1)** southern Chile, an area where the socio-political and economic implications of maize’s late arrival have tenuously been assessed before; **2)** northern Chile, an area where maize was a staple and a major crop used in alcoholic beverage production during the Inka state occupation. The results of this work indicates that maize's economic and political significance is more complicated than assumed, and that more focus should be taken into the significance of the large diversity of resources found in western South America.

Cambia…. todo cambia….

Julio Numhauser

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Author declaration

I declare that this thesis is a presentation of original work and I am the sole author. Chapters 4 has already been published in a peer-reviewed journal, while Chapters 5 and 6 have been prepared for publication in a peer-reviewed journal. Although these works have been achieved by collaborating with several co-authors, I declare that I am the main author of these publications. This work has not previously been presented for a degree or other qualification at this University or elsewhere. All sources are acknowledged as references.

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Montalvo-Cabrera, J. A., Uribe, M., González, J., Gajardo, J., Lucquin, A., Colonese, A. C., Talbot, H. M. and Craig, O. E.The ceremonial use of maize in the expansion of the Inka state in Tarapacá, Northern Chile. *In review by the co-authors.*

- **Author contribution: Montalvo-Cabrera, J. A.**, Uribe, M., González, J., Gajardo, J.Colonese, A. C. and Craig, O. E., designed the research. **Montalvo-Cabrera, J. A.,** undertook the lipid extraction and stable isotope analysis. **Montalvo-Cabrera, J. A.**, Craig, O. E. and Lucquin, A., worked on the interpretation of the data. **Montalvo-Cabrera, J. A.,** Talbot, H. M. and Lucquin, A., undertook sample preparation and technical tasks for GC-MS and GC-c-IRMS analysis. Gajardo, J. and Uribe, M., did the pottery selection. **Montalvo-Cabrera, J. M.**, Uribe, M., González, J. and Gajardo, J., worked on context interpretation and revision.

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Sincerely,

Javier Andrés Montalvo Cabrera

Chapter 1: Introduction

Since the beginning of the colonial period in the Americas, maize has received outstanding attention from the Western world. It was depicted by European colonisers as a primary staple of great importance in the indigenous diet, economy, and socio-political life, ultimately leading to its designation as the "wheat of the Americas" or the "wheat of the Indies" (Molina 1776, 133; de León Pinelo 1636, 57). Early colonial documents described the diverse culinary use of maize by local groups in several preparations such as tortillas, alcoholic beverages, gruel, and as an ingredient in different types of stews (Cortés, 1971, 104; Columbus, 1969, 214; Gay 1865, 97). After Columbus' second voyage in 1493, maize was integrated into the agrarian European economy, quickly becoming a staple of great economic significance (Revilla et al., 2021). The familiarity of maize with "Old World" crops and the strong reliance of the European economy on crop agriculture conditioned European preference for maize over other plants from the Americas (Guedes, 2019; Earle, 2012, 149-150). These preferences would likely influence the historical recognition of maize as the most prominent and representative plant of the so-called New World.

In archaeology, the identification of domesticated grain crops has long been considered an indicator of the onset of agriculture and the development of social complexity. Maize, as a primary crop, has played a pivotal role in shaping these ideas in the archaeology of the Americas (Schwarcz, 2016; Lesure et al., 2014). The focus on maize as a prominent resource has encouraged advancements in archaeobotanical research to improve its identification in the archaeological assemblage (Musaubach, Plos and Babot, 2013; Pearsall, 2007). The possibility of identifying the consumption of C_4 plants through stable isotope analysis of carbon ($\delta^{13}C$) in human remains has further supported the representation of maize in the archaeological record, highlighting its use as a staple in various contexts (Schwarcz, 2006). However, the amount of research done on maize compared to other plants has led to biases towards its predominant role in the development of complex societies, overshadowing the potential impact of other resources on fostering significant socio-political and economic processes in the Americas (Arroyo-Kalin, 2010; Iriarte, 2009).

Current debates on non-agricultural groups and their recognition as viable economies to explain social complexity in archaeology have challenged the dominant focus on grain agriculture. The exploitation of a broad and diverse range of wild resources available to non-

agricultural groups has been considered a long-standing and successful strategy compared to the sole reliance on farming (Scott 2017, 63; Zeder, 2011). The complementarity between hunting, gathering, and food production has contradicted the idea of farming as a synonym for progress, complexity and civilisation, bringing into discussion the importance of alternative economic strategies that could support complex and diverse societies (Guedes, 2019; Smith, 2001).

Archaeologists studying the Americas have reinterpreted concepts such as agriculture and domestication, considering that these deviate from the traditional grain-focused perspective characteristic of European and Asian contexts (Clement et al., 2021; Iriarte et al., 2020; Fausto and Neves, 2018). Plant domestication in the Americas seemed to occur in scattered and separated points, comprising not only grains but a variety of tubers, trees, legumes, and vegetables (Iriarte et al., 2020; Piperno, 2011). Landscape modifications included cultivating maize and other domesticated plants and managing various wild resources (Roa Solís et al., 2024; Maezumi et al., 2018; McRostie et al., 2017). This complementarity of wild and domesticated plants seems to have been part of complex socio-economic systems that did not necessarily rely on agriculture. How maize fits in this broad spectrum of available resources is a question that needs to be assessed in its context.

1. The domestication, spread, and adoption of maize in the Americas

Theories on the origin of maize have abounded in literature for decades. In 1939, George Beadle rejected the idea of maize originating from an extinct predecessor and proposed that teosinte, a wild grass native to Mesoamerica, was its ancestor (Beadle, 1939). Further research has challenged Beadle´s theory by indicating the hybridisation between teosinte *Zea diploperennis* and gamagrass (*Tripsacum* sp.) as the main driver for maize domestication (Eubanks, 2001, 1995). However, the phenotypical characterisation of a 6250 calibrated years before the present (cal BP) maize cob from Oaxaca, Mexico, supported the direct relation of maize with teosinte (Benz, 2001; Piperno and Flannery, 2001). Nowadays, genetic and molecular research confirms that maize was domesticated in southwest Mexico from a single ancestor, teosinte subspecies *Zea mays* ssp*. parviglumis*, around 9000 years ago (Doebley, 2004; Matsuoka et al., 2002). Further studies have also indicated that the introgression of teosinte subspecies *Zea mays* ssp*. mexicana,* contributed to maize evolution

and allowed its adaptation and spread into the drier and cooler conditions of the Mexican highlands (van Heerwaarden et al., 2011).

The domestication of maize differentiated it radically from its wild ancestor (Figure 1.1). These differences resulted from selection pressures on several features during early maize domestication, reducing allelic variability in maize compared to teosinte (Chen et al., 2020; Jaenicke-Després et al., 2003). Several genes have been recognised to participate in characteristic domestication features in maize. For example, *teosinte branch 1* (*tb1*) gene is responsible for the branched plant architecture in teosinte, whereas a maize-type allele represses the development of secondary branches, which leads to the unbranched look in maize (Doebley, 2004; Jaenicke-Després et al., 2003). Variability on the *thick tassel dwarf1 (td1)* gene allowed the development of the characteristic female inflorescence or ear in maize, exhibiting extra rows of kernels compared to teosinte (Bommert et al., 2005). A single nucleotide difference in *teosinte glume architecture1* (*tga1*) gene is responsible for the elimination of the cupulate fruitcase enclosing kernels in teosinte, leading to the characteristic free kernel in maize (Wang et al., 2015).

Domesticated and wild-type gene alleles were identified in 5000 cal BP maize cobs from Teotihuacan, Mexico, indicating the existence of a diverse population of semi-domesticated maize types in Mesoamerica (Ramos-Madrigal et al., 2016; Vallebueno-Estrada et al., 2016). While specific genes have been found to have an effect on maize´s domestication features, most of its morphological characteristics are likely the consequence of the additive effect of various genes (Chen et al., 2020; Doebley, 2004). Thus, far from being an abrupt event, maize domestication seemed to have been a gradual and complex process involving the progressive fixation of a mosaic of maize-like and wild-type alleles (Ramos-Madrigal et al., 2016).

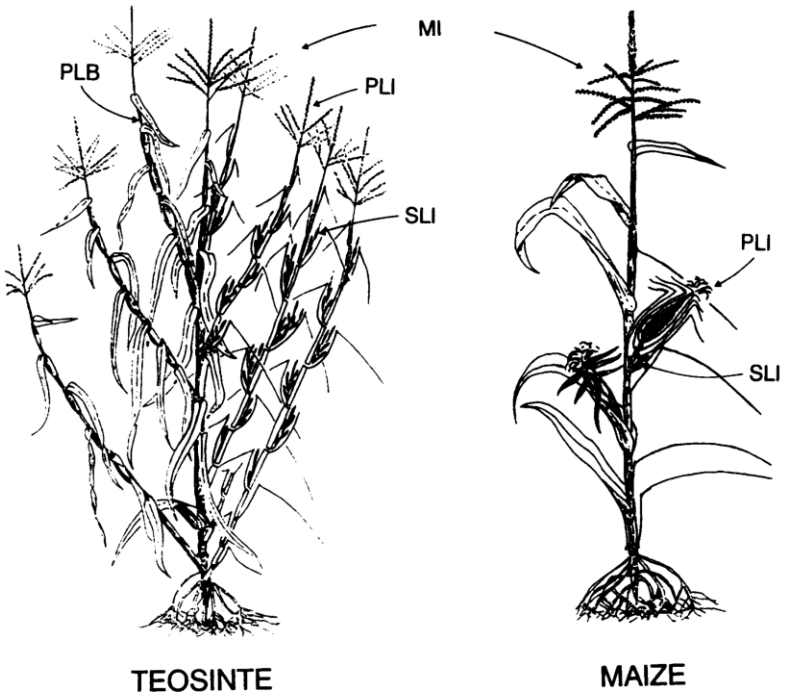


Figure 1.1: Comparison between teosinte and maize architecture showing their respective main inflorescence (MI), primary lateral inflorescence (PLI), secondary lateral inflorescence (SLI), which is only present in early maize types, and the primary lateral branch (PLB) in teosinte. Image taken from Doebley et al., (1990).

Archaeobotanical evidence suggests the exploitation of maize by groups of the Balsas River Valley in southwest Mexico as early as 8700 cal BP (Piperno et al., 2009). However, stable isotope and ancient DNA analysis on human bone remains suggest that maize did not become a staple in Mesoamerica until 4000 cal BP, which might have involved the introduction of new maize varieties from southern regions (Kennett et al., 2022, 2020). Maize consumption varied among groups, indicating a more complex scenario regarding maize reliance and agriculture in Mesoamerica (Rosenswig et al., 2015; Somerville, Fauvelle and Froehle, 2013; Seinfeld, von Nagy and Pohl, 2009).

The introduction of maize in South America was pivotal in its evolutionary history. South America became a secondary improvement centre for maize domestication, leading to new landraces and significant genetic diversity (Kistler et al., 2020, 2018). Maize reached lowland South America around 6900 cal BP (Lombardo et al., 2020), 2000 years earlier than the Teotihuacan semi-domesticated specimen, which likely indicates that maize arrived as a partially domesticated species (Kistler et al., 2018). Southwest Amazonia was the centre for

maize improvement in South America (Kistler et al., 2018), spreading into different points of the region (Costa et al., 2022). Southwestern Amazonia is one of South America's largest seasonally flooded areas, which possibly supported early maize cultivation (Brugger et al., 2016). Experimentation with plant domestication and management was already in place by the time maize arrived in lowland South America (Watling et al., 2018). Maize production was expanded and enhanced by constructing raised fields, which also included cultivating other plant products (Iriarte and Dickau, 2012). Maize came to form part of complex Amazonian polyculture systems that comprised domestic and wild plant species (Iriarte et al., 2020; Maezumi et al., 2018). Stable isotope analysis on human remains has indicated that maize was important for several groups in the Amazonia; however, its significance varied among them, reflecting the co-existence of different complex socio-economic strategies (Müller et al., 2022; Colonese et al., 2020; Hermenegildo et al., 2017).

Parallelly, the Central Andes in western South America also comprised an important maize cultivation and experimentation centre. Genetic analysis on early archaeobotanical remains from Paredones, Perú (6700 - 5000 cal BP) suggests a direct migration from Mexico to the Andes, indicating an independent dispersal route to the on followed by the lowland South American maize (Vallebueno-Estrada et al., 2023; Costa et al., 2022; Freitas et al., 2003). Archaeobotanical evidence and $\delta^{13}C$ and $\delta^{15}N$ analysis on human remains indicate that maize was adopted by preceramic groups (Pezo-Lanfranco and Colonese, 2024; Tung et al., 2020; Grobman et al., 2012); however, it did not become a staple until much later (Pezo-Lanfranco and Colonese, 2024; Santana-Sagredo et al., 2021; Alfonso-Durruty et al., 2017). The level of maize reliance among these groups was variable; for example, maize was a secondary resource in the Peruvian sites of Huaca Prieta and La Galgada, showing high dependence on marine and terrestrial resources, respectively (Dillehay et al., 2022; Washburn et al., 2020; Dillehay et al., 2012). On the contrary, Paredones, located near Huaca Prieta, saw a higher reliance on maize and animal meat, with minimal consumption of marine products (Tung et al., 2020).

In the Central Andes maize has been considered to hold high significance in the formation and expansion of pan-Andean states during the Middle Horizon (1400 - 950 cal BP) such as Wari (Finucane, 2009; Finucane, Agurto and Isbell, 2006) and Tiwanaku (Somerville et al., 2015; Goldstein, 2015). The movement of people and workforce outside the core of these Andean states has been associated with the need for territorial expansion to cultivate highly prestigious crops, principally maize (Bélisle, 2015; Goldstein, 2015). During the Late Period (500 - 418 cal BP), or the expansion of the Inka state throughout the Andean regions, maize held important socio-political and economic significance as a tribute and prestigious

commodity from which sacred beverages were prepared (Bray, 2009; Finucane, 2007). Maize became the Inka state´s main crop, and its production was promoted throughout its provinces (Hastorf and Johannessen, 1993; Murra 1980, 95).

However, discussions on the consolidation of pan-Andean states have demonstrated a different perspective regarding maize´s importance. For example, $\delta^{13}C$ and $\delta^{15}N$ analysis of human remains from Chavin de Huantar, considered to be the onset of Andean complex civilisations that flourished between 2900 - 2350 cal BP, demonstrated that, during this period, maize did not have a significant role in driving the coalescence of Andean groups (Tykot, van der Merwe and Burger, 2006; Burger and van der Merwe, 1990). Moreover, several examples from Wari and Tiwanaku states provinces have demonstrated a higher reliance on diverse products, while maize was merely considered a secondary resource (Suero et al., 2023; Kellner and Schoeninger, 2008). Similarly, during the Late Period, the Inka promoted maize as the state´s crop; however, examples in northwest Argentina have demonstrated that maize did not show to be more significant than local resources (Giovannetti, 2021; Lantos et al., 2015).

While maize held socio-political and economic significance for various pre-colonial groups, for others, it was not more than a secondary product. Maize was integrated into different environmental, cultural, political, and economic settings throughout the Americas, becoming part of complex and diverse pre-colonial trajectories. These contexts were embedded in various environments comprising a remarkable biodiversity of animals and plants widely exploited by local groups.

2. Biodiversity, social complexity and foodways in western South America

Western South America holds terrestrial and aquatic biodiversity hotspots which sustained pre-colonial populations long before the arrival and adoption of maize (Miloslavich et al., 2011; Myers et al., 2000). This region has been the setting for pre-colonial populations highly adapted to various environmental conditions with access to an extensive diversity of coastal and terrestrial resources (Pezo-Lanfranco and Colonese, 2024; Dillehay et al., 2022; Beresford-Jones et al., 2018; Marquet et al., 2012). Early European chronicles described the importance of a wide range of available resources, indicating extensive use of both terrestrial

and aquatic plants and animals, along with various cultivated plants (Cobo, 1979, 28; de Cieza de León, 1945, 276; Mariño de Lobera, 1595, cited in Escobar, 1865, p. 124). Archaeobotanical research and stable isotope analysis on human remains have attested to the importance of several products in sustaining significant socio-political and economic processes while indicating that maize might have only been part of economic strategies that comprised a wide range of wild and domesticated resources (Pezo-Lanfranco and Colonese, 2024; Pearsall et al., 2020; Beresford-Jones et al., 2018; McRostie et al., 2017).

The limited amount of studies in various areas from western South America due to poor preservation of archaeobotanical and human remains and the lack of archaeological research has led to the extrapolation of evidence from well-documented areas where maize was potentially exploited, leading to a bias against the possible contribution of other resources (i.e. C_3 plants, terrestrial and aquatic animals). In some cases, the identification of archaeobotanical remains preserved as residues adhered to vessel containers and stone tools has allowed assessing the culinary importance of several artefacts in the cooking and processing of various food products, including maize, providing direct evidence of their integration into local diets and foodways (Zarrillo et al., 2008; Chandler-Ezell et al., 2006; Staller and Thompson, 2002). However, this evidence is not always successfully found due to exposure to post-depositional degradation agents. While stable isotope analysis of human remains has highlighted the contribution of maize to the past human diet, this method is limited in addressing the significance of maize in specific past socio-cultural practices such as feasts and ceremonies, where other resources potentially also had a major contribution.

Food encompasses multiple dimensions involving the political, economic, dietary and ideological life of past populations; such a definition is contained within the concept of foodways, primarily employed in addressing the complex nature of food at every stage of its procurement, production, consumption, storage, serving and discard (Peres 2023, 3; Twiss 2012, 2). The analysis of pottery employment in cooking, storing, and serving food is a valuable way to inquire into past foodways, not only because of ceramics' endurance throughout time but also because of the capacity that pottery has to inform about past social practices (Peres, 2023, 70). In South America, ceramic vessels have played a prominent role in preparing fermented beverages consumed in socio-political contexts, where various plants, including maize, have been described to be used (Giovannetti, 2021; Bray, 2009; Cobo, 1979, 27). Pottery has also allowed for the preparation of large quantities of food consumed in ceremonial and congregational contexts, as well as in domestic settings (Núñez de Pineda y Bascuñán, 1673, cited in Cayuqueo, 2022, p. 50; Lantos et al., 2015; Rosales, 1674, Barros Arana, 1877, p. 145). The production of ceramic vessels with various morphofunctional and

decorative characteristics sometimes blends with pottery's symbolic and political significance, determining which foods are cooked, stored, and served in specific containers and which are not (Lantos et al., 2017; Bray, 2009, 2003). Therefore, identifying the original contents of pre-colonial vessels could foster our understanding of the economic and socio-political significance of the foods chosen by past pre-colonial groups in various local contexts.

The analysis of organic residues extracted from the porous matrix of ceramic containers allows the identification of the original contents preserved in archaeological vessels and their use-life in the past (Peres 2023, 60; Lucquin et al., 2018, 7932). Studies of this nature have been conducted previously in South America, primarily in Brazil and Argentina (Chaile et al., 2018; Miyano et al., 2017; Colonese et al., 2014; Hansel et al., 2004). A few publications have used organic residue analysis to address the employment of pre-colonial pottery for cooking, storing and serving maize products (Admiraal et al., 2023; Lantos et al., 2015). On the other hand, studies based on organic residue analysis of ceramic containers are nearly nonexistent on the western side of the Andes (e.g., Chile and Peru). In this thesis, I will present three case studies from southwest South America, two from southern Chile and one from northern Chile, regarding the integration of maize into local foodways.

Southern Chile has the southernmost limit for the dispersal of maize cultivation in the Americas (Campbell et al., 2020; Roa et al., 2018). The earliest evidence for maize in southern Chile dates back to the transition between the Early Ceramic Period (**ECP**: 1550 - 1000 cal BP), and the Late Ceramic Period (**LCP**: 950 - 400 cal BP), possibly associated with hunter-gatherer groups from the Pitrén cultural complex (Adán and Mera, 2011). However, several studies have indicated that it was not until the Late Ceramic Period that maize was well-integrated and extensively cultivated by farming groups from the El Vergel cultural complex, leading to its spread throughout the territory (Campbell et al., 2020; Roa et al., 2018). Its arrival into southern Chile coincided with a moment of demographic growth, socio-political changes towards social differentiation, intensification of food production and colonisation of several nearshore Islands (Campbell, 2015; Campbell and Quiroz, 2015; Dillehay, 2007). However, little is known about whether maize had a significant role in these socio-political and economic changes or if other resources were denoting specific socio-economic strategies that fostered these processes throughout the Ceramic Period.

While southern Chile saw a late arrival of maize, in northern Chile, particularly in the region of Tarapacá, maize was adopted around 3000 cal BP; however, it did not become a staple until 1000 cal BP (Santana-Sagredo et al., 2021). During the Inka state occupation of Tarapacá maize was part of daily dietary intake. At the same time, its use in ceremonial and

feasting contexts, mainly in the form of an alcoholic beverage known as *chicha*, has been assumed based on historical records and the ubiquitous presence of maize remains in Inka archaeological sites (Bray, 2009; Dillehay, 2003; Hastorf and Johannessen, 1993). However, historical records have denoted that several other products were used to produce *chicha* by the Inka state (Cobo, 1979, 27; de Cieza de León, 1945, 275). Moreover, archaeobotanical remains in several Inka sites from Tarapacá have shown the significance of other products possibly employed in producing *chicha* (Zori 2011, 385, 399). The view of maize as a prominent resource invested with ideological solid and symbolic significance supports the assumption of its employment as a primary product consumed during Inka ceremonial feastings. However, the direct analysis of the original content of the vessels employed in the consumption of food during Inka ceremonial contexts in Tarapacá has never been assessed before.

This thesis tackles the hegemonic view of maize as a major driver for socio-political and economic processes in South America. I will discuss the culinary use of maize in different case studies focusing on southwestern South America, mainly on the pre-colonial foodways of south and northern Chile to assess the socio-political and economic significance of maize in this territory. To further address and guide the main discussions of this thesis, I have presented the following objectives and research questions:

3. Main objective

Western South America possess an outstanding diversity of local resources that sustained past pre-colonial groups before maize's adoption. However, maize has traditionally been acknowledged as a socio-political and economic driver of pre-colonial groups, which has incentivised considerable research focus. Considering South America's territorial extension and complex archaeological history, I have centred my main objective on **evaluating maize's socio-political and economic significance in southwestern South America through organic residue analysis from pre-colonial pottery artefacts from southern and northern Chile.** I will be focusing first on southern Chile, considered to be the southernmost point for maize dispersal in the Americas that saw the late arrival of maize, and where maize´s significance has tenuously been discussed. On the other hand I have included a case study from northern Chile as an example of a context where maize exploitation is higly presumed due to the presence of the Inka state. For this, I have presented three case studies, two from southern Chile which comprise most of the samples analysed in this work, and one from

northern Chile. These two areas show different cultural, environmental and chronological settings where maize was exploited, serving as an interesting contrast to better evaluate maize's socio-political and economic significance in western South America.

4. Specific objectives

- To identify maize through a multiproxy approach combining the molecular and carbon isotopic composition of organic residues, particularly lipids, extracted from pre-colonial pottery from southern and northern Chile.

- To assess whether maize shows a competitive advantage compared to marine resources in supporting island colonisation by pre-colonial farmers from southern Chile.

- To evaluate the significance of maize in fostering socio-political and economic changes throughout the Ceramic Period in the Central Valley of the Auraucanía, southern Chile.

- To examine the socio-political importance of maize in a ceremonial context from the Atacama Desert during the Inka state occupation of Tarapacá, northern Chile.

5. Research questions

- Is it possible to identify the use of maize in archaeological pottery through organic residue analysis?

- Did maize play a prominent socio-political and economic role in southern Chile?

- Did maize hold prominent socio-political importance during feasts and ceremonies celebrated under the Inka occupation of Tarapacá, northern Chile?

6. Thesis structure

To better approach my objectives and research questions, I will first present the archaeological and chronological context of southern Chile, followed by the methodological approach I have used to assess the identification of maize in archaeological pottery. Each case study is aimed at fulfilling specific objectives and answering my previously listed research questions. For this, I have structured my thesis in the following order:

Chapter 2: The Araucanía in southern Chile has seen a lack of archaeological studies, most of which have concentrated on specific areas (e.g. Mocha Island, Puren-Lumaco Valley). However, this region is the southernmost point of pre-colonial maize cultivation in the Americas. It has evidenced important socio-political and economic changes throughout its pre-colonial occupation by hunter-gatherers and farming societies. Furthermore, the Araucanía is currently considered a biodiversity hotspot, indicating its potential economic significance for pre-colonial groups that adapted and used the various available resources. In this chapter, I present a thorough review of the archaeology and environmental setting of the Araucanía to better contextualise two of the three case studies presented in this thesis.

Chapter 3**:** The extraction and analysis of organic residues from pottery artefacts has allowed the identification of the main content of archaeological ceramic containers permitting further discussions on past foodways. While this methodology has been used in several archaeological contexts worldwide, its use in South American archaeology is limited to few case studies. Moreover, its employment in addressing questions towards maize consumption by pre-colonial groups is way more limited. In this section, I thoroughly describe the use of organic residue analysis from pottery artefacts and its main methodological approaches. I also present a classification of the main compounds comprising the large and diverse lipid family. A review of the main works on organic residue analysis in South America is also presented to give an idea of the field's current state of the research in the region. I also indicate current works done on organic residue analysis to identify maize from pre-colonial pottery artefacts. This thesis presents the first case studies using organic residue analysis in pottery artefacts from pre-colonial Chile.

Chapter 4: Previous studies have discussed that during the late Ceramic Period in southern Chile, the colonisation of Mocha Island was possible after the intensification of food production. Relative carbon ^{13}C enrichment on human remains has been interpreted as the partial consumption of maize, indicating that farming activities were more important than using

marine products to sustain the permanent settlement of El Vergel farmers on Mocha Island. Through the extraction and analysis of organic residue from El Vergel pottery from Mocha Island I aim to evaluate whether maize and farming practices were more important than marine resources in the colonisation process of Mocha Island during the Late Ceramic Period. This case study is the first research of its kind on the Pacific coast of South America.

***Chapter 5*:** The transition from the Early (1550 - 950 cal BP) to the Late (950 - 400 cal BP) Ceramic Period in southern Chile saw important socio-political and economic changes, such as the emergence of social differentiation, demographic growth, intensification of food production and colonisation of nearshore Insular territories. The identification of archaeobotanical remains denoting the widespread presence of maize in the Araucanía and its identification in complex agricultural raised fields from the Central Valley coincides with this transitional moment. However, the influence that maize might have had on fostering these processes has tenuously been addressed before. By analysing and extracting organic residues from Early and Late Ceramic Period pottery artefacts from two sites located in the Central Valley of the Araucanía, in this chapter, I evaluate the significance that maize might have had during important socio-political and economic changes throughout the Ceramic Period of the Araucanía.

***Chapter 6*:** This case study is set in a different context and comprises a much smaller number of samples. At first, due to the amount of Inka pottery and jars associated with a period of intense maize agriculture, this case study was supposed to represent a context where pottery containers were most likely used for storing maize products. However, after analysing the results, I realised that most pottery yielded alkanoic acids with a carbon isotopic signal showing a high contribution of C_3 products; only 5 samples showed to be likely related to the storing of maize products. By presenting an example of the Inka occupation of northern Chile that contrasts considerably with the southern case studies, I aim to discuss maize's significance in a socio-political context where the prominent use of maize is expected. While southern Chile has been poorly studied, northern Chile is the area that holds the most research studies in Chile, which strongly supports preconceived notions on maize's culinary significance under the Inka presence in the region. I will assess the reason why maize is not represented in the pots as expected for a site denoting the presence of the Inka state within a feasting context.

***Chapter 7*:** Here, I discuss maize's significance in western South America based on the previously mentioned case studies. I aim to refer to the rich diversity of socio-political and economic processes and strategies found in western South American archaeology and

discuss other significant areas from South America as examples of important socio-economic trajectories that might have fostered critical socio-political and economic processes in the subcontinent. I also refer to the possibilities for improvement and how important it is to work on further considerations to generate suitable approaches that can enrich our discussions in the future.

7. Supporting information

The data generated in this research is presented in the text, appendices and supplemental information. The former is present in the following CSV files:

C. Catalanes Early Ceramic Period ORA data.CSV
C. Catalanes data Late Ceramic Period.CSV
SMR5 ECP Data.CSV
SMR5 LCP Data.CSV
Iluga Túmulos Data.CSV

Chapter 2: Archaeological and ecological background of the Araucanía in southern Chile

1. Introduction

Southern Chile is considered the southernmost point of pre-colonial food production in the Americas (Campbell et al., 2020; Roa et al., 2018) and a hotspot for biodiversity and endemism (Bannister et al., 2012; Myers et al., 2000). It is defined as a geocultural region located between the Itata River in the north (37º09' Lat S) and the "Los Lagos" administrative region in the south (41º42' Lat S) (Figure 2.1) (Roa et al., 2018; Campbell and Quiroz, 2015; Aldunate, 1989). Beyond its northern end, there is a lack of archaeological evidence until the proximity to *Santiago de Chile*. Its southern limit defines the frontier with the Chilean Patagonia, a region characterised by a late adoption of pottery in several areas and the presence of hunter-fisher-gatherers with no pre-colonial evidence for the use of ceramic technology (Reyes et al., 2023, 2022b).



Figure 2.1: Map of Chile and its main areas. Figure created by the author.

Southern Chile is divided at the Mahuidanche-Lastarria mountain cordon (39º20' Lat S) into a northern and southern sector (Figure 2.2), showing well-defined environmental particularities (Aldunate, 1989). Its northern portion is characterised by a transition between the Mediterranean and Temperate macroclimates, with a predominance of *Nothofagus* trees, along with a mix of sclerophyll and temperate evergreen forests, with the presence of *Araucaria araucana* forests in high altitudes (Amigo and Ramirez, 1998). In contrast, southward of the Mahuidanche-Lastarria mountain range, more temperate conditions prevail, with a predominance of evergreen species composing the dense and impenetrable Chilean temperate forest (Aldunate, 1989).

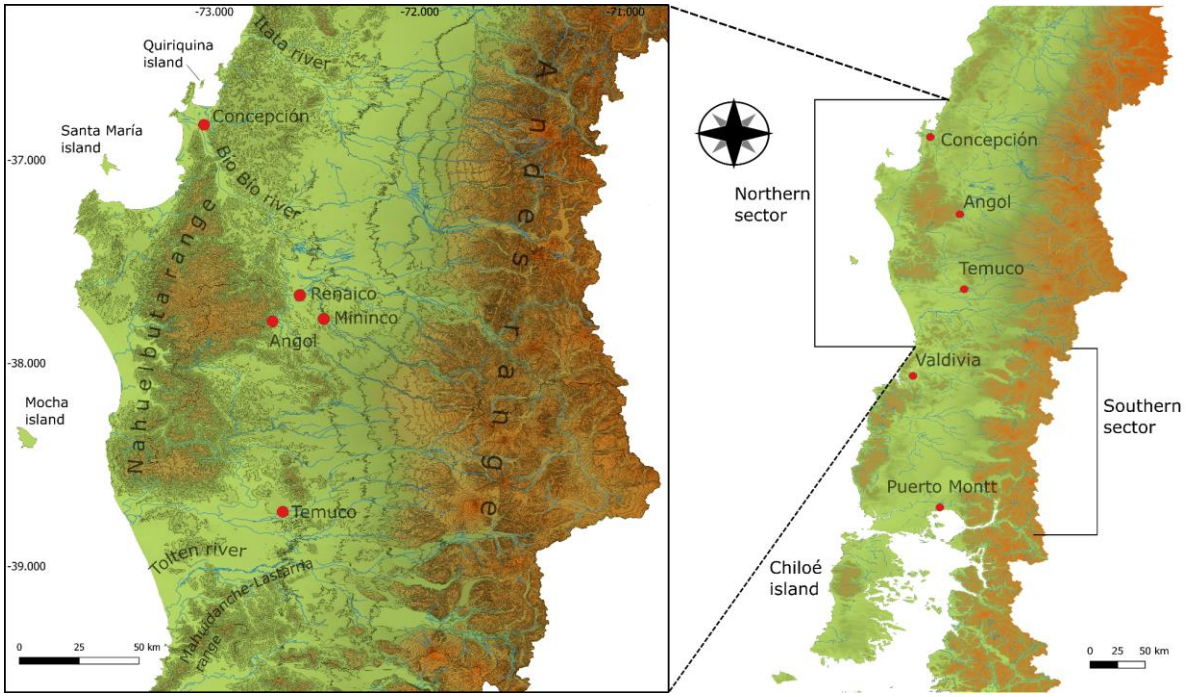


Figure 2.2: Map of southern Chile and a close-up of the Araucanía territory. Figure created by the author.

Between the Bío Bío and Toltén Rivers, in the northern sector of southern Chile, is located the historical region of the Araucanía, which comprises the Bío Bío and La Araucanía administrative regions (Figure 2.2). It was first defined by the Spanish conquerors who realised that southward the Bío Bío River (*Fütraleufu* or great river due to its extension), a dense and bellicose population inhabited a prosperous and amenable territory (Bengoa 2007, 171; Bibar, 1558, cited in Leonard, 1966, p. 153; Molina, 1776: 13). They called the people inhabiting this area *Araucanos*, based on a small Indigenous region southward of the Bío Bío River called Arauco (Molina, 1776:13).

Pre-colonial groups of the Araucanía evidenced a progressive intensification of food production throughout the Ceramic Period (1550 - 400 cal BP), accompanied by the continuity of hunting and gathering practices (Campbell et al., 2020; Roa et al., 2018). This description contrasts with the socio-economic strategies seen beyond the Andes, deep into the Argentinian northern Patagonia and Pampa regions; here, hunting and gathering represented the primary socio-economic practices, with no clear evidence for plant management and food production (Saghessi et al., 2023; Musaubach and Berón, 2016). At the same time, beyond the Mahuidanche-Lastarria limit in southern Chile, an intensification of plant management and food production has merely been characterised, as no robust evidence supports a

development similar to what is described for the northern sector (Reyes et al., 2022b; Belmar et al., 2021; Silva, González and Popovic, 2017).

Throughout the Ceramic Period, the Araucanía experienced Important demographic growth attributed to an intensification of food production (Gayo et al., 2019; Campbell and Quiroz, 2015; Campbell, 2015). However, the diversity of local resources, both terrestrial and aquatic, was also an essential part of pre-colonial foodways (Roa Solís et al., 2024; Adán and Mera, 2011; Quiroz and Sánchez, 2010). From the coast to the Andes range, local communities used the different available resources while maintaining economic complementarity and communication networks between them (Bengoa 2007, 82).

Therefore, it is impossible to approach the demographic, political and socio-economic events that shaped the Araucanía without considering the diversity of resources available to pre-colonial groups. The following section briefly describes the environmental characteristics of the principal ecological zones found in the region.

2. Regional setting

Throughout most of Chile´s geography, its ecological settings can be assessed based on a longitudinal description of the main geographical areas, from west to east **a)** the Andes range; **b)** Central Valley; **c)** Coastal range; **d)** coast (Figure 2.3). Changes in their configuration are evident when comparing the different administrative regions from north to south. In the case of the Araucanía, these geographical settings hold a diversity of terrestrial and aquatic ecosystems.

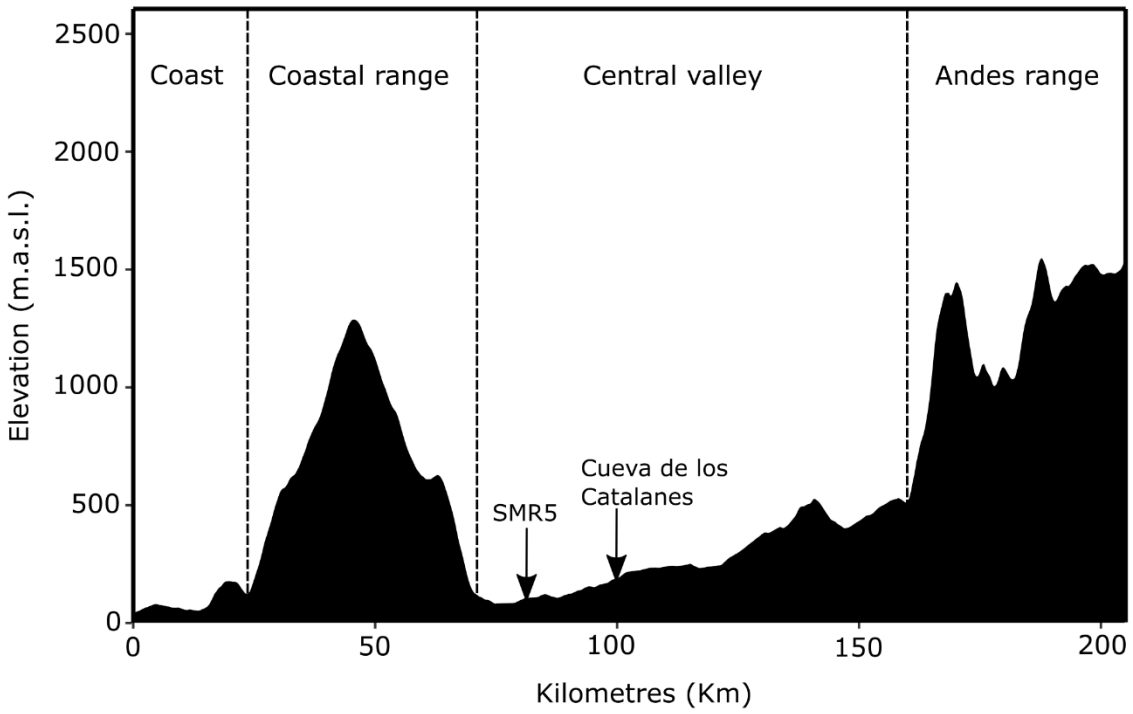


Figure 2.3: Elevation profile map of the Araucanía in metres above sea level (m.a.s.l.). The figure also indicates the location of the sites from the Central Valley that were considered for this research. Data was taken around latitude 37°8'S, based on digital elevation models (DEM) from the "Ministerio de Bienes Nacionales de Chile" (Chilean Ministry of National Assets). Figure created by the author.

2.1. The Andes range

The Andes range is divided into two main areas: the high and low valleys. The former, known as *veranada*, is located above 1300 - 1400 metres above sea level (m.a.s.l) and consists of two main vegetational formations, wet prairies and steppe, with a shared predominance of grasses and shrubs and a less representation of edible fruits of the Rosaceae family (Huiliñir-Curío, 2010; Hauenstein et al., 2003). The *veranada* is localised between two mountainous ranges, one to the east delimiting the frontier with Argentina, with altitudes below 2000 m.a.s.l, and one to the west, consisting of the principal volcanoes of the region, above 2000 m.a.s.l (Fuentes-Ramírez, Pauchard and Hauenstein, 2011). This high Andean valley ecosystem has socio-economic importance for local Mapuche-Pewenche people as grazing areas for their livestock (Hauenstein et al., 2003). The Andean frontier with Argentina is a communicational area with communities from the eastern Andes sector,

allowing the transactions of materials and goods since pre-colonial times (Huiliñir-Curio, 2020; Salazar and Berón, 2013).

At this altitude, temperate conditions sustain the predominance of araucaria (*Araucaria araucana*), or monkey puzzle tree, and lenga beech (*Nothofagus pumilio*) forests (Gutiérrez et al., 2020). The araucaria forest is a sacrade place for the Pehuenche indigenous communities, while the gathering and consumption of araucaria nuts or *piñones* comprises a significant part of their economy and diet (Huiliñir-Curío, 2020). The temperate forest of the Andean range holds a diverse avian fauna (González and Contreras, 2008). Large trees from the *Araucaria araucana* and *Nothofagus* forest are constantly used as nesting areas by different avian species of woodpeckers (e.g. *Colaptes pitius*, *Veniliornis lignarius, Campephilus magellanicus*), along with *Tachycineta meyeni* (Chilean swallow), *Pygarrhichas albogularis* (white-throated treerunner), and *Enicognathus ferrugineus* (austral parakeet), *Scelorchilus rubecula* (chucao) and *Scytalopus magellanicus* (magallanic tapaculo) (Ibarra et al., 2010). Andean wetlands show a high abundance and richness of avian species, with a significant representation of the tagua (*Fullica armillata*) and blanquillo (*Podiceps occipitalis*) (González and Contreras, 2008).

In the lower valley, lenga beechesalso compose the vegetational landscape, along with other trees from the *Nothofagus* genus, such as *Nothofagus antarctica* (ñirre), *Nothofagus dombeyi* (coigüe), *Nothofagus alpina* (raulí), and *Nothofagus obliqua* (hualle) (Fuentes-Ramírez et al.,2011; Huiliñir-Curío, 2010). These forests are associated with a diversity of edible mushrooms and a complex undergrowth ecosystem composed of shrubs and Andean bamboo species of culinary significance (Ibarra et al., 2018; Cordero, Abello and Galvez, 2017; Fuentes-Ramírez et al., 2011). In association with these forest compositions, several Andean lakes were formed from melting glaciers, allowing access to an extensive reservoir of freshwater and freshwater plant and animal species.

The Andean sector is also the habitat for several large and medium-sized mammals. The pudú deer (*Pudu puda*) inhabits the temperate evergreen forest at an altitude limit of 1200 m.a.s.l (Meier and Merino, 2007). The puma (*Puma concolor*) is pudú's primary predator and the principal native carnivore of Chile (Rau and Jiménez, 2002). Although it inhabits the temperate forest of the Andean and Coastal cordillera, it is possible to see them hunting in wetlands and private farms in the lower valleys, where it feeds from domestic game (Rau and Jiménez, 2002; Muñoz-Pedreros et al., 1995). According to historical records, the Andean deer huemul (*Hyppocamelus bisculus*) used to frequent the Araucania's Andean sector (Vila

et al., 2006). Its population has been reduced and is currently restricted to specific areas of the Andes range (Povilitis, 1998).

2.2. Central Valley

The main rivers of the Araucanía initiate their course from glacier lakes located in the Andes range, stretching to the sea, and spreading into the territory through the three main river basins, the Bío Bío, Imperial, and Toltén, creating a well-connected riverine system that supplies the region with freshwater resources. The natural course of these rivers runs through an extensive system of valleys with a prosperous agricultural richness. Currently, this area has the highest rate of native flora replacement, as intense agricultural and agroforestry activities have contributed to the introduction of exotic species, directly impacting the landscape and local ecosystems (Amigo and Rodríguez-Guitián, 2011). However, it is possible to find areas where elements of the native forest are still present.

The location of the central valley between the Andes and Coastal (or *Nahuelbuta*) ranges allows the extension of the drier Mesomorphic ecosystem from central Chile, reducing precipitations and permitting dry seasons (Gutiérrez et al., 2020; Amigo and Ramirez, 1998). Species typical of the Mediterranean sclerophyll forest, such as boldo (*Peumus boldus*), peumo (*Cryptocaria alba*), quillay (*Quillaja saponaria*), lingue (*Persea lingue*), litre (*Lithraea caustica*), co-exist with an abundant deciduous forest from the *Nothofagus* genus, mainly *Nothofagus obliqua* (Patagonian oak or *hualle*) and *Nothofagus alpina* (raulí) (Amigo and Rodríguez-Guitián, 2011; Gutiérrez et al., 2020). Elements of temperate laurifolia evergreen forests are present due to the transition from a Mediterranean to aTemperate macroclimate, characteristic of the Valdivian temperate forest. Part of these species are laurel (*Laurelia sempervirens*), avellano (*Genuvia avellana*), olivillo (*Aextoxicon punctatum*), arrayán (*Luma apiculata*), ulmo (*Eucryphia cordifolia*), coigüe (*Nothofagus dombeyi*), tineo (*Weinmannia trichosperma*), and canelo (*Drimys winteri*). This transitional characteristic has sustained a mixed forest with one of the highest levels of endemism and richness of plant species seen in Chile, many of which hold important culinary and economic significance for local indigenous communities (Cordero et al., 2017; Bannister et al., 2012; Smith-Ramírez, 1998).

The low drainage surrounding the main river basins forms extensive wetlands, such as swamps dominated by marshy vegetation and forested wetlands or *hualves* composed of Myrtaceae species like *Myrceugenia exsucca* (pitra), *Blepharocalyx cruckshanksii* (temo), and

Drimys winteri (canelo) (Figueroa, Contreras and Saavedra, 2018; Hauenstein et al., 2005). These productive ecosystems are important nesting areas for many aquatic birds (Parra-Coloma, Urra and Aguayo-Molina, 2014; Victoriano, González and Schlatter, 2006). Freshwater mammal species such as coypo (*Myocastor coypus*) and the Chilean otter (*Lontra provocax*) also inhabit these wetlands (Figueroa et al., 2018). Freshwater fish such as *Percichthys trucha* (creole perch) and *Diplomystes nahuelbutaensis* (tollo), are native to the Araucania river basins, while freshwater molluscs and crustaceans are also found in these ecosystems (Valdovinos and Parra, 2006; Habit et al., 2006).

Large and medium-sized local mammals are frequently found in this area. Guiña cat (*Leopardus guigna*) is a forest specialist wild feline mainly inhabiting the temperate forest of the Araucanía (Fleschutz et al., 2016). Pudú deers and pumas are also found in this area, as well as the South American fox zorro chilla (*Lycalopex griseus*) and the quique grison (*Galictis cuja*) (Zúñiga, Muñoz-Pedreros and Fierro, 2009; Meier and Merino, 2007). Associated with the undergrowth ecosystem, it is possible to find Chile's only marsupial species, the *Dromiciops gliroides* or *monito del monte* (Cortez Parra and Torres-Fuentes, 2021). An enigmatic case was the chilihueque or hueque, a small guanaco (*Lama guanicoe*) that used to inhabit Southern Chile. Due to the introduction of European game, the population of chilihueque diminished considerably until its complete extinction in the region (Gay, 1847, 154). According to Claudio Gay, by the 17th century, there were only a few specimens left (Gay, 1847, 154); however, Tomás Guevara reported that by the end of the 18th there were still several *chilihuques* inhabiting the Araucanía (Guevara 1927, 303).

2.3. Coastal range (The Nahuelbuta range)

In the Araucanía, the Coastal range reaches altitudes up to 1500 masl. It acts as a natural barrier, mitigating the oceanic influences in the Central Valley, allowing drier seasons and reducing precipitations. Species of the *Nothofagus* forest dominate its west and east slope, while forests of *Nothofagus pumilio* and *Araucaria araucana* appear on its high altitudes (Gutiérrez et al., 2020). Due to the more temperate conditions of its high altitudes, it holds a diversity of evergreen species characteristic of the Valdivian temperate forest, along with species associated with Magellanic moorland herbs (Villagrán, 2001; Cisternas et al., 1999). These particularities allow for a similar avifauna as seen for the Andean sector, while other species, such as pumas, pudú and foxes, also inhabit these highlands.

2.4. Coast and insular territory

Between the mouth of the Bío Bío river (36º49' Lat S) and Tirúa (38º20' Lat S) runs the Arauco-Cañete coastal plane with a north-south extensión of around 170 km and west-east extension of around 25 km (Quiroz and Sánchez, 2004). Further south, the Coastal range runs in direct contact with the seashore through an extensive system of abrupt cliffs that limit the presence of coastal plains and terraces (San Martin et al., 1992).

Coastal wetlands such as estuaries and tidal marshes emerged due to rivers flowing into the sea (Ramírez et al., 2014; San Martin et al., 1992). At the same time, the definitive settlement of the coastline during the Holocene Epoch reduced soil drainage, which led to the formation of lakes, lagoons and coastal swamps with a prominent diversity of avian species (Pellet and Cornejo, 2021; Figueroa et al., 2018; Sánchez, 2005). These ecosystems are associated with a terrestrial vegetational composition characteristic of the sclerophyll-temperate mixed forest (Gutiérrez et al., 2020; Moreno-Chacón et al., 2018).

The coast of the Araucanía holds a high productivity of marine resources due to the influence of the Humboldt upwelling system and the number of estuaries that input rich freshwater resources into the sea (Miloslavich et al., 2011; Stuardo and Valdovinos, 1989). Different fish, including diverse pelagic species such as *Merluccius gayi*, *Engraulis ringens*, *Trachurus murphyi* are commonly found (Neira et al., 2014; Montecino and Lange, 2009). Communities of whales such as sperm whales (*Physeter catodon*), fin whales (*Balaenoptera phisalus*) and blue whales (*Balaenoptera musculus*) inhabit these coasts, along with the common sea lion (*Otaria flavescens*) (Neira et al., 2014). At the same time, different types of molluscs and crustaceans can be found on the shores (Neira et al., 2014; Montecino and Lange, 2009). There are numerous species of seaweed in this region, of which an important portion holds culinary and economic importance for local coastal communities. Some of these species are the cochayuyo (*Durvillaea antarctica*), pelillo (*Gracilaria chilensis*), chicorea of the sea (*Chondracanthus chamissoi*), red luga (*Gigartina skottsbergii*), and black luga (*Sarcothalia crispata*) (Lasserre Peralta, 2019; Donoso et al., 2016).

In front of the coast are the Quiriquina, the Santa María, and the Mocha Island. Quiriquina Island, located in front of Concepción Bay, is the closest and smallest. During the mid-Holocene Epoch, it formed part of a system of fjords until the coastline's stabilisation, after which it remained separated from the mainland (Link et al., 2019; Torres, Silva and Lucero, 2007; Seguel, 1969). Santa María Island is 10 km away from the locality of Punta Lavapié,

whereas Mocha Island is the largest and furthest of the three insular territories. These three Islands have been the landscape of human population settlement and interactions since pre-colonial times (Campbell, 2015).

3. Inhabitants of the Araucanía

Southern Chile exhibits a large diversity of available marine and terrestrial resources exploited by human populations before the arrival of the European colonisers in the 16th century. Early hunter-gatherer groups used the different ecological zones, exhibiting efficient adaptation strategies that supported further socio-economic developments during the Ceramic Period. The intensification of food production throughout the onset of farming and the integration of exotic plants such as maize have been argued to have brought significant socio-economic and political changes to local groups. To further assess these statements, reviewing the archaeological background of the Araucanía is crucial.

3.1. Early human evidence in southern Chile

Southward of the Mahuidanche-Lastarria range, the sites of Monte Verde, located 55 km from the city of Puerto Montt, and Pilauco, situated 100 km north of Monte Verde, exhibited evidence suggesting the presence of hunter-gatherer groups between 18500 and15000 cal BP (Dillehay et al., 2015; Pino et al., 2013; Dillehay et al., 1982). Monte Verde consisted of a residential site with evidence of wooden foundations, wooden tools, and an industry of stone artefacts, consisting of grinding stones, bifacial and unifacial flacked tools associated with extinct megafauna (Dillehay et al., 2015, 2019; Pino Quivira and Dillehay, 1988; Dillehay et al., 1982). In the case of Pilauco, the finding of a human footprint and industry of stone tools associated with Pleistocene fauna were recovered from a context dated to around 15000 cal BP, showing to be contemporaneous with Monte Verde (Pérez-Balarezo et al., 2022; Moreno et al., 2019; Pino et al., 2013). The identification of plant, animal and seaweed remains indicate that local groups relied on a diversity of local resources, possibly reflecting a mixed diet (Dillehay et al., 2008; Dillehay and Collins, 1988). Despite the longstanding research referring to what might show to be the earliest human settlements of the Americas, the veracity and chronological accuracy of the data are part of an ongoing debate (Dillehay et al., 2022a; Fiedel, 2022; Politis and Prates, 2021; Fiedel, 1999).

3.2. Archaic Period in the Araucanía (11000 - 2000 cal BP)

The earliest evidence of human presence in the Araucanía dates back to the Early Archaic Period, *ca* 10000 cal BP, and it was found in the Andes range, particularly in the northern portion of the Calafquén Lake, at the rock shelter Marifilo-1 site (Mera and García, 2005; Adán et al., 2004). This site was intermittently used throughout the Archaic Period by highly mobile hunter-gatherers practising a mixed economy based on hunting wild mammals, such as pudú deers, huemul, and chilla foxes, along with gathering freshwater molluscs and local edible nuts and fruits from the temperate forest (Lehnebach et al., 2008; Mera and García, 2005; Velázquez and Adán, 2002). A subsistence strategy based on a mixed economy was replicated throughout the Archaic Period in different localities of the Araucanía's Andean sector, showing considerable differences regarding the ecological landscape, available resources, and technological innovations (Navarro Harris, Dillehay and Adán Alfaro, 2011; Adán, García and Mera, 2010; García, 2009; Adán, Reyes and Mera, 2001).

The lower altitudes of the Andes range in this area allowed the formation of trans-Andean paths currently transited by local Indigenous communities (Coña, 2017; Salazar and Berón, 2013). Archaic groups used these paths as part of their highly mobile strategies, allowing constant interactions between both sides of the Andes (Adán, García and Mera, 2010; Adán, Reyes and Mera, 2001).

The characteristics of the Central Valley gave rich and diverse conditions for early human settlement; however, the archaeological record is scarce, mainly because of intense anthropogenic impact and the lack of academic interest. Scarce evidence has been identified in the Puren-Lumaco Valley, indicating an almost continuous human occupation since ca. 8000 cal BP (Dillehay, 2007). Around 4000 cal BP, the rock shelter Quillen-1 was used as a habitational and lithic production site, showing a continuous occupation until the Late Ceramic Period (Valdés et al., 1985; Sánchez et al., 1984). Hunter-gatherers exploited freshwater resources from nearby rivers, hunted local fauna, and collected edible plants, reflecting a subsistence strategy based on a mixed economy (Valdés et al., 1985). Nearby, the rock shelter Quino-1 site exhibited a similar occupation by hunter-gatherers practising a subsistence strategy based on exploiting freshwater and terrestrial resources (Quiroz, Vásquez and Sánchez, 1997). Interpretations of the archaeological assemblage indicate an early use of ceramic containers by these hunter-gatherer groups based on thermoluminescence dating of pottery fragments (Quiroz, Vásquez and Sánchez, 1997);

however, given the current discussions and discrepancies regarding thermoluminescence dating, caution must be taken when referring to these results (Marsh et al., 2021).

Most Archaic period research has focused on the coast (Campbell and Quiroz, 2015). Several archaeological sites have been discovered throughout the Arauco-Cañete coastal plain; however, Early Archaic Period sites are non-existent on the coast as post-Pleistoscene progressive warmer conditions led to raised sea levels, resulting in the current submergence of most early coastal occupations (Quiroz and Sánchez, 2004). By around 8000 cal BP, maritime transgressions and regressions shaped the current coastline, giving access to new environments and landscapes occupied by coastal Archaic hunter-gatherers (Torres et al., 2007). The stabilisation of the sea level allowed a proliferation of Archaic sites, showing a dense and continuous occupation of the coast of southern Chile from the Mid-Archaic Period (Campbell and Quiroz, 2015). These populations developed an intense adaptation for exploiting marine resources through hunting, fishing and gathering local marine species. The freshwater nutrients brought into the sea by numerous estuarine ecosystems on the Araucanía coast allowed a rich diversity of malacological resources (Quiroz and Sánchez, 2004; Bustos and Vergara, 2001). The identification of dense shell middens and associated coastal technology indicated intense exploitation by coastal foragers and a constant occupation of the coast since 8000 cal BP (Torres et al., 2007; Bustos and Vergara, 2001; Seguel, 1969).

Archaeological sites southward of the Arauco-Cañete coastal plain are scarce due to the extension of abrupt cliffs stretching from the Coastal range. One exception is the Monkul-1 shell midden, dated around 2000 cal BP (Van Meurs and Gordon, 1989). The site is set on a marine terrace associated with the estuarine ecosystem of the Moncul River; here, local hunter-gatherers practised intense exploitation of coastal resources, primarily of the Chilean mussel *Mytilus chilensis* (Van Meurs and Gordon, 1989).

Close to the Concepción Bay, the Quiriquina Island was intensively occupied by pre-colonial groups. Although there are no associated carbon-dated materials from the archaeological assemblage, researchers suggest that it was first inhabited by highly adapted coastal Archaic hunter-gatherers (Quiroz and Sánchez, 2004; Seguel, 1970). The short distance between the Island and the mainland possibly allowed a fluent and continuous movement of coastal hunter-gatherers (Campbell, 2015); however, the current occupation of the Island by the Chilean Navy has limited further systematic research.

Current evidence indicates that Mocha Island was the first inhabited insular territory in the Araucanía. Around 3000 cal BP, coastal hunter-gatherers reached Mocha Island,

employing specialised technology to exploit marine resources (Quiroz and Sánchez, 2004; Vasquez, 1997). After this occupation, a chronological hiatus from 3000 to 2250 cal BP showed no human population on the coast of the Araucanía; the reasons for this event are still poorly understood (Campbell, 2015). After at least 800 years of coastal occupational discontinuity, a proliferation of sites was evident on the coast, Central Valley and Andes range, which reflected progressive demographic growth in the region (Figure 2.4) and a more constant occupation of Santa María and Mocha Island, which led to their permanent settlement during the Late Ceramic Period (Campbell, 2015; Massone et al., 2012; Vasquez, 1997).

However, several challenges in radiocarbon dating can hinder our interpretations of archaeological events (Becerra-Valdivia et al., 2020). One of these difficulties regards the non-linearity between radiocarbon and calendar dates which highly impacts the construction of calibration curves. Constant atmospheric ^{14}C values throughout extensive periods of calendar years lead to the formation of plateaus in the calibration curve (Hogg et al., 2019). Radiocarbon measurements situated within these plateaus will likely exhibit lower levels of precision, especially for short-duration archaeological sites, which are frequently found in southern Chile (Dreshaj, Raemaekers and Dee, 2023; McDonald and Manning, 2023; Campbell and Quiroz, 2015). The brief occupation of these sites restricts the possibility of wiggle-matching sequential archaeological events, which can help to improve the precision of radiocarbon measurements (Dreshaj et al., 2023; Galimberti, Bronk Ramsey and Manning, 2004). Therefore, caution must be taken when using the summed probability density of radiocarbon dates to interpret past human events.

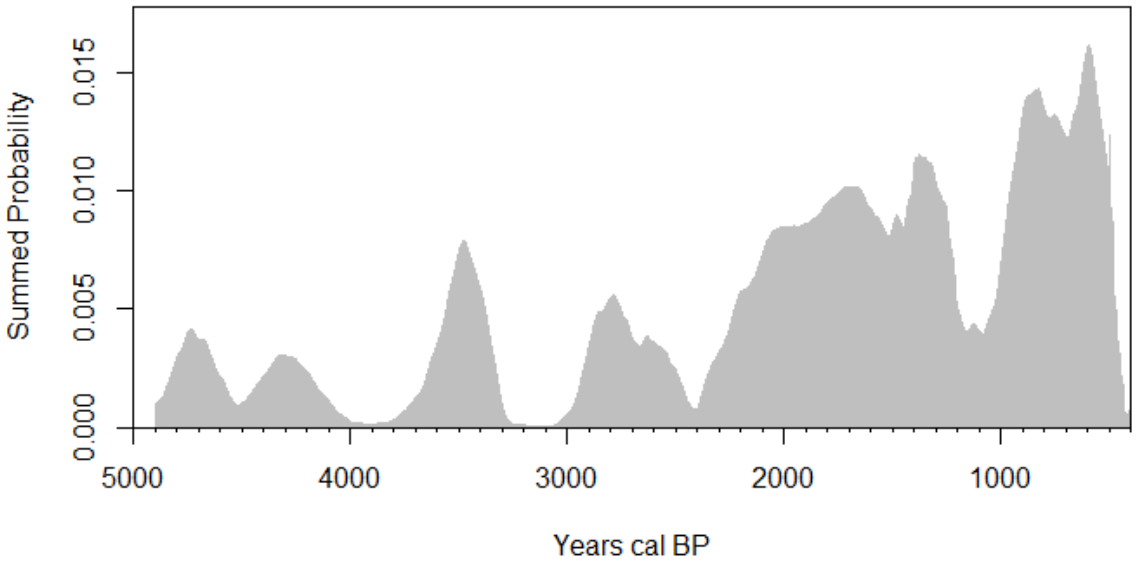


Figure 2.4: Summed probability density distribution using bins to merge ^{14}C dates. Data from sites in the Araucanía were obtained from Campbell & Quiroz (2015) and Campbell et al. (2020). Carbon dates were re-calibrated using the Southern Hemisphere curve (SHCal20) (Hogg et al. 2020). Only terrestrial organic materials and individuals showing a terrestrial diet based on stable isotope analysis data were considered to avoid any reservoir effect. All data was plotted using the "rcarbon" package in R 4.2.2 (Crema and Bevan, 2021). Only samples with a sigma (σ) value below 100 years were considered. Figure created by the author.

3.3. The Ceramic Period

3.3.1. Initial ceramic evidence (2000 - 1600 cal BP)

The earliest pottery evidence in the Araucanía has been attributed to the last moments of the Late Archaic Period. Although most evidence is based on thermoluminescence dating, a few sites have shown ceramic containers associated with ^{14}C-dated materials, indicating an early adoption of pottery during the Late Archaic Period (Marsh, 2017; Campbell and Quiroz, 2015). Radiocarbon dates from southern Chile were re-calibrated from Campbell and Quiroz (2015) and Campbell et al, (2020) (Appendix 14). The southern hemisphere calibration curve

SHCal20 was used (Hogg et al., 2020), following the R package “rcarbon” (Crema and Bevan, 2021).

Early pottery evidence associated with ^{14}C dated materials have been found in only two sites from the Andean sector, the *Flor del Lago-1* site, located on the northern border of the Villarica lake (Beta 253960, charcoal, 2110 ±40 BP, median calibrated 2038 BP, two-sigma 2284 – 2280 cal BP; Beta 273148, no material provided, 2080 ±40 BP, median calibrated 1999, two-sigma 2095 – 1918 cal BP), and the site *Pichipehuenco-2* located in the Galletué-Liucura area (Beta 273150, no material provided, 2190±40 BP, median calibrated 2124 cal BP, two-sigma 2297 - 2263 cal BP) (Adán and Mera, 2011; García, 2009). On the coast, the site P25-1 in Mocha Island showed an early presence of pottery, associated with two ^{14}C dates on charcoal deposits (Gd 9197, 1940±180 BP, median calibrated 1839 BP, two-sigma 2313 - 2226 cal BP; Gd 10007, 1760±130 BP, median calibrated 1633 BP, two-sigma 1923 - 1353 cal BP) (Sánchez, 1997). Considering the high laboratory error of the Mocha Island samples (±100), the results must be taken cautiously. Large laboratory errors over ± 100 are likely associated with poorly preserved samples, which can lead to potential interpretable biases (Mendez-Quiros et al., 2023).

The economy of these groups has been associated with hunting and gathering strategies described for the Late Archaic Period (Campbell and Quiroz, 2015). However, during this period, palynologic analyses on the Central Valley have indicated vegetational clearance for the cultivation of maize from 2000 cal BP (Abarzúa et al., 2014). This evidence has no correlative data to support an early cultivation of maize in the Araucanía; therefore, it has to be cautiously considered.

The earliest evidence on maize cultivation is associated with posterior ceramic groups that demonstrated significant socio-economic changes compared to previous Archaic groups. Until now, the most representative and accepted evidence for a noticeable use of ceramics in the Araucanía is associated with two main cultural complexes: the Pitrén and the El Vergel.

3.3.2. The Early Ceramic Period (1550 - 950 cal BP) and the Pitrén pottery

During the Late 1950’s Osvaldo Menghin and co-workers undertook several excavations in the area of Pitrén, located in the southwest portion of the Calafquén Lake (Menghin 1962, 25). Here the archaeologist found a cemetery denoting a particular ceramic style that, according to him, definitely represented a cultural and chronological complex of its

own (Menghin 1962, 27). The identified pottery encompassed different types of containers, mainly used in the storage and serving of beverages and food such as bowls and jars, some of which showed an anthropomorphic and zoomorphic modelling, and were likely used within the funerary rituals displayed at the Pitrén cemetery; according to Menghin, there were no cooking pots identified in this context (Menghin 1962, 27).

The Pitrén pottery assemblage includes symmetric and asymmetric jars, bowls, cooking pots and mugs (Figure 2.5) (Adán, 2000). Diagnostic decorative elements consist of a reddish-slip surface over the modelled vessel, in some cases including incised decoration and negative painting of leave imprints and geometric shapes (Pérez, Reyes and Hermann, 2012; Adán, 2000). Anthropomorphic, zoomorphic and phytomorphic modelled pieces are also characteristic elements of the Pitrén cultural complex (Adán, 2000).



Figure 2.5: Pitrén pottery: A) Cooking pot; B) Jar; C) Bowl; D) Phytomorphic (Curcubitaceae); E) Decorated; F) Anthropomorphic. Figure created by the author. Photopraphs by Roxana Torres, Daniel Quiroz and Josefa Krstulovic from the Natural History Museum of Concepción, Chile, and Erika Palacios from the University of Chile.

The recurrent finding of Pitrén pottery in pre-colonial cemeteries of southern Chile led to its interpretation as representative of a regional funerary complex (Aldunate, 1989). However, posterior systematic analyses of several pottery assemblages defined Pitrén as the earliest ceramic cultural complex of southern Chile (Dillehay, 1990b). Pitrén cultural complex is the main cultural-historic category of the Early Ceramic Period (ECP) and is represented by semi-sedentary hunter-gatherer groups that employed pottery technology, practised early plant management and horticulture, and managed to make use of each major ecological zone (Adán and Mera, 2011, 1997). These groups maintained contact with trans-Andean groups from the Nequén and Río Negro Provinces in Argentina, evidenced by the finding of diagnostic Pitrén pottery in several archaeological contexts (Prates et al., 2019; Musaubach and Berón, 2016; Salazar and Berón, 2013; Hajduk, 1978). Furthermore, the evidence for early metalwork and textile confection has been interpreted to reflect important socio-political differentiation and complexity within the social organisation of these hunter-gatherer groups (Mera and Munita, 2021; Mera et al., 2015).

Pitrén groups inhabiting the Andes range maintained similar socio-economic strategies as described for the previous Archaic Period (Adán and Mera, 2011; Velázquez and Adán, 2002; Adán, Reyes and Mera, 2001). The identification of *Chenopodium* sp. grains inside a Pitrén pottery possibly indicates the exploitation of a wild type of quinoa (Adán and Reyes, 2000).

Although hunting wild fauna and gathering local plant resources remained the primary socio-economic activities among Pitrén communities of the Central Valley these groups (Roa Solís et al., 2024; Mera and Munita, 2021; Adán and Mera, 2011), domesticated plants, mainly beans (*Phaseolus vulgaris*) and quinoa (*Chenopodium quinoa*) were identified in the Central Valley, possibly indicating early evidence of horticultural practices (Campbell, Roa and Santana-Sagredo, 2018; Roa et al., 2018). One possible maize caryopsis was identified at the Cueva de los Catalanes site in association with the ECP; however, this evidence is still not well supported to ensure an early use of maize on this site (Roa Solís et al., 2024). However, close to the Villarrica Lake, maize remains found in the Villarrica W10 site showed ^{14}C dates of around 950 cal BP, potentially indicating that maize was likely introduced during the transition between the Early and Late Ceramic Period (Roa et al., 2018; Campbell and Quiroz, 2015; Adán and Mera, 2011).

The identification of plant macro remains and wood charcoal suggests the succession of different forest compositions, indicating that local ECP groups were possibly managing the landscape to habilitate open areas for plant cultivation (Roa Solís et al., 2024; Lequesne,

Villagrán and Villa, 1999). However, most plant macro remains phytoliths found in Early Ceramic Period sites show a predominance for exploiting local wild plants (Roa Solís et al., 2024; Roa Solís and Zurro, 2022; Mera and Munita, 2021). According to Roa et al. (2024), landscape modifications during the ECP were possibly the result of wild plant cultivation and management rather than a unique intensification of the cultivation of domesticated plants (Roa Solís et al., 2024, 179).

Coastal Pitrén groups exploited marine products while collecting and hunting resources from the mainland and the coastal range forests (Quiroz and Sánchez, 2010; Contreras et al., 2005). These groups kept a more constant but intermittent occupation of Santa María and Mocha Island, where they practised a mixed economy integrating terrestrial and marine products into their diet (Montalvo-Cabrera et al., 2024; Campbell et al., 2020; Quiroz and Sanchez, 2005).

Notably, further north beyond the Lebu River, research on early ceramic groups has denoted the presence of hunter-gatherers exhibiting differences compared to Pitrén (Quiroz and Sánchez, 2010; Bustos and Vergara, 2001; Seguel, 1969). The possible identification of a different ceramic tradition raised the need to understand better the diversity and variability of the groups inhabiting the Araucanía during the Early Ceramic Period (Adán and Mera, 2011).

3.3.3. The Late Ceramic Period (950 - 400 cal BP) and the El Vergel farmers

On the records of the first soldiers arriving in southern Chile with Pedro de Valdivia around the year 1550, they described crossing a fertile territory southward of the Bío Bío River, rich in a diversity of grains tubers and fruits, managed by a population that practised the cultivation of maize, beans and potatoes (Bibar, 1558, cited in Leonard, 1966, p. 153; Mariño de Lobera, 1595, cited in Escobar, 1865, p. 124). These groups were the ancestors of local Mapuche indigenous groups and the descendants of the Late Ceramic Period (LCP) farmers of the El Vergel cultural complex (Arango-Isaza et al., 2023; Dillehay and Rothhammer, 2013).

The el Vergel cultural complex was first characterised as a funerary tradition as large urn containers (Figure 2.6) employed in the inhumation of the deceased were found in the Central Valley of the Araucanía (Aldunate, 1989; Bullock, 1970). Although its pottery maintained modelling and decorative features from the previous Pitrén cultural complex, the

use of red and black slip and red and black geometric motives over a white slip surface (Figure 2.6) became part of the diagnostic features for its identification (Adán et al., 2005).

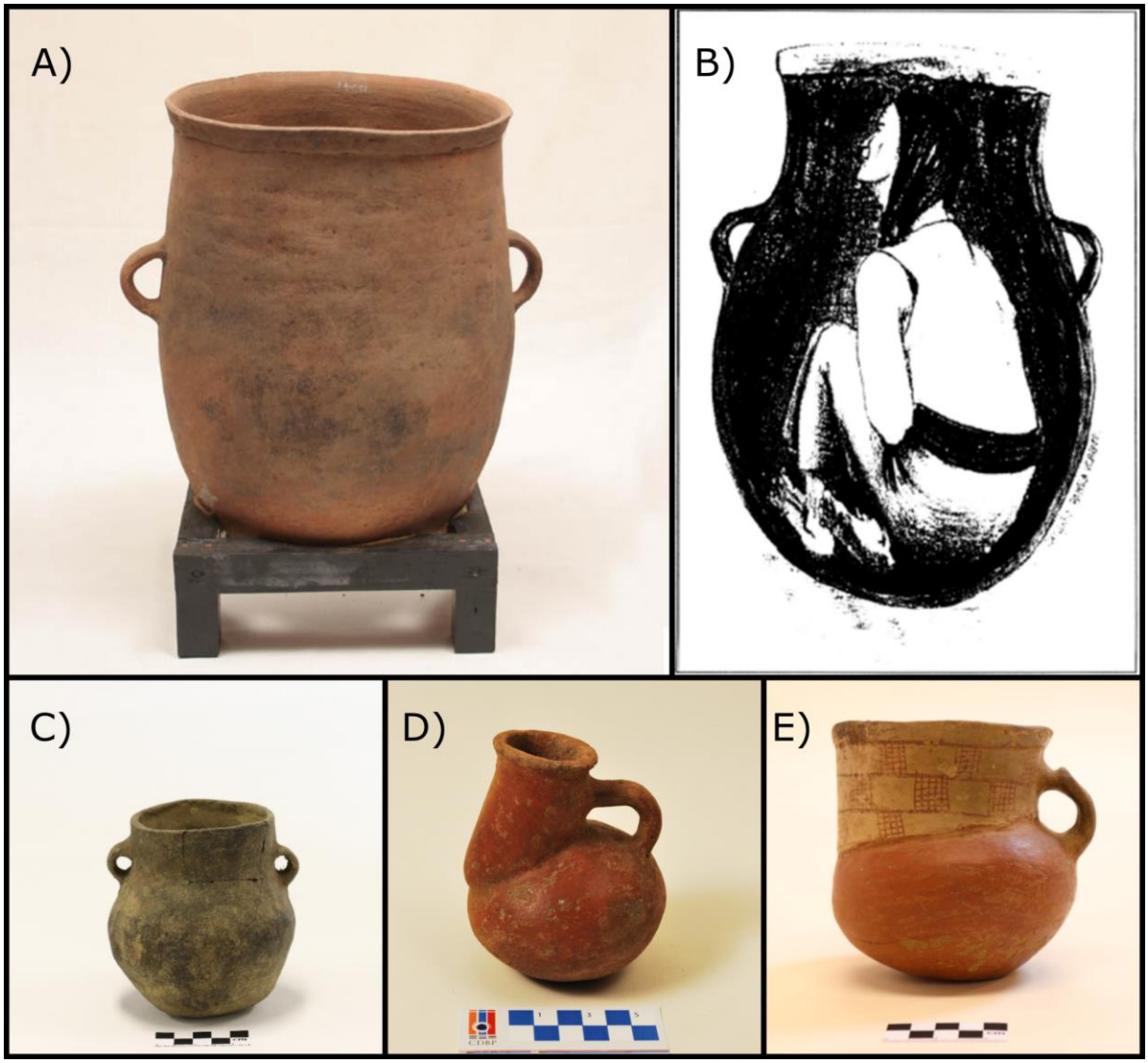


Figure 2.6: El Vergel pottery: ***A)*** *Urn-type pot* ***B)*** *Representation of an El Vergel funerary urn* ***C)*** *Cooking pot* ***D)*** *Jar* ***E)*** *Jar with geometric motifs. Figure created by the author. Photographs by Juan Cesar Astudillo and Patricia Muñoz from the Cañete Museum and Roxana Torres from the National History Museum of Concepción, Chile.*

Further research identified the El Vergel cultural complex in the coast, central valley and the Andes (Quiroz and Sánchez, 2010; Navarro and Aldunate, 2002; Adán, Reyes and Mera, 2001). The proliferation of El Vergel sites in these ecological zones exhibits differences

in how these groups inhabited these environments. While most evidence comes from the coast and Central Valley, where the El Vergel farmers intensified food production, the evidence in the Andes range is scarce. During the Late Ceramic Period, people inhabiting this area maintained a hunting-gathering strategy that persisted until the colonial Period (Roa et al., 2018; Adán, Reyes and Mera, 2001).

During the LCP, several mounds known as *kuel* were built for funerary and ceremonial events (Campbell and Pfeiffer, 2017; Dillehay, 1986, 2007). Organising large groups of people to construct public ceremonial spaces and their employment during important festivities and gatherings denotes significant socio-political changes during the LCP (Campbell, 2020; Dillehay and Saavedra Zapata, 2014b; Dillehay, 2007). At the same time, long-distance relations with trans-Andean groups and between the different ecological zones were intensified, denoting a fluent movement of goods, people and knowledge (Campbell et al., 2018; Bengoa, 2007; Becker, 1997a).

The El Vergel farmers inhabiting the Central Valley and the coast practised the cultivation and management of domesticated plants, principally beans, quinoa (*Chenopodium quinoa*), maize (*Zea mays*), and pumpkin (*Cucurbita sp*), and possibly the taming of South American camelids (Roa et al., 2018; Roa, Silva and Campbell, 2015; Iriarte, 2014; Silva, 2010, 2014a; Contreras et al., 2005). During this period, maize was incorporated as part of the foodways of local farmers, spreading throughout the Central Valley and the coast (Roa et al., 2018). However, a limited number of analyses on carbon ($\delta^{13}C$) and nitrogen ($\delta^{15}N$) stable isotopes from human bone remains have noted a potential preference for C_3 products, possibly indicating that maize was not a staple during the Late Ceramic Period (Campbell et al., 2020). On the other hand, the management and gathering of a diversity of local plants such as madi (*Madia sativa*), wild tubers (*Solanum spp*), grains (*Bromus* sp.), fruits and local nuts (e.g. *Aristotelia chilensis* (maqui), *Fragaria chiloensis* (wild strawberry), *Cryptocaria alba* (peumo), chilean hazelnut (*Gevuina avellana*), and *Rubus* sp.) were likely maintained as prominent socio-economic practices (Roa Solís et al., 2024, Roa et al., 2018; Silva, 2014b; Dillehay et al., 2007).

The construction of raised fields and drainage canals in the area of the Puren-Lumaco Valley reflects an intensification of food production based on the management of domesticated and wild plants to feed an expanding population (Dillehay et al., 2020; Dillehay, 2007; Dillehay et al., 2007). An intensification of wild and domesticated plant management has also been suggested in other parts of the Araucanía based on possible evidence for vegetation clearance to dispose of open areas for plant cultivation (Roa Solís et al., 2024).

Hunting local wild animals was maintained as part of the primary socio-economic activities (Campbell et al., 2020). The identification of textiles, potentially coming from camelids, presumes a certain level of animal management (Navarro and Aldunate, 2002). Although it has been stated that textiles were made from lama wool (*Lama glama*), genetic studies on chilihueque bone remains from Mocha Island demonstrated that camelids from the Araucanía were kindred to wild Patagonian guanaco (*Lama guanicoe*) (Westbury et al., 2016).

Evidence from the coast of the Araucanía indicated the presence of tools for textile production associated with camelid bone remains (Contreras et al., 2005). The translocation of chilihueques from the mainland to Mocha Island potentially suggests their possible tame by local farmers (Campbell et al., 2020; Sánchez, Quiroz and Massone, 2004; Becker, 1997a); however, the high frequency of juvenile camelid remains found in several coastal sites have been interpreted as the result of maladaptation to human management, possibly indicating that chilihueques were not fully domesticated (Contreras et al., 2005; Sánchez, Quiroz and Massone, 2004).

Furthermore, the finding of poultry remains in the el Arenal-1 site was interpreted as a possible early use of domesticated chicken (*Gallus* sp.) in the coast of the Araucanía (Contreras et al., 2005). Radiocarbon dates associated these remains with the LCP, while ancient DNA (aDNA) analyses indicated kinship with Polynesian chicken, denoting pre-colonial trans-Pacific contact between southern Chile and Polynesia (Storey et al., 2013, 2008, 2007). However, while the evidence has not been exempt from debate (Thomson et al., 2014), new excavations at the el Arenal-1 site did not find evidence supporting a trans-oceanic connection with Polynesia while discussing the possible pre-colonial presence of Polynesian chicken in the Araucanía (Buhring et al., 2024).

Farming seemed to have represented a prominent aspect of coastal El Vergel groups' economy; however, fishing, gathering and hunting marine species showed different levels of reliance throughout several coastal sites (Quiroz and Sánchez, 2010; Bahamondes, Silva and Campbell, 2006; Contreras et al., 2005; Massone, 2005). Particularly at Mocha Island, relative ^{13}C enrichment on human bone remains from the Late Ceramic Period has been associated with a mixed diet based on the consumption of maize, C_3 organisms and a limited intake of marine resources (Campbell et al., 2020), despite the abundance of local marine products available on the Island.

4. Conclusion

The environmental history of southern Chile helped to shape the trajectories of past pre-colonial groups from the Araucanía. People inhabiting the coast, Central Valley and the Andes range followed different socio-economic trajectories reflected by exploiting several available resources. However, by 950 cal BP, important socio-political and economic changes occurred in the Araucanía, mainly represented by the intensification of food production and the emergence of social differentiation, and the colonisation and permanent settlement of El Vergel farming communities in nearshore Islands. These processes coincided with the introduction of maize cultivation in the coast and Central Valley. The role that maize had in fostering socio-political and economic processes occurring on the coast and Central Valley of the Araucanía has tenuously been addressed before.

Southern Chile is a fascinating territory with a complex archaeological history. Unfortunately lack of research interest and the intense anthropogenic influence (e.g. industrial agroforestry and extensive modern agriculture) have limited further investigations and the possibility to assess these processes extensively.

Chapter 3: Organic residues and archaeology

1. Introduction

For most of the 20th century, archaeological pottery studies focused on ceramics' typological and physical characterisation. Archaeologists have used pottery to build typological classifications and associated these with past human societies' spatial and chronological distribution (Ford, 1962; Wheat, Gifford, and Wasley, 1958; Nelson, 1916). The analysis of pottery's physical composition has also allowed archaeologists to unravel past societies' economic, culinary and technological aspects while associating the movement of craft materials with long-distance interaction and exchange (Orton and Hughes, 2013, 238; Whitbread, 2015, 28; Shepard, 1965, 348-358). In the late 1970s, Renfrew (1977) referred to the importance of developing new approaches towards pottery studies. The author presented a list of possible archaeological methodologies, indicating the work of Condamin et al. (1976) on organic residue analysis (ORA) of ancient pottery (Renfrew, 1977). In this pioneering article, Condamin et al. (1976) described how chemical analysis and the employment of gas chromatography-mass spectrometry (GC-MS) techniques can help to identify the commodities stored in archaeological vessels.

After Condamin et al. (1976)'s publication, organic residue analysis of archaeological artefacts developed quite slowly (Heron and Evershed, 1993; Patrick, Koning and Smith, 1985; Passi et al., 1981). It was in the 1990s that this field saw a significant expansion. Innovation in the use of new methodologies permitted a better insight into the chemical characterisation of organic residues, allowing the identification of different compounds and linking these to potential commodities processed in ancient pots, such as animal and plant resources (Dudd, Evershed and Gibson, 1999; Evershed et al., 1992; Evershed, Heron and Goad, 1991). Furthermore, the integration of carbon stable isotope analysis ($\boldsymbol{\delta}^{13}$C) on specific compounds by gas chromatography-combustion-isotope ratio mass spectrometry (GC-c-IRMS) complemented the biomolecular data (Mottram et al., 1999; Evershed et al., 1997c, 1994). The improvement of ORA was reflected in the number of publications using this novel methodology, expanding to different contexts outside Europe (Shoda et al., 2017; Dunne et al., 2016; Colonese et al., 2014; Craig et al., 2011).

2. Application of ORA in archaeology

Under certain conditions, organic matter can be found adhered to the surface of archaeological artefacts as evidence of past human activities. This evidence has been largely reported to be found associated to stone tools (Galanidou, 2006; Rafferty, 2002; Quigg et al., 2001), organic artefacts (Brettell et al., 2017; Robinson et al., 1987), and human remains (Buckley and Evershed, 2001; Buckley, Stott and Evershed, 1999). Furthermore, the use ofceramic containers to process different types of commodities can lead to the formation of organic residues as evidence of past culinary practices (Regert, 2011; Regert et al., 2003b).

Organic residues can be preserved as food crusts attached to the walls of pottery containers after several uses (Oudemans and Boon, 1991; Patrick et al., 1985); however, this evidence is not always present as food crustscan readiliy be lost in the burial environment. On the other hand, t the heating (Heron et al., 2013a; Charters et al., 1993), grinding (Cramp, Evershed and Eckardt, 2011) and storing (Pecci et al., 2013; Stern, 2000; Evershed et al., 1997a) of different commodities inside a pottery container can lead to the migration or absorption of organic residues into the porous matrix of unglazed ceramic vessels. Organic residues are trapped within the pottery porous matrix, which reduces their exposure to external degradation agents, allowing their preservation throughout time (Evershed, 2008a). The source of information that these residues contain has great value for archaeologists. However, due to the amorphous shape or invisible recognition of organic residues, they cannot be physically classified and identified; therefore they need to be analysed chemically (Regert, 2011; Heron and Evershed, 1993).

Within the biomolecular evidence preserved in organic residues, the identification and extraction of lipids have been traditionally favourable due to their physico-chemical characteristics that allow their preservation throughout time (Evershed, 1993). Their hydrophobicity and almost non-polarity make them only soluble in organic solvents and non-soluble in water, which prevents them from being leached out by water (Voet and Voet, 2011; Charters et al., 1993). These characteristics may have allowed lipids to endure post-depositional degradation factors better than other biomolecules such as DNA, proteins, and carbohydrates (Eglinton et al., 1991). Lipids comprise a large group of molecular compounds, some likely to be extracted from pottery artefacts and identified through GC-MS. Therefore, knowing their classification is necessary to understand their characteristics better.

3. Lipids classification

Lipids are found in all living organisms and have different functions and characteristics (Evershed et al., 1992). They act as the main component of the cell membrane and have an important role in energy storage and signalling pathways during metabolic processes (Fahy et al., 2011). Regarding their high diversity is that in 2005 the International Lipid Classification and Nomenclature Committee, funded by the LIPIDS MAPS Consortium, developed a classification system of eight different categories, which were improved and updated in 2009 (Fahy et al., 2009, 2005). The eight groups' category consists ofFatty Acyl lipids (FA), Glycerolipids (GL), Glycerophospholipids (GP), Sphingolipids (SP), Saccharolipids (SL), Polyketides (PK), Sterol lipids (ST), and Prenol Lipids (PR) (Fahy et al., 2011). They can be differentiated by their biosynthetic pathways, being these last two categories, sterol lipids (ST) and prenol lipids (PR), of a different origin compared to the rest of the lipid groups (Fahy et al., 2009).

3.1. fatty acyl lipids (FA)

This category mainly consists on fatty acids and fatty acid derivatives such as alkanes, fatty alcohols, wax esters, eicosanoids and docosanoids (Fahy et al., 2009; Ratnayake and Galli, 2009).

3.1.1 fatty acids

Fatty acids are mainly composed of a hydrophobic hydrocarbon chain, which can be linear, branched or cyclic, bound to a slightly charged carboxylic group (Bond et al., 2016, 187; Nelson and Cox 2013, 360; Evershed et al., 1992). They can be classified according to the number of carbon atoms of their hydrocarbon chain as small chain fatty acids ($C_{3:0}$-$C_{7:0}$), medium chain fatty acids ($C_{8:0}$-$C_{13:0}$), long chain fatty acids ($C_{14:0}$-$C_{20:0}$), and very long chain fatty acids ($\geq C_{21:0}$) (Ratnayake and Galli, 2009).

Saturated fatty acids are known to have a single bond between each hydrocarbon unit. In contrast, unsaturated fatty acids have at least one double bond at any point of the hydrocarbon chain (Figure 3.1) (Nelson and Cox 2013, 357). Following this division, the

standard nomenclature for saturated fatty acids would be the number of carbon atoms followed by a ´´0´´, which stands for the absence of double bonds in its composition (Berg, Tymoczko and Stryer, 2012, 359). For example, stearic acid (*n*-Octadecanoic acid) has 18 carbon atoms and no double bond; therefore, it is written as $C_{18:0}$ (Berg et al., 2012, 359). On the other hand, the nomenclature of unsaturated fatty acids indicates the number of double bonds in the hydrocarbon chain. Therefore, the number will specify whether it consists of a mono-unsaturated fatty acid (MUFA) or poly-unsaturated fatty acid (PUFA) (Voet and Voet, 2011, 387). For example, the mono-unsaturated fatty acid oleic acid (*cis*-9-octadecenoic acid) has 18 carbon atoms and one double bond in the 9th carbon, and it is written as $C_{18:1}$; whereas in the case of a poly-unsaturated fatty acid such as linoleic acid (*cis*-, *cis*-9,12 octadecadienoic acid), numbers 9 and 12 would refer to the position that each double bond of $C_{18:2}$ has in the hydrocarbon chain (Nelson and Cox, 2013, 358). The *cis*- denomination refers to one of the two molecular isomers or configurations, *cis*- or *trans*-, that an unsaturated fatty acid can have regarding their double bond position (Heipieper, Meinhardt and Segura, 2003). The *cis*-configuration is the most common to occur in living organisms; however the *trans*-configuration, although less frequent, can also occur in nature (Sommerfeld, 1983).

Double bonds make the hydrocarbon chain curve, which avoids a straight interaction between fatty acids, as it would be for saturated fatty acids (Voet and Voet 2011, 388). This particular hydrocarbon chain disposition reduces van der Waals interactions between the molecules, leading to a reduction of their melting point; thus, unsaturated fatty acids can be found in a liquid phase at room temperature (Nelson and Cox 2013, 359). However, unsaturated fatty acids in the *trans-configuration* have a straighter hydrocarbon chain disposition, increasing their melting point at room temperature (Marchand, 2010; Nelson and Cox 2013, 359).

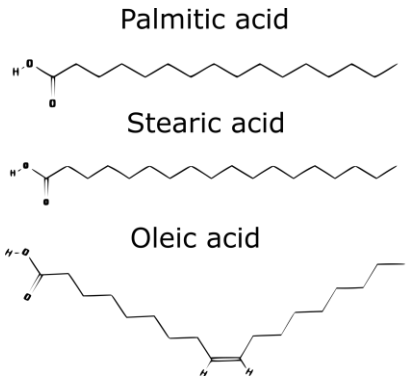


Figure 3.1: Palmitic ($C_{16:0}$), stearic ($C_{18:0}$) and oleic ($C_{18:1}$) acids.

3.1.2. alkanes

Another compound category comprises alkanes. These compounds are characterised by being composed only of carbon and hydrogen atoms (Figure 3.2), bonded by a single bond between them; thus, it is possible to refer to them as hydrocarbons (Clayden, Greeves and Warren, 2012). When a hydrogen atom is removed from an alkane, this changes into an alkyl configuration, allowing other compounds to be formed, such as alcohols (Bruice 2007, 74).

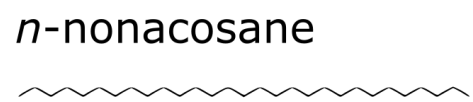


Figure 3.2: Alkane n-nonacosane (C_{29}) showing a straight hydrocarbon chain.

3.1.3. fatty alcohols

Alcohols consist of a saturated carbon chain bonded to a hydroxyl functional group (-OH) (Figure 3.3). Those fatty alcohols ranging from one carbon atom to eight carbon atoms are still soluble in water (Mudge, Belanger and DeLeo, 2018). However, alcohols with a carbon number above eight exhibit hydrophobicity and are classified as fatty alcohols (Mudge et al.,2018).

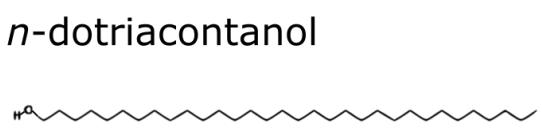


Figure 3.3: Fatty alcohol n-dotriacontanol (C_{32}).

3.1.4. wax esters

The esterification of a fatty acid with a fatty alcohol builds a wax ester (Figure 3.4) (Metz et al., 2000). This biomolecule is used as energy storage and as a building block for different molecular structures of plant and animal species (Lardizabal et al., 2000). The presence of fatty acids and fatty alcohols makes them a very hydrophobic compound (Nelson and Cox 2013, 362).

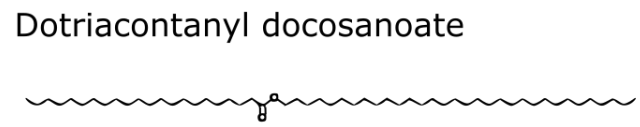


Figure 3.4: Wax ester dotriacontanyl docosanoate (C_{54}), as the result of the ester bond betwen $C_{22:0}$ fatty acid and C_{32} fatty alcohol.

3.1.5. eicosanoids and docosanoids

Eicosanoids and docosanoids are signalling lipids essential in the inflammatory response (Figure 3.5), synthesised by the oxidation of arachidonic acid and other related poly-unsaturated fatty acids (Dennis and Norris, 2015).

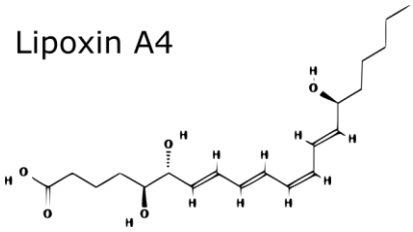


Figure 3.5: Eicosanoid lipoxin A4.

3.2. glycerolipids (GL)

This category includes those lipids bounded to a glycerol molecule (Fahy et al., 2005). Within this category, acylglycerols are composed of a glycerol molecule bound to one to three fatty acids, each through an ester linkage between the carbon atom of the fatty acid carboxylic head and a hydroxyl group from the glycerol molecule (Figure 3.6) (Nelson and Cox 2013, 360). It will depend on whether the acylglycerol comprises one, two or three fatty acids to be considered mono-, di, or triacylglycerol (Voet and Voet, 2011).

Triacylglycerols are well-represented in living organisms for their role as energy storage molecules and in insulation (Nelson and Cox, 2013, 360). In plants, they are mainly concentrated in fruits, pollen and oil-rich seeds (Schmid, 2016), which in the latter represents one of the main components necessary for seed growth and development, previous to photosynthetic autonomy (Schmid, 2016; Wiesman and Chapagain, 2009). Triacylglycerols are effectively stored in the adipose tissue of mammals as fatty acid reservoirs for energy obtainment through fatty acid **β**-oxidation (Berg et al. 2012).

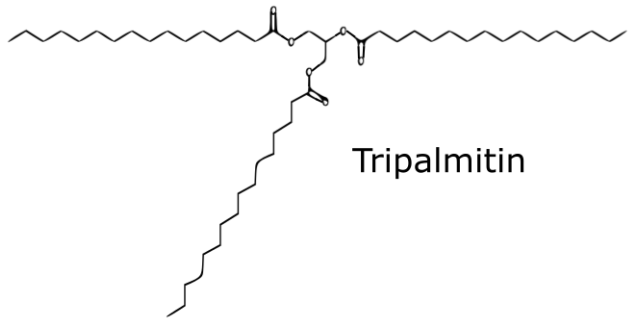


Figure 3.6: Acylglycerol tripalmitin, highly present in most organisms.

3.3. glycerophospholipids (GP) and sphingolipids (SP)

Both lipid categories are essential components of the cellular membrane due to their amphipathic structure (Futerman, 2016). Glycerophospholipids, as glycerolipids, are based on the esterification of fatty acids with hydroxyl groups of a glycerol molecule; however, in the case of glycerophospholipids, a diacylglycerol is esterified by its free -OH group to a phosphate molecule, being the latter the hydrophilic portion of the lipid (Figure 3.7) (Fahy et al., 2005; Hishikawa et al., 2014).

Although sphingolipids are also an essential constituent of the cellular membrane (30%) (Futerman, 2016), they do not have a similar structure to glycerophospholipids. Instead of a glycerol molecule as the backbone of its structure, sphingolipids contain a long-chain sphingoid base, which bonds to a fatty acid to form a ceramide (Figure 3.7) (Young et al., 2012). From a single hydrogen atom, polar molecules can be attached to this ceramide structure to form an amphipathic sphingolipids (Merrill et al., 2007).

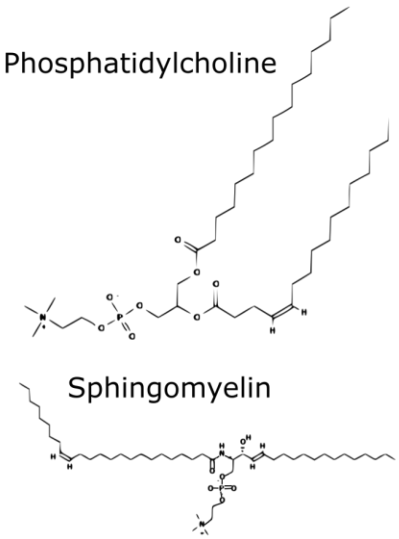


Figure 3.7: Glycerophospholipid phosphatidylcholine and shpingolipid sphingomyelin. Both compounds are important components of the plasma membrane of eukaryotic cells.

3.4. saccharolipids (SL)

These compounds also comprise an essential component of the cellular membrane (Dowhan, Bogdanov and Mileykovskaya, 2016). It consists of a fatty acyl bound to a saccharide or sugar molecule (Figure 3.8), which has been differentiated from the term glycolipid due to different linkage characteristics (Fahy et al., 2005).

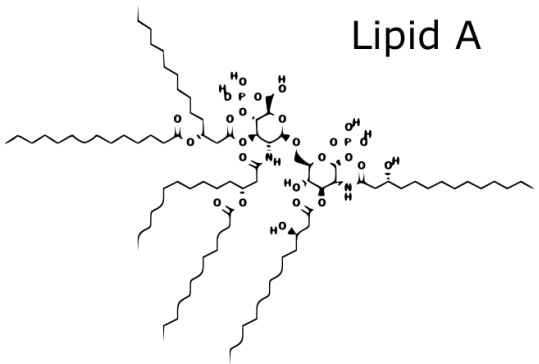


Figure 3.8: Saccharilipid lipid A showing the glucosamine disacharide bounded to fatty acids.

3.5. polyketides (PK)

They are a large family of compounds, mainly present in plants, fungi and bacteria (Figure 3.9) (Miyanaga, 2017). These compounds are built by chain elongation of acetyl-CoA or propionyl CoA, and malonyl-CoA or methylmalonyl-CoA blocks (Baerson and Rimando, 2007).

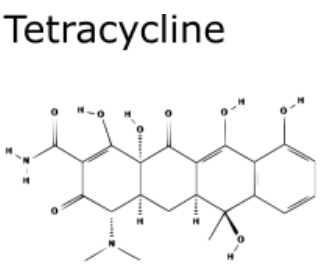


Figure 3.9: Polytetide tetracycline.

3.6. sterol lipids (ST), and prenol lipids (PR)

Sterol and prenol lipids differentiate from the rest due to their biosynthetic pathway. Sterol lipids and prenol lipids, have their origin in the synthesis of isoprenoid units by the polymerisation of isopentenyl pyrophosphate and dimethylallyl pyrophosphate, having mevalonic acid as a precursor (Fahy et al., 2011; Clayden, Greeves and Warren, 2012; Chatzivasileiou et al., 2019), or pyruvate and glyceraldehyde 3-phosphate as part of a non-mevalonate pathway (Chatzivasileiou et al., 2019). The remaining lipid categories are based on synthesising fatty acyl lipids by thioester condensation, which would suffer different modifications regarding each group (Fahy et al., 2011).

Sterols comprise a large family of lipids characterised bya rigid structure of four fused rings, to which a hydrocarbon and an alcohol group are attached (Figure 3.10) (Ratnayake and Galli, 2009). Sterols can be found as free molecules, as well as esterified to other compounds such as long-chain fatty acids (Goodwin, 1985; Jones and Glomset 1985, 95). They are an essential component of the eukariotik cellular membrane (Guan et al., 2009).

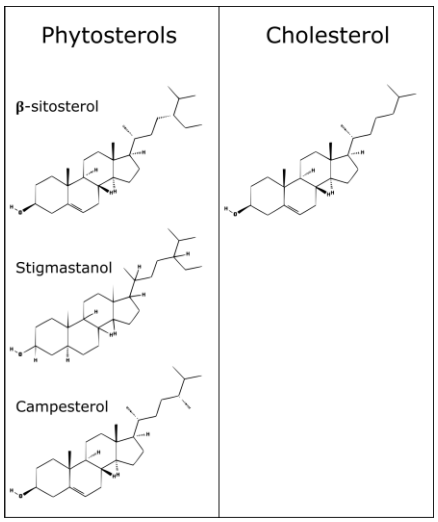


Figure 3.10: Plant and animal sterols. Figure created by the author.

As for prenol lipids, although they have a similar biosynthetic pathway to sterols, they have functional and structural differences (Fahy et al., 2011). Prenol lipids are simpler and can have a linear configuration of isoprenoid units (Ratnayake and Galli, 2009). This configuration will conform the basis for the synthesis of terpenoids, which are commonly present in nature (Figure 3.11) (Gershenzon and Dudareva, 2007). Quinone compounds such as vitamin E, vitamin K and ubiquinones are also part of these isoprenoid-based compounds (Ratnayake and Galli, 2009).

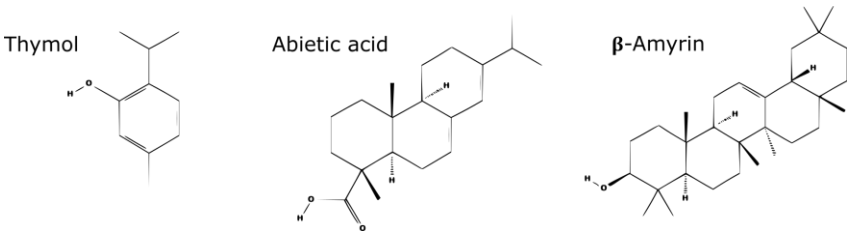


Figure 3.11: Examples of prenol lipids showing monoterpenoid thymol, highly found in thyme oil; diterpenoid abietic acid, which is a main constituent of pine resin; and triterpenoid β-Amyrin highly found in several plants. Figure created by the author.

4. Lipid analysis in archaeological ceramic artefacts

The classification presented above demonstrated how diverse lipids can be. However, several degradation factors will make unlikely for lipids to be preserved in their original state. Lipids survival throughout time has been approached under the concept of biomarkers. Although this concept can be applied to any biomolecule (Evershed, 2008b), it has been traditionally employed for describing lipid preservation in archaeology.

4.1. Lipid preservation in the archaeological record

In geochemistry, a biomarker or biological marker is regarded as the carbon skeleton of organic compounds that survived after the decay and degradation of organic matter throughout time (Philp and Oung, 1988; Brassell et al., 1985). Their presence in the geological record has been used to track back the primary organic sources contributing to soil formation (Mackenzie et al., 1982).

For example, the survival of free n-alkanes in the geological record, predominantly very long odd number carbon chain, diterpenoids and C_{29} sterols have been argued to indicate a terrestrial organic input in soil formation (Madureira, Conte and Eglinton, 1995; Comet et al., 1981; Cranwell, 1981). Whereas the presence of sterols such as dinosterol, and shorter carbon chain n-alkanes would account for a contribution from aquatic organisms (Brassell, 1984; Comet et al., 1981).

Although the biomarker concept emerges from geochemistry, a broader use has been applied to analyse organic residues from archaeological pottery for a better understanding of past human activities (Evershed, 2008b, 1993). Whether lipids are exposed to high temperatures due to different pottery usage and cultural practices or to natural and environmental factors through post-depositional periods after pottery discard, their molecular content is likely to be degraded (Regert et al., 2003b; Evershed, Heron and Goad, 1990). Practices such as cooking (Lucquin et al., 2016a; Cramp et al., 2014), craft production (Colombini, Modugno and Ribechini, 2005; Regert et al., 2003a), and using pottery as lamps (Heron et al., 2013a; Copley et al., 2005) have involved the application of high temperatures to organic matter, provoking the formation of different biomarkers as a consequence (Drieu, Lepère and Regert, 2019; Evershed et al., 2008; Hansel et al., 2004; Raven et al., 1997).

The exposure of lipids to high temperatures during cooking events will likely lead to the formation of oxidising agents (Malainey, Przybylski and Sherriff, 1999; Regert et al., 1998). Lipid oxidation (Figure 3.12) occurs when oxidising molecules attack lipids, especially carbon-carbon double bonds in unsaturated fatty acids, leading to their degradation (Ayala, Muñoz and Argüelles, 2014). However, for an oxidising agent to act on a lipid compound, the latter must be reactive (Falade and Oboh, 2015; Choe and Min, 2007). For this, the exposure to heat or light will cleavage the weakest C-H bond in an unsaturated lipid, forming a radical compound with unpaired electrons (Ghnimi, Budilarto and Kamal-Eldin, 2017). The formed radical lipid attracts oxidising agents such as oxygen, which are prone to attack back the radical lipid, leading to the formation of new oxidising agents such as hydroperoxides in a process known as auto-oxidation, leading to the propagation of hydroperoxides to continue the oxidation of other compounds (Ghnimi et al., 2017; Hamilton et al., 1997).

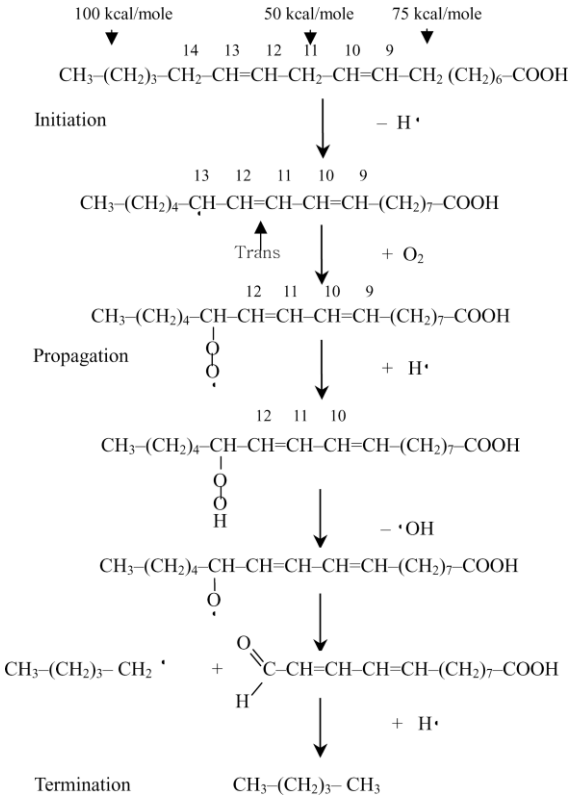


Figure 3.12: Main steps for lipid auto-oxidation (Choe and min, 2007).

The formation of radical lipids by hydrogen abstraction can also enhance polymerisation (Figure 3.13), as the radical carbon tends to react with other lipids, leading to dimerisation and polymerisation (Choe and Min, 2007). Oil polymerisation increases with

temperature, leading to more hydrogen abstraction, thus increasing the formation of radical lipids (Choe and Min, 2007).

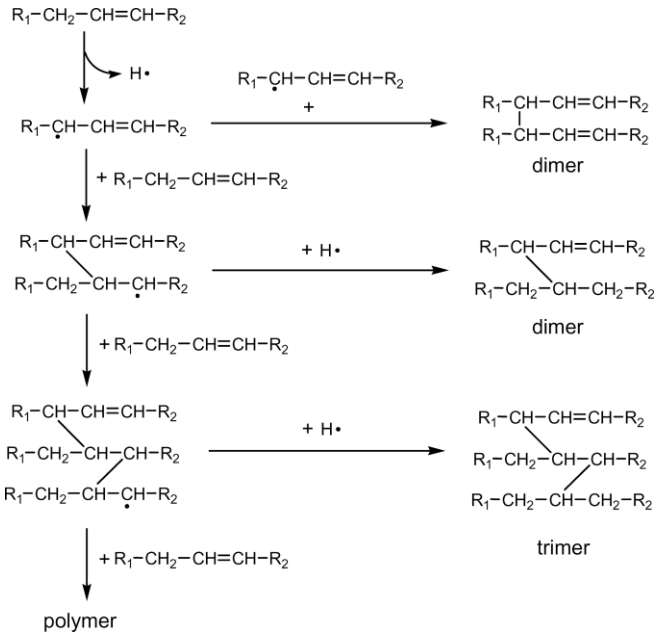


Figure 3.13: Lipid polymerisation after hydrogen abstraction (Choe and min, 2007).

When exposed to high temperatures, lipids can experience a process of structural and geometric reconfiguration of their atoms known as isomerisation (Dugave and Demange, 2003; Brassell et al., 1985). Isomers can express differences in their physical characteristics, as seen in the isomerisation of *cis*- double bonds to a more stable trans-configuration in unsaturated fatty acids exposed to high temperatures (Li et al., 2019). The latter has been stated to be more resistant to oxidative processes (Ghnimi et al., 2017), which delays food degradation and rancidity (Hamilton et al., 1997). An acidic or alkaline environment generated by metal ions can also catalyse lipid isomerisation as well as ring aromatisation of organic compounds, leading to the formation of different biomarkers (Varma, 2002; Brassell et al., 1985; Hussler et al., 1981).

When discarded, pottery enters into the burial environment where other degradation factors can act on lipid residues from processed organic matter (Figure 3.14). Ester bonds present in acylglycerols, such as triacylglycerols, can be cleaved by water to form mono- and diacylglycerols and free fatty acids during a process known as hydrolysis (Regert et al., 1998; Evershed et al., 1992). Acylglycerol hydrolysis can also be enhanced after potsherd discard

by the enzymatic action of soil microorganism lipases (Sugimori and Utsue, 2012). Carbon is an essential nutrient for soil microorganisms' growth; thus, organic molecules, in this case lipids, become a principal target for degradation processes such as hydrolysis and fatty acid β-oxidation by microorganisms (Kallenbach, Frey and Grandy, 2016; Sugimori and Utsue, 2012). The consequences of this are degradation and microbial lipids forming in the burial environment (Kallenbach et al., 2016; Sugimori, Nakamura and Mihara, 2002). Lipids from the soil environment can migrate into the porous matrix of potsherds (Dudd, Regert and Evershed, 1998). According to Heron, Evershed and Goad. (1991), it would be possible to discriminate between lipids from the soil environment with those preserved in the pottery matrix; however, some soil microorganism lipids, such as branched *iso* and *anteiso* fatty acids, can also be found in aquatic an ruminant products, thus their interpretation must be taken with caution (Kaneda, 1991; Hauff and Vetter, 2010).

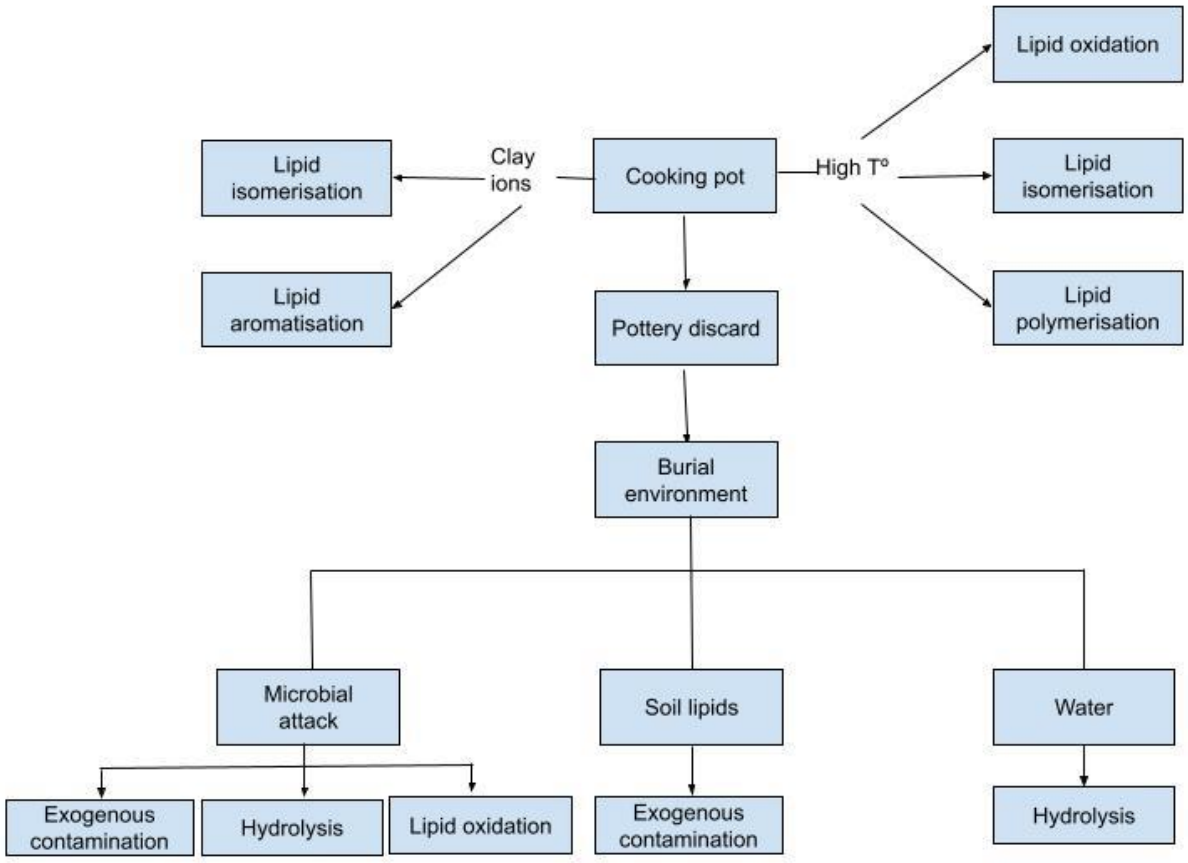


Figure 3.14: Summary of degradation factors during cooking events and after the potsherd has enter the soil environment. Figure created by the author.

4.2 Instrumentation

Lipids are separated, identified, and quantified by gas chromatography (GC) and gas chromatography-mass spectrometry (GC-MS). The former separates compounds as the extract enters the GC through an injector port where thermally stable volatile compounds are vaporised before their separation through the GC column (Qian, Peterson and Reineccius, 2017). A carrier gas, such as helium, hydrogen or nitrogen, takes the volatile compounds through the column while an oven controls the different temperatures needed during the process (McMaster, 2008; AL-Bukhaiti et al., 2017). According to the compound's affinity to the column's stationary phase, a retention time will be displayed (AL-Bukhaiti et al., 2017). After compound separation, their quantification can be achieved through a Thermal Conductivity Detector (TCD) or by Flame Ionisation Detector (FID) (Sobrado et al., 2016; AL-Bukhaiti et al., 2017). The employment of a mass spectrometer (MS) will lead to the ionisation of the compounds by an ionisation source, allowing their subsequent separation and detection according to their mass/charge ratio (*m/z*)(McMaster, 2008; Gross 2017, 14). The mass spectrometer will generate a chromatogram showing several compounds eluting according to their *m/z* at a specific retention time (min), and the ordinate indicating the relative abundance of each compound to provide further quantification (Gross 2017, 15).

5. Lipid interpretation in ceramic artefacts

5.1. Animal products

Based on the Regert's approach (2011), this section will be divided into three principal sources of animal fat which are prone to be identified in archaeological pottery: **a)** subcutaneous fat of ruminant and non-ruminant animals, **b)** dairy products, and **c)** aquatic animals.

5.1.1 Subcutaneous fat of ruminants and non-ruminants

Apart from some marine animals which use wax esters as their primary energy storage, most animals use triacylglycerol for this purpose (Gurr and Harwood, 1991). The evidence for animal fats can be seen in a mixture of triacylglycerols (T_x) between T_{40} and T_{54}, with T_{50} and

T_{52} as the most abundant (Evershed et al., 2002), and a distribution of mono- and diacylglycerols accompanied by free fatty acids, as the product of fatty acid hydrolysis from triacylglycerols (Dunne et al., 2019; Evershed et al., 2002; Regert et al., 1998).

At the same time, cholesterol is an essential constituent of the cellular membranes of most animal tissues (Brown and Sharpe, 2016; Galea and Brown, 2009). In the blood, they are esterified with lipoproteins for transportation (Larsson, 1994). Cholesterol may suffer degradation while heating animal fat or due to post-depositional processes (Evershed et al., 1992); therefore, it is likely to be identified from the residues along with bio-hydrogenated, dihydrated and oxidation derivatives (Figure 3.15) (Whelton et al., 2021; Hammann et al., 2018; Gea et al., 2017; Kimpe et al., 2004).

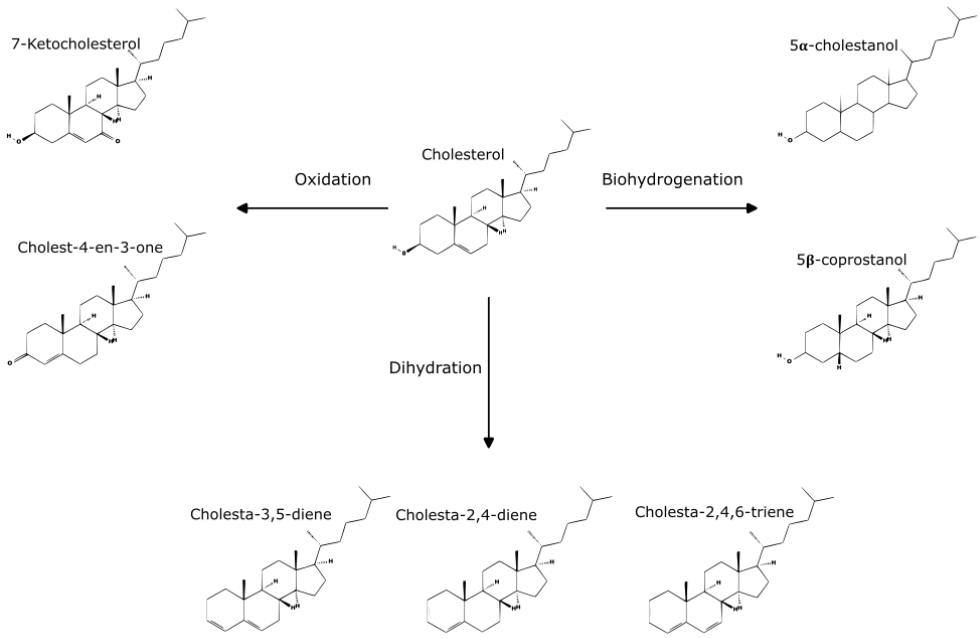


Figure 3.15: Degradation products of cholesterol. Figure is based on data from (Hammann et al., 2018; Whelton et al., 2021). Figure created by the author.

Palmitic ($C_{16:0}$) and stearic ($C_{18:0}$) acids are commonly found in animal and plant resources (Spangenberg, Jacomet and Schibler, 2006; Copley et al., 2001). However, terrestrial animals tend to have a ratio between palmitic and stearic acids (P/S) closer to one, contrasting with plant commodities, which show a considerably higher abundance of $C_{16:0}$ over

$C_{18:0}$ fatty acid (Dunne et al., 2016; Evershed et al., 1997c). More specifically, a relatively higher abundance of $C_{18:0}$ over $C_{16:0}$ in the residue has been considered to indicate an origin from ruminant fat, whereas a relatively higher contribution of $C_{16:0}$ to the residue might indicate the presence of monogastric animals such as pigs (Evershed et al., 2002; Kimpe, Jacobs and Waelkens, 2002). Although Dudd et al. (1998) have argued that $C_{18:0}$ and $C_{16:0}$ alkanoic acids are not likely to be degraded by microorganisms, Den Dooren De jong, Dauvilier and Roman, (1961) demonstrated that soil bacteria could attack oleic acid to form palmitic acid during the process of adipocere formation in human tissue. Furthermore, the higher solubility of $C_{16:0}$ over $C_{18:0}$ in water may also alter the P/S in archaeological materials (Whelton et al., 2021). Therefore, interpretations of the P/S to assess the residue's origin needs to be taken cautiously.

The distribution of triacylglycerols in animal fat shows differences between ruminant and non-ruminant adipose, likely identified in residues extracted from pottery artefacts. Ruminant adipose fat comprises a broad triacylglycerol distribution, ranging from T_{42} to T_{54}, whereas non-ruminants have a narrower distribution comprising a range from T_{44} to T_{54} (Regert, 2011; Dudd et al., 1999). Moreover, the abundance of T_{54} is relatively higher in ruminants than in non-ruminants (Regert, 2011; Evershed et al., 1997c). However, triacylglycerols do not always survive post-depositional conditions as fatty acid moieties are easily hydrolysed, leading to the sole identification of the glycerol backbone and free fatty acids (Dudd and Evershed, 1998a).

Identifying branched fatty acids $C_{15:0}$ and $C_{17:0}$ in their *iso* and *ante-iso* configuration might also indicate the presence of ruminant carcass fat in the residues (Dunne et al., 2012; Gurr and Harwood, 1991). The distribution of these compounds has been shown to differ between ruminant products and aquatic organisms by showing a preference for the *ante-iso* configuration over the *iso-branched* fatty acid isomer in ruminants (Hauff and Vetter, 2010). However, soil microorganisms can also synthesise branched fatty acids, accounting for possible exogenous lipid contamination in the residues instead of indicating an origin from ruminant fat (Dudd et al., 1998).

5.1.2. Dairy products

Compared to animal adipose, triacylglycerols in fresh milk comprise a distribution between T_{26} to T_{54} (Dudd and Evershed, 1998a). Short to long-chain fatty acids ($C_{4:0}$ - $C_{20:0}$) are the most common components of dairy triacylglycerols (Carrer et al., 2016; Dudd et al.,

1998; Kuksis, Marai and Myher, 1973). However, short to medium-chain fatty acids are more susceptible to being hydrolysed from triacylglycerols during post-depositional events than longer-chain homologues (Dudd et al., 1998). Free short-chain fatty acids' high solubility makes them prone to being lost or highly altered in the burial environment, which limits their preservation and further recovery from archaeological contexts (Craig et al., 2005; Evershed et al., 2002). These diagenetic factors will lead to the degradation of triacylglycerols from milk, generating a triacylglycerol distribution that can easily resemble that of a ruminant adipose origin (Dudd and Evershed, 1998a; Dudd et al., 1998).

5.1.3 Marine products

The evolutionary history of aquatic and terrestrial animals has led to adaptive differences that can be recognised in their lipid content. On one hand, terrestrial animals' adipose tissue has a high abundance of saturated over unsaturated fatty acids, whereas aquatic animals are richer in unsaturated fatty acids (Samuel and Worthy, 2004; Passi et al., 2002). The distribution of unsaturated fatty acids varies among species, and can be influenced by several external factors such as temperature, habitat and season (Samuel and Worthy, 2004; Hall, Parrish and Thompson, 2002; Vlieg and Body, 1988).

Monounsaturated fatty acids are abundant in aquatic animals (Osman, Suriah and Law, 2001; Strandberg et al., 2008; Passi et al., 2002). While monounsaturated fatty acids are readily obtained from the diet, several aquatic organisms can undergo Δ-9 desaturation of $C_{16:0}$ and $C_{18:0}$ fatty acids to form monounsaturated aliphatics $C_{16:1}$ (n-7) and $C_{18:1}$ (n-9) (Parrish, 2013; Osman et al., 2001; Henderson, 1996). Fatty acid desaturation can also occur in terrestrial mammals but at a lower rate than in aquatic animals (Guerrero and Rogers, 2019).

The absence of Δ-12 and Δ-15 desaturase enzymes in all vertebrates restricts the conversion of monounsaturated fatty acids into linoleic ($C_{18:2}$) and ***α***-linolenic ($C_{18:3}$) fatty acids and, therefore, must be obtained from the diet (Tocher, 2003). These compounds are the main precursors for synthesising complex polyunsaturated fatty acids in several organisms. $C_{18:2}$ and $C_{18:3}$ undergo a Δ-4, Δ-5, and Δ-6 desaturation leading to the synthesis of arachidonic acid ($C_{20:4}$), eicosapentaenoic acid ($C_{20:5}$) and docosahexaenoic acid ($C_{22:6}$) (Monroig, Tocher and Navarro, 2013; Tocher, 2003; Gurr and Harwood, 1991). $C_{18:2}$ and $C_{18:3}$ desaturation to these complex polyunsaturated fatty acids occurs at high levels in aquatic organisms, especially freshwater fish and marine flagellates and diatoms from where polyunsaturated fatty acids enter into the marine food chain; therefore, it is expected to find high levels of polyunsaturated

fatty acids $C_{20:4}$, $C_{20:5}$, $C_{22:6}$ in most aquatic animals (Guerrero and Rogers, 2017; Strandberg et al., 2008; Samuel and Worthy, 2004; Tocher, 2003).

Mono and polyunsaturated fatty acids are likely to be degraded when exposed to high temperatures and due to post-depositional events, which makes them almost undetectable in organic residues from archaeological pottery (Evershed et al., 1992). However, identifying several unsaturated fatty acid degradation products can support further interpretations.

Monounsaturated fatty acids can produce $\boldsymbol{\alpha}$,-dicarboxylic acids as well as hydroxy- and dihydroxy fatty acids from the oxidation of monounsaturated fatty acid double bonds (Regert et al., 1998). The carbon chain length of $\boldsymbol{\alpha}$,-dicarboxylic acids indicates the exact position where the double bond was in the original monounsaturated fatty acid, whereas hydroxy- and dihydroxy fatty acids retain the carbon number of their precursor (Colombini et al., 2005; Copley et al., 2005). Hydroperoxides formed by polyunsaturated fatty acid auto-oxidation can react with monounsaturated fatty acids to form dihydroxy fatty acids (Hansel and Evershed, 2009). The formation of C_{16} to C_{22} carbon chain length dihydroxy acids indicates the degradation of monounsaturated fatty acids of similar carbon chain length (Hansel and Evershed, 2009). A distribution of monounsaturated fatty acids of $C_{16:1}$ - $C_{22:1}$ are not typical of terrestrial animals and plants (Beare-Rogers, Dieffenbacher and Holm, 2001); however, they are abundant in aquatic animals (Strandberg et al., 2008; Passi et al., 2002; Osman, Suriah and Law, 2001). Therefore, identifying dihydroxy fatty acids of C_{16}, C_{18}, $C_{20,}$ and C_{22} carbon chain length can indicate the processing of aquatic products in archaeological pottery (Hansel and Evershed, 2009).

Matikainen et al. (2003) demonstrated that the alkali-isomerisation of methyl linolenic acid ($C_{18:3}$) followed by a 1,5 hydrogen shift could lead to a fully conjugated $C_{18:3}$. The constant exposure to a high temperature would allow the continuity of the isomerisation process, leading to the formation of cyclohexadienoic structures and their subsequent aromatisation, resulting in the synthesis of $\boldsymbol{\omega}$-(*o*-alkylphenyl) octadienoic acid (APAA-C_{18}). Notably, Hansel et al. (2004) indicated that a 1,7 hydrogen shift may promote the formation of a wider distribution of APAA isomers (Figure 3.16).

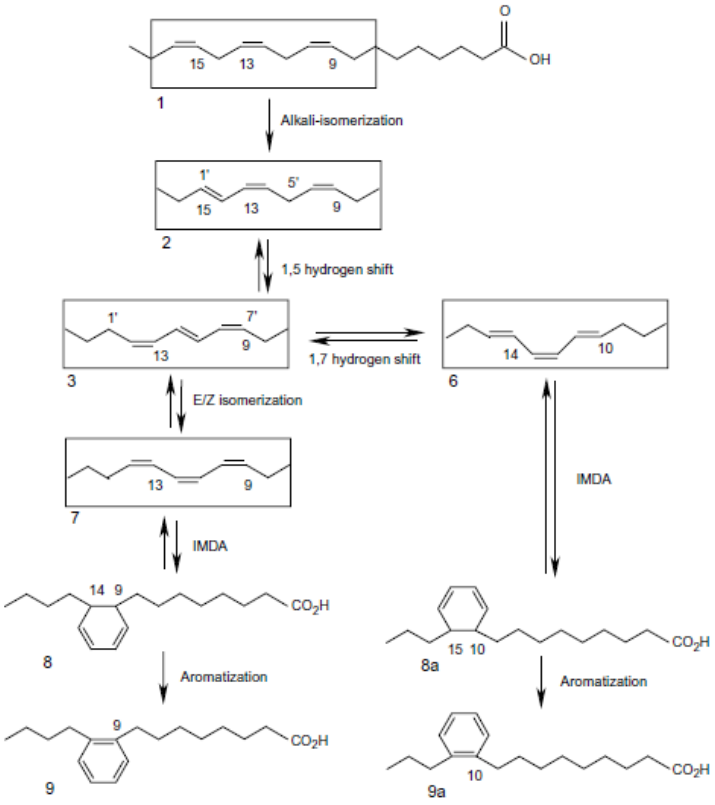


Figure 3.16: Isomerisation of ω-(o-alkylphenyl) alkanoic acids (Hansel et al., 2004).

Further research demonstrated that a homologous series of C_{16}, C_{18}, C_{20} and C_{22} ***ω***-(*o*-alkylphenyl) alkanoic acids (APAAs) could be formed from $C_{16:x}$ - $C_{22:x}$ mono- and polyunsaturated fatty acid precursors when exposed to the catalytic action of ion metals from the ceramic clay under high temperatures (Bondetti et al., 2020; Evershed et al., 2008; Copley et al., 2004; Hansel et al., 2004). C_{16} and C_{18} APAAs result from heating products rich in $C_{16:x}$ and $C_{18:x}$ unsaturated fatty acids, such as vegetable and aquatic oils, inside a ceramic vessel (Hansel et al., 2004). However, the abundance of $C_{20:x}$ and $C_{22:x}$ mono and polyunsaturated fatty acids in aquatic products can lead to the formation of C_{20} and C_{22} APAAs when exposed to high temperatures during cooking events (Admiraal et al., 2020; Shoda et al., 2017; Evershed et al., 2008). According to Evershed et al. (2008), identifying a homologous series of C_{18}, C_{20} and traces of C_{22} in the residue is necessary to confirm the use of pottery to cook aquatic products (Figure 3.17).

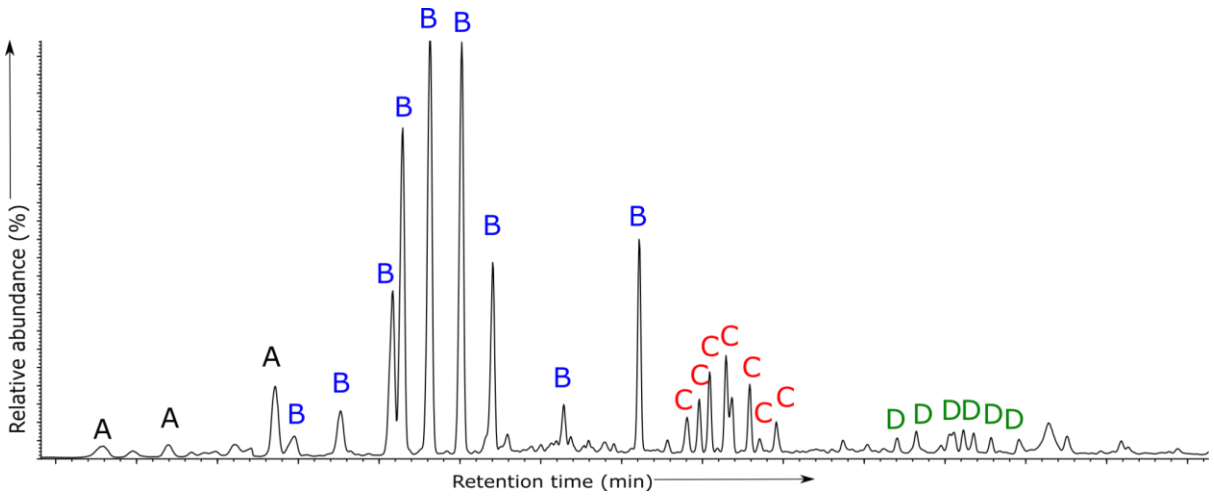


Figure 3.17: Partial chromatogram (m/z 105) of sample P25-1.10 from Mocha Island (This study) exhibiting ω-(o-alkylphenyl) alkanoic acids homologous serie of ***A)*** *C_{16},* ***B)*** *C_{18},* ***C)*** *C_{20},* ***D)*** *C_{22}. Figure created by the author*

Although APAA-C_{20} is more likely to be formed from aquatic resources, the low quantity of $C_{20:x}$ unsaturated fatty acids in various terrestrial animals and plants can also yield APAA-C_{20} (Bondetti et al., 2020). Therefore, an APAA-C_{20}/APAA-C_{18} ratio above 0.06 has been determined to confirm the presence of aquatic products in organic residues from pottery artefacts, while all values below are considered to indicate an origin from terrestrial animals and plants (Bondetti et al., 2020).

Furthermore, isoprenoid fatty acids, comprising phytanic acid (3,7,11,15 tetramethylhexadecanoic acid), pristanic acid (2,6,10,14-tetramethylpentadecanoic acid), and 4,8,12-trimethyltridecanoic acid (4,8,12-TMTD) are diagnostic biomarkers for identifying the exploitation of aquatic resources in archaeological pottery (Figure 3.18) (Courel et al., 2020; Taché and Craig, 2015). These highly branched molecules are likely to be found in aquatic animals (Ackman, Hooper and Ke, 1971; Ackman and Hooper, 1970, 1968), and their structure makes them resistant to degradation (Cramp and Evershed, 2014).

Zamene and phytadene are unsaturated hydrocarbons highly found in zooplankton; if liberated into the ocean, they will likely undergo **β**-oxidation of their double bond, leading to the formation of 4,8,12-TMTD. The latter will likely enter the marien food chain; thus, its identifcifation is highly associated with aquatic organisms (Ackman and Hooper, 1968). Although 4,8,12-TMTD can be synthesise from pristanic acid **β**-oxidation in the rumen of

polygastric animals, this compound is found in a lesser abundant in relation to aquatic organisms (Hansen, 1969).

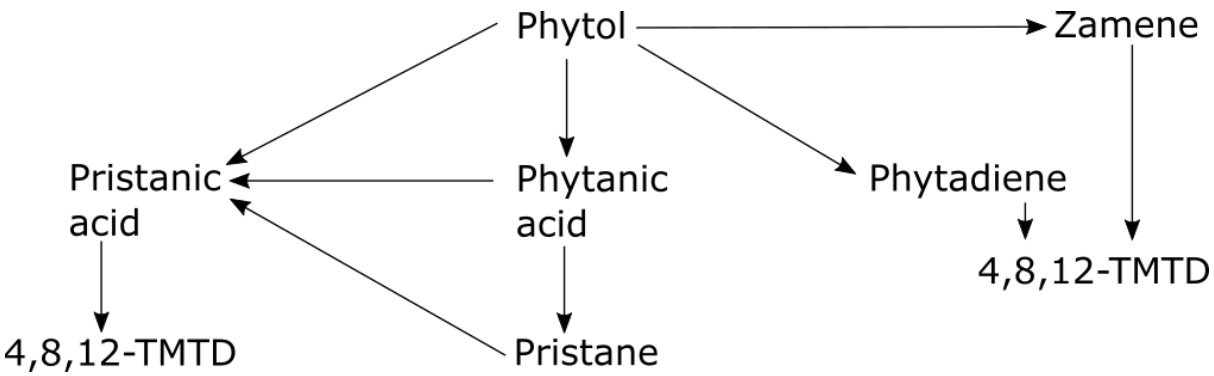


Figure 3.18: Scheme modified from Ackman and Hooper (1968) showing the general synthesis path for the main sioprenoid fatty acids. Figure created by the author.

Phytanic acid is not only found in aquatic animals, as it is also found in ruminant fat (Lough, 1977). Phytol is present in phytoplankton chlorophyll, consumed by zooplankton and then metabolised to phytanic acid, allowing its entrance into the food chain of aquatic animals (Wanders, Komen and Ferdinandusse, 2011). However, phytol is also an important constituent of terrestrial plants chlorophyll; when consumed by ruminants, phytol is metabolised into phytanic acid by bacteria from the rumen (Wanders et al., 2011; Hansen, 1966). The metabolisation of phytol to phytanic acid leads to the formation of two diastereomers, 3S, 7R, 11R-phytanic acid (SRR) and 3R, 7R, 11R-phytanic acid (RRR). The proportions of these two diastereomers vary according to their origin from aquatic animals or ruminants (Ackman and Hansen, 1967). Further studies indicated that a higher abundance of the SRR-phytanic acid over the RRR-phytanic acid diastereomer (i.e., 76% ±16.6) is characteristic of aquatic animals; in contrast, a lower representation of the SRR-phytanic acid diastereomer (<64%) likely indicates an origin from ruminant fat (Dolbunova et al., 2023; Lucquin et al., 2016b). It has also been noted that the employment of C_4 fodder in ruminants can alter SRR-phytanic acid diastereomer contribution towards values resembling those described for an aquatic origin (Kaffarnik et al., 2014; Schröder and Vetter, 2011). This evidence must be considered, especially in archaeological contexts where the possible use of C_4 plants as fodder for local livestock is suggested.

Aquatic resources can be identified through organic residue analysis from archaeological pottery when identifying several biomarkers. For this, Evershed et al. (2008) consider that a full set of C_{16}, C_{18}, C_{20} and C_{22} carbon chain $\boldsymbol{\omega}$-(*o*-alkylphenyl) alkanoic acids and at least one of the primary isoprenoid fatty acids, is required for the identification of aquatic resources in pottery. Several publications have integrated phytanic acid diastereomer contributions and dihydroxy fatty acids to complement the evidence from APAAs and isoprenoid acids and support the identification of aquatic products (Dolbunova et al., 2023; Admiraal et al., 2020; Courel et al., 2020; Cramp et al., 2014). However, past cooking events might have not always reached the minimum temperature needed for APAA formation; moreover, the low abundance of APAAs makes them hard to preserve in the archaeological record (Lucquin et al., 2018, 7933). Therefore, interpreting the presence of aquatic products in organic residues from pottery artefacts is a challenging task that requires complementing several molecular evidence to support further statements.

5.2. Plant resources

The low lipid concentration found in plants can be easily masked when mixed with other resources such as animal products, thus making identifying plant commodities through organic residue analysis a challenging task (Shoda et al., 2018; Reber and Evershed, 2004b). However, several characteristics can help to indicate the presence of plant products in the residues.

A high abundance of $C_{16:0}$ over $C_{18:0}$ fatty acid, with a P/S above 4, is unlikely to be found in terrestrial animals and is likely derived from plant commodities (Dunne et al., 2016; Kimpe et al., 2002). P/S values over 1.3 have also been considered when supportive evidence indicates the presence of plant products in the pots (Dunne, 2021). Notably, a higher abundance of lauric ($C_{12:0}$) and myristic ($C_{14:0}$) acids over $C_{16:0}$ and $C_{18:0}$ fatty acids has been identified in coconut and palm oils (Orsavova et al., 2015; Copley et al., 2001), whereas plant leaves and stem waxes tend to show a distribution of very long chain fatty acids normally absent in plant oils (Dunne et al., 2016; Gurr and Harwood, 1991).

Several authors have used fatty acid distribution and ratios as a central approach to indicate the origin of the residues from various plant and animal products (Eerkens, 2005; Malainey et al., 1999; Patrick et al., 1985). However, fatty acid solubility is directly associated with their specific carbon chain length, leading to differential preservation of the fatty acids in

the residues and possible biases when solely relying on fatty acid ratios (Whelton et al., 2021; Evershed et al., 2002).

Plants are rich in unsaturated fatty acids and are a primary dietary source of linoleic acid and ***α***-linolenic acid for several vertebrates (De Caterina and Basta, 2001). The identification of C_9 ***α***,-dicarboxylic acid (azelaic acid) has been associated with the oxidation of $C_{18:1}$ from plant commodities (Dunne et al., 2016; Regert et al., 1998). Moreover, the identification of hydroxy and dihydroxy fatty acids 12-hydroxy-9-octadecenoic and 9,12-dihydroxyoctadecenoic acid could indicate the presence of castor oil, while 11,12-dihydroxyeicosanoic, 13,14-dihydroxydocosanoic and 15,16-dihydroxytetracosanoic acid has been associated with Brassicaceae seed oil (Colombini et al., 2005; Copley et al., 2005; Regert et al., 1998).

Bondetti et al. (2020) demonstrated that the distribution of the different APAA-C_{18} isomers produced from the thermal degradation of $C_{18:x}$ unsaturated fatty acid could help indicate the origin of the residue from plant or animal products. The authors indicated that the ratio between the E and H APAA-C_{18} isomers (E/H) helped to further differentiate between 1) cereals, fruits and non-leafy plants; 2) terrestrial and aquatic animals; 3) leafy plants (Figure 3.19). Further analysis by Dolbunova et al. (2023) indicated that E/H values above 4 were likely associated with cereal, fruits and non-leafy vegetables.

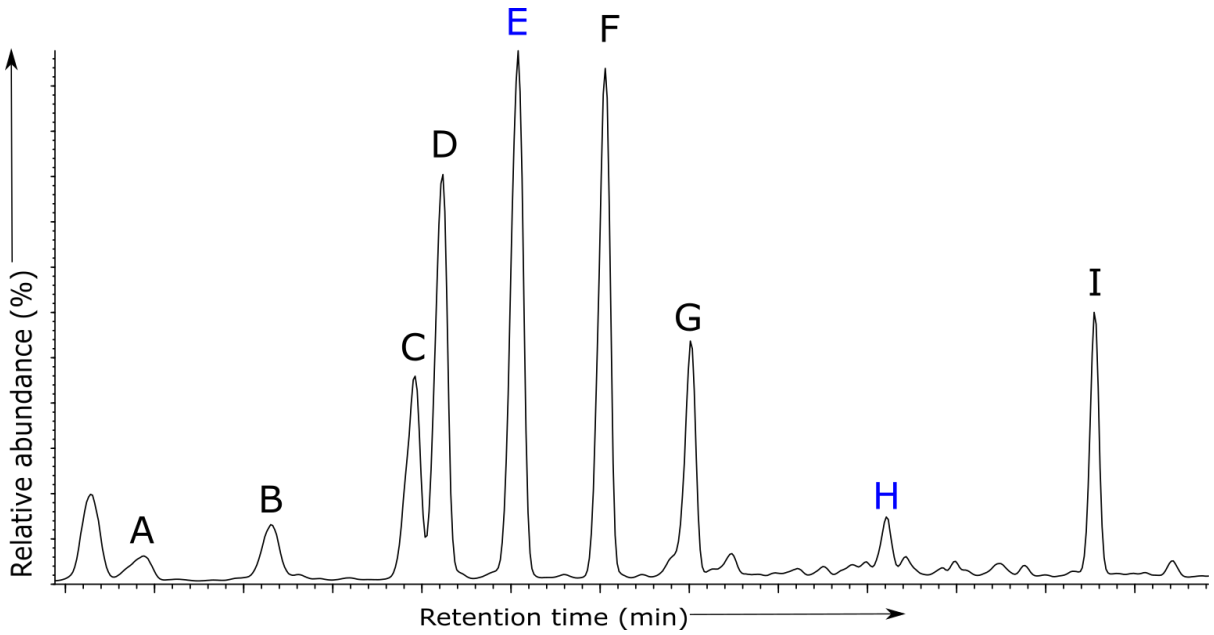


Figure 3.19: Partial chromatogram for APAA C_{18} (m/z 290) from sample P25-1.10 from Mocha Island (this study). The letters (A-I) indicate the APAA C_{18} isomers, from which the ratio between the E and H isomers helps to discriminate the residue's origin. APAA- C_{18} isomers are differentiated by the number of unsaturations and their positions in the carbon chain. Figure created by the author.

The identification of plant sterols, known as phytosterols, in the residues strongly supports the processing or storage of plant commodities in the pots (Evershed et al., 1992). Phytosterols comprise several compounds from which the most common phytosterols are **β**-sitosterol, stigmasterol and campesterol (Schmid, 2016; Scovell, Behrman and Gopalan, 2005).

5.2.1. Plant wax

The epicuticular wax of higher plants, which serves as protection against desiccation, ultraviolet light and pathogens, is composed of a mix of different hydrophobic compounds (Schmid, 2016; Lee and Suh, 2013), likely to be identified through organic residue analysis from archaeological pottery (Evershed, Heron and Goad, 1991). This mixture consists of a predominance of odd over even very- long chain *n*-alkanes (C_{21}-C_{37}), ketones, aldehydes, wax esters, and a predominance of even over odd long-chain alcohols (C_{20}-C_{34}), and very-long chain fatty acids (C_{20}-C_{34}) (Schmid, 2016; Lee and Suh, 2013; Dove and Mayes, 2006).

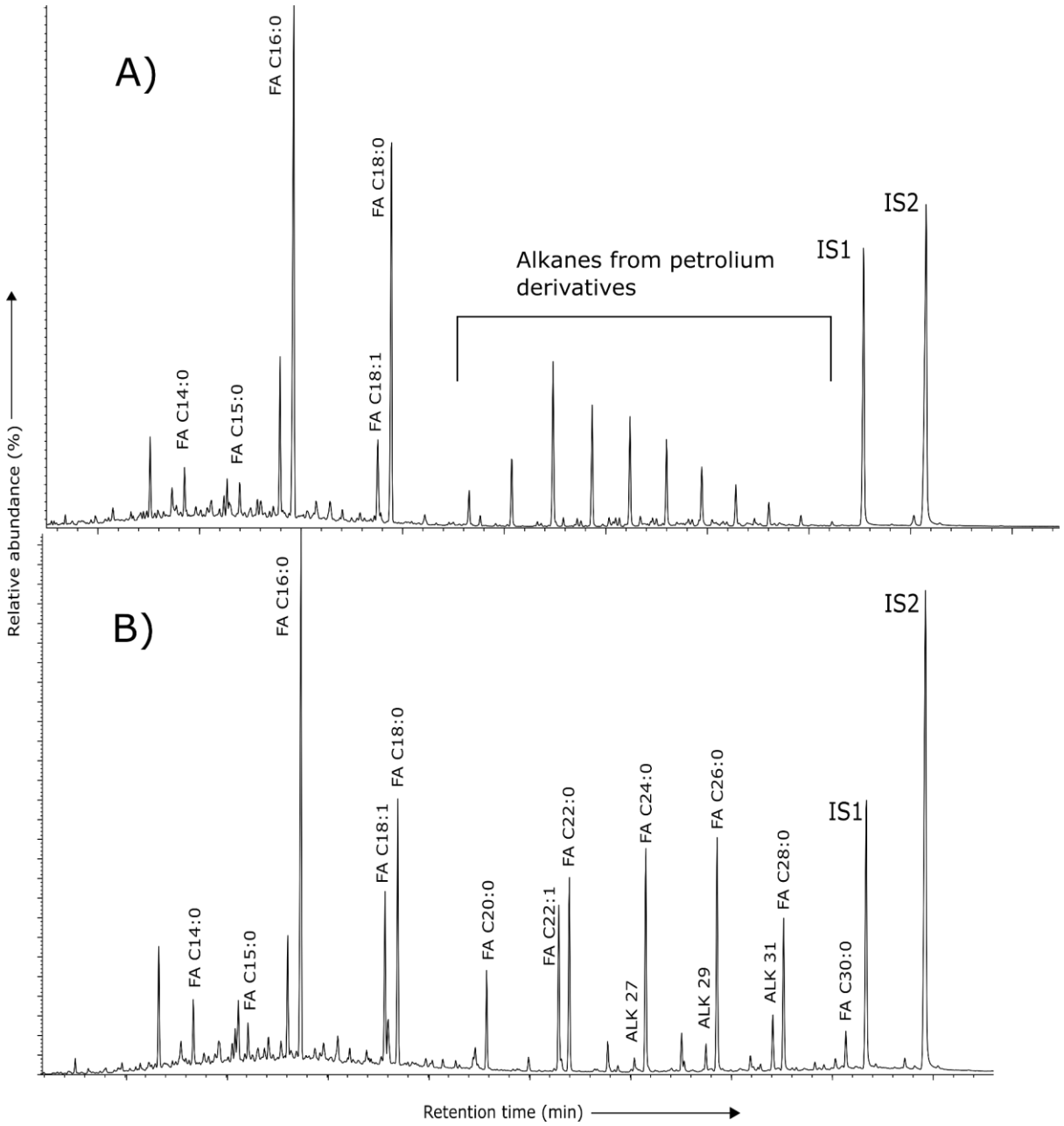


Figure 3.20: A) Chromatogram of sample C.C.ECP-49 from Catalanes Cave, southern Chile, exhibiting a characteristic n-alkane distribution from petroleum derivatives. B) Chromatogram showing a characteristic long-chain alkane and fatty acid distribution characteristic of plantwaxes. Sample C.C.LCP-22 from Catalanes Cave, southern Chile. Figure created by the author.

The use of petroleum derivatives in museum artefacts can alter *n*-alkane distributions found in the residues and their use as a plant biomarker (Whelton et al., 2021). The distribution of *n*-alkanes in petroleum derivatives exhibits a characteristic distribution consisting on the abundance of even carbon chain *n*-alkanes (Figure 3.20). To differentiate an *n*-alkane origin between petroleum and plant organisms the employment of the carbon preference index (CPI) has been useful. Diefendorf et al. (2011) presented the following equation that considers both, the abundance of odd and even carbon chain n-alkanes to assess their distribution:

$$CPI = 0.5\left[\frac{\Sigma_{odd}(C_{25} - C_{33})}{\Sigma_{even}(C_{26} - C_{34})}\right] + \left[\frac{\Sigma_{odd}(C_{25} - C_{33})}{\Sigma_{even}(C_{24} - C_{32})}\right]$$

Further assessments on aliphatic compound distributions have shown the usefulness of indicating the average carbon length (ACL) index of *n*-alkanes, long chain fatty acis and fatty alcohols to evaluate possible vegetational composition changes in geochemistry (Cerda-Peña and Contreras, 2022; Bush and McInerney, 2013). For this, the equation has been described as:

$$ACL = \frac{\Sigma(C_n\ x\ n)}{\Sigma C_n}$$

Where *n* indicates the carbon chain length (fatty acids= C_{24}-C_{34}; *n*-alkanes= C_{25}-C_{35}; fatty alcohols C_{24}-C_{30}) and C_n accounts for the abundance of the specific alkane. An ACL index indicating the predominance of C_{31} and C_{33} n-alkanes has been associated to the presence of C_4 grasses, while shorter ACL values are linked to C_3 plants (Rommerskirchen et al., 2006).

The incorporation of mid-chain *n*-alkanes to identify plant origin has helped to differentiate between emerged and terrestrial plants from submerged and floating-leaved plants based on a specific equation described by Ficken et al. (2000):

$$P_{aq} = \frac{(C_{23} + C_{25})}{(C_{23} + C_{25} + C_{29} + C_{31})}$$

Values <1 are concordant with an origin from terrestrial and emerged plants, while a P_{aq} value between 0.1 and 0.4 corresponds to emerging macrophytes, while values between 0.4 and 1 are likely from a submerged plant origin (Ficken et al., 2000). The above-mentioned indexes have proved to be useful in organic residue analysis of archaeological pottery, indicating possible origins for plant waxes (Dunne et al., 2016; Dunne, 2021).

5.2.2 Resine, wood tars and pitches

Plants, mainly woody plants, produce resin, a secretion rich in terpenoids and phenolic compounds, primarily employed in their protection against microorganisms and herbivores (Gershenzon and Dudareva, 2007; Langenheim, 2003; Phillips and Croteau, 1999). Its preservation through time has been represented by its fossilised form as amber (Langenheim, 1990). However, several organic products have been historically produced from resins which, due to Its chemical characteristics, are likely to be preserved and identified in the archaeological record (Regert et al., 2008; Colombini et al., 2005; Stern et al., 2003).

Tars are prepared by the destructive distillation of woody plant resins and wood, while the protracted boiling of woody tars will lead to the formation of pitches (Regert et al., 2003a; Robinson et al., 1987). Their exposure to high temperatures will result in dehydrogenation, decarboxylation, aromatization and demethylation, of main terpenoids found in several resins and resinous materials, forming new compounds likely to be used as biomarkers (Colombini et al., 2005; Colombini et al., 2003). For example, abietic acid is the main terpene found in conifer resins, from which further oxidation will form dehydroabietic acid and 7-oxo-dehydroabietic acid, likely also found in conifer resins and wood (Colombini et al., 2003; Regert and Rolando, 2002; Robinson et al., 1987). Continuous exposure to high temperatures during the production of tar and pitches will result in the formation of retene, norabietatriene, tetrahydroretene and methyl-dehydroabietic acid (Colombini et al., 2005; Colombini et al., 2003). The identification of these compounds in archaeological pottery has been related to the use of tars and pitches as adhesive agents or in the waterproofing of pottery containers (Correa-Ascencio et al., 2014; Colombini et al., 2005; Regert et al., 2003a).

5.2.3. Starchy plants

Starchy plants, such as tubers and grains, hold great nutritional and economic importance worldwide as high productive yields and strong environmental resilience allow human access to a generally stable and constant food supply stock (Jones et al., 2011). Their identification in the archaeological record through starch granules collection has allowed researchers to assess plant management, domestication and population contacts from different contexts (Ciofalo, Sinelli and Hofman, 2020; Bonomo et al., 2011; Piperno et al., 2009, 2004). However, starchy granules can sometimes be lost in the burial environment; moreover, discussions have indicated the likeness of post-depositional degradation factors to

be mistaken as evidence for anthropogenic practices such as cooking (Collins and Copeland, 2011; Barton and Torrence, 2015; Mercader et al., 2017).

Starchy plants are represented by a very low lipid content compared with a high percentage of carbohydrates, the latter likely to be lost in the archaeological record due to their water solubility (Colonese et al., 2017a). This low lipid content leaves almost no trace in the archaeological record, which makes starchy plant identification quite challenging through organic residue analysis (Shoda et al., 2018). However, several attempts have been made to identify starchy plants from archaeological pottery.

Heron et al. (2016) demonstrated that after undergoing experimental cooking events using millet, specifically broomcorn millet (*Panicum miliaceum*), a pentacyclic triterpene methyl ester known as miliacin, was absorbed in low quantities into the matrix of replica vessels. This biomarker was absent in finger millet (*Eleusine coracana*), barnyard millet (*Echinochloa esculenta*) and foxtail millet (*Setaria italica*), which were also tested in this experiment. Posteriorly, its identification in archaeological pottery and food crust from Poland and the Korean peninsula demonstrated the possibility for milician to survive and be identified in the archaeological record.

Although not as straightforward as millet, the identification of wheat and rye has been achieved through the use of GC-MS to identify 3,5-dihydroxy phenolic compounds known as alkylresorcinols. These compounds consist of five different homologues with an odd-numbered, mainly saturated, alkyl chain ranging from $C_{15:0}$ to $C_{25:0}$, present in considerable quantities in the bran fraction of wheat, rye, barley and triticale cereals (Ross, 2012). The ratio between $C_{17:0}$ and $C_{21:0}$ homologues has allowed to differentiate between whole grain wheat and rye in cereal products, as $C_{17:0}/C_{21:0}$ in wheat would result on a value of 0.1, whereas in rye the ratio of both homologous would be of 1.0 (Chen et al., 2004).

This methodology was applied by Colonese et al. (2017a) on a residual substance attached to the central surface of an Early Bronze Age wooden container found at the summit of the Lötschenpass in Switzerland. The identification of plant lipids through acid extraction (AE) such as phytosterols and their degradation products, and the lack of animal biomarkers would suggest that the residue originated from plant commodities. This was also supported by the archaeobotanical evidence that showed the presence of wheat and rye remains.

The results exhibited the presence of alkylresorcinols from $C_{17:0}$ to $C_{25:0}$, with a ratio between C_{17} and $C_{21:0}$ of 0.02, closer to the value for wheat. At the same time the

predominance of $C_{19:0}$ and $C_{21:0}$, the two dominant homologues for wheat (Chen et al., 2004) would underpin the idea of a higher representation of wheat in the container. These results were complemented by proteomic analyses, supporting the idea of the presence and predominance of wheat in the wooden container.

While the analysis of alkylresorcinols through GC-MS has shown to be possible, more sensible analytical methods have allowed for better discrimination of the compounds identified from various sources (Ross, 2012; Ross et al., 2003). Further analyses on organic residues from pottery artefacts demonstrated that gas chromatography quadrupole time-of-flight mass spectrometry (GC-Q-ToF-MS) proved to be a more sensitive method in the identification of alkylresorcinols (Hammann et al., 2022; Hammann and Cramp, 2018).

Underground storage plant organs such as roots, tubers, and rhizomes have had an important economic and dietary significance worldwide. Their identification through organic residue analysis has focused on the distribution of long chain $\boldsymbol{\alpha}$,-dicarboxylic acids above C_{20} carbon chain as characteristic for suberin, a polymer found in most underground storage plant organs (Dunne et al., 2022; Pollard et al., 2008). Identifying these compounds has allowed interpretations on the use of archaeological pottery to process underground storage plant organs in western Africa (Dunne et al., 2022). However, the possibility of finding long chain $\boldsymbol{\alpha}$,-dicarboxylic acids in the soil from root degradation (Mendez-Millan et al., 2010) must be considered when interpreting them as evidence for past culinary practices. The distribution of long chain $\boldsymbol{\alpha}$,-dicarboxylic acids does not allow specific taxonomic identification (e.g. differentiate between potato and wild tubers), indicating that further studies are needed to improve this approach.

5.3. Bacterial lipids and fermentation

Most references on bacterial lipids are associated with their potential identification from contamination and how this can undermine further interpretations (Heron et al., 1991; Dudd et al., 1998). On the other hand, the identification of hopanes as bacterial hopanoid degradation products has been used to indicate the employment of pottery in the preparation of alcoholic beverages (Rageot et al., 2019; Correa-Ascencio et al., 2014). Hopanoids act as rigidifers of the plasma membrane of several bacteria, including the ethanol-producer *Zymomonas mobilis* (Blumenberg et al., 2006; Sahm and Bringer-Meyer, 1987). This gram-negative bacteria has been identified as one of the microorganisms involved in the fermentation of maguey sap to

produce pulque beverage, traditionally consumed in Mexico (Romero-Luna, Hernández-Sánchez and Dávila-Ortiz, 2017). While hopanoids can be found in bacteria, they do not comprise all bacteria involved in fermentation which limits their use as a potential indicator for the use of pottery in fermentation (Bassi et al., 2020; Kuchta, Kubinec and Farkas, 1998). Moreover, using bacterial biomarkers as evidence for fermentation can lead to biases due to the involvement of various microorganisms in postdepositional organic diagenesis (Craig, 2021).

6. Lipid $\delta^{13}C$ analysis in archaeological pottery

The integration of stable isotope analysis on organic residues has significantly enhanced the identification of archaeological pottery's original content, underscoring its crucial role in unravelling past culinary practices. Summarily, isotopes are different versions of a specific chemical element, with the same number of protons and electrons but differing in the number of neutrons. The change in neutron number does not alter the element's reactivity; however, the different isotope masses will likely impact several reactions, leading to isotopic separation or fractionation in the resultant product (Peterson and Fry, 1987). Fractionation is represented by the ratio between the heaviest and lightest isotope of a given element (e.g. $^{13}C/^{12}C$ for carbon) (van der Merwe, 1982); however, to measure the isotopic signal of a specific sample, this ratio needs to be compared to a reference standard of known isotopic value (Slater, Preston and Weaver, 2001), as shown in the following equation:

$$\delta X = \left[\frac{R_{sample}}{R_{standard}} - 1\right] x1000\ (‰)$$

Where X indicates the heaviest isotope (e.g. ^{13}C for carbon), R_x indicates the ratio between the heaviest and the lightest isotopes in the sample and standard. In the case of carbon, the isotopic ratio of a specific sample is compared to the Vienna Pee Dee Belemnite (VPDB) international standard, which is based on a designated carbonate of known isotopic signal (Coplen, 1995; Schipilliti et al., 2013). The standard's $\delta^{13}C$ is considered to be the zero point (Hélie et al., 2021); thus, $\delta^{13}C$ values closer to zero will indicate an enrichment on the heaviest isotope, while $\delta^{13}C$ with more negative values will indicate an enrichment of the lightest isotope (or depletion of the heaviest isotope) (O'Leary, 1988). Values are always expressed in parts per thousand or *per mill* (‰) due to the slight deviation of the isotopic ratio of the sample compared to the international standard (van der Merwe, 1982).

The use of stable isotope analysis on organic residue has mainly been approached through single-compound $\delta^{13}C$ analysis of the main alkanoic acids $C_{16:0}$ and $C_{18:0}$ due to their high abundance in living organisms. For this purpose, gas chromatography - combustion - isotope ratio mass spectrometry (GC-c-IRMS) has been traditionally employed in archaeology (Dudd and Evershed, 1998b; Evershed et al., 1997b, 1994). This method measures the abundance of carbon isotopes first by separating the compounds through a GC for their further oxidation and conversion into gas CO_2 through a combustion furnace (Zhang et al., 2012; Evershed et al., 1994). Water is removed from the resultant combustion gases as it can form HCO_2^+ which can distort isotopic fractionation (Evershed et al., 1994). After this step, ionised CO_2 is collected in a mass spectrometer for $^{13}C/^{12}C$ measurements for further comparison to the VPDB standard to ensure instrument's accuracy (Schipilliti et al., 2013).

$\delta^{13}C$ isotopic fractionation is different between several plant species, terrestrial and marine animals. These variations can be used in archaeology for evaluating the use of resources by past human societies. By analysing the $\delta^{13}C$ signal of preserved lipids from archaeological pottery, it is possible to discriminate the use of these commodities in archaeological containers (Figure 3.21).

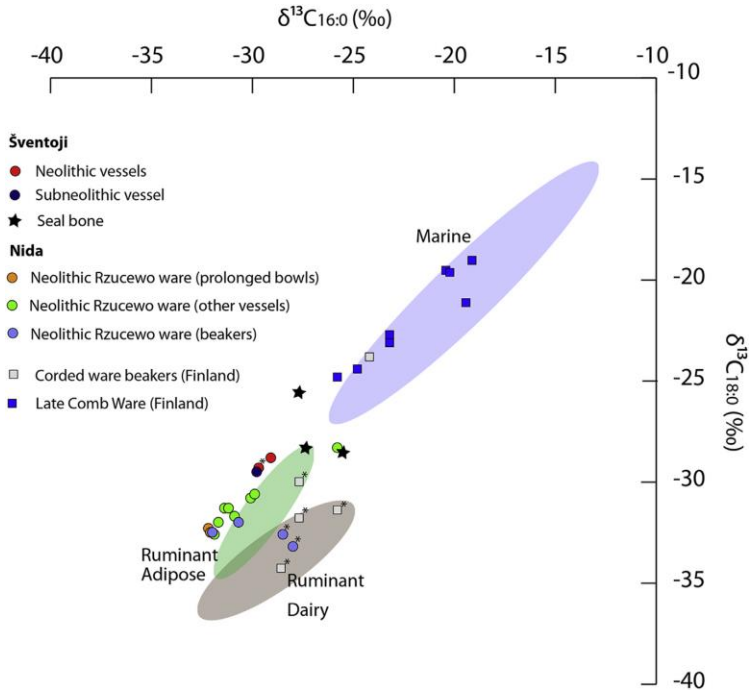


Figure 3.21: Characteristic plot for single compound $\delta^{13}C$ analysis of the main alkanoic acids $C_{16:0}$ and $C_{18:0}$. The example used $\delta^{13}C$ values from modern references to construct 95% confidence interval ellipses. Example from (Heron et al., 2015).

The following section presents a general characterisation of $\delta^{13}C$ fractionation in terrestrial and aquatic organisms.

6.1. Terrestrial resources

6.1.1 Plants

In a terrestrial environment, carbon is fixed into plants from atmospheric CO_2 during photosynthesis (Tieszen, 1991). Plants based on the Calvin cycle incorporate a higher amount of the lighter ^{12}C isotope, resulting in relatively more negative $\delta^{13}C$ values compared to plants following the Hatch-Stack cycle, where plants exhibit a higher preference for the heavier isotope ^{13}C, showing more positive $\delta^{13}C$ values (Spangenberg and Ogrinc, 2001). Since the initial metabolite during CO_2 fixations in plants following the Calvin cycle is 3-phosphoglycerate, a compound of three carbon atoms, they are called C_3. In contrast, plants following the Hatch-Stack cycle have a four-carbon atom compound, oxaloacetate, as their

initial product, therefore, are known as C_4 (Spangenberg, Macko and Hunziker, 1998). Plants such as succulents and several desert plants can alternate between both photosynthetic pathways throughout the day, which has been known as the crassulacean acid metabolic pathway or CAM (O'Leary, 1981, 1988). While CAM plants can exhibit a broad distribution of $\delta^{13}C$ values, several $\delta^{13}C$ measurements on C_3 and C_4 plants worldwide have indicated that both groups show little overlap (Figure 3.22), thus allowing their differentiation through carbon stable isotope analysis (O'Leary, 1988).

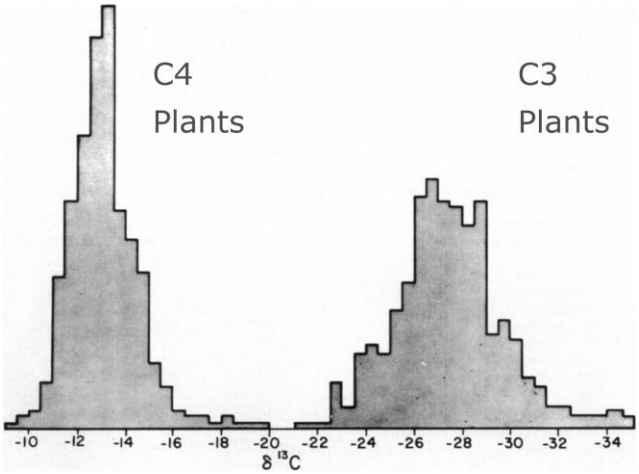


Figure 3.22: Plant $\delta^{13}C$ measurements from around 1000 plant species showing a bimodal distribution according to a C_4 or C_3 photosynthetic pathway. Obtained and modified from O'Leary (1988).

This difference regarding carbon fixation pathways has been used in archaeology to identify the processing of plant commodities in archaeological pottery. In their pioneer research, Evershed et al. (1994) described the possibility of analysing the $\delta^{13}C$ signal of preserved lipids, in this case, long-chain alkanes and ketones, from early medieval pottery to identify the exploitation of plant resources. Based on soil samples, modern wyld-type *Brassica oleracea* lipid extracts, and isotopic references, the authors suggested that C_3 plants, possibly from the genus *Brassica*, were processed in the pots.

Different C_4 plants can be identified through $\delta^{13}C$ analysis, where C_3 species dominate local carbon isotopic ecology. Heron et al. (2016) complemented the identification of millet biomarker miliacin with the $\delta^{13}C$ analysis of the main alkanoic acids $C_{16:0}$ and $C_{18:0}$. The authors

supported the identification of millet, a C_4 grass, by incorporating carbon isotope data from the residues extracted from archaeological pottery from the Korean Peninsula and Poland.

6.1.2. Terrestrial animals

Carbon isotope fractionation in plants is transferred through the food chain (Tieszen, 1991; Tieszen et al., 1983). Hervibours' $\delta^{13}C$ values will reflect the isotopic signal of the plants they incorporate into their diet and the resultant fractionation from particular metabolic pathways (DeNiro and Epstein, 1978). Carbon stable isotope fractionation differences are evident between ruminant and non-ruminant adipose tissue (Figure 3.23a), even when these are fed under similar conditions (Evershed et al., 1997c).

Ruminants synthesise $C_{16:0}$ from *de novo* by incorporating carbon from dietary carbohydrates, whereas $C_{18:0}$ will partially routed from the hydrogenation of $C_{18:x}$ unsaturated fatty acids obtained from their diet (Craig et al., 2012; Copley et al., 2003). Since carbohydrates have a more positive $\delta^{13}C$ value than lipids, $C_{16:0}$ will exhibit a higher ^{13}C enrichment than $C_{18:0}$. According to Copley et al. (2003), this difference will produce a negative $\Delta^{13}C_{18:0\text{-}C16:0}$ offset ($\Delta^{13}C$), where values below -1‰ are likely to be from ruminant adipose or carcass fat (Figure 3.23b). Moreover, since $C_{18:0}$ cannot be synthesis *de novo* in the mamarian gland, it will totally be sourced from the hydrogenation of $C_{18:x}$ unsaturated fatty acids in the rumen. Therefore, dairy producst will likely produce an even more negative $\Delta^{13}C$ value; according to the authors, an offset below -3.3‰ from the residues will indicate an origin from dairy products. On the other hand, monogastric animals exhibit a $\Delta^{13}C$ value closer to zero (Mottram et al., 1999; Evershed et al., 1997c).

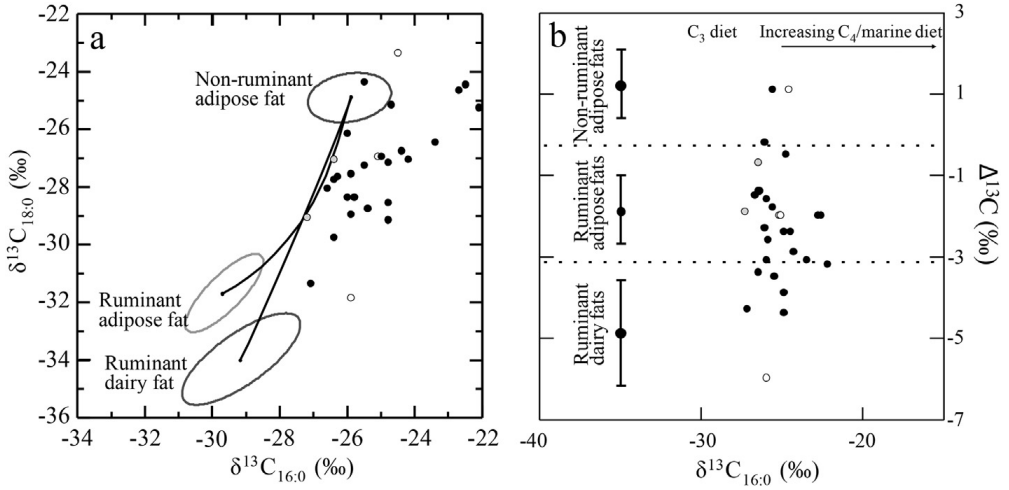


Figure 3.23: The example from (Nieuwenhuyse et al., 2015) shows the ***a)*** *$\delta^{13}C$ values for the main alkanoic acids $C_{16:0}$ and $C_{18:0}$ against modern references for several animal products. The use of the $\Delta^{13}C_{C18:0-C16:0}$ proxy allowed to identify in* ***b)*** *the use of pottery for the processing of dairy products during the Late Neolithic Period in Syria, and the possible use of C_4 plants as animal fodder.*

According to Craig et al. (2012) $\Delta^{13}C$ offsets in wild ruminants' carcass fat, mainly from deer (*Cervus elaphus*), shows values between -2.7‰ and -4.3‰, which considerably overlap with dairy and carcass fat $\Delta^{13}C$ from domestic ruminants. Therefore, the authors indicated the need to consider a threshold from -4.3 ‰ to ensure the identification of dairy products from organic residues.

6.2. Aquatic resources

Marine bicarbonate has a 7‰ ^{13}C enrichment compared to air CO_2, accounting for a ^{13}C enrichment in marine organisms, as carbon from dissolved bicarbonate is fixed by phytoplankton, thus entering the marine food chain (Chisholm, Nelson and Schwarcz, 1982). This isotopic difference has helped to indicate the exploitation of marine resources in several archaeological contexts (Shoda et al., 2017; Lucquin et al., 2016a; Craig et al., 2013). However, the identification of marine products through carbon stable isotopes could be confused by the presence of ^{13}C enriched terrestrial organisms such as C_4 plants (Santana-Sagredo et al., 2015), and animals fed with a diet based on C_4 fodder (Szpak et al., 2013).

As for freshwater organisms, the isotopic variability present in freshwater environments makes specific references necessary (Dufour, Bocherens and Mariotti, 1999). Works on pottery organic residues have shown that depletion of ^{13}C is frequent in freshwater fish when compared to marine organisms (Lucquin et al., 2016a; Craig et al., 2007); however this trend can be variable and in certain cases can overlap with terrestrial species (Dufour, Bocherens and Mariotti, 1999).

Courel et al. (2020) analysed hunter-gatherer foodways from different areas of the Baltic Region. Eastern Baltic hunter-gatherers practised the exploitation of aquatic resources; however, relatively ^{13}C depleted residues relative to marine references indicated an origin more likely from freshwater species; the evidence aligns with the identification of aquatic biomarkers in pottery from sites located away from the sea coast.

6.3. Mixing commodities

Single-compound $\delta^{13}C$ analysis has allowed researchers to identify the origin of the commodities processed or stored in archaeological pottery. However, the potential use of ceramic vessels for mixing different types of commodities might influence the overall measured $\delta^{13}C$ signal, undermining the possibility of identifying the residue's origin. Several publications have shown that the mixing of different commodities can result in $\delta^{13}C$ values resembling that of other commodities. For example, Hendey et al. (2018) noticed that the increasing contribution of C_3 plant lipids in pots used to process dairy products will deviate dairy $\Delta^{13}C_{18:0\text{-}C16:0}$ values towards a more positive $\Delta^{13}C$ signal. Thus, the mixing of dairy products with C_3 plants would likely result in a $\Delta^{13}C$ value that can easily be mistaken with ruminant adipose fat.

In a posterior publication by Cramp et al. (2019) the authors indicated that a negative $\Delta^{13}C$ offset in samples where aquatic biomarkers were identified was probably the result of mixing aquatic products with ruminant adipose or dairy fat. Due to the high abundance of $C_{18:0}$ in ruminants compared to aquatic organisms, the former will likely contribute more to the residue's measured $\delta^{13}C$ signature (Cramp et al., 2019). Simultaneously, the input of ^{13}C enriched $C_{16:0}$ from marine products to the residue will result in a negative $\Delta^{13}C$ value, resembling the carbon isotopic signature of ruminant fat. This possibility was confirmed by employing a simple linear model using average $\delta^{13}C$ values of modern references connected by hypothetical mixing lines (Figure 3.24). The resultant $\Delta^{13}C$ values reflected the contribution

of two commodities to the measured $\delta^{13}C$ signature based on the proportion of the $C_{16:0}$ and $C_{18:0}$ alkanoic acids from each product. The model indicates that the mixing of marine or freshwater products with ruminant adipose fat or dairy will likely generate a negative $\Delta^{13}C$ value, even when less than 50% of ruminant fat is included in the mixing.

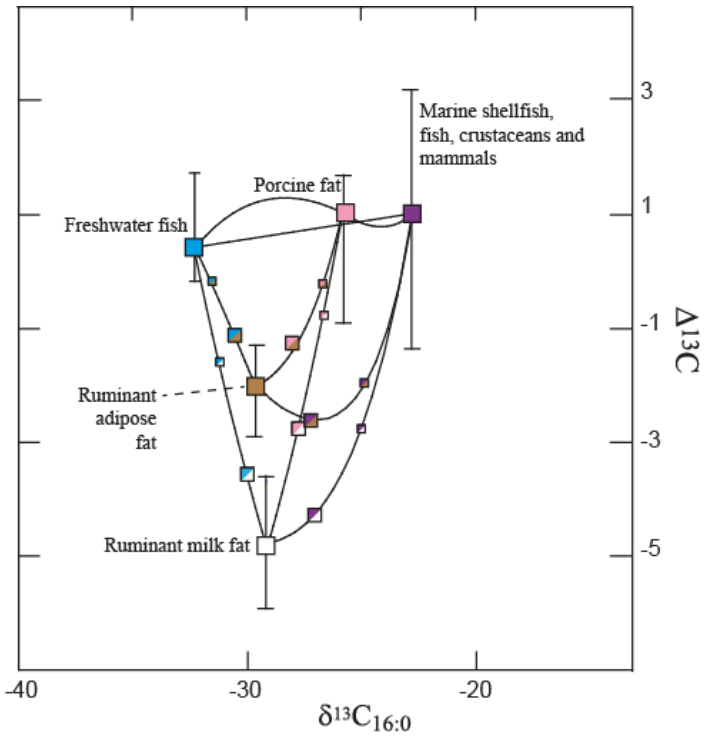


Figure 3.24: Simple linear mixing model from Cramp et al. (2019) (Figure S2 from the supplementary data), showing several averaged $\delta^{13}C$ end-points for various commodities connected by hypothetical mixing lines.

As for aquatic products, millet has a $\Delta^{13}C$ value closer to zero (Taché et al., 2021). Therefore, the identification of milliacin, a millet biomarker especially for broomcorn millet (*Panicum miliaceum*), in several samples denoting a negative $\Delta^{13}C$ value concordant with a ruminant adipose and dairy product origin (Figure 3.25a) brought the need to employ this model to assess the possible mixing of C_4 plants with ruminant fats (Taché et al., 2021). The results demonstrated that large negative $\Delta^{13}C$ values of samples showing evidence for the use of millet can be reached when mixing it with ruminant dairy and adipose from specimens fed on C_3 and C_4 plantsFigure (Figure 3.25b) (Taché et al., 2021).

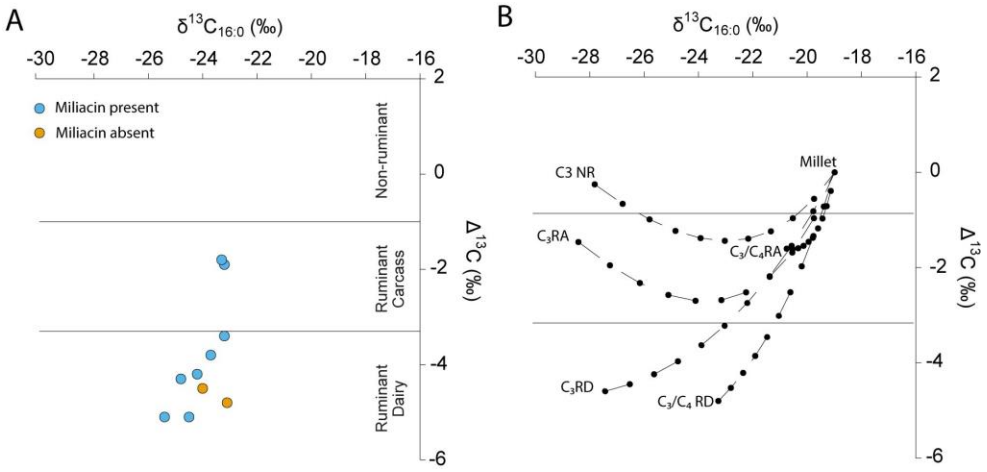


Figure 3.25: A) Scatter plot of samples showing the absence and presence of milliacin biomarkers. B) Simple linear mixing model using millet and several ruminant adipose and dairy fat references based on specimens fed on C_3 and C_4 plants (Taché et al., 2021).

A similar work by Admiraal et al. (2023) evaluated how $\Delta^{13}C$ values responded when mixing maize with several C_3 products, including C_3 plants and several wild and domesticated ruminants fed on C_3 plants. The authors identified considerably negative $\Delta^{13}C$ values resembling those describing the processing of dairy products in the pots (Figure 3.26). Since dairy was not practised in South America during pre-colonial times, the authors evaluated the possibility of mixing maize with ruminant fat, considering the low abundance of $C_{18:0}$ in plant commodities.

A simple linear mixing model provides practical guidance for understanding the $\delta^{13}C$ signature when two different commodities are mixed in the pottery (Fernandes et al., 2018). However, this model is limited when more than two commodities are considered. For this purpose, a Bayesian mixing model has been used to assess the fat contribution from various products to the residue (Fernandes et al., 2018). Probabilistic inferences are based on the $\delta^{13}C$ signal of the main alkanoic acids from several local products likely used in the archaeological pottery; thus, access to a solid reference of products is considerably important (Fernandes et al., 2018). The model considered different uncertainties, mainly based on carbon isotopic measurements and fatty acid concentration in each of the products included in the model. However, the carbon isotope proximity between several commodities has shown to be problematic for building the model (Fernandes et al., 2018).

While the original model was built based on the carbon isotope measurement of the main alkanoic acids, several publications have considered incorporating a third proxy to support probabilistic interpretation of the data. Lucquin et al. (2018) included the $C_{16:0}$ and $C_{18:0}$ fatty acids and the phytanic acid-SRR diastereomer values to evaluate the contribution of aquatic resources to the content of archaeological pottery from Japan. Moreover, Admiraal et al. (2023), in an attempt to better characterise the contribution of maize in pre-colonial pottery, the authors applied a Bayesian mixing model by including the carbon isotopic signal of $C_{18:1}$, which is highly abundant in maize and in other plant species. Only a few samples had a minimal abundance of $C_{18:1}$ necessary for injection and did not show a co-elution of $C_{18:1}$ with other compounds. The results supported a high contribution of maize lipids to the residue, compared to the input of C_3 plants and ruminant fat.

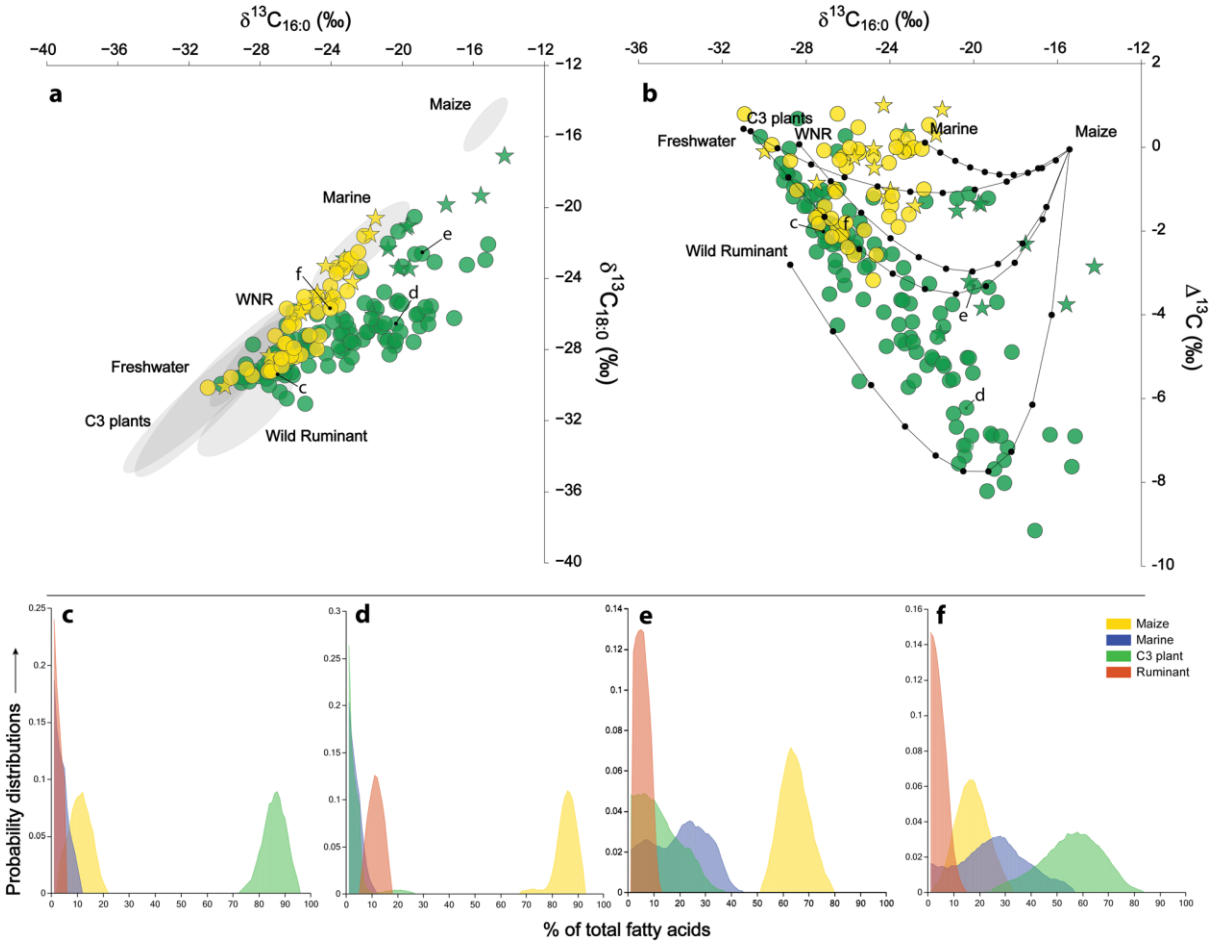


Figure 3.26: ***a)*** *and* ***b)*** *show single compound δ^{13}C of the main alkanoic fatty acids $C_{16:0}$ and $C_{18:0}$ extracted from Guaraní and Itararé pottery, showing a progressive use of maize in the ceramic containers.* ***c-f)*** *shows four examples of mixing models where $C_{16:0}$, $C_{18:0}$ and $C_{18:1}$ were used as variables for generating a Bayesian distribution to assess the mixing of maize with several local products. Image from Admiraal et al. (2023).*

Mixing models to evaluate the original content of archaeological pottery better is a nuanced approach. The need for a solid reference baseline normally limits its potential use in several contexts. More work is needed to improve these approaches in the future.

7. Organic residue analysis in South America

Since the early 2000s, the number of publications on organic residue analysis of archaeological pottery has increased considerably. Its employment in archaeological research

has extended worldwide, reaching contexts in Europe (Cramp et al., 2019; Craig et al., 2011), North Africa (Dunne et al. 2016, 2012), North America (Admiraal et al., 2020; Taché and Craig, 2015), and Asia (Lucquin et al. 2018, Shoda et al. 2017). Several researchers have applied ORA in local contexts in South America, with significant potential for its further development and application. However, its employment is still in its early stages.

In two previously mentioned pioneering investigations led by Fabricio Hansel (Hansel and Evershed, 2009: Hansel et al., 2004;), organic residues were analysed to study pre-colonial pottery from the Itararé culture, Santa Catarina, Brazil. However, more than addressing specific archaeological questions, Hansel and collaborators focused on discussing methodological aspects towards the identification of marine products through organic residues from pottery.

However, several years later Colonese et al. (2014) analysed pottery from the same area, focusing on population diet and the exploitation of aquatic resources by pre-colonial societies. The authors undertook organic residue analysis on one of the 4 studied sites, Galheta IV (G-IV), from which they were able to identify aquatic biomarkers and undertake single-compound $\delta^{13}C$ analysis of the main alkanoic acids, concluding that early pottery was employed in the processing of marine resources.

Continuously, in the Province of Santa Catarina, Admiraal and collaborators (2023) applied GC-MS and GC-c-IRMS to residues extracted from 224 pottery sherds to assess how the Guaraní expansion to southern Brazil influenced the local cuisine of coastal groups. The authors applied the first organic residue analysis on Guaraní pottery, indicating an intensification of maize agriculture in the region, which influenced coastal foodways towards a higher reliance on food production.

Pioneer investigations have also been conducted by different researchers from Argentina, focusing on two main areas: The Argentinian Patagonia and northwest Argentina. In the area of Neuquén in north Patagonia, the distribution of fatty acids and *n*-alkanes has been used to identify the exploitation of plant resources in pre-colonial hunter-gatherer pottery (Cordero and March, 2013). However, most research in the Argentinian Patagonia has been based on using fatty acids as fingerprints for identifying the residue's origin. This approach has led to the conclusion of the specialised use of pottery by hunter-gatherers to mainly cook animal fat (Chaile et al., 2018; Stoessel, Martínez and Constenla, 2015). The identification of complex polyunsaturated fatty acids in coastal sites from the Argentinian Patagonia has also indicated the employment of pottery technology to cook marine products (Stoessel, Martínez

and Constenla, 2015; Gómez Otero, Constenla and Schuster, 2014). However, polyunsaturated fatty acids do not survive in the archaeological record due to intense exposure to oxidation; furthermore, the sole use of fatty acids as fingerprints to track back the residue's origin has proved to be limited, needing to be supported by more robust molecular evidence, and other methodologies such as single-compound $\delta^{13}C$ analysis (Whelton et al., 2021).

Most $\delta^{13}C$ analysis on residues extracted from pottery has been approached through bulk $\delta^{13}C$ from attached food crusts by employing elemental analysis- isotopic ratio mass spectrometry (EA-IRMS) (e.g. Chaile et al., 2018; Lantos et al., 2017; Miyano et al., 2017). Limited access to GC-c-IRMs has restricted the use of single-compound $\delta^{13}C$ analysis in Argentina. However, in nortwest Argentina, Lantos et al. (2015) implemented single-compound $\delta^{13}C$ analysis of the main alkanoic acids to better distinguish the origin of residues extracted from Inka and pre-Inka pottery. Their analysis on cooking pots and presumably alcoholic beverage (*chicha*) storage pots, and the use of experimental cooking vessels, and local ruminant and plant references, allowed them to compare lipid and $\delta^{13}C$ data. Their results presumed that cooking pots were used for preparing stews, where local ruminant meat, such as Llama (*Lama glama*), was mixed with C_4 (maybe maize), and C_3 plants, presumably beans (*Phaseolus vulgaris*) and *algarrobo* (*Prosopis* spp.). By undertaking $\delta^{13}C$ analysis on specialised *chicha* storage and serving pots, known as aryballos, they realised that two types of *chicha*, one made presumably of maize, and another from *algarrobo*, were served, and occasionally mixed.

Publications from nortwest Argentina have demonstrated methodological innovations compared to the Patagonia. Not only the use of GC-c-IRMS by Lantos and collaborators, but the implementation of liquid chromatography coupled to electrospray ionisation and quadrupole time-of-flight mass spectrometry (HPLC-ESI-Q-TOF) has allowed to improve specificity on the molecular identification. In a work by Lantos et al. (2020), HPLC-ESI-Q-TOF allowed to identify the presence of triacylglycerols from the residue of pre-colonial pottery, indicating that animal fat was the main product cooked in ceramic containers from El Colorado site, Catamarca, Argentina.

In the western side of the Andes, GC-MS, LC-MS has been used to identify the organic content of human hair, smoking pipes, and several other utensils destined for the consumption of psicoactive products (Miller et al., 2019; Echeverría, Planella and Niemeyer, 2014; Ogalde, Arriaza and Soto, 2010, 2009). On the other hand, the analysis of organic residues from pottery artefacts has been limited. Research on ceramic containers from Arica in northern Chile has been the only example of organic residues analysis from pre-colonial ceramic

vessels in Chile, with no conclusive interpretations for the identification of fatty acids (Fernández Murillo, 2016). Generally speaking, organic residues analysis on pre-colonial pottery in western South America has been limited.

8. Maize identification from pottery artefacts

Most works on organic residues from South America have focused on the identification of terrestrial animal fat and marine products, with few examples using this methodology to identify plant commodities, particularly maize (e.g. Admiraal et al., 2023: Lantos et al., 2017: Lantos et al., 2015). The low lipid content found in maize, as in most starchy plants, presents its identification from organic residues to be a challenging task. The lipid fraction of maize kernels contain a high abundance of unsaturated fatty acids, mainly represenetd by linoleic ($C_{18:2}$), and oleic ($C_{18:1}$) acids, and a lower abundance of saturated fatty acids, primary palmitic ($C_{16:0}$), stearic ($C_{18:0}$), and eicosanoic ($C_{20:0}$) acids (Ogalde, Arriaza and Cuellar, 2020; Carrillo et al., 2017). Fatty acid distributions above $C_{20:0}$ can also be found; however, in much lower quantities (Bianchi and Avato, 1984).

Reber and Evershed (2004) performed cooking experiments with whole maize kernel and cornmeal and demonstrated that most lipid content was rapidly lost within the first six months of potsherd deposition. After undergoing cooking experiments, replica pots were broken and exposed to different deposition contexts. The pots that performed a single cooking event did not exhibit any lipids after six months, whereas the pot that underwent several cooking events did contain preserved lipids, but in a very low quantity. Although biomolecular preservation did vary between contexts, poor lipid preservation was a common characteristic in each case due to high oxidation of unsaturated fatty acids and relatively low lipid content in maize. The authors indicated that the resultant lipid profile was not different from those described for general starchy plant products; this profile can be reflected by a high P/S ratio, a low representation of very long chain fatty acids, and the absence of long chain alkanes (Dunne, 2021; Lantos et al., 2015).

The work by Bondetti et al. (2020) look promising for the identification of grains and seeds, based on the degradation of unsaturated fatty acids from plant products. Maize kernels would likely show a high E/H ratio, above four, indicating an origin from cereals, fruits and non-leafy vegetables (Bondetti et al., 2020; Dolbunova et al., 2023). However, this assumption has not been tested.

Several works have indicated the abundance of fatty alcohol *n*-dotriacontanol (C_{32}-OH) in C_4 grasses like maize, which might help for further identification of maize from organic residues (Rommerskirchen et al., 2006; Reber and Evershed, 2004b; Bianchi and Avato, 1984). Although C_{32}-OH is rarely found in C_3 grasses, its identification cannot be used as a biomarker for maize identification as it can also be found in several poaceas from the panicoid subfamily (Reber and Evershed, 2004b). Its identification needs to be supported by solid evidence to suggest its origin from the processing of maize in archaeological pottery.

The use of carbon stable isotopes has shown to be essential for maize identification in environments dominated by C_3 plants, as no specific biomarker, as seen for millet, have been identified to assess the presence of maize in archaeological pottery. Reber et al. (2003) measured the $\delta^{13}C$ signature of C_{32}-OH to analyse 130 pottery sherds from pre-colonial contexts from the Mississippi Valley to assess the culinary use of maize in ceramic containers. From all the samples, only 16 had identifiable quantities of C_{32}-OH , from which only 10 demonstrated reliable isotopic results, plotting between -19‰ and -21‰, thus associated to a C_4 plant origin.

Since n-dotriacontanol is not always identified in organic residues analysis, single compound $\delta^{13}C$ analysis of the main alkanoic acids $C_{16:0}$ and $C_{18:0}$ have been employed to assess the presence of maize in pre-colonial pottery from the Americas (Reber, 2022; Reber and Evershed, 2004a). In Admiraal et al. (2023), the authors indicated an intensification of maize production from Guaraní pottery from Southern Brazil by around 1000 cal BP. Most evidence is supported by the use of GC-c-IRMS, where a clear progressive incorporation of maize into local foodways can be identified, as samples show a continuous ^{13}C enrichment towards maize references (Figure 3.25). The authors claim that a negative $\Delta^{13}C$ might reflect the mixing of ruminant carcass fat with maize, as dairy was not practised until the European colonisation. This study is the first in South America to include a large set of samples showing clear evidence for the progressive increment in the use of maize in pre-colonial pottery through organic residue analysis. The identification of fatty alcohol distributions in several samples up to and above C_{32} also supports the origin of the residues from panicoid grasses, likely to be maize.

Despite the conclusive support given by single-compound $\delta^{13}C$ to the identification of C_4, plants, likely maize, from organic residues, possible overlapping with marine animals is always a possibility. Furthermore, maize is not always the only C_4 plant found in several pre-colonial contexts; for example, other edible C_4 plants from South America such as *Amaranthus*

caudatus, were largely consumed by Andean pre-colonial groups (Ugalde et al., 2021; McRostie et al., 2019; Cadwallader et al., 2012). However, a clear lipid distribution indicating a plant origin for the residues, and the association of *n*-dotriacontanol to C_4 grasses from the panicoid subfamily might allow the differentiation of maize from marine organisms and C_4 non-grasses. Therefore, it is crucial to complement the isotopic data with robust molecular evidence that can support a plant origin of the residues and, from there, to present evidence as precisely as possible for a better taxonomic identification.

9. Conclusion

The extraction and analysis of organic residues from pottery artefacts shows promising possibilities to assess past culinary practices and foodways in archaeology. The assessment of these residues might allow the identification of a wide range of commodities such as marine, plant and terrestrial animal products. Organic residue analysis of pottery containers can provide further complementation to other methodologies that might present certain limitations to identify several products.

This approach has been applied in several archaeological contexts worldwide; however, its use in South Ameican archaeology has been limited. Its extensive incorporation into South American archaeological might be beneficial to better understand the use of pottery and the significance of several commodities in local foodways. In this sense, the use of organic residue analysis to identify maize from pre-colonial pottery has been poorly addressed. This approach might help to complement other methods used in the identification of maize from past foodways, such as stable isotope analysis and archaeobotany.

Chapter 4: Unravelling Island economies through organic residue analysis: the case of Mocha Island (southern Chile)

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Statement: This work has already been published; however, in this thesis I have included more details and figures about the sites, as well as more details regarding the samples analysed in this work.

2. Abstract

Biophysical conditions played a fundamental role in early human colonisation of insular territories, particularly in food-producing societies dealing with limited resources and the challenges of maintaining a sustainable carrying capacity. Studies on past human colonisation of small oceanic islands thus offer insights into economic plasticity, ecological impacts, and adaptation of early food-producing groups. On the coast of southern Chile, early evidence is dated to 950 cal BP of island colonisation by coastal populations with mainland subsistence systems based on the exploitation of marine resources, along with gathering, managing, and cultivating plants and hunting terrestrial animals. Strikingly, the extent to which these mixed

economies contributed to insular colonisation efforts is largely unknown. Here we used organic residue analysis of ceramic artifacts to shed light on the subsistence of populations on Mocha Island in southern Chile. We extracted and analysed lipids from 51 pottery sherds associated with the El Vergel cultural complex that flourished in southern Chile between 950 and 400 cal BP. Chemical and stable isotope analysis of the extracts identified a range of food products, including C_3 and C_4 plants and marine organisms. The results reveal the central role of mixed subsistence systems in fueling the colonisation of Mocha Island.

Keywords: pottery; organic residue analysis; island colonisation; southern Chile; El Vergel; mixed economies

3. Introduction

The socioecological drivers of early human colonisation of oceanic islands have been the subject of a contentious debate in archaeology (Terrell, 2023; Braje et al., 2017). Insular biogeographic conditions, distance from mainland enclaves, seafaring technology, and the flexibility of a subsistence economy were some of the interplaying factors conditioning island-colonisation efforts (O'Connor et al., 2019; Crowther et al., 2016; Leppard, 2014; Takamiya, 2006). Over the preceding several decades, genetic, archaeological, and linguistic studies have increased our understanding of these processes in regions such as the west and northeast Pacific Ocean (McFadden et al., 2021; Horsburgh and McCoy, 2017; Lepofsky et al., 2015; Walworth, 2014; Potter and White, 2009). Despite extensive archaeological research along the western coast of South America (Reyes et al., 2022a; Beresford-Jones et al., 2018; King et al., 2018; Stothert, Piperno and Andres, 2003), large geographic areas of the Chilean coast remain poorly understood.

On the Pacific coast of South America, the productive Humboldt upwelling system (3°24′ S–54°55′ S) has sustained coastal adapted populations since the Late Pleistocene. Throughout the Holocene, coastal communities developed mixed economies with distinct degrees of reliance on marine and terrestrial based resources (Dillehay et al., 2022b; Pearsall et al., 2020; Knudson, Peters and Cagigao, 2015). In southern Chile the intensification of food production during the Late Ceramic period (950–400 cal BP) led to sedentarism and a remarkable demographic upsurge (Campbell and Quiroz, 2015). During this time, farming groups from the El Vergel cultural complex migrated and permanently settled on small offshore islands (Campbell, 2015; Campbell and Quiroz, 2015). From here, the occupation of Mocha

Island, the largest, most isolated island, lying 35 km offshore, is an intriguing case. The proliferation of El Vergel residential sites and the construction of monumental architecture by a large workforce seem to suggest that a dense, complex, and permanently settled society was established here (Campbell and Pfeiffer, 2017; Campbell, 2015). However, Mocha Island's biogeographic characteristics denote limited terrestrial resources, making for unsuitable conditions for supporting large groups of people (Campbell, 2015; Pefaur and Yáñez, 1980).

The translocation of domestic plants and tamed camelids is argued to have been a prerequisite for the permanent settlement of Mocha Island by the El Vergel groups (Roa et al., 2021; Godoy-aguirre, 2018; Campbell, 2015; Roa, Silva and Campbell, 2015; Becker, 1997b). Preliminary results on lipid distributions extracted from Mocha Island pottery fragments indicated a preferential use of terrestrial resources, including the exploitation of terrestrial animals (Roa et al., 2021). Carbon ($\delta^{13}C$) and nitrogen ($\delta^{15}N$) stable isotope analyses of El Vergel individuals from Mocha Island suggest a diet largely based on terrestrial resources with only a low contribution of marine proteins (Campbell et al., 2020). The results clearly show the predominance of a mixed diet. ^{13}C enrichment in the individuals is attributed to the consumption of locally cultivated maize (Campbell et al., 2020), while marine resources are claimed to have played a marginal role in the foodways and adaptive process of El Vergel farmers, despite the island's location within one of the most productive upwelling systems of the world (Roa et al., 2021; Campbell et al., 2020).

Attributing human bone collagen ^{13}C enrichment to the sustained consumption of C_4 plants such as maize is problematic when marine resources are involved because of the possible overlapping of $\delta^{13}C$ values (Corr et al., 2005). Moreover, mollusc remains were found at every residential site on Mocha Island, along with marine mammal and fish bones; in some cases, they outnumbered terrestrial animal remains (Roa et al., 2021; Power, 2013). The identification of net sinkers and fishing hooks in some sites indicates the use of specialised technology for exploiting local marine resources (Martínez, 2015, 2013; Quiroz and Sanchez, 2005). The clear evidence for the use of marine commodities and the possible methodological biases suggest that the role of marine products in El Vergel farmers' foodways on Mocha Island is not well understood and needs further assessment.

Here, we present the first organic residue analysis from the Pacific coast of South America using single-compound stable carbon isotope composition and molecular evidence extracted from pottery sherds to evaluate the use of marine resources by El Vergel coastal farmers settling on Mocha Island. This analysis enabled us to assess local foodways, the use

pottery might have had for processing marine resources, and whether this reflects the adaptive and socioeconomic strategies developed during Mocha Island's colonisation.

4. Mocha Island: Environmental Setting

Mocha Island is an offshore small insular territory of 52 km^2 that is located 35 km from the mainland (Figure 4.1). It originates as the highest section of an emerging ridge from the continental plaque, continuously rising above sea level for the last 6000 years (Melnick et al., 2003; Nelson and Manley, 1992). It comprises two main mountain ranges of 390 m.a.s.l and is covered by a dense evergreen forest that is surrounded by a coastal plain of beaches and meadows (Bahlburg and Spiske, 2015; Prieto, 1997). Annual precipitation of around 1,350 mm forms small rivers and lakes supplying the island with fresh drinkable water (Prieto, 1997; Pefaur and Yáñez, 1980).

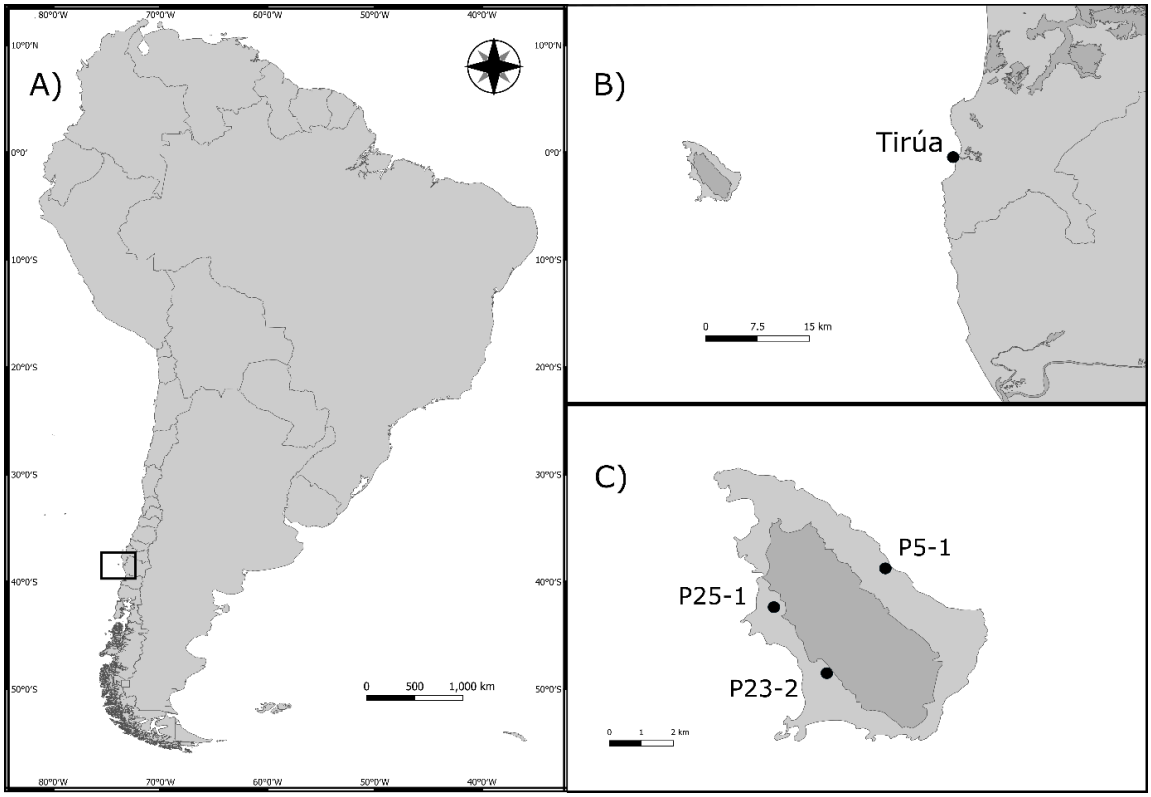


Figure 4.1: ***(A)*** *Mocha Island in South America;* ***(B)*** *Mocha Island facing the coast of Chile;* ***(C)*** *archaeological sites at Mocha Island. Modified from Montalvo-Cabrera et al. (2024). Figure created by the author.*

The composition of its flora has remained relatively unchanged for the last 2000 years: it is mainly dominated by evergreen species such as *Aextoxicon punctatum*, *Drimys winteri*, and *Azara lanceolata* and a variety of Myrtaceae (Lequesne, Villagrán and Villa, 1999). This landscape also contains a range of wild edible plants, mainly *Aristotelia chilensis* (Chilean wineberry), *Fragaria chiloensis* (Chilean strawberry), *Rubus geoides* (wild raspberry), *Gevuina avellana* (Chilean hazelnut), and *Madia sativa* seeds (Roa et al., 2021; Roa et al., 2015).

The native fauna comprised various small rodents, birds, reptiles, and amphibian species; no large terrestrial mammals were present before the translocation of mainland animals by humans (Saavedra, Quiroz and Iriarte, 2003; Pefaur and Yáñez, 1980; Reiche, 1903). Local marine fauna are abundant and include molluscs, crustaceans, marine mammals, and different fish species (Rebolledo, 2013; Báez, 1997; Gálvez, 1997; Reiche, 1903).

5. The El Vergel Cultural Complex and Pottery Production

The El Vergel cultural complex consisted of farming groups that cultivated domestic crops, tamed wild animals, and engaged in hunting-and-gathering practices (Roa et al., 2018; Contreras et al., 2005; Becker, 1997a). The main characteristic element of the El Vergel cultural complex is its pottery. Decoration using red or black geometric motifs over a white slip surface is a distinctive feature of the El Vergel pottery found in funerary contexts (Bahamondes, Silva and Campbell, 2006; Adán et al., 2005; Aldunate, 1989). Large ceramic containers were common, some of which functioned as funerary urns; however, these containers were also found in residential settings (Navarro and Aldunate, 2002; Gordon, 1978; Bullock, 1970). Ethnographic investigations within the Mapuche people, descendants of the El Vergel cultural complex, described the use of similar large containers for culinary purposes, mainly the storage and preparation of fermented maize or wheat beverages known as *chicha* or muday (Alvarado, 1997). Other types, such as nondecorated monochrome vessels and pots with a red slip surface, are frequently found in residential sites (Adán et al., 2005; Quiroz, 2001). These pots correspond to various vessel types such as jars, cooking pots, mugs, and dishes, presenting specific morphological characteristics that denote a long-standing pottery tradition in southern Chile (Lopez, 2017; Palma, 2013; Adán et al., 2005; Dillehay, 1990a; Aldunate, 1989).

On Mocha Island, diagnostic elements found in residential sites confirm the presence of the El Vergel cultural complex (Roa et al., 2021; Campbell, 2020; Campbell and Pfeiffer, 2017). Its population comprised autonomous communities with equal access to local and exotic resources (Campbell, 2020). Camelids or chilihueque, the name for local guanaco (*Lama guanicoe*), were translocated from the mainland and exploited at every residential site for fur and meat (Roa et al., 2021; Westbury et al., 2016; Becker, 1997b). Domesticated crops, specifically quinoa (*Chenopodium quinoa*), maize (*Zea mays*), and beans (*Phaseolus vulgaris*), were cultivated along with gathering wild local plants (Roa et al., 2021; Roa et al., 2015; Rojas and Cardemil, 1995). The presence of molluscs, marine birds and mammals, and fish remains at every residential site confirms generalised access to coastal foraging, hunting, and fishing, mainly nearshore fishing (Roa et al., 2021; Martínez, 2015, 2014; Power, 2013; Rebolledo, 2013; Gálvez, 1997;).

The sites selected for this study are located at different points of the island (Figure 4.1). The P5-1 site is one of the largest on the island. It is located on a marine terrace in its northeast portion (Campbell 2020). The site faces the continental coast and is close to a kuel or monumental mound dating back to the start of the Late Ceramic Period (Campbell and Pfeiffer, 2017). In the case of P23-2, the site is situated on the island's southwest side, over a marine terrace facing the open sea, similar to P25-1, which is also located on the south side of the island, over a marine terrace, and around 2 km from the shore (Campbell, 2020)..

Roa et al. (2021) assessed the ubiquity of the archaeobotanical evidence, which indicated variable representation of several plant species throughout these sites. The evidence indicated a high presence of quinoa seeds, while using other domesticated plants such as maize and beans was more variable. A diversity of wild edible plants such as *Aristotelia chilensis*, *Madia sativa*, *Fragaria chiloensis* and *Rubus* sp. were also exploited in these sites, which comprised an important part of the archaeobotanical assemblage (Figures 4.2 and 4.3.

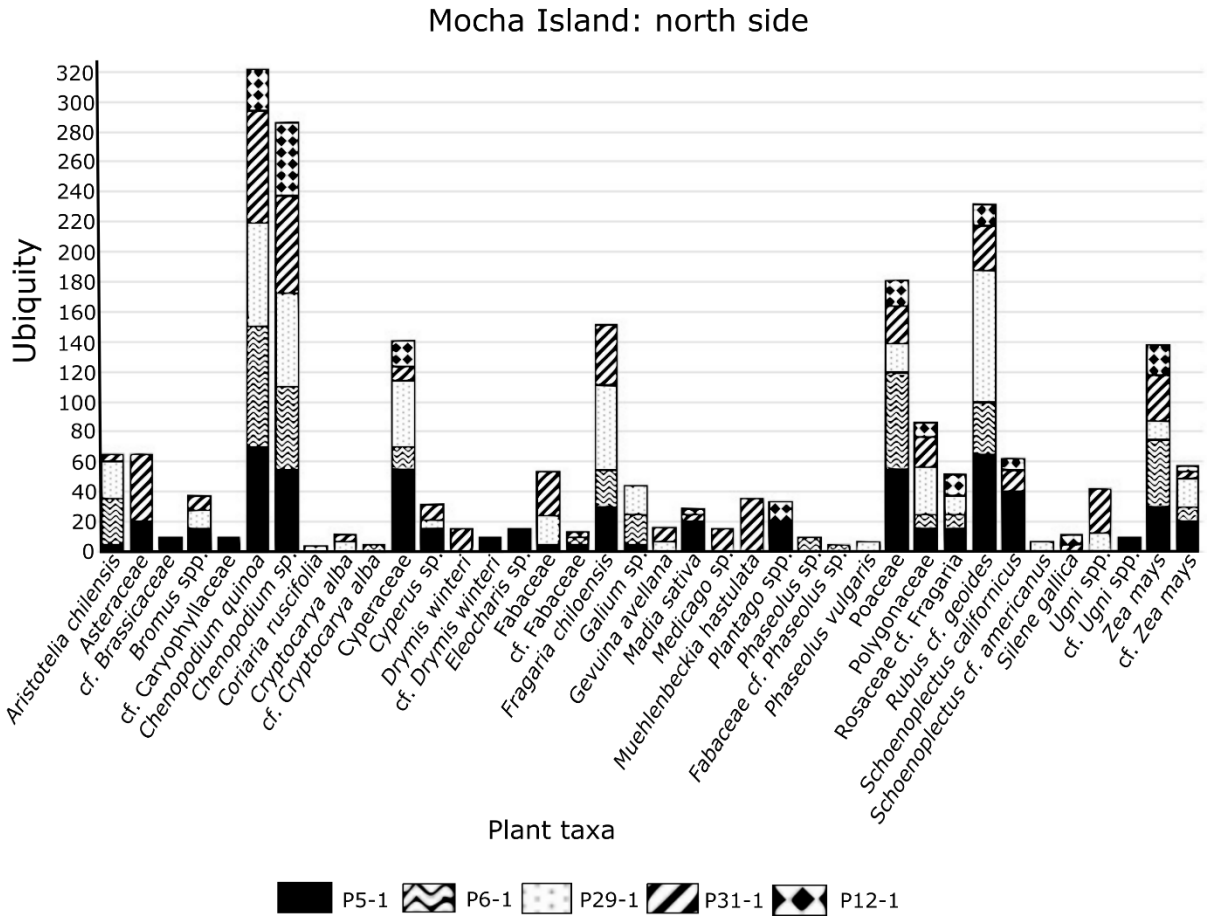


Figure 4.2: Bar chart showing the ubiquity of carpological remains for several domesticated and wild plant species. Samples were recovered from several sites located in the north side of the island. The chart was obtained and adapted from Roa et al. (2018).

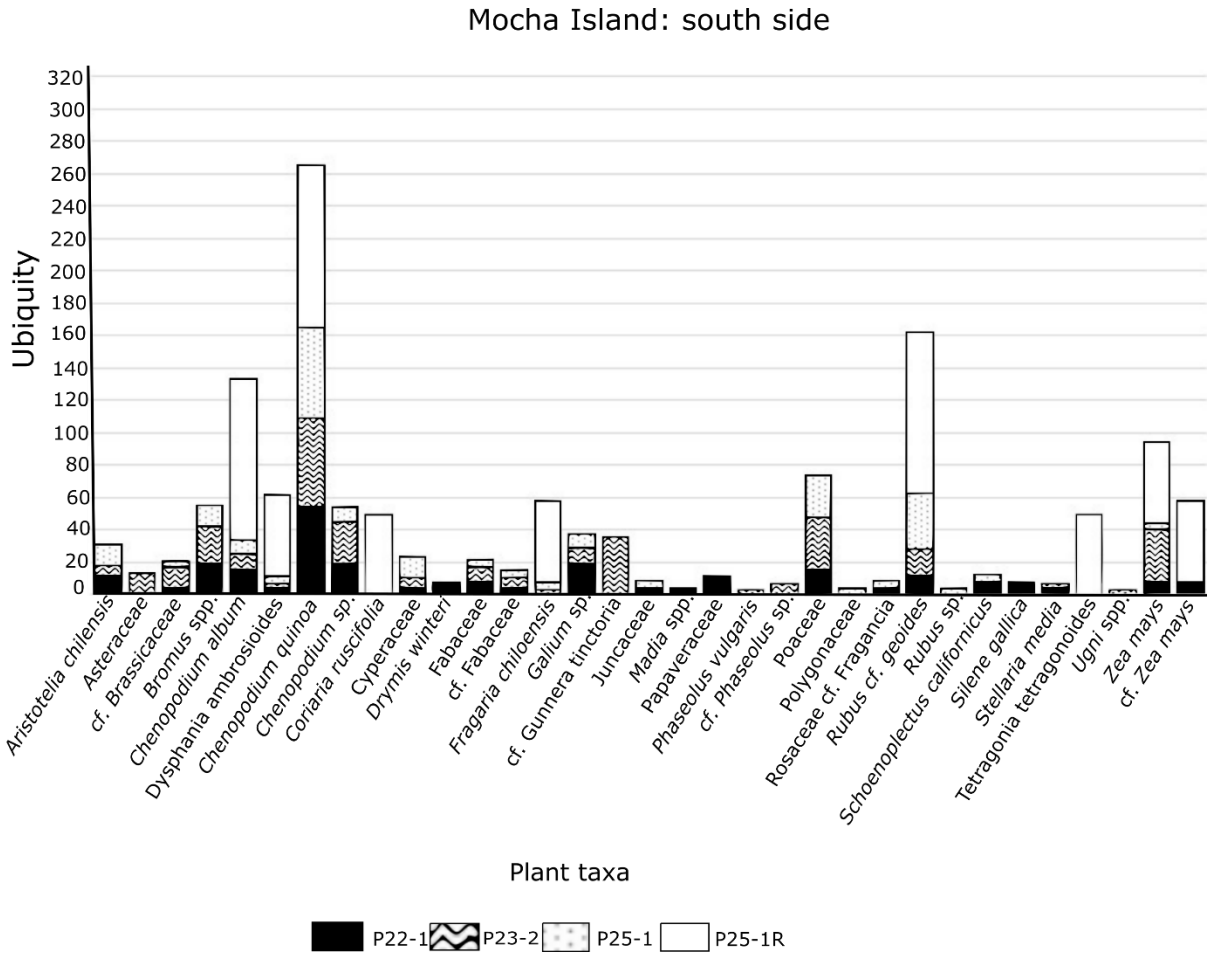


Figure 4.3: Bar chart showing the ubiquity of carpological remains for several domesticated and wild plant species. Samples were recovered from several sites located in the south side of the island. The chart was obtained and adapted from Roa et al. (2018).

The zooarchaeological remains identified in these three sites also demonstrated variable ubiquity (Roa et al., 2021). The evidence indicates a high presence of mammal bones, especially from camelids, South American deer Pudu puda, marine mammals and rodents (Figure 4.4). Various bird species such as penguins, seagulls, and cormorants, were identified by Roa et al. (2021), showing variable representation in every site (Figure 4.5). The identification of fish bones denoted a higher presence of nearshore species such as *Aphos porosus*; however, pelagic fish such as chondrichthyes were also identified (Figure 4.6). Evidence for the exploitation of invertebrates was present in every site (Roa et al., 2021); however, systematic analysis on invertebrate zooarchaeological remains were held in a limited number of sites, from which P5-1 is the only one that is part of this study (Figures 4.7 and 4.8).

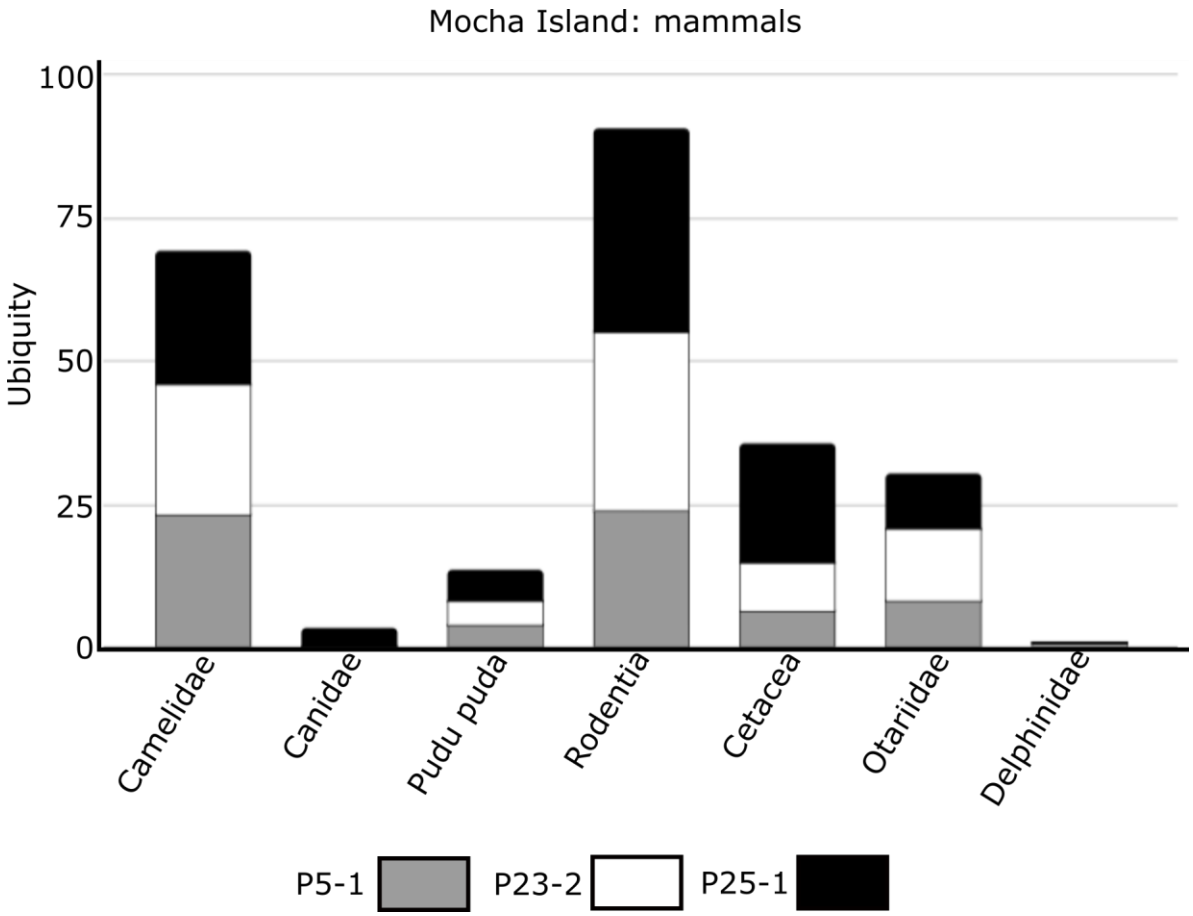


Figure 4.4: Ubiquity of mammal bone remains identified in Mocha Island. Data was obtained from Roa et al. (2021). Figure created by the author.

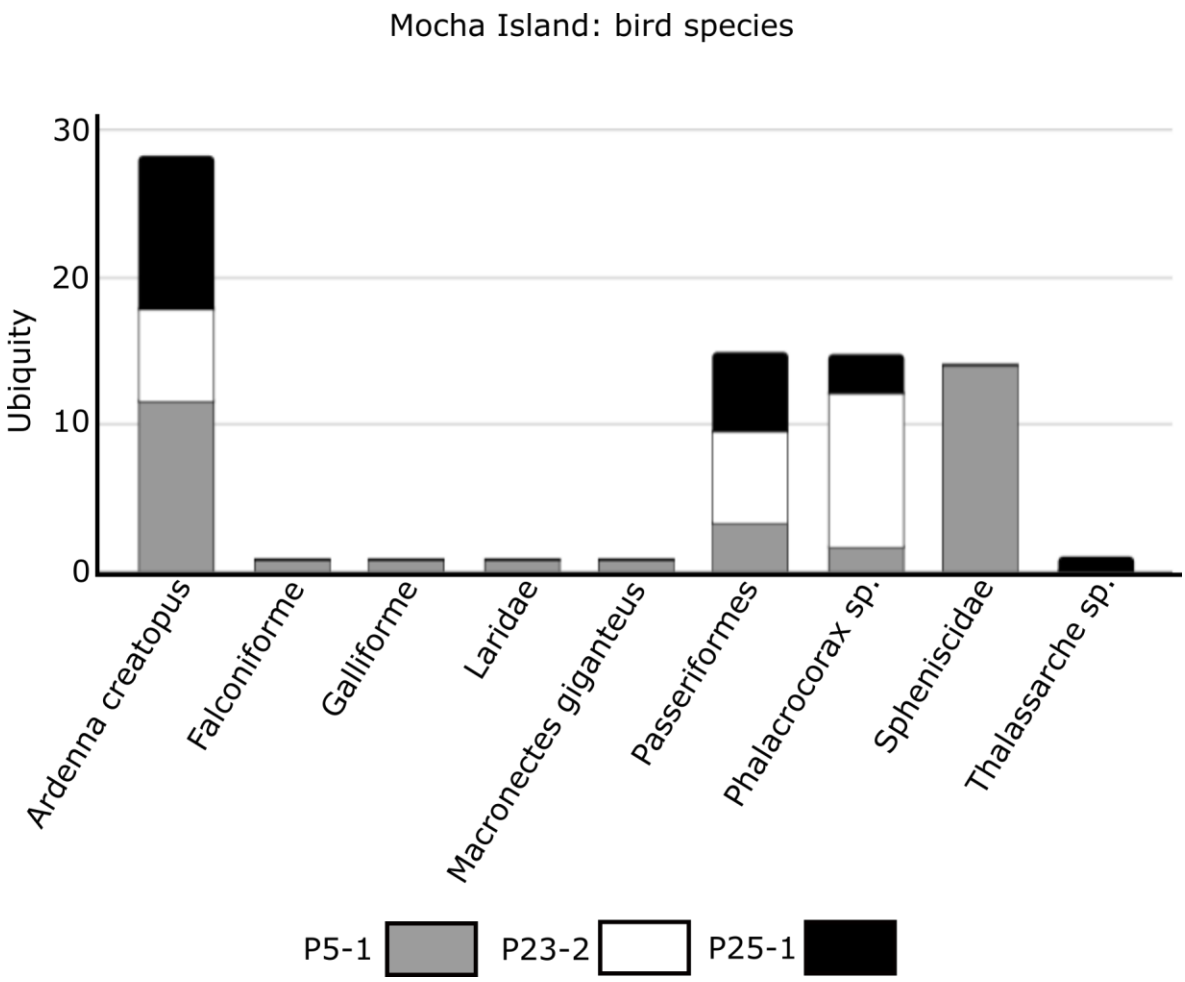


Figure 4.5: Ubiquity of bird bone remains identified in Mocha Island. Data was obtained from Roa et al. (2021). Figure created by the author.

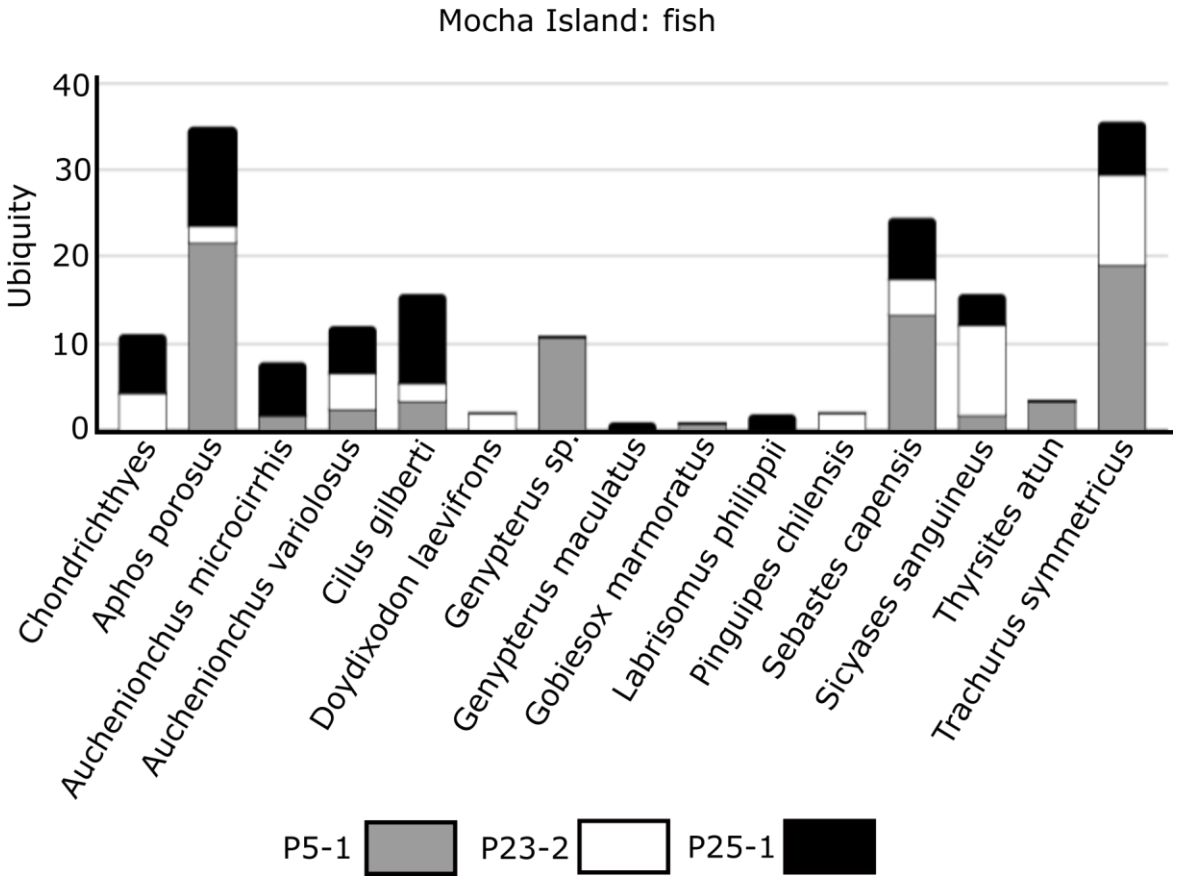


Figure 4.6: Ubiquity of fish bone remains identified in Mocha Island. Data was obtained from Roa et al. (2021). Figure created by the author.

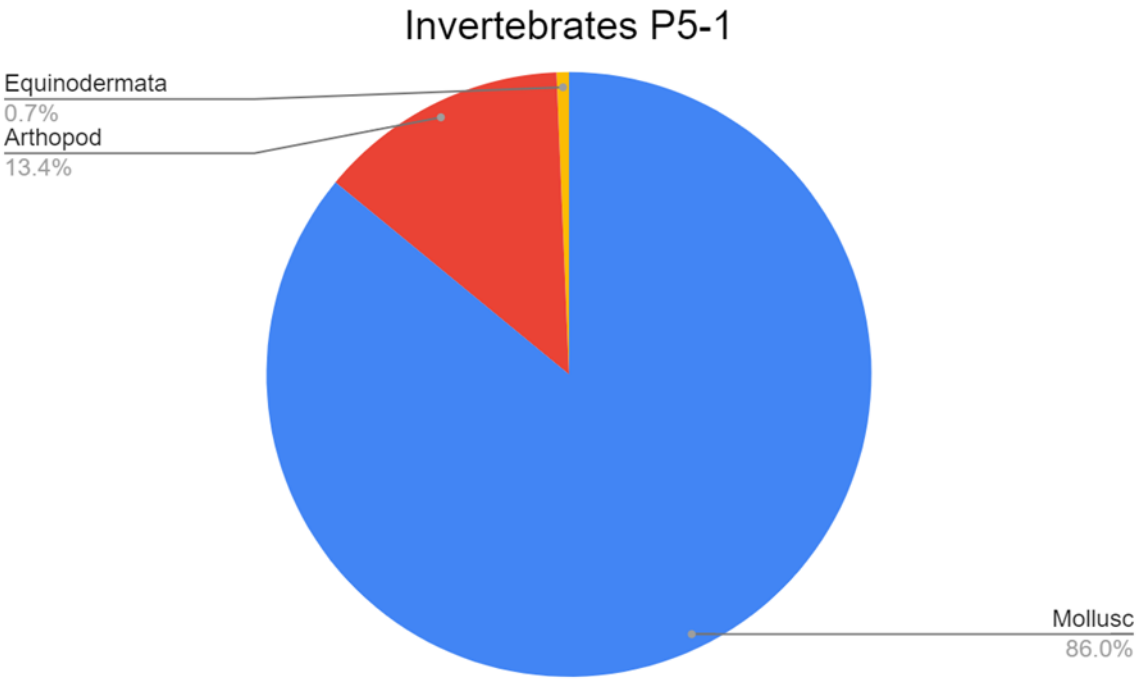


Figure 4.7: Frequency of invertebrate zoorachaeological remains for P5-1 site. Data was obtained from Power (2013). Figure created by the author.

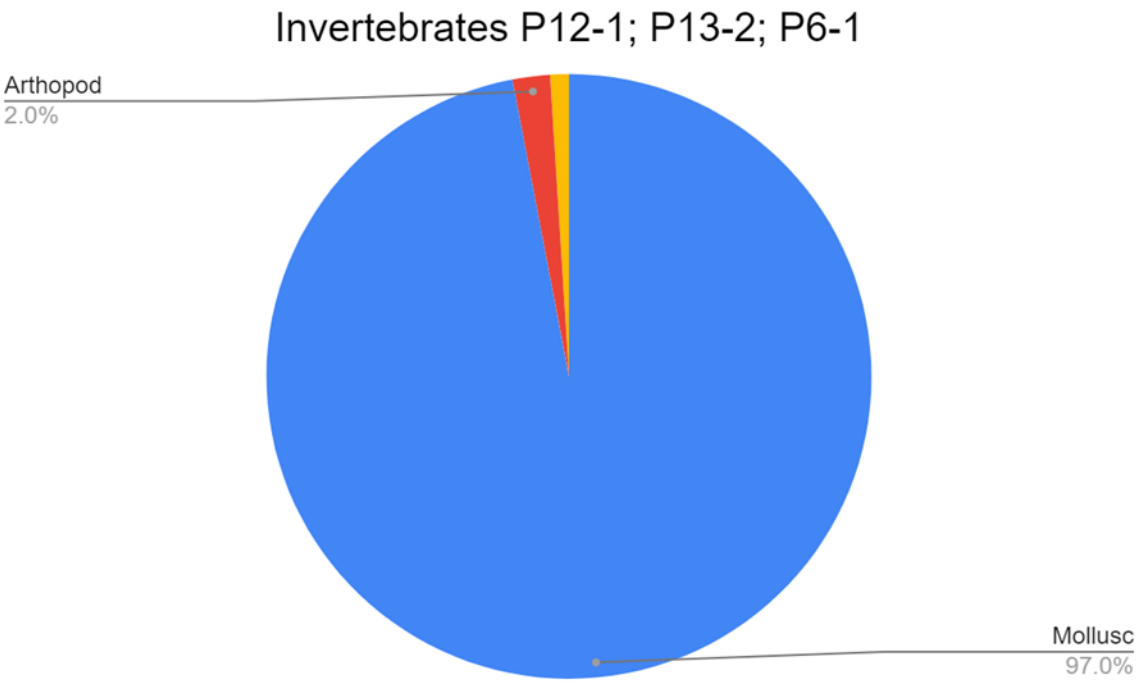


Figure 4.8: Frequency of invertebrate zooarchaeological remains from sites P12-1, P13-2, and P6-1. Data obtained from Power (2014). The report did not specify the frequency of taxa per site. Figure created by the author.

6. Materials and Methods

We selected 51 pottery sherds chronologically associated with the Late Ceramic Period from these three residential sites (Table 4.1).

6.1. Potsherds from P5-1

Potsherds were recovered from excavation unit 05.02.03, which contained a high density of ceramic materials (Figure 4.9). Of the 2594 recovered potsherds, only 17 were analysed for the P5-1 site.

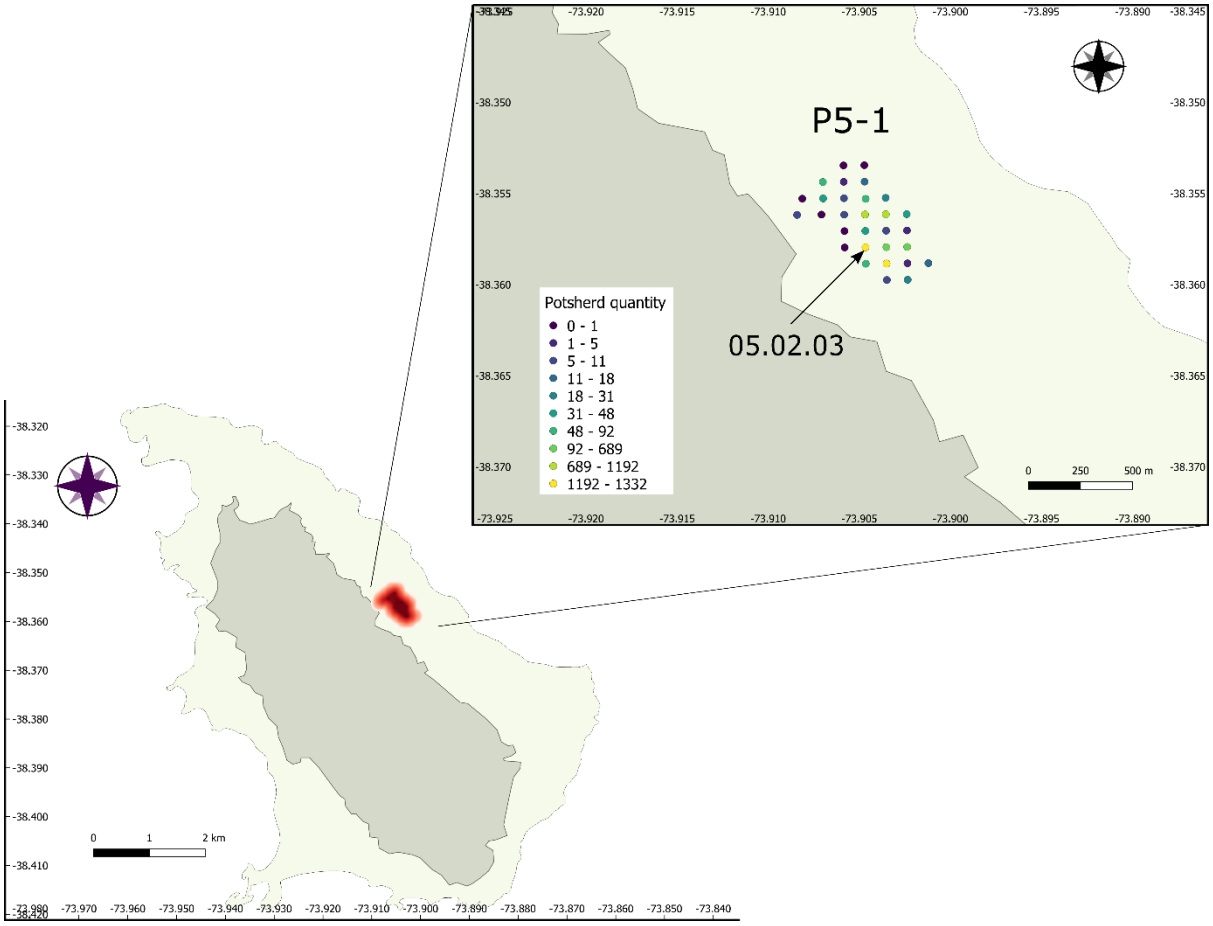


Figure 4.9: Map of P5-1 boreholes. Leyend indicates the number of potsherds recovered from each borehole, based on Campbell (2016a). Figure created by the author.

6.2. Potsherds from P23-2

Six boreholes were excavated in 2015 (Figure 4.3), from which a total of 2118 potsherds were recovered. Borehole 23.03.01 comprised most of the potsherds recovered from the P23-2 site, yielding 1160 pottery fragments. Fifteen potsherds from this borehole were selected for organic residue analysis.

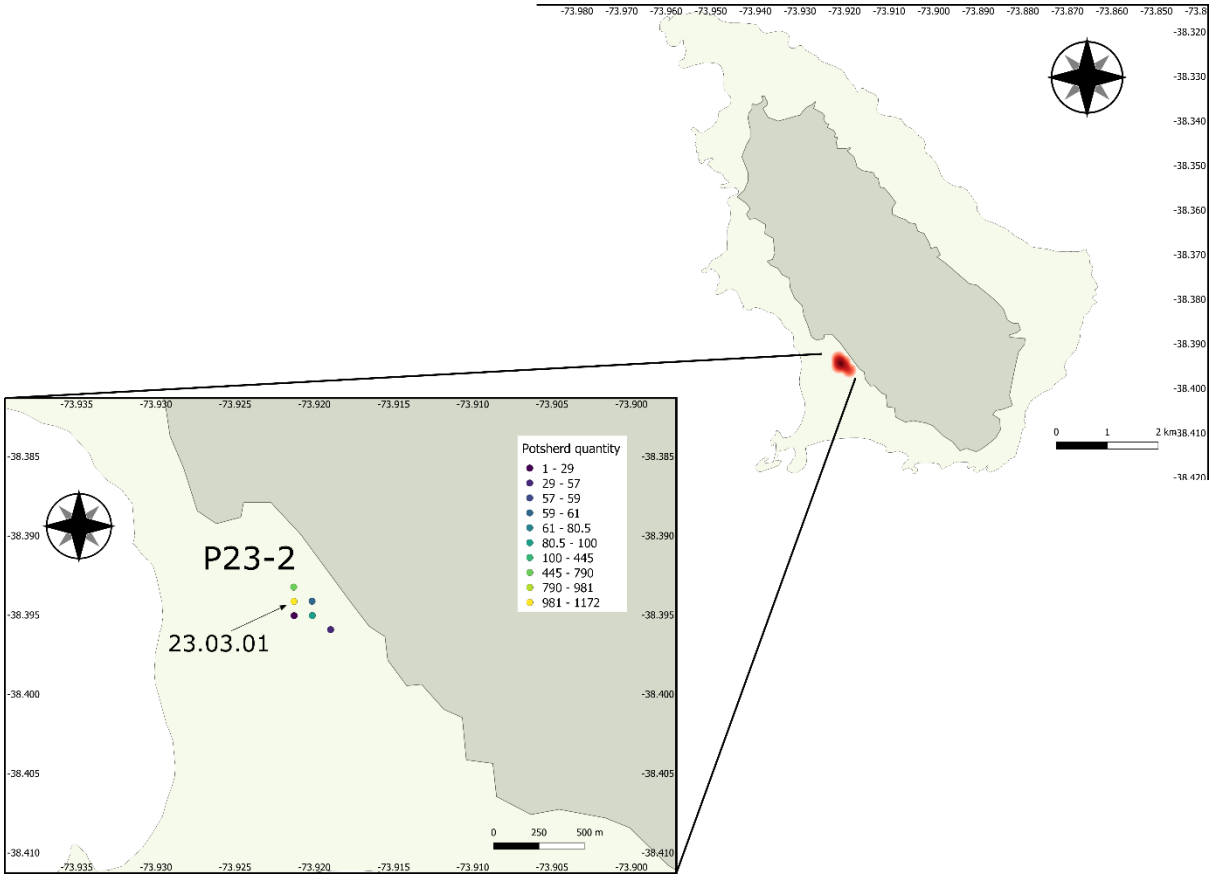


Figure 4.10: Map of P23-2 boreholes. Leyend indicates the number of potsherds recovered from each borehole, based on Campbell (2016b). Figure created by the author.

6.3. Potsherds from P25-1

Around 15 boreholes were excavated in 2015, from where it was possible to recover 4105 potsherds. Samples obtained for organic residue analysis came from 5 boreholes, from which different potsherd quantities were possible to recover (Figure 4.4).

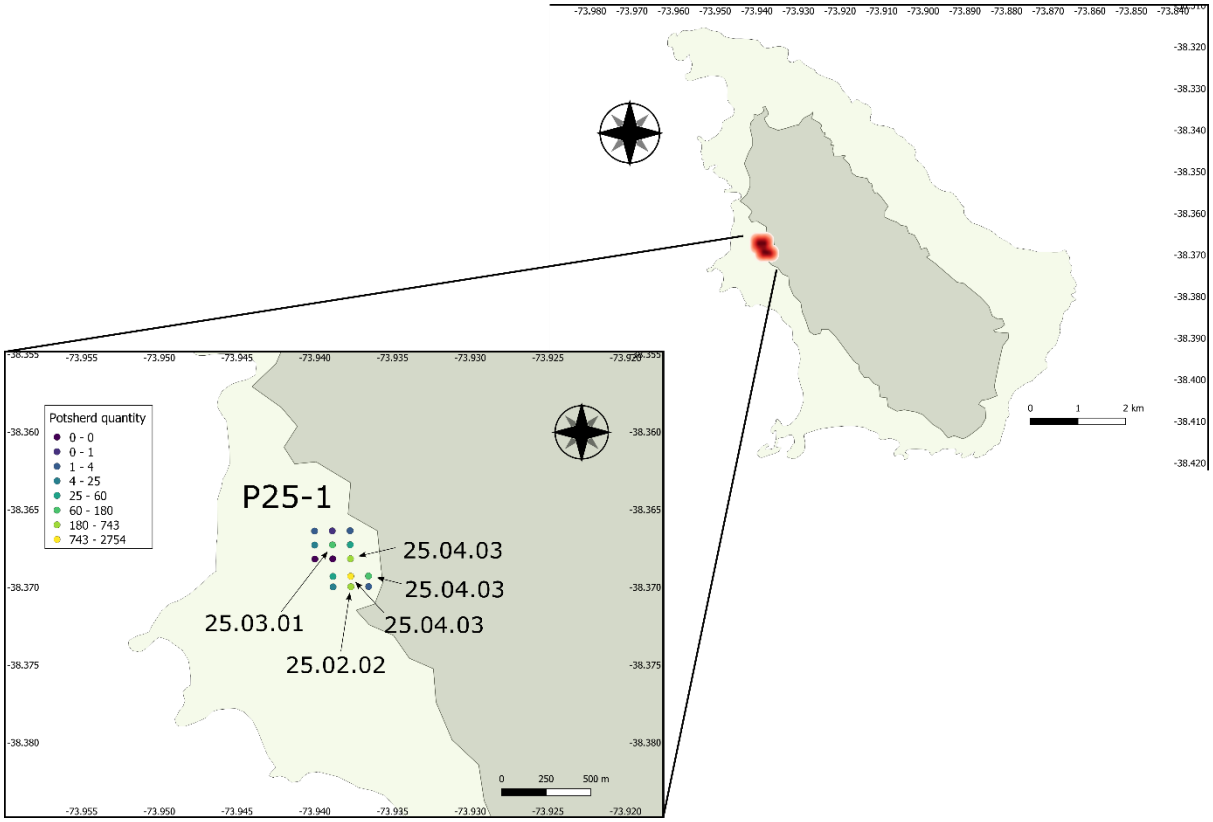


Figure 4.11: Map of P23-2 boreholes. Leyend indicates the number of potsherds recovered from each borehole, based on Campbell (2017). Figure created by the author.

The selected sherds comprised polished, smoothed, and eroded monochrome pottery fragments from the rim, neck, body, and base (Appendix 6). The fragmentary nature of the samples and the lack of complete vessels, has prevented us from associating the potsherds with a specific type of vessel (Appendix 6). However, characteristics such as fragment shape and a thickness above 7.5 mm have been associated with pot uses for cooking and storing commodities; thus, only samples with a thickness greater than 7.25 mm were selected. The identification of soot marks in some of them potentially indicates their exposure to a heating source, possibly during cooking events. Based on these characteristics, the selected fragments likely represent cooking pots and large containers, mainly used for processing food resources (Albán, Palma and Delgado, 2013).

Table 4.1: ^{14}C Dates from associated material, Mocha Island

Site	Nº of Samples	Laboratory Code	Excavation Unit	Material	Taxa	^{14}C Age BP	Median Probability cal yrs BP
P5-1	17	UB-26214	05.02.03	Kernel	*Zea mays*	552 ± 26	530
		UB-26216	05.02.03	Bone (Phalange)	Camelidae	605 ± 26	554
		UB-26215	05.02.03	Cobb	*Zea mays*	635 ± 25	607
P23-2	15	UB-29290	23.03.01	Kernel	*Zea mays*	1108 ± 28	961
		UB-29289	23.03.01	Seed	*Phaseolus vulgaris*	679 ± 25	603
P25-1	19	UB-29287	25.03.04	Kernel	*Zea mays*	644 ± 35	605
		UB-29288	25.03.04	Kernel	*Zea mays*	661 ± 30	606

Table 4.1: *Data from (Campbell and Pfeiffer, 2017). Table created by the author.*

To obtain molecular and $\delta^{13}C$ references of local resources, cooking experiments were conducted using seven nontempered replica vessels (Bondetti et al. 2020; see Appendix 2.2). Each commodity was cooked in a specific container, with no mixing between resources.

6.4. Organic Residue Analysis

A small section of the inner surface of the 51 pottery fragments was cleaned before drilling 2–3 mm into the potsherds to obtain 1 g of pottery powder for acid extraction (AE). A one-step acidified methanol protocol was followed (Craig et al., 2013). A blank was included as a control to assess contamination throughout the extraction process. An internal standard (n-tetratriacontane (C34): 10 µg) was incorporated in each tube. Methanol (4 mL) and sulphuric acid (800 µL) were added and the mixture was subsequently sonicated and centrifuged before being placed on a heating block for four hours at 70°C. Three successive

extractions using n-hexane allowed the lipids' separation before their neutralisation using potassium carbonate. Subsequently, lipids were concentrated and resuspended in n-hexane at appropriate dilution for gas chromatography-mass spectrometry (GC-MS) analysis. An internal standard (n-hexatriacontane (C36): 10 µg) was added at the end of the extraction to infer lipid yield and concentration.

Another 1 g was prepared from nine potsherds and subjected to solvent extraction (SE). The extraction procedure followed a protocol similar to that described by Colonese et al. (2017b, 181). Lipids were extracted by sonication from the ceramic powder using dichloromethane:methanol DCM:MeOH 2/1 v/v three times. A blank was also prepared to assess contamination. After the centrifugation, the liquid fraction was transferred into clean and labelled hydrolysis vials, and their content was evaporated to complete dryness under a gentle stream of nitrogen. One aliquot of the sample was resuspended in 50 µl of n-hexane and derivatised by adding four drops of N, O-bis (trimethylsilyl) trifluoroacetamide with 1% trimethylchlorosilane (BSTFA+TMCS, 99:1). Samples were placed on a heating block for one hour at 70°C and then dried under a gentle stream of nitrogen. Subsequently, samples were resuspended in n-hexane for their analysis by GC-MS.

Seven acid extracted samples were also derivatised with BSTFA+TMCS, 99:1, to identify very long-chain fatty alcohols. Samples were transferred from their respective GC-vial inserts by adding 100 µl of n-hexane into each vial and transferring it into a clean hydrolysis vial (x3). Samples were dried and resuspended in 50 µl of hexane, and four drops of BSTFA+TMCS, 99:1, and a flush of nitrogen were added before they were placed on a heating block at 70°C for 1 hour. Samples were concentrated and then transferred to a new GC-vial with a conical insert for their analysis within 48 hours.

Around 1 g of pottery powder was obtained from each of the seven replica vessels used in the cooking experiments. An extraction protocol using acidified methanol was also followed. Lipid extracts were screened and quantified using an Agilent 7890B high-temperature gas chromatograph (Agilent Technologies, Cheadle, Cheshire, UK) equipped with a flame ionisation detector (HT-GC-FID). A 100% Dimethylpolysiloxane DB-1 column (15 m × 320 µm × 0.1 µm; J&W Scientific, Folsom, California, USA) was used. A splitless injector was used to inject 1 µL of acidextracted samples into the GC at 300°C. The carrier gas was helium, with a constant flow rate of 2 mL min-1. The temperature of the oven was set at 100°C for two minutes and then increased to 20°C min^{-1} up to 325°C, holding for three minutes.

A splitless injection was also used to inject 1 µL of solvent-extracted samples at 350°C. The temperature of the oven was set at 50°C for two minutes and then increased 10°C min^{-1} up to 375°C, settling for 10 minutes.

Acid-extracted and solvent-extracted samples were analysed using an Agilent 7890A series chromatograph attached to an Agilent 5975C inert XL mass selective detector with a quadrupole mass analyser (Agilent Technologies). The column used was a methylpolysiloxane (5%-phenyl) DB-5ms (30 m × 0.25 mm × 0.25 µm; J&W Scientific). Samples were injected (1 µL) using a splitless injector at 300°C. Helium was used as the carrier gas, at a constant flow rate of 2 mL min^{-1}. The temperature of the oven was set at 50°C for two minutes and then rose 10°C min^{-1} to 325°C, where it settled for 15 minutes. The mass spectrometer ionisation energy was 70 eV, obtaining a spectrum by scanning ions between m/z 50 and 800.

Preliminary results using HT-GC-FID indicated the possible presence of triacylglycerols in some samples (Roa et al. 2021). We used high-temperature gas chromatography-mass spectrometry (HT-GC-MS) to assess their presence in the pots. TMS solvent extracts were analysed using a Perkin Elmer Clarus 690 gas chromatograph coupled to a SQ8-T mass spectrometer. The column was a DB5-HT column (30 m × 0.25 mm × 0.1 µm). Helium was used as the carrier gas at a constant flow rate of 1mL min^{-1}. The temperature of the oven was set at 50°C for two minutes and then increased to 10°C min^{-1}, reaching a maximum temperature of 375°C held for 15 minutes.

The identification of isoprenoid acids and ω-(o-alkylphenyl) alkanoic acids (APAAs) as aquatic biomarkers was performed using a GC-MS equipped with a 50% cyanopropyl-methylpolysiloxane DB-23 column (60 m × 0.25 mm × 0.25 µm; J&W Scientific) as in the study by (Shoda et al., 2017). A splitless injector was used to inject 1µL of the acidified methanol extracts into the GC at 300°C. Helium was used as the carrier gas, with a flow rate of 1.5 mL min^{-1}. The temperature of the oven was set at 50°C for two minutes and then rose 10°C min^{-1} to 100°C, after which it increased 4°C min^{-1} up to 140°C then 0.5°C min^{-1} up to 160°C, and finally by 20°C min^{-1}, reaching 250°C where it settled for 20 minutes. The mass spectrometer was operated in single ion monitoring (SIM) mode to achieve higher sensitivity of target compounds. Selected ions allowed the identification of the three main isoprenoid acids (m/z 74, 101, 171, and 326 for phytanic acid; m/z 74, 88, 101, and 312 for pristanic acid; and m/z 74, 87, 213, and 270 for 4, 8, 12-trimethyltridecanoic acid [4, 8, 12-TMTD]), and APAAs of carbon length C16–C22 (m/z 74, 105, 262, 290, 318, 346).

MSD Chemtation F.01.03.2357 software was used to compute the GC-MS results. Compounds were identified according to retention time and mass spectrum and by comparison with the National Institute of Standards and Technology (NIST) library. Peaks were integrated using Agilent Mass Hunter Quantitative Analysis software version B.07.01/ Build7.1.524.0 for GC-MS.

6.5. GC-c-IRMS and single-compound $\delta^{13}C$ analysis

We selected 49 samples for $\delta^{13}C$ analysis of the main alkanoic acids $C_{16:0}$ and $C_{18:0}$ based on a minimal alkanoic acid quantity needed for injection. These samples were analysed using a Delta V Advantage Isotope Ratio Mass Spectrometer (Thermo Fisher Scientific, Bremen, Germany), coupled to a Trace Ultra 1310 Gas Chromatograph (Thermo Fisher) with a GC IsoLink II interface (with a CuO combustion reactor held at 850°C). The column was an ultra-inert fused-silica DB-5 ms UI (60 m × 0.25 mm × 0.25 µm; J&W Scientific), into which 1 µl of each sample was injected for analysis. Ultra-high-purity grade helium was used as the carrier gas with a flow rate of 2 mL min^{-1}, from which a small part of the flow was diverted to an ISQ mass spectrometer (Thermo Fisher) for parallel acquisition of molecular data. The temperature of the oven was set at 50°C for 0.5 min, and then increased 25°C min^{-1} to 175°C and then 8°C min^{-1} until it reached 325°C and then settled for 20 minutes.

Eluted compounds were ionised in the mass spectrometer through electron impact. The intensities of ions m/z 44, 45, and 46 were recorded to automatically compute the $^{13}C/^{12}C$ ratios of each peak in the extracts. The software used for the computation were Isodat (Thermo Fisher Scientific) and LyticOS (Isoprime, Cheadle, UK). Computation was based on the comparison with the repeatedly measured standard reference gas (CO_2). Calculated $\delta^{13}C$ values are presented in parts per mil (‰), relative to the Vienna PeeDee Belemnite (V-PDB) international standard.

Batches were calibrated using a linear calibration curve (average R2 = 0.996 ± 0.002 in 16 batches) based on expected versus measured $\delta^{13}C$ values of n-alkanes and n-alkanoic acid esters from international standards (Indiana A7 and F8-3 mixtures). The accuracy of the instrument was determined on n-alkanoic acid methyl esters of known isotopic composition (Indiana F8-3, 18 measurements). The mean and standard deviation of these compounds were −29.89 ± 0.1‰ for $C_{16:0}$ (reported mean value vs, V-PDB −29.90 ± 0.03‰) and −23.41 ± 0.06‰ for $C_{18:0}$ (reported mean value vs. V-PDB −23.24 ± 0.13‰). The precision was based

on a laboratory standard mixture regularly injected between samples (108 measurements). Alkanoic acids mean ± SD values were −30.70 ± 0.15‰ for $C_{16:0}$ methyl ester and −26.44 ± 0.15‰ for $C_{18:0}$ methyl ester. After this analysis, values were corrected for the methylation of the alkanoic acids that occurred during acid extractions. Corrections were performed using a mass balance formula to compare the values with a standard mixture of $C_{16:0}$ and $C_{18:0}$ fatty acids of known isotopic composition, which was already included in each batch during the acid extractions. Modern references were adjusted according to the atmospheric $\delta^{13}C$ variations between the Early Holocene Epoch and the present to better interpret the archaeological data (Hellevang and Aagaard, 2015).

7. Results

Lipid concentrations from the acid extracted fraction ranged from 3 to 360 μg g^{-1}, with a mean of 57.4 μg g^{-1}. Only one sample (P25-1.19) yielded lipids below the interpretable limit of >5 μg g^{-1} (Evershed, 2008a) and was excluded from further analysis (Supplemental Table 3). Lipid profiles exhibited a mixture of aliphatic compounds comprising a distribution of saturated fatty acids ($C_{8:0}$–$C_{30:0}$), monounsaturated fatty acids ($C_{14:1}$–$C_{24:1}$), fatty alcohols (C_{12}–C_{32}), α, ω-dicarboxylic acids (C_8–C_{14}), and branched and linear fatty acids $C_{15:0}$ and $C_{17:0}$. Overall, from the lipid distributions and biomarker identification it was possible to distinguish between the processing of aquatic and plant commodities in the El Vergel pots.

7.1. The El Vergel pottery and its use for processing aquatic organisms

Four El Vergel pots (8%) had a full range of aquatic biomarkers comprising at least one of the three main isoprenoid acids (4, 8, 12-trimethyltridecanoic acid, pristanic, or phytanic acid), and a homologous series of C_{18}, C_{20}, and, in one sample, C_{22} APAAs. These biomarkers are established indicators for the processing of aquatic resources (Lucquin et al., 2016b; Evershed et al., 2008). APAAs are only formed by the protracted heating ⩾ 200°C of $C_{18:x}$, $C_{20:x}$, and $C_{22:x}$ unsaturated fatty acids that are found in plants and terrestrial animals but mainly in aquatic commodities (Appendix Figure 15.1) (Bondetti et al., 2020; Evershed et al., 2008; Hansel et al., 2004) and therefore must be derived from cooking events. The ratio between the two phytanic acid diastereomers, 3S, 7R, 11R, 15–phytanic acid (SRR), and 3R,7R,11R,15–phytanic acid (RRR), helps discriminate between ruminant and aquatic products. A high contribution of the SRR–diastereomer (i.e., 76% ± 16.6) is likely derived from

aquatic animal tissue (Appendix 5) (Lucquin et al., 2016b). An SRR–phytanic acid diastereomer contribution higher than 70% in three of these four samples further supports an aquatic origin. The absence of freshwater commodities on the Island during the Late Ceramic period likely reflects a marine origin for the aquatic resources.

7.2. Molecular evidence for terrestrial commodities

Plant commodities were the most representative resources identified in the El Vergel pots. Nineteen samples (38%) had a distribution of even over odd-numbered very long-chain fatty acids and odd over even-numbered middle- and long–chain alkanes, characteristic of the epicuticular wax of plant leaves and stems (Dunne, 2021; Diefendorf et al., 2011; Dove and Mayes, 2006). Very long-chain fatty alcohols with an even over odd-numbered carbon chain distribution, characteristic of plant commodities (Dove and Mayes, 2006), and plant sterols were identified in several samples.

Several pots (n = 16, 32%) contained APAAs with 18 carbon atoms (APAA-C_{18}) but lacked the C_{20} and C_{22} ω-(o-alkylphenyl) alkanoic acids. APAA-C_{18} can form from a wide range of plant and animal products, but the distribution of isomers (A-I) can provide further discrimination (Appendix Figure 15.1) (Bondetti et al., 2020). Samples in Figure 4.12 had an APAA-C_{18} E/H isomer ratio, possibly reflecting **(1)** cereals, fruits, and non-leafy vegetables; **(2)** terrestrial and aquatic animals; and **(3)** leafy plants (Dolbunova et al., 2023; Bondetti et al., 2020). Mixing cereals, fruits, and non-leafy vegetables with leafy plants may produce an intermediate E/H ratio similar to an animal origin (Bondetti et al. 2020). Only one of six samples with intermediate E/H values had a complete set of aquatic biomarkers; the rest lacked any evidence for processing aquatic or terrestrial animal products. The identification of plant epicuticular wax lipid distributions in four of these samples might indicate a plant origin.

Three samples with a full set of aquatic biomarkers had high APAA-C_{18} E/H ratios similar to those found in heated local reference samples of cereals, fruits, and non-leafy vegetables (Figure 4.12). This evidence may indicate the use of aquatic and plant products in the same container.

A palmitic over stearic acid ratio (P/S) $\leqslant 1$ identified in 46% of the samples may indicate that terrestrial animals were exploited in the El Vergel pots (Romanus et al., 2007). However, the higher solubility of $C_{16:0}$ compared to $C_{18:0}$ may alter the P/S ratio in animal tissue (Whelton

et al., 2021). Monoacylglycerols and diacylglycerols were present in a small number of solvent-extracted samples, whereas no triacylglycerols were identified. A distribution of $C_{15:0}$ and $C_{17:0}$ branched and linear fatty acids was found in most of the El Vergel samples. Their presence is associated with bacterial lipids from the rumen of polygastric animals (Evershed et al., 2002). However, they can also be linked to soil bacteria contamination, making them unspecific to ruminant fat (Dudd et al., 1998). An SRR-diastereomer contribution below aquatic values may indicate the processing of ruminants in only one El Vergel pot (Lucquin et al., 2016b). Cholesterol was detected in some samples in small amounts; however, we cannot ascertain its archaeological origin due to the absence of clear bio-hydrogenated and oxidation derivatives (Hammann et al., 2018).

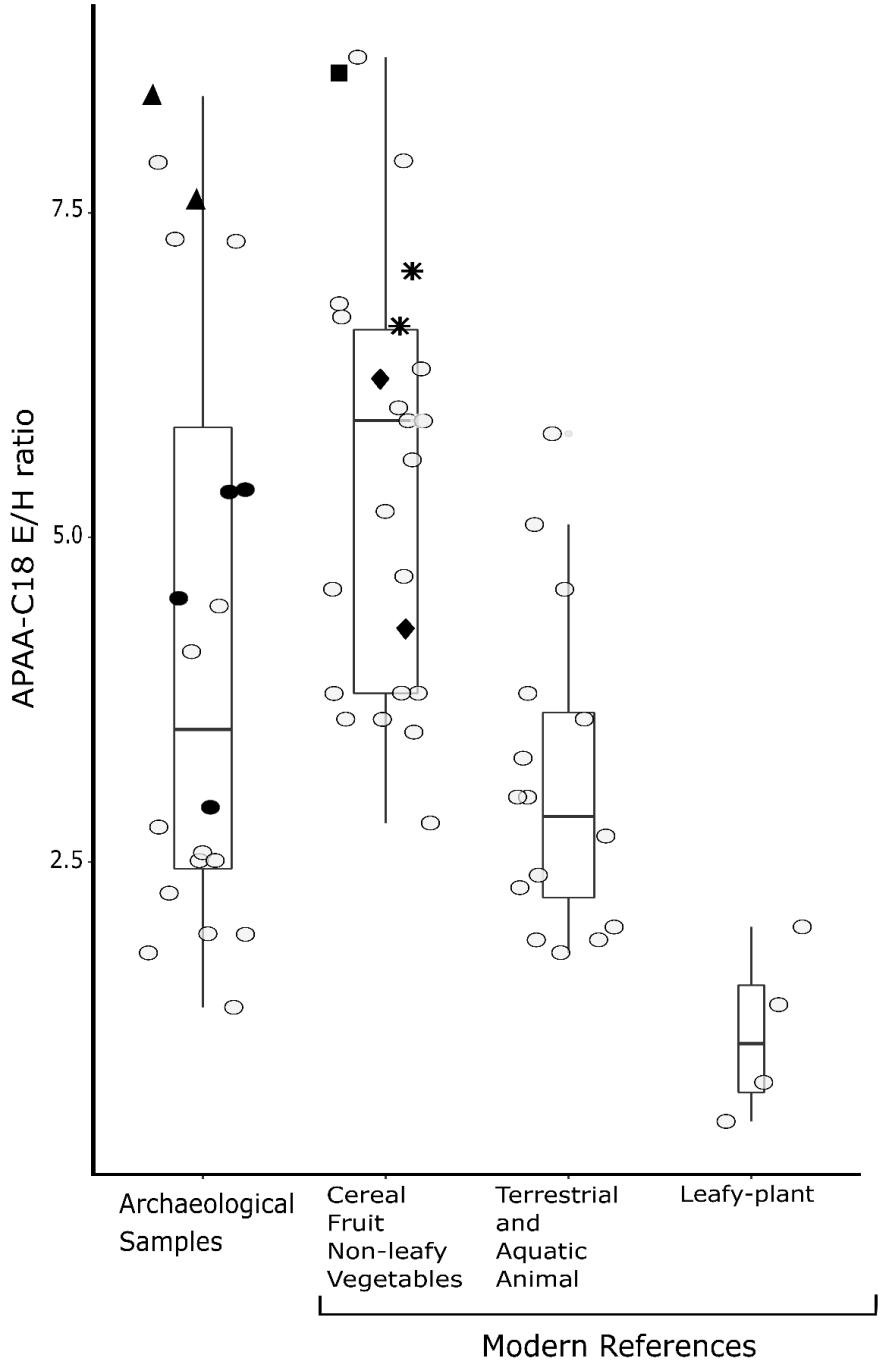


Figure 4.12. Boxplots of the APAA–C18 E/H ratio of Mocha Island samples. Black circles represent samples with a full set of aquatic biomarkers. The black triangles are samples showing ^{13}C enrichment of their residues consistent with a C_4 origin. Black square, asterisks, and diamonds correspond to modern references for Chilean hazelnut, maize, and quinoa, respectively. References are based on Bondetti and others (2020) and this study (Appendix 4). A jitter plot is used to avoid overlapping and improve the visualisation of the data. Figure created by the author.

7.3. Single-compound stable carbon isotope analysis

To further identify the commodities' origin, we measured the stable carbon isotope values of the palmitic ($\delta^{13}C_{16:0}$) and stearic ($\delta^{13}C_{18:0}$) alkanoic acids extracted from the pots and then compared these values to modern and archaeological references of marine, C_3 plants, maize, and ruminant animals, including South American camelid *Lama glama* (Appendix 3).

Most pots (74%) had $\delta^{13}C_{16:0}$ and $\delta^{13}C_{18:0}$ values plotting within the ranges for C_3 plants. Three of the four samples showing a complete set of aquatic biomarkers were more depleted in ^{13}C relative to marine references, plotting close to C_3 plant values (Figure 4.13). The E/H ratios of these samples coincided with the use of cereals, fruits and non-leafy vegetables, probably indicating that plants and aquatic products were used in these pots.

It is likely that maize was processed in some pots with high $\delta^{13}C$ values relative to C_3 plant references (Figure 4.13). Extracts from these pots had high APAA-C18 E/H ratios coinciding with cereals, fruits, and non-leafy vegetables; a high P/S ratio; and a fatty alcohol distribution characteristic of plant commodities. The identification of C_{32} alcohol (C_{32}-OH) in a high relative abundance can be associated with panicoid grasses like maize (Reber and Evershed, 2004b). Although C_{32}-OH was found in low abundance in one sample, its association with panicoid plants is debatable (Appendix Figure 15.2). The different $\delta^{13}C$ values in these samples might indicate the incorporation of other commodities, possibly C_3 plants, in varying proportions.

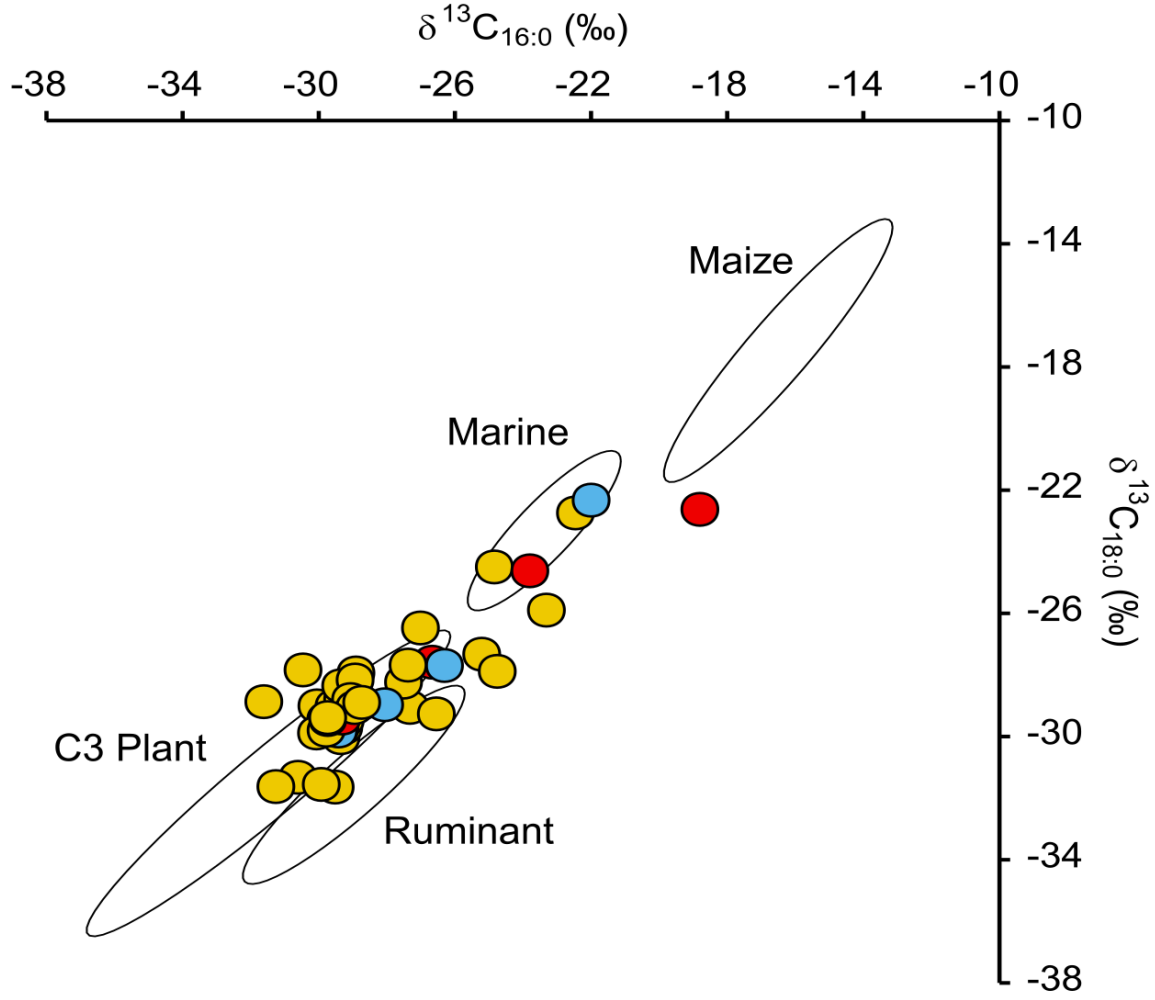


Figure 4.13. Scatterplot of compound-specific $\delta^{13}C$ analysis of the main alkanoic acids (X-axis = $\delta^{13}C_{16:0}$; Y-axis = $\delta^{13}C_{18:0}$) extracted from the El Vergel pots. Blue circles indicate the four samples with a full range of aquatic biomarkers. The red circles highlight samples with a high APAA-C_{18} E/H associated with cereals, fruits, and non-leafy vegetables (Bondetti et al. 2020). The 68% confidence ellipses are based on reference values published in the literature and from this study (Appendix 3). Figure created by the author.

Nine samples had $\Delta^{13}C(C_{18:0–C16:0})$ offsets coinciding with ruminant carcass fat references (Dolbunova et al., 2023; Copley et al., 2003). These samples yielded plant lipids and some aquatic biomarkers; however, they lacked compounds typically documented in ruminant fats, such as SRR% <64 and acylglycerol distributions. They possibly reflect the mixing of plant and animal products that can result in low $\Delta^{13}C(C_{18:0–C16:0})$ values (Taché et al., 2021; Cramp et al., 2019; Hendy et al., 2018). To investigate further, we applied a simple

linear mixing model based on averaged $\delta^{13}C$ values of modern reference endpoints, adopting a progressive 10% increment (Figure 4.14). The hypothetical mixing lines indicate the possible use of marine and terrestrial animal products or C_3 and C_4 plants in these pots.

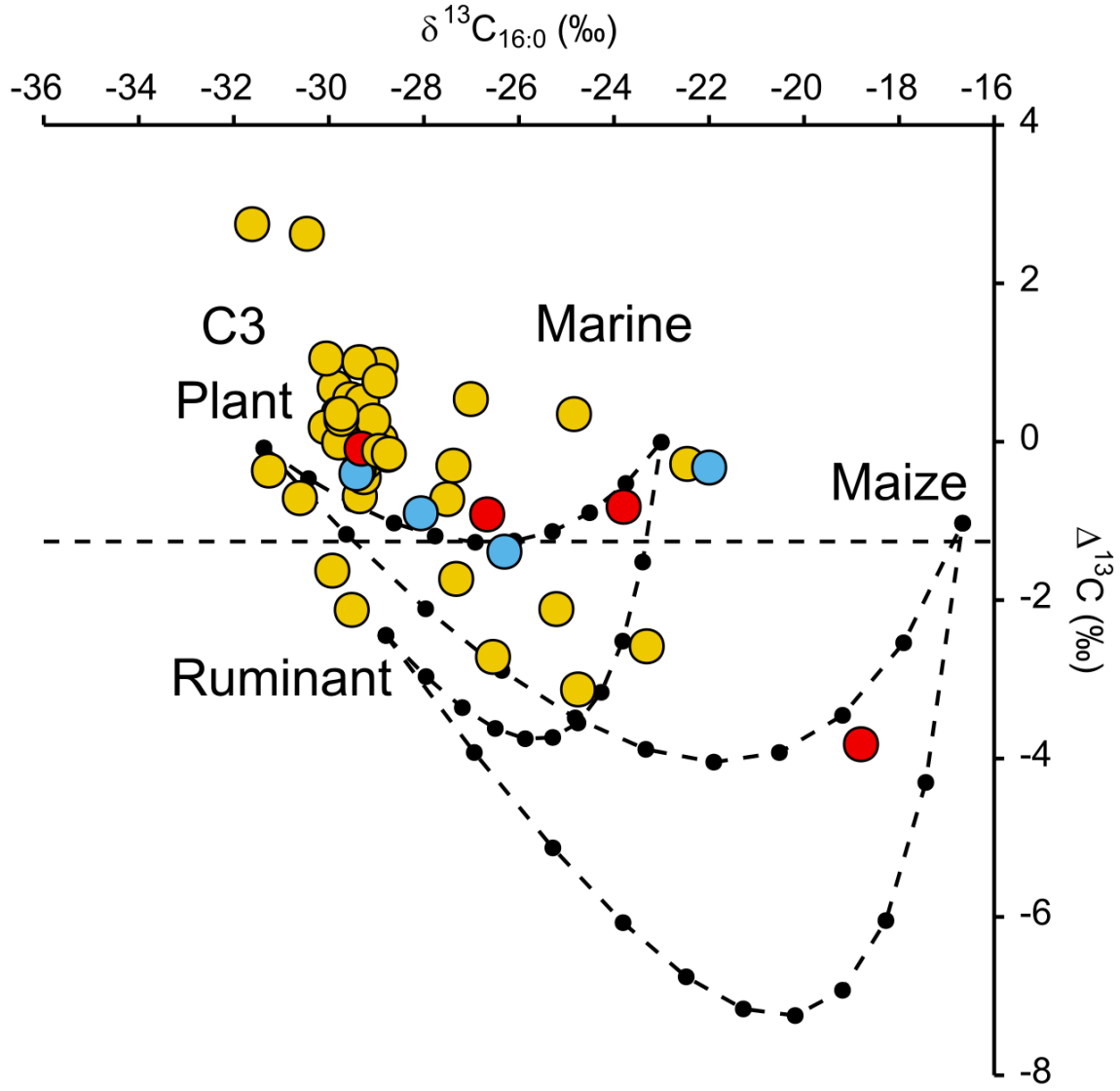


Figure 4.14: Compound-specific $\delta^{13}C$ analysis of Mocha Island samples plotted according to their $\delta^{13}C_{16:0}$ value against $\Delta^{13}C_{(C18:0-C16:0)}$. Blue circles indicate the four samples with a full set of aquatic biomarkers. The red circles highlight the samples with an APAA-C_{18} E/H ratio associated with cereals, fruits, and non-leafy vegetables (Bondetti et al. 2020). Average $\delta^{13}C$ isotopic endpoints were built using modern and archaeological references from published data and this study (Appendix 3). Mixing lines with 10% increments connecting the endpoints were calculated based on the mean relative amount of each alkanoic acid present in the products. Data were gathered from the USDA database. The dashed line indicates a −1.26‰ threshold set for the identification of ruminant carcass fat, as in Dolbunova et al., (2023). Figure created by the author.

8. Discussion

Organic residues from pottery artefacts demonstrated that local marine products were certainly part of the El Vergel farmers' foodways, which aligns with other evidence of marine resources exploitation on the island, such as faunal remains and fishing gear (fish hooks and net sinkers). This coastal adaptation gave the inhabitants access to a rich source of animal protein, compensating for the lack of large mammals on the island. Together our results highlight the overall importance of marine resources in the region and as a critical component of early island colonisation efforts. Coastal adaptation was essential for island colonisation and perhaps was more widespread than previously thought, as evidenced in the mainland El Arenal-1 site in Punta Lavapié (Contreras et al., 2005).

The single-compound $\delta^{13}C$ analysis and more robust molecular evidence allowed us to reinterpret previous results indicating the use of pots for processing terrestrial animal fat (Roa et al., 2021). The exploitation of translocated ruminants was underrepresented in the El Vergel pots, contradicting the abundance of camelid remains found on Mocha Island (Roa et al., 2021). According to the chronicles of the first Europeans arriving on Mocha Island and southern Chile, camelids were important for local communities, which would have limited their availability for trading and consumption (Van Meurs, 1993; Van Noort, 1600, cited in Ijzerman 1926:57–58; Rosales, 1674, cited by Barros Arana, 1877, p. 324-325). Their exploitation as a source of animal protein cannot be ruled out because other culinary practices, such as roasting and drying the meat, were possibly used.

Marine products were processed in the pots alone or mixed with C_3 plants. The $\delta^{13}C$ values of these samples demonstrate that C_3 plants were exploited intensively, given that oily and lipid-rich marine products are likely to increase $\delta^{13}C$ values. Our results thus reveal that plants were the basis of the foodways of Mocha Island inhabitants. The use of leaves and stems and non-leafy plants, such as cereals and fruits, and non-leafy vegetables in the pots are concordant with the diversity of plants exploited by local Mocha Island farmers (Godoy-aguirre, 2018; Roa et al., 2015). Evidence for the epicuticular wax of leaves and stems was present in most El Vergel pots. The Mapuche people from southern Chile traditionally use the leaves and stems of some plant species found on Mocha Island for medicinal purposes. However, the culinary use of leaves with other commodities such as cereals and animal meat has also been documented in the past, including the preparation of various stews and other local dishes (Gay 2018, 197; Rosales, 1674, cited by Barros Arana, 1877, p. 153–154).

The lipid distribution of grains, seeds, and fruits is characterized by a low abundance of very long-chain fatty acids and alkanes, along with a P/S ratio >1.3 (Dunne, 2021; Dunne et al., 2016). Mixing these products with leaves and stems will likely produce a distribution similar to that seen for plant epicuticular wax, hence overshadowing their presence. The APAA-C_{18} E/H ratio and $\delta^{13}C$ values revealed the exploitation of C_3 cereals, fruits, and non-leafy vegetables and their mixing with leafy plants; one may presume that the former group was overshadowed in the pots by the latter. The whole spectrum of cereals, fruits, and non-leafy vegetables may have included domestic and wild plants traditionally used in southern Chile. Chilean wineberry, Chilean strawberries, Chilean hazelnut, and *Madia sativa* seeds are wild plants used in pottery containers for roasting, preparing alcoholic beverages, and extracting oil (Pino Ramos et al., 2019; Godoy-aguirre, 2018; Schmeda-Hirschmann, 1995). In the case of domestic plants, this group was dominated by beans, maize, and quinoa. However, chronicles mentioned the consumption of potatoes by Mocha Island inhabitants (Van Meurs, 1993; Van Noort, 1600, cited in Ijzerman 1926:56–58), although this tuber has not yet been identified in the archaeological record of the island.

Quinoa seeds were the most abundant plant remains found at every Mocha Island site (Roa et al., 2021), and quinoa likely comprises a major part of the C_3 plant residues found in the pots. Its use in southern Chile was reported since the Early Ceramic Period (1550–950 cal BP) from wild-type quinoa managed by hunter-gatherer groups (Roa et al., 2018; Campbell and Quiroz, 2015; Adán and Mera, 2011). Its ability to adapt to different environments without much maintenance investment may have facilitated its early management and incorporation into local foodways (Planella, 2019).

The recent arrival of maize on Mocha Island and southern Chile (around 950 cal BP) would have required a period of experimentation and adaptation to the local temperate conditions. This might have influenced the degree to which the El Vergel farmers exploited maize; it was possibly restricted to the production of fermented beverages. The two pots used for processing maize likely also included C_3 plants. The different $\Delta^{13}C$ offsets between the two samples possibly indicate the mixing of maize with C_3 plants with varying proportions of $C_{16:0}$ and $C_{18:0}$ alkanoic acids. Incorporating other starchy plants into the pot may have enhanced the fermentation process for producing maize-based beverages (Arriaza et al., 2016). From the residue data, we can argue that maize was not consumed as a staple food by Mocha island farmers; otherwise, we would expect more samples showing high $\delta^{13}C$ values, possibly with a negative $\Delta^{13}C$ offset due to the mixing of C_3 and C_4 plants, as indicated by our simple linear model. Therefore, we suggest that the high $\delta^{13}C$ and relatively low $\delta^{15}N$ values for

human bone collagen described in previous studies might not necessarily represent a preferential consumption of maize over aquatic resources. Low trophic-level marine organisms such as molluscs might also have been preferentially consumed, an interpretation supported by the faunal evidence from the island.

9. Conclusion

The permanent settlement of Mocha Island by the El Vergel farmers required the translocation of domestic crops and tamed animals. Previous studies referred to the exploitation of marine resources as only a secondary activity and stressed the continuity of farming to support the dense populations on the island. Our organic residue analysis revealed that the El Vergel pottery of Mocha Island was mainly used for exploiting plant commodities and marine products. Plant commodities were the basis of the local diet; however, maize was not a staple and might have been mainly destined for the production of fermented beverages. Plant products were mixed with aquatic resources, although different types of plants were also simultaneously exploited. Marine commodities were a source of animal protein and may have compensated for the limited availability of terrestrial animal products during certain seasons and complemented the exploitation of translocated ruminants.

The use of pottery indicates that local and translocated resources were an important part of the foodways of the El Vergel coastal farmers from Mocha Island. Local marine resources complemented translocated domestic plant commodities such as maize and quinoa, sustaining a mixed economy where farming, coastal foraging, hunting, and fishing were practised. Perhaps it was only when this mixed economy developed that the dense populations could be supported on Mocha Island following its settlement by the El Vergel farmers. More studies focusing on mainland sites are needed to characterise better the foodways and socioeconomic strategies of the El Vergel farmers inhabiting coastal and inland areas.

10. Acknowledgments.

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Chapter 5: Evaluating the culinary significance of maize in the Araucanía, southern Chile: evidence from organic residue analysis of pottery.

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2. Abstract

Maize, one of the primary crops cultivated in South America, has achieved significant interest in regional archaeology. However, the study of maize in regions considered peripheral to major centres of agricultural production, such as southern Chile, has received minimal attention. Southern Chile is the southernmost point for the dispersal of maize cultivation in the Americas, with archaeological evidence dating back *ca*. 950 cal BP. Despite the manifest presence of maize, our knowledge of its culinary and economic importance for pre-colonial societies from Southern Chile is scarce. In this study, we extracted and analysed organic residues from 188 pottery sherds to explore the potential significance of maize in local foodways over the transition from the Early (1550 - 950 cal BP) to the Late Ceramic (950 - 400 cal BP) Period.

Biomolecular and carbon-stable isotope ($\delta^{13}C$) analyses of lipids indicate that maize was not a staple. Instead, our findings suggest that maize had a potentially prominent role in preparing fermented beverages consumed in important socio-political gatherings and feasts.

Keywords: Maize; Organic residues analysis; pottery; South America; feasts; fermented beverages

3. Introduction

The significance of maize (*Zea mays*) production in pre-colonial Western South America has traditionally been a major focus of archaeological research. Evidence from Paredones and Huaca Prieta sites in the Peruvian Pacific coast indicates that maize was first adopted ca. 6700 cal BP (Vallebueno-Estrada et al., 2023; Grobman et al., 2012), being one of the earliest evidence for maize adoption in South America, after the Llanos del Mojo maize in lowland Bolivia, dating back ca. 6900 cal BP (Iriarte et al., 2020; Lombardo et al., 2020). Maize was incorporated as part of western South American foodways, showing different levels of reliance and becoming a staple in several regions ca. 2000 cal BP (Delgado, 2021; Washburn et al., 2020; Alfonso-Durruty et al., 2017; Logan, Hastorf and Pearsall, 2012; Falabella, Planella and Tykot, 2008; Bonzani and Oyuela-Caycedo, 2006; Chávez and Thompson, 2006; Staller and Thompson, 2002). Nevertheless, while current research has primarily concentrated on well-documented centres of maize production, peripheral regions, such as Southern Chile, the southernmost limit of maize cultivation in the Americas, are poorly understood.

The area between the Bío Bío and Toltén rivers in Southern Chile, historically known as the Araucanía (36º - 39º Lat S), saw the late arrival of maize ca. 950 cal BP evidenced by the presence and carbon-dating of botanical remains (Roa et al., 2018 Adán and Mera, 2011). At this time, the intensification of food production, population growth and the emergence of social differentiation defined the transition from hunter-gatherer societies of the Early Ceramic Period (ECP: 1550 - 950 cal BP), mainly represented by the Pitrén cultural complex, to Late Ceramic Period (LCP: 950 - 400 cal BP) farming societies from the El Vergel cultural complex, along the coast (Roa et al., 2021; Campbell, 2020; Campbell and Pfeiffer, 2017; Campbell, 2015; Silva, 2010; Quiroz and Sánchez, 2010; Contreras et al., 2005) and Central Valley (Silva, 2014b; Adán and Mera, 2011; Rossen, 2011; Dillehay, 2007; Dillehay et al., 2007). Notably, the Central Valley, located between the Coastal and Andean ranges (Figure 5.1),

exhibits ideal conditions for maize agriculture (Sánchez, 2020). Early chronicles from the 16th century described this area as densely populated by farming communities engaged with the cultivation of maize and other major crops (Bibar, 1558, cited in Leonard, 1966, p. 153; Mariño de Lobera, 1595, cited in Escobar, 1865, p. 124). However, the direct evidence for pre-colonial consumption and early adoption of maize in the Central Valley is constrained to a low abundance of macro remains (Roa et al., 2018; Adán and Mera, 2011; Dillehay et al., 2007) and the sporadic finding of micro plant remains (Ramírez Funes, 2023, 2022; Godoy-Aguirre, 2017; Iriarte, 2014).

The poor preservation of organic remains due to soil acidity and climatic conditions in the Araucanía has limited the potential of carbon ($\delta^{13}C$) and nitrogen ($\delta^{15}N$) stable isotope analyses of human remains, thus restraining our understanding of maize intake in southern Chile (Campbell et al., 2020). In addition, only a few individuals from the Early Ceramic Period in the Central Valley have been analysed for stable isotopes, revealing a diet sustained on terrestrial animals and C_3 plants (Campbell et al., 2020). No $\delta^{13}C$ and $\delta^{15}N$ analyses on human remains exist for the Late Ceramic Period in the Central Valley. These constraints prevent a more detailed exploration of the significance of maize in local foodways by bioarchaeological means.

The extraction of organic residues from ceramic containers is a viable approach to directly look into vessel content and assess past foodways, even under conditions of poor organic preservation (Roffet-Salque et al., 2017). Indeed, the extraction and analysis of lipids from pre-colonial pottery from south and north America has helped to elucidate the use of maize in local foodways (Admiraal et al., 2023; Lantos et al., 2015; Reber and Evershed, 2004a, 2004b; Reber et al., 2003). Particularly, in southern Chile, the analysis of organic residues from Late Ceramic Period potsherds from Mocha Island indicated that maize was not a staple food and that its use was possibly confined to the preparation of fermented beverages (Montalvo-Cabrera et al., 2024). The ubiquitous presence of pottery sherds in archaeological contexts from southern Chile makes the analysis of organic residues an attractive approach to reassessing the relevance of maize in local foodways.

By analysing a total of 188 Early (ECP) and Late (LCP) Ceramic Period potsherds from two domestic sites located in the Central Valley of the Araucanía (Figure 5.1), we aim to clarify the importance that maize had in local foodways since its adoption and whether it played a significant role in the socio-political and economic changes characterising the transition from the Early to the Late Ceramic Period. We hypothesise that maize was not included as a staple food because available wild resources and C_3 domesticated crops might have had a higher

significance on local foodways. This is the first time organic residue analysis has been used on pottery artefacts from the Central Valley of the Araucanía to assess maize's significance in local foodways of pre-colonial groups.

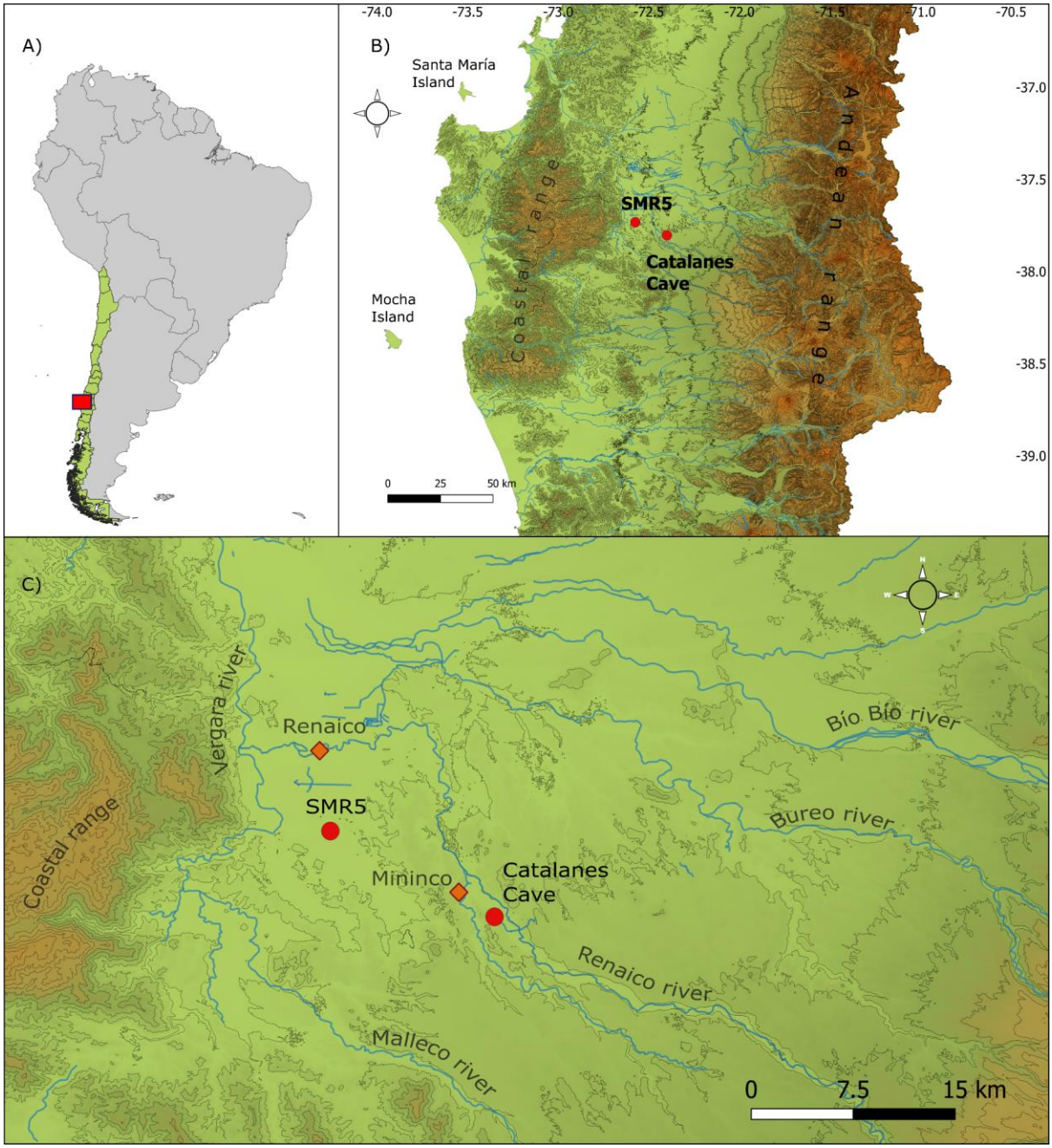


Figure 5.1: ***A-B)*** *Study region showing the position of Cueva de los Catalanes and SMR5 in Southern Chile. Both sites are located in the Central Valley, between the Coastal and Andean range.* ***C)*** *Sites and associated rivers and nearby cities of Mininco and Renaico. Figure created by the author.*

4. The Central Valley and the adoption of maize

The geomorphological characteristics of the Araucanía have allowed particular environmental conditions in the Central Valley. The high altitude of the Coastal range attenuates oceanic influences from the Pacific, consequently yielding warmer seasons and overlapping Mediterranean and Temperate macroclimates (Amigo and Ramirez, 1998). These conditions permit the development of a mixed forest with a wide diversity of plant and animal species (Gutiérrez et al., 2020).

The mixed forest gives access to a diversity of edible plant organs, composed of various wild tubers, fruits, grains, nuts, shoots, and leaves, which are still part of local culinary traditions maintained by the Mapuche people (Bannister et al., 2012; Montecino, 2005, 2009; Myers et al., 2000; Smith-Ramírez, 1998). Hunter-gatherers from the Early Ceramic Period, mainly represented by the Pitrén cultural complex, exploited resources from the mixed forest while experimenting with plant management (Roa Solís et al., 2024; Mera and Munita, 2021; Campbell, Roa and Santana-Sagredo, 2018b). Beans (*Phaseolus vulgaris*) and quinoa (*Chenopodium quinoa*) were initially managed by these groups, and likely maize as well (Roa Solís et al., 2024; Campbell et al., 2018b; Roa et al., 2018; Adán and Mera, 2011). However, it was not until the Late Ceramic Period that maize seemed to become well integrated into the foodways of farming groups of the El Vergel cultural complex (Roa et al., 2018). During this period, food production was intensified, leading to demographic growth, increased sedentarism and socio-political changes towards the formation of a trans-egalitarian society and the emergence of social differentiation (Campbell, 2020; Campbell and Quiroz, 2015; Dillehay, 2007)**.**

Southern Chile is the southernmost point for maize dispersion and cultivation in the Americas (Campbell et al 2020; Roa et al 2018). Given that maize is inherently suited to warmer climates, its propagation into southern Chile meant its adaptation to more temperate and wet environmental conditions. However, the influence of the warmer Mediterranean macroclimate in the Central Valley of the Araucanía offers suitable conditions for agricultural developments (Amigo and Ramirez, 1998). These circumstances are believed to have facilitated maize cultivation in the region, subsequently enabling its spread to more temperate areas in southern Chile.

In the Central Valley of the Araucanía, maize was integrated into the foodways of ceramic-using communities who engaged in the cultivation of domestic plants such as quinoa, beans, pumpkin (*Curcubita* spp.), hunting, and the gathering and management of wild plants from the mixed forest (Roa Solís et al., 2024; Campbell et al., 2020; Roa et al., 2018; Iriarte, 2014). Historical and ethnographic records have indicated that pottery containers were used in the preparation of different dishes including maize (Montecino, 2005). Some of these preparations included a type of polenta known as *chuchoca* (Molina 1776, 134), as well as a range of stews in which maize, alongside other domesticated and wild plants, was cooked with meat, possibly sourced from guanaco (*Lama guanicoe*), pudú (*Pudu puda*), birds and freshwater resources (Gay, 1873, cited in Jean and Milos, 2018, p. 197; Coña, 2017, 46-47; Rosales, 1674, cited in Barros Arana, 1877, p.153; Gay 1865, 97) . The preparation of maize-based humitas was also described, which involved wrapping ground maize grains in the husk and boiling them subsequently within a pot (Núñez de Pineda y Bascuñán, 1673, cited by Cayuqueo, 2022, p. 46; Molina 1776, 134).

However, maize was most often documented historically for its role in the production of fermented beverages known as *chicha* (beer) or muday. *Chicha* denoted significance in gatherings and ceremonies where socio-political relations were mediated through its consumption (de Ercilla, 1578, 42; Núñez de Pineda y Bascuñán, 1673, cited in Cayuqueo, 2022, p. 50; Bibar, 1558, cited in Leonard, 1966, p. 193). Large pottery containers, mentioned in early chronicles as "*tinajas*", were used in the storage of *chicha*, while smaller pots were implemented for communal consumption (Núñez de Pineda y Bascuñán, 1673, cited in Cayuqueo, 2022, p. 53; Rosales, 1674, cited in Barros Arana, 1877, p. 143). Ethnographic studies on the Mapuche people have noticed the resemblance between large containers or *fütrametawe*, with characteristic Late Ceramic Period pottery of the El Vergel cultural complex, possibly denoting a pottery tradition related to the production of alcoholic beverages (Alvarado, 1997). Ceramic containers were essential not only for serving and consuming *chicha* but also for its preparation, which involved cooking and fermenting previously grounded kernels (e.g. Gay, 1873, cited in Jean and Milos, 2018, p. 200-201; Coña 2017, 149; Rosales, 1674, cited in Barros Arana, 1877, p. 155). While maize-based *chicha* was frequently mentioned in early colonial documents, the significance of beverages produced from wild fruits and grains was notable within local communities (Núñez de Pineda y Bascuñán 1673, cited in Cayuqueo, 2022, p. 53; Rosales, 1674, cited in Barros Arana, 1877, p. 155).

5. Sites

In this study we investigate the presence of maize through the analysis of ceramic vessels from Cueva de los Catalanes and San Miguel de Renaico 5 (SMR5), both sites located in the Central Valley of the Araucanía. Cueva de los Catalanes is separated from SMR5 by 15 km, about 3.4 km southeast of the locality of Mininco, and 800 m west of the Renaico River (37º48' Lat S; 72º49' Long W) (Figure 5.1). The cave has an extension of 33 m, divided into an anterior and posterior chamber, with a secondary cave located *ca.* 3.5 m high at the side of the main cave (Figure 5.2) (Campbell et al. 2018b).

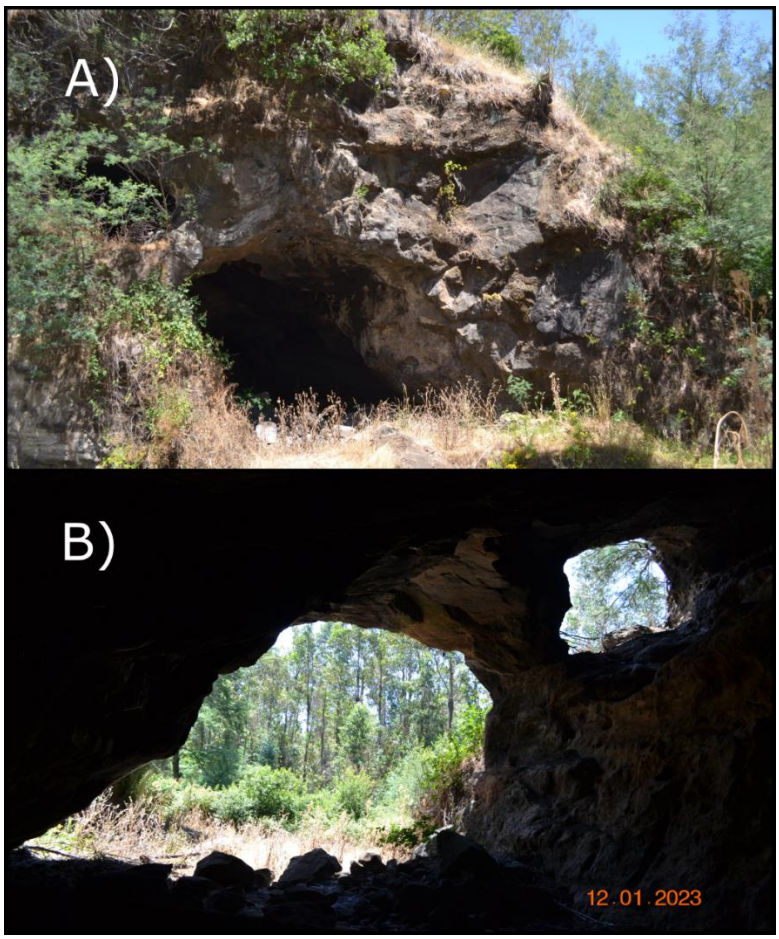


Figure 5.2: Cueva de los Catalanes. The figure shows A) a frontal picture of the cave, and B) a picture from the inside of the cave, exhibiting the main entrance and an alternative small cave situated ca. 3.5 m high. Original photography by the author. Figure created by the author.

Berdichewsky (1968) described the cave as a residential site occupied throughout the Ceramic Period. However, Menghin (1962) referred to its possible use during ceremonies and feasts. Ethnographic evidence underscores the religious significance of caves in ceremonies and gatherings and as places inhabited by mythical figures (Menghin 1962, 24; Latcham 1924, 226; Philippi 1886, 5). The identification of rock art, human and camelid bone remains, possibly from *Lama guanicoe*, and smoking pipe fragments might attest to the cave's importance as a ceremonial and ritualistic site; however, its use as a domestic setting cannot be ruled out. Smoking pipes are primarily associated with various socio-political and religious manifestations (Herreros, 2004); whereas historical records highlight the importance of camelids in the Araucanía, with their sacrifice and consumption reserved for specific celebrations (Guevara 1927, 303; Rosales 1674, cited in Barros Arana, 1877, p. 145; Hawkins 1622, cited in Bethune, 1847, p. 98).

New excavation campaigns in 2016 allowed the identification of a large quantity of monochrome pottery, indicating the possible presence of the Pitrén and El Vergel cultural complex; whereas the obtainment of radiocarbon dates attested to the cave's occupation during the Early and Late Ceramic Period (Table 5.1)(Campbell, 2018; Santana-Sagredo, 2018; González, 2017). Soot marks and charcoal remains denoted the possible use of pottery for cooking practices (Godoy-Aguirre, 2017). Plant macro remains and phytoliths recovered from the cave's sediment demonstrated a continuous use of local wild plants throughout the Ceramic Period (Roa Solís et al., 2024; Roa Solís and Zurro, 2022; Roa et al., 2018). A single bean was identified inside the cave and carbon-dated to the Early Ceramic Period, while quinoa grains were present throughout the site's occupation (Campbell et al., 2018b; Roa et al., 2018). A single maize cob was identified in the upper stratigraphic levels, with ^{14}C dates assigned to the Late Ceramic Period, as well as evidence for the use of chilli (*Capsicum* sp.) during the LCP (Figure 5.3) (Roa Solís et al., 2024; Campbell et al., 2018b; Roa et al., 2018).

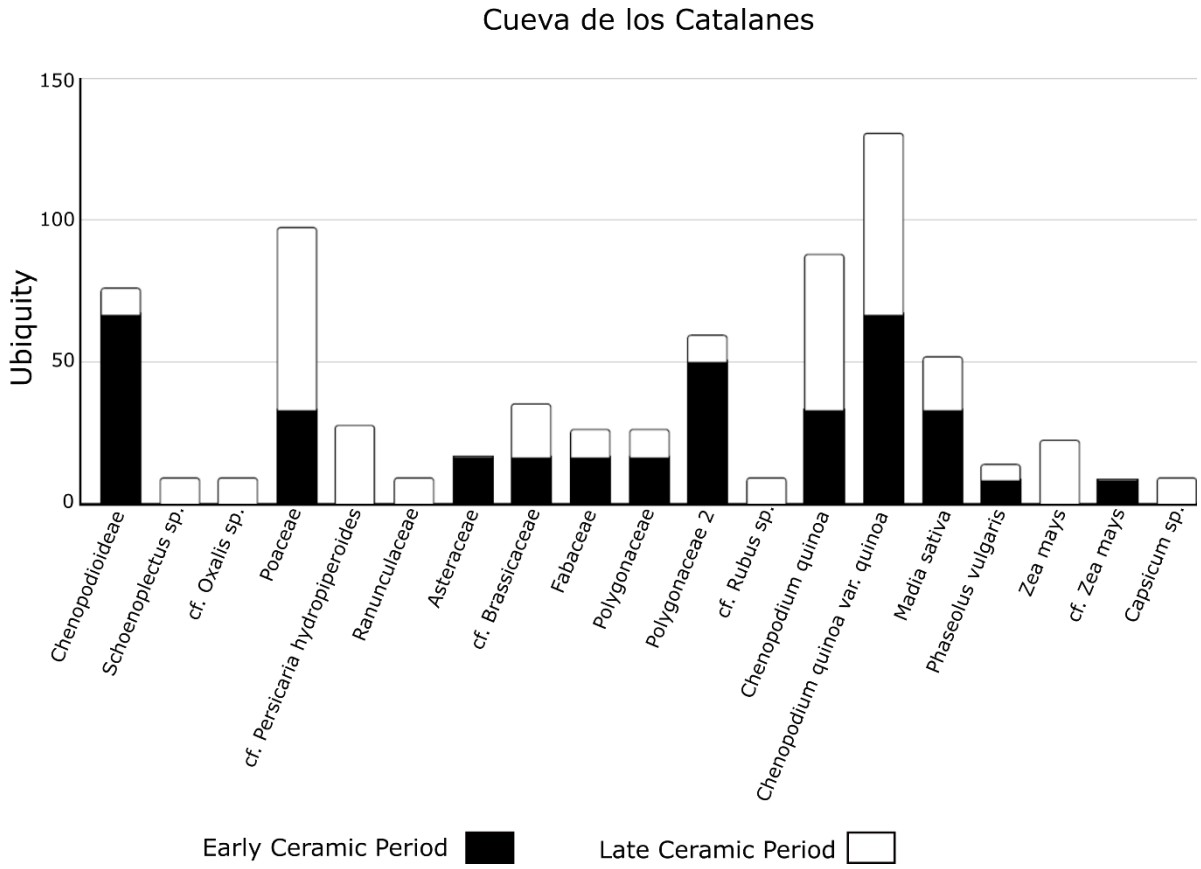


Figure 5.3: Bar chart showing the ubiquity of carpological remains for several domesticated and wild plant species. Data obtained from Roa Solís et al. (2024). Figure created by the author.

Table 5. 1: ^{14}C Dates from associated material from Cueva de los Catalanes.

Test pit	Depth (cm)	Laboratory code	Year	Material	Taxa	14C Age BP	Median probability cal yrs BP
P1	90-100	UGAMS 27385	2016	Seed	*Zea mays*	660± 20	590
	150-160	AA 111538	2018	Bone	Camelidae	1527±24	1392
	180-190	AA 111539	2018	Bone	*Canis lupus familiaris*	1547±24	1417
P2	130-140	UGAMS 27386	2016	Seed	*Phaseolus vulgaris*	1300± 25	1226
	160-170	UGAMS 27387	2016	Bone	Camelidae	1410± 25	1316
	130-140	AA 110999	2018	Bone	*H. sapiens*	972±23	853
	60-70	AA111000	2018	Bone	*H. sapiens*	178±23	183
	60-70	AA111537	2018	Bone	*Pudu puda*	242±37	199

Note: Data obtained from Campbell et al. (2020). Table created by the author.

In addition to the identification of camelid bones, medium-sized mammals such as *pudú* deer (*Pudu puda*) and Chilean fox (*Lycalopex* sp.) were also found, albeit in smaller quantities (Figure 5.4) (Zuñiga, 2018). Despite the site's proximity to the Renaico River, evidence for the exploitation of freshwater resources was scarce (Berdichewsky 1968, 78). The $\delta^{13}C$ and $\delta^{15}N$ values from one adult human bone from the Early Ceramic Period showed a diet mainly based on terrestrial resources; however, the lack of more evidence does not allow a proper interpretation of the population's diet (Campbell et al., 2028; Santana-Sagredo, 2018).

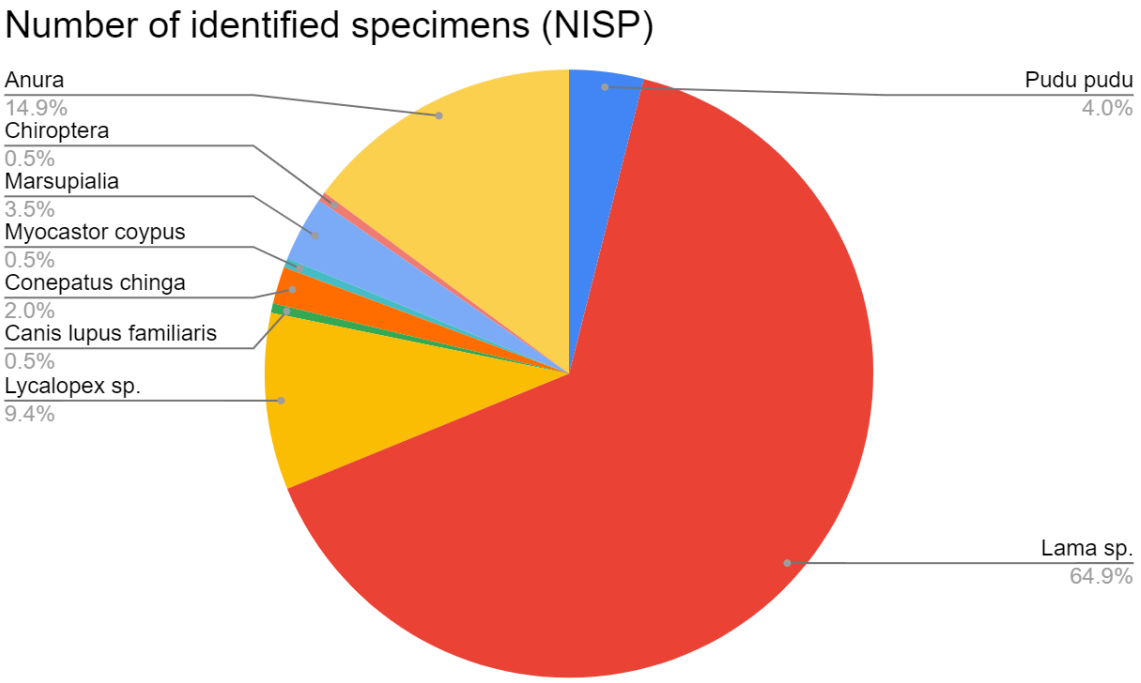


Figure 5.4: Number of identified specimens (NISP) from Cueva de los Catalanes. The ubiquity index was not available for this site. Figure created by the author.

San Miguel de Renaico 5 (SMR5) is one of the few open-air large residential sites systematically excavated in the Araucanía. It is 5.8 km South East from the locality of Renaico, and about 3.7 km East from the Vergara River (37º73' Lat S; 72º57' Long W). It encompasses an area of 1300 m^2, from which *ca.* 10% has been excavated, yielding large quantities of pottery fragments (Cortés, Dávila and Palma, 2018; Dávila et al., 2016). Diagnostic elements allowed the identification of pottery from the Pitrén and the El Vergel cultural complex, likely indicating a continuous occupation throughout the Ceramic Period (Dávila et al., 2016). However, bad organic preservation due to soil acidic conditions limited the possibility of obtaining radiocarbon dates (Campbell et al., 2018).

The identification of several smoking pipe fragments also indicates that ceremonies and social gatherings were possibly held on site (González, 2017; Herreros, 2004). This evidence aligns with early historical accounts describing the celebration of socio-political gatherings, religious ceremonies, and communal work festivities within or near residential sites (Núñez de Pineda y Bascuñán, 1673, cited in Cayuqueo, 2022, p. 46), and in specially designed areas (Mariño de Lobera, 1595, cited in Escobar, 1865, p. 124).



Figure 5.5: Map showing the location of San Miguel de Renaico 5 (SMR5), Araucanía Region, Chile. The area is currently used for agricultural production. Figure created by the author.

6. Materials and methods

A total of 128 potsherds from Cueva de los Catalanes (64 samples from each period) and 60 from SMR5 (30 from each period) were selected for organic residue analyses. Samples from Cueva de los catalanes were recovered from two test pits, P1 and P2 (Figure 5.6; Figure 5.7; Appendix 8); whereas in SMR5, samples were recovered from boreholes P17, P24, P28, P29, P30, P31, P32, P33, P34, and P35 (Figure 5.8; Appendix 10).

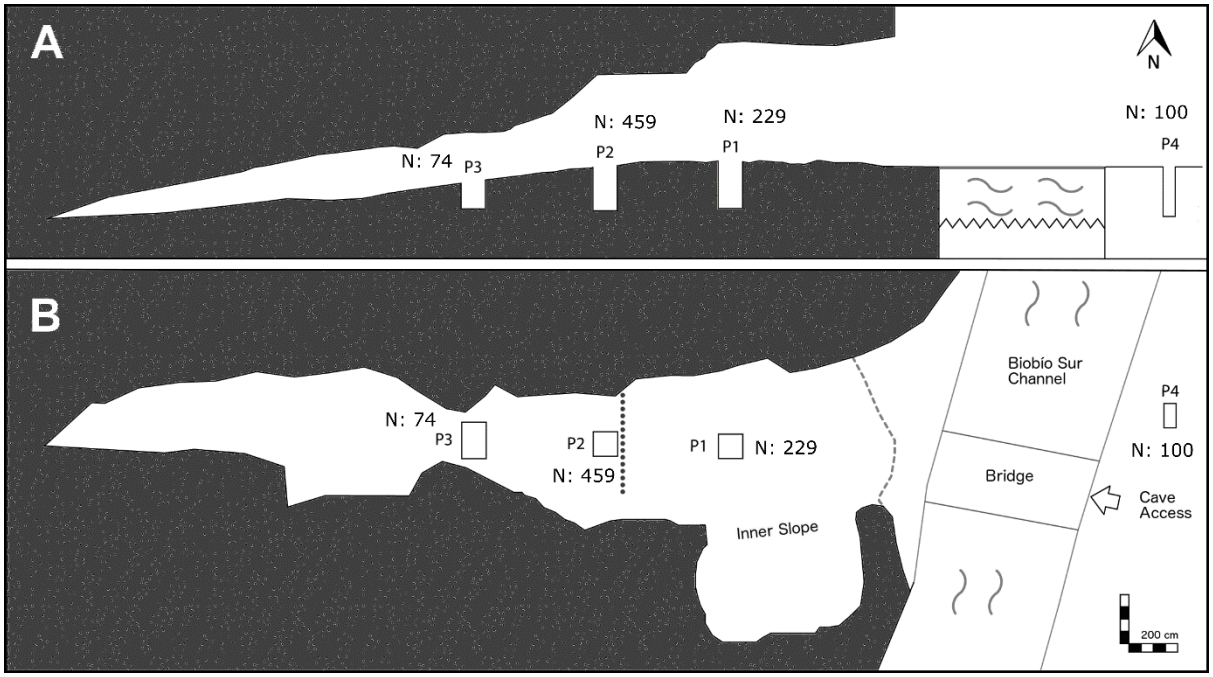


*Figure 5.6: Cueva de los Catalanes **A)** cross view and **B)** top view. N: indicates the number of potsherds recovered from each borehole. Image obtained and modified from Campbell et al. (2018).*

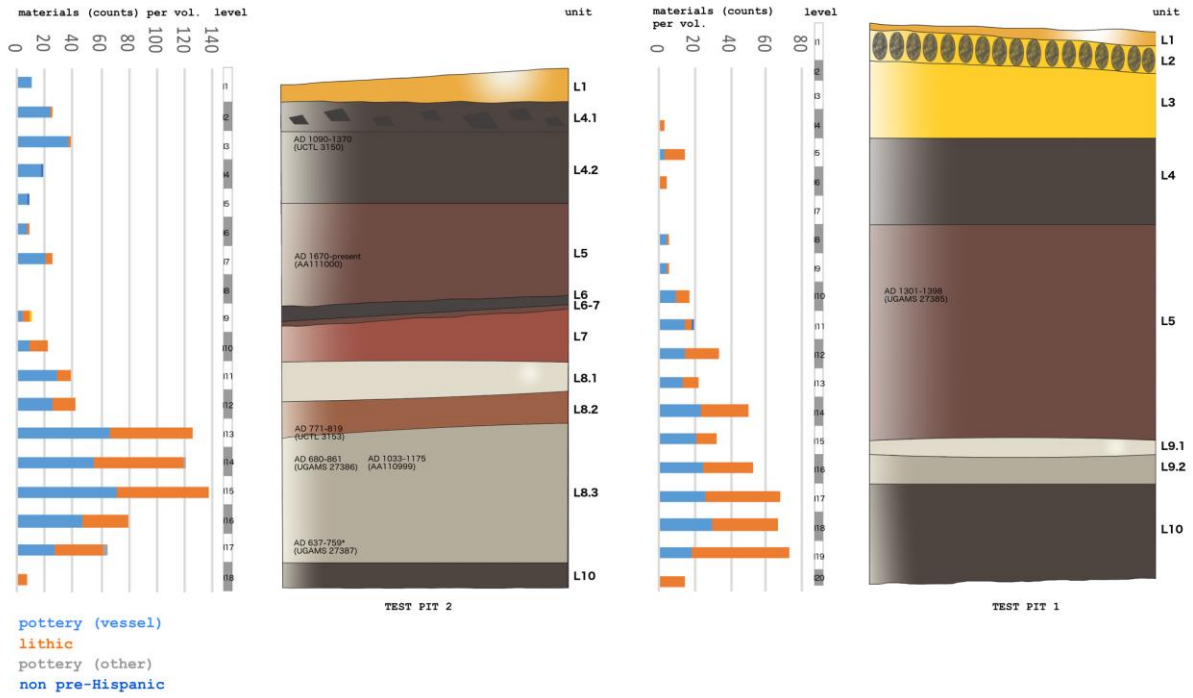


Figure 5.7: Stratigraphy of pit tests P1 and P2 indicating the abundance of material (pottery in blue), in each excavation level. The figure was obtained from Roa (2018).

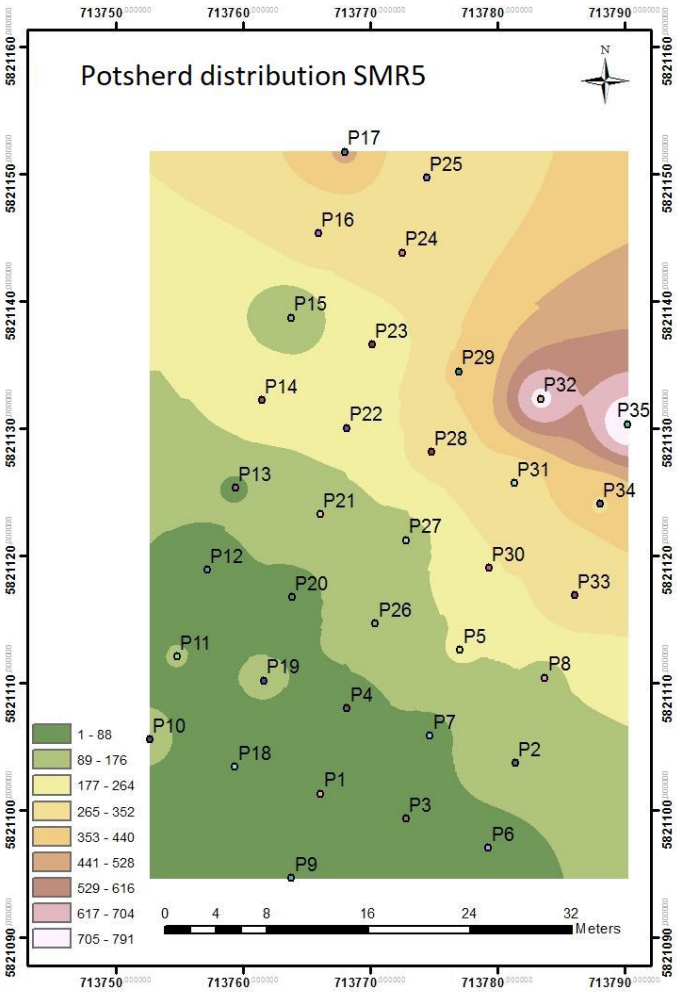


Figure 5.8: Potsherd density throughout SMR5 site. Potsherds from this study were recovered from boreholes P17; P24; P28; P29; P31; P32; P33; P34; P35. The map was obtained and modified from Dávila et al. (2016).

The samples comprised rim, body and base fragments of polished, smoothed and eroded monochrome pottery (Appendix 7). However, the fragmentary nature of the potsherds did not allow us to determine a specific pottery types (Appendix 7). Only samples with a thickness greater than 5 mm were selected due to their possible association with cooking pots (Dávila et al., 2016; González, 2017). The presence of soot marks on some of these sherds indicates their exposure to fire, possibly during cooking events.

6.1. Organic residue analysis

Around 1 g of pottery powder was obtained by drilling *ca*. 2 - 3 mm of the inner surface of the potsherds, after removal of a small surface layer (ca. 1 mm) to avoid contamination. Pottery powder was kept sealed in a hatch tube for no more than 24 hours before being submitted to lipid extraction.

The extraction and methylation of lipids followed a one-step acidified methanol (AE) protocol (Craig et al., 2013). A blank tube was included in each extraction batch to monitor possible contamination. An internal standard (*n*-tetratriacontane (C_{34}): 10 µg) was incorporated in each sample. Methanol (4 mL) was added to all samples, standards and blanks, after which they were sonicated for 15 min. 800 µL of concentrated sulphuric acid was added to each sample, standard and blank before placing them on a heating block at 70ºC for 4 h. Lipids were separated from the mixture by three successive extractions using *n*-hexane (2 mL). Extracts were neutralised using potassium carbonate and subsequently transferred into a new and clean tube. Lipids were concentrated under a gentle stream of nitrogen and, subsequently, resuspended in *n*-hexane at appropriate dilution for gas chromatography-flame ionization detector (GC-FID) and gas chromatography-mass spectrometry (GC-MS) analysis. An internal standard (n-hexatriacontane (C_{36}): 10 µg) was included in each sample to infer lipid concentration.

A subset of these samples (n = 147, 78.2%) were derivatized with N,O-bis(trimethylsilyl) trifluoroacetamide with 1% trimethylchlorosilane (BSTFA+TMCS, 99:1) to identify long-chain fatty alcohol distributions. Samples were resuspended in 100 µL of *n*-hexane, taking only half of the extract for derivatization. Fractions were transferred into a new hydrolysis vial and dried completely under a gentle stream of nitrogen. Lipids were resuspended in 100 µL of hexane. Extracts were derivatized by adding six to eight drops of BSTFA+TMCS, 99:1. Samples were placed on a heating block for 1 hr at 70ºC. Subsequently, lipids were concentrated and transferred into a new GC-vial with a conical insert and analysed within 48 h.

Another 2 g of powder was obtained from eleven samples and subsequently submitted to a solvent extraction (SE), as in (Colonese et al., 2017b). Lipids were extracted from the ceramic powder by sonication using 5 mL of dichloromethane:methanol (DCM:MeOH 2/1 *v/v)* three times. A blank tube was included in each batch to assess contamination. An internal standard (C_{34}: 10 µg) was incorporated in each sample. Samples were centrifuged and

transferred into a clean hatch tube. Subsequently, samples were dried under a gentle stream of nitrogen, and lipids were resuspended in 1 ml of DCM: MeOH 2/1 *v*/*v* and transferred to a clean hydrolysis vial. Samples were completely dried and then resuspended in 500 µL of DCM. A 100 µL aliquot was transferred into a new hydrolysis vial, drying and saving the remaining 400 µL for further extractions. The fraction was dried entirely, and lipids were resuspended in 100 µL of *n*-hexane. Samples were derivatised by adding four to five drops of BSTFA+TMCS (99:1). All samples were placed on a heating block for 1 h at 70ºC. Subsequently, the content of each tube was dried under a gentle stream of nitrogen and resuspended in *n*-hexane at appropriate dilutions. Internal standard (C_{36}: 10 µg) was included in every sample to asses lipid concentration. Samples were analysed via GC-FID and GC-MS within 48 h.

An Agilent 7890B high-temperature gas chromatograph (Agilent Technologies, Cheadle, Cheshire, UK) equipped with a flame ionization detector (HT-GC-FID) was used for lipid screening and quantification. The column used was a 100% Dimethylpolysiloxane DB-1 HT (15 m X 320 µm X 0.1 µm; J&W Scientific Scientific, Folsom, CA, USA). A splitless injector was used to inject 1 µL of acid-extracted samples into the GC at 300°C. Helium was used as a carrier gas at a constant flow rate of 2 mL min^{-1}. The oven temperature was set at 100°C for 2 minutes, subsequently rising 20°C min^{-1} up to 325°C, then held for 3 min. Solvent-extracted samples were injected in splitless mode (1 µL) at 350°C. The oven temperature was set at 50°C, settling for 2 minutes, subsequently rising at 10°C min^{-1} up to 375°C, where it held for 10 minutes.

All samples were analysed using an Agilent 7890A series chromatograph attached to an Agilent 5975C inert XL mass selective detector with a quadrupole mass analyser (Agilent Technologies, Cheadle, Cheshire, UK), and a Trace Ultra 1310 GC, coupled with an ISQ MS (Thermo Fisher, Bremen, Germany). Single ion monitoring (SIM) mode was employed for targeting specific ions (*m/z* 71, 75, 299, 327, 355, 383, 411, 439, 467, 495, 523, 551) to identify long-chain fatty alcohols.

A methylpolysiloxane (5%-phenyl) DB-5ms (30 m X 0.25 mm X 0.25 µm; J&W Scientific, Folsom, CA, USA) column was used onto which 1 µL of each sample was injected in splitless mode at 300ºC. A constant flow rate of 2 mL min^{-1} of helium was used as a carrier gas. The temperature of the oven was set at 50ºC for 2 min, subsequently rising 10ºC min^{-1} to 325ºC, holding for 15 min. The ionisation energy of the mass spectrometer was 70 eV, from which a spectrum by scanning ions between m/z 50 and 800 was obtained.

The identification of Isoprenoid acids and ω-(*o*-alkylphenyl) alkanoic acids (APAAs), used for assessing the presence of several commodities, was achieved through GC-MS analysis using a 50% cyanopropyl-methylpolysiloxane DB-23 column (60 m x 0.25 mm x 0.25 µm; J&W Scientific, Folsom, CA, USA) as in Shoda and others (2017). Acidified methanol extracts were injected into the GC (1µL) using a splitless injector at 300ºC. The carrier gas was helium at a flow rate of 1.5 mL min^{-1}. The temperature of the oven was set at 50ºC for 2 min, increasing 10ºC min^{-1} to 100ºC, after which it rose 4ºC min^{-1} up to 140ºC, and subsequently, 0.5ºC min^{-1} to 160ºC, to finally rise 20ºC min^{-1}, reaching 250ºC where it settled for 20 min. Single Ion Monitoring (SIM) mode was used to target specific compounds, in which selected ions were used for the identification of the three main isoprenoid acids (*m/z* 74, 101, 171, 326 for phytanic acid; m/z 74, 88, 101, and 312 for pristanic acid, *m/z* 74, 87, 213 and 270 for 4, 8, 12 - trimethyltridecanoic acid (4, 8, 12 - TMTD)), and APAAs of carbon length C_{16} to C_{22} (*m/z* 74, 105, 262, 290, 318, 346).

GC-MS results were computed by using MSD Chemstation F.01.03.2357 software. Criteria for compound identification were based on retention time, mass spectrum and comparison with the National Institute of Standards and Technology (NIST) library. Integration of peaks was performed using Agilent Mass Hunter Quantitative Analysis software version B.07.01/ Build7.1.524.0 for GC-MS.

6.2. GC-c-IRMS and Single Compound $\delta^{13}C$ Analysis

A total of one hundred and forty-seven samples were suitable for $\delta^{13}C$ analysis of $C_{16:0}$ and $C_{18:0}$ alkanoic acids (Catalanes Cave ECP= 45; Catalanes Cave LCP=48; SMR5 ECP=24; SMR5 LCP= 30), based on a minimal alkanoic acid quantity needed for injection (> 30 ng). Alkanoic acids $C_{24:o}$, $C_{26:0}$, $C_{28:0}$, and alkane C_{29} were also measured for one eligible sample with a sufficient quantity for injection. All samples were analysed using a Delta V Advantage Isotope Ratio Mass Spectrometer (Thermo Fisher Scientific, Bremen, Germany), coupled to a Trace Ultra 1310 Gas Chromatograph (Thermo Fisher) with a GC IsoLink II interface (with a CuO combustion reactor held at 850ºC). Samples were injected (1 µl) into an ultra-inert fused-silica DB-5ms UI column (60 m x 0.25 mm x 0.25 µm; J&W Scientific, Folsom, CA, USA). Ultra high-purity grade helium was used as the carrier gas with a flow rate of 2 mL min^{-1}. A small part of the flow was derived to an ISQ mass spectrometer (Thermo Fisher) for parallel acquisition of molecular data. The temperature of the oven was set at 50ºC for 0.5 min, rising 25ºC min^{-1} to 175ºC, then 8ºC min^{-1}, reaching 325ºC, settling for 20 min.

Eluted compounds were ionised in the mass spectrometer through electron impact. The intensities of ions *m/z* 44, 45 and 46 were recorded to automatically compute the $^{13}C/^{12}C$ ratios of each peak in the extracts. The software used for the computation were Isodat (Thermo Fisher Scientific, Bremen, Germany) and LyticOS (Isoprime, Cheadle, UK). Computation was based on the comparison with repeatedly measured standard reference gas (CO_2). Calculated $\delta^{13}C$ values are presented in parts per mil (‰), relative to the Vienna PeeDee Belemnite (V-PDB) international standard.

Batches were calibrated using a linear calibration curve (average $R^2 = 0.997 \pm 0.001$ in 30 batches) based on expected versus measured $\delta^{13}C$ values of n-alkanes and n-alkanoic acid esters from international standards (Indiana A7 and F8-3 mixtures). The accuracy of the instrument was determined on *n*-alkanoic acid methyl esters of known isotopic composition (Indiana F8-3, 18 measurements). The mean and standard deviation of these compounds were -29.91 ± 0.16‰ for $C_{16:0}$ (reported mean value vs. V-PDB -29.90 ± 0.03‰) and -23.24 ± 0.17 ‰ for $C_{18:0}$ (reported mean value vs. V-PDB -23.24 ± 0.13‰). The precision was based on a laboratory standard mixture, regularly injected between samples (145 measurements). Alkanoic acids mean ± SD values were -30.65 ± 0.16‰ for $C_{16:0}$ methyl ester and -26.18 ± 0.16‰ for $C_{18:0}$ methyl ester. Subsequent to the analysis, values were corrected for the methylation of the alkanoic acids that occurred during acid extractions. Corrections were performed using a mass balance formula to compare the values with a standard mixture of $C_{16:0}$ and $C_{18:0}$ fatty acids of known isotopic composition, already included in each batch during the acid extractions. Modern references were adjusted according to the atmospheric $\delta^{13}C$ variations between the Early Holocene Epoch and the present to better interpret the archaeological data (Hellevang and Aagaard, 2015).

6.3. $\delta^{13}C$ reference for South American camelid fat

The measurement of $\Delta^{13}C_{(C18:0\text{-}C16:0)}$ is a robust proxy for the identification of ruminant fats in the residues as differential carbon routing and fatty acid biosynthesis in ruminants differ from those characterising monogastric animals (Copley et al., 2003; Craig et al., 2012). Carbon stable isotope references from South American camelid meat (specifically llama (*Lama glama*) and guanaco (*Lama guanicoe*) from Lantos et al. (2015), Admiraal et al. (2023), and this study showed considerably negative $\Delta^{13}C$ values compared to domestic ruminants

from outside the Americas. A $\Delta^{13}C$ threshold was set at -2.4 ‰ (2 s.d from mean) to identify South American camelid fat from the residues.

6.4. Statistical analysis

All statistical analyses were performed using R 4.2.2 (R Core Team 2024).

7. Results

Interpretable lipid concentrations were obtained in every sample (>5 µg g^{-1}) (Evershed, 2008a), ranging from 6.5 to 1204 µg g^{-1} for Cueva de los Catalanes (mean= 49.9 µg g^{-1}) and 9.3 to 483.1 µg g^{-1} for SMR5 (mean= 49 µg g^{-1}). Lipid extracts showed a mixture of aliphatic compounds comprised of saturated fatty acids ($C_{8:0}$ to $C_{32:0}$), monounsaturated fatty acids ($C_{14:1}$ - $C_{24:1}$), fatty alcohols (C_{18} - C_{34}) and α,ω-dicarboxylic acids (C_6 - C_{28}).

The ratio between the palmitic ($C_{16:0}$) and stearic ($C_{18:0}$) acid (P/S) showed differences between terrestrial animals and plant commodities (Dunne et al., 2016; Evershed et al., 1997b). A P/S above 1.3, found in most samples (Catalanes Cave: ECP=85.9%, LCP=93.75%; SMR5:ECP=93.3%; LCP=100%), is unlikely to come from terrestrial animals, though possibly from plant products (Dunne, 2021). The higher solubility of $C_{16:0}$ compared to $C_{18:0}$ makes it more susceptible to leaching out in humid and wet environments such as southern Chile, thus altering the original plant P/S (Whelton et al., 2021). Caution must be taken when interpreting P/S as evidence for plant commodities.

Phytosterols such as stigmastanol and β-sitosterol and characteristic plant terpenoids α- and β-amyrin were identified in a limited number of pots (5.8%). The predominance of even over odd-numbered very long-chain fatty acids and odd over even-numbered long-chain alkanes, highly found in the epicuticular wax of plant leaves and stems, was identified in most samples (Cueva de los Catalanes: ECP=39.68%, LCP=67.18%; SMR5: ECP=76.66%; LCP=70%) (Dunne, 2021; Dove and Mayes, 2006).

Distribution of C_6 - C_{15} α,ω-dicarboxylic acids (diacids) in ECP and LCP samples from both sites showed a high abundance of C_9, probably from the oxidation of unsaturated fatty acids from plant resources (Dunne et al., 2016; Regert et al., 1998). Moreover, several

samples also exhibited a distribution of long-chain diacids ($\geqslant C_{20}$). These aliphatic compounds constitute the principal monomers of suberin (*ca.* 23 - 46 %), a natural polyester mainly found in underground storage plant organs such as tubers, roots and rhizoids (Pollard et al., 2008).

The exposure of unsaturated fatty acids $C_{18:x}$, $C_{20:x}$ and $C_{22:x}$ from plant and animal resources to prolonged heating $\geqslant$ 200ºC during cooking events can form homologous series of C_{18}, $C_{20,}$ and C_{22} ω-(*o*-alkylphenyl) alkanoic acids (APAAs) (Bondetti et al., 2020; Evershed et al., 2008). APAA-C_{18} and APAA-C_{20} were identified in several samples from Catalanes Cave (ECP: APAA-C_{18}, n=19, APAA-C_{20}, n=4; LCP: APAA-C_{18}, n=27, APAA-C_{20}, n=9;) and SMR5 (ECP APAA-C_{18}, n=4, APAA-C_{20}, n=1; LCP APAA-C_{18}, n=4, APAA-C_{20}, n=3). The ratio between APAAs C_{20} and C_{18} supports a terrestrial origin for these residues (Appendix Figure 15.4) (Bondetti et al., 2020).

Most samples showed a distribution of APAA-C_{18} isomers (A-I), possibly formed from the cooking of various plant and animal products. The ratio between the E and H APAA-C_{18} isomers (E/H) has been shown to help discriminate between **1)** cereals, fruits and non-leafy vegetables, **2)** animals, and **3)** leafy plants (Bondetti et al., 2020). The identification of C_{18}.APAA isomers in modern references for maize kernels, quinoa grains, and local wild seeds and nuts supported the idea that a high E/H value can also confirm an origin from cereals and seed references from southern Chile (Figure 5.9). The majority of the samples from Cueva de los Catalanes had a high E/H value, suggesting an origin from cereals, fruits and non-leafy vegetables (Figure 5.2) and showing no differences between the ECP and the LCP (Catalanes Cave: ECP mean= 4.32; LCP mean= 4.16; t.test, t=0.27, p-value= 0.78). At SMR5, only a few samples yielded interpretable APAA quantities, mostly showing an E/H value concordant with an origin from cereals, fruits, and non-leafy vegetables. The lack of APAAs in SMR5 pottery might be related to the fact that APAAs are only sometimes formed during cooking events, and when formed, they are readily lost in the environment (Lucquin et al., 2018). The acidic soil of the SMR5 site and its exposure to open-air conditions might have boosted APAA degradation.

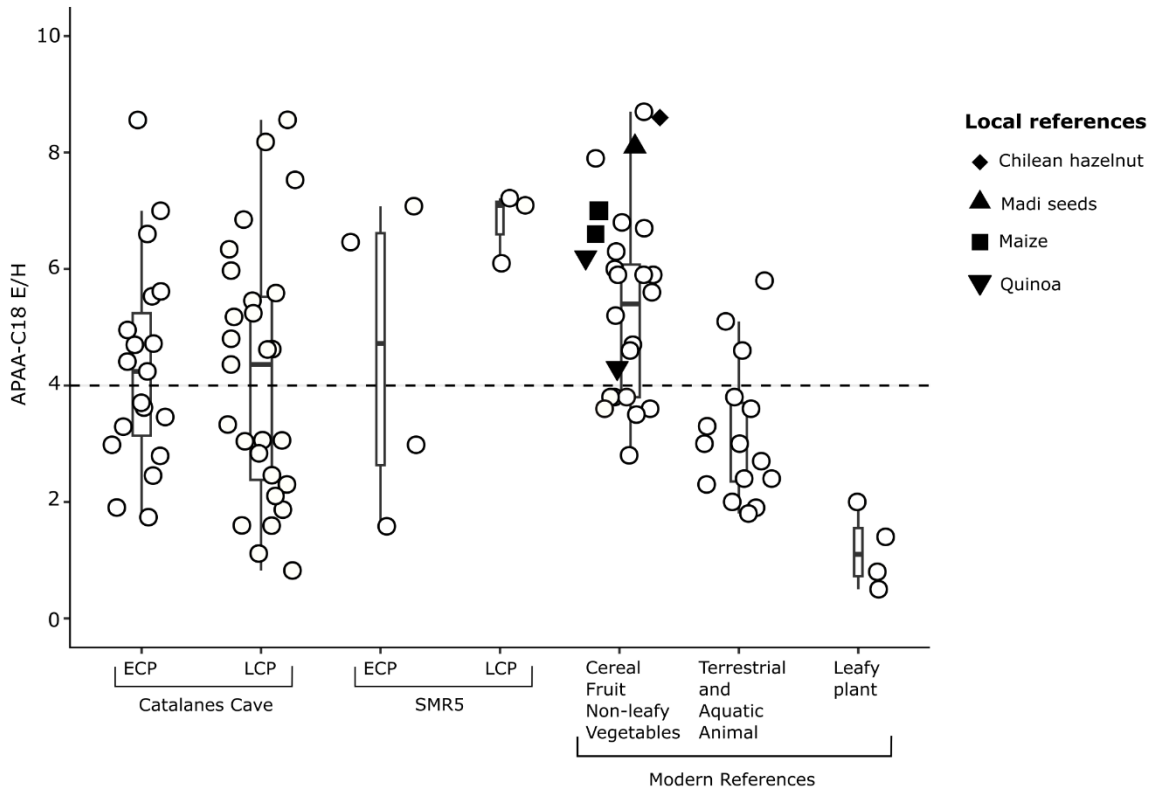


Figure 5.9: Boxplot of Cueva de los Catalanes and SMR5 Early and Late Ceramic Period samples showing APAA-C_{18} E/H values for the ECP and LCP in both sites. Values >4 are more likely to indicate an origin from cereals, fruits and non-leafy vegetables (Dolbunova et al., 2023; Bondetti et al., 2020). Modern references from Bondetti et al. (2020) and this work were plotted. References for South American plants are differentiated by shape and fill. Figure created by the author.

Carbon stable isotope analyses of the main alkanoic acids $C_{16:0}$ and $C_{18:0}$ demonstrated that most of the plant residues identified in the pots have a C_3 origin (Figure 5.10). However, more positive $\delta^{13}C$ values in several LCP samples from both sites indicate the use of C_4 plants, with maize being the most likely candidate (Campbell et al., 2020).

Long-chain fatty alcohol distributions up to C_{32}-OH or above are commonly found in C_4 panicoid grasses like maize while being rare in C_3 grasses (Rommerskirchen et al., 2006; Reber and Evershed, 2004b; Bianchi and Avato, 1984). Panicoid grasses in southern Chile consist of around 15 native and endemic species (Finot et al., 2009), none of which have a culinary use; therefore, fatty alcohol distributions up to C_{32}-OH found in several samples are likely associated with maize. Such lipid profiles were identified in several ECP and LCP samples, some of which showed a positive $\delta^{13}C$ signal concordant with a C_4 origin. Maize has

been characterised as having a predominant abundance of C_{32}-OH over the remaining fatty alcohols (Bianchi and Avato, 1984). The abundance of C_{32}-OH in the samples was not shown to be higher than the remaining fatty alcohols. The possible mixing of C_3 and C_4 plants in the pots might have altered the relative proportion of C_{32}-OH expected for maize residues.

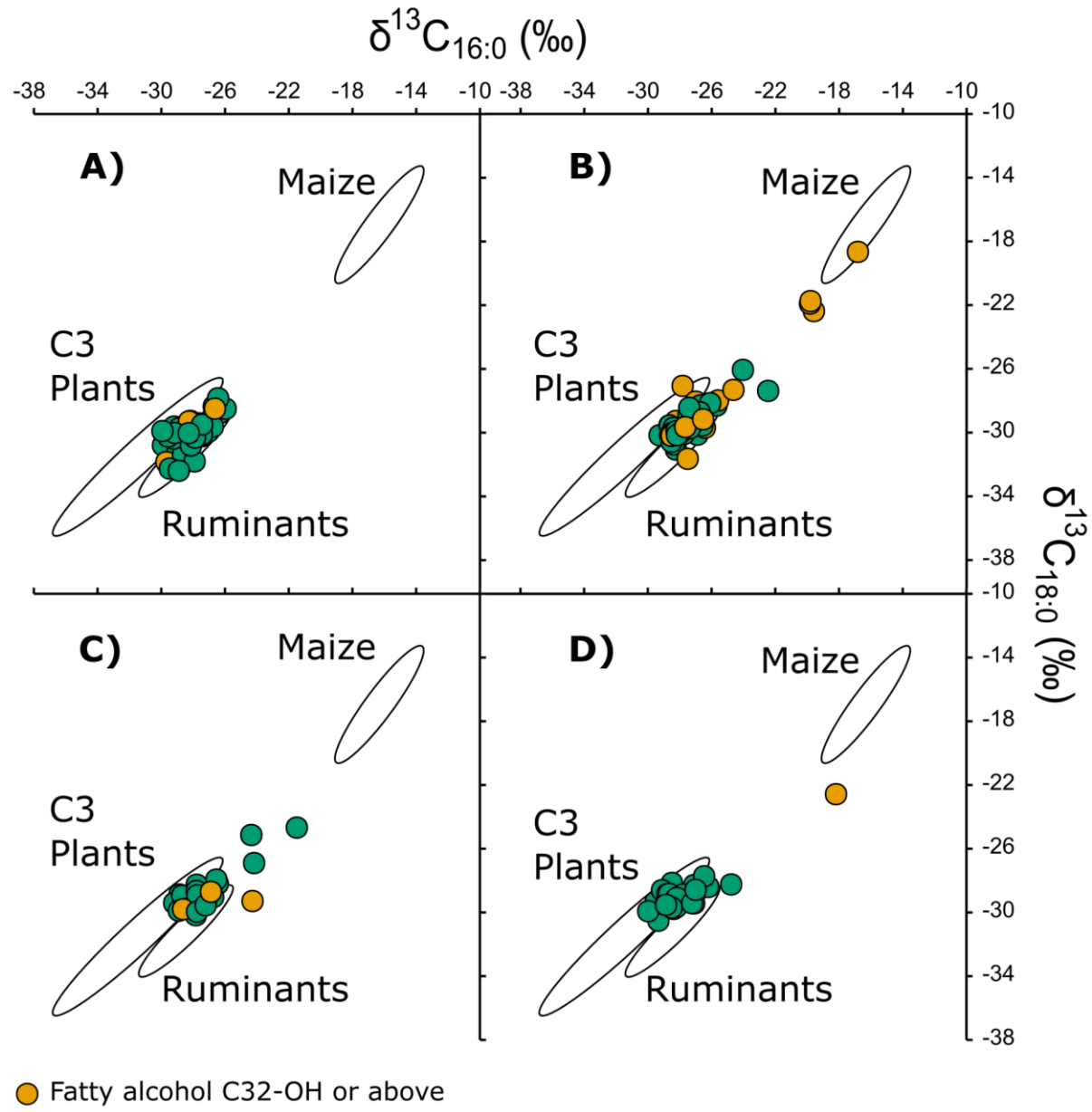


*Figure 5.10: Carbon stable isotopes ($\delta^{13}C$) of the main alkanoic acids from residues extracted from **A)** Early Ceramic Period (ECP) and **B)** Late Ceramic Period (LCP) from Catalanes Cave, and **C)** ECP and **D)** LCP from SMR5. Confidence ellipses (P = 68%) were built using modern references from the literature and this study. Figure created by the author.*

Several ECP and LCP samples from both sites showed $\delta^{13}C$ values between maize and C_3 plant references, likely indicating the mixing of both products in the pots. The identification of a fatty alcohol distribution above C_{32}-OH in some of these samples might also suggest that C_3 and C_4 plants were used in the same container.

Mixing C_3 and C_4 plants in the same vessel can lead to low $\Delta^{13}C_{(C18:0 - C16:0)}$ ($\Delta^{13}C$) values when there is a higher contribution of $C_{18:0}$ fatty acid from C_3 products (Admiraal et al., 2023). Conversely, a higher input of $C_{18:0}$ from C_4 plants will likely generate a positive $\Delta^{13}C$ instead. To assess this possibility, we applied a simple linear mixing model using averaged $\delta^{13}C$ endpoints built on modern references of maize, C_3 plants and ruminants, connected by hypothetical mixing lines. Figure 5.11 shows samples with a high $\delta^{13}C$ signal and a fatty alcohol distribution, likely indicating an origin from maize. A negative $\Delta^{13}C$ in these samples possibly reflects a higher contribution of $C_{18:0}$ fatty acids from C_3 plants. Very long-chain fatty acids and alkanes with a C_3 origin were identified in one sample likely used for processing maize, strongly suggesting mixing C_3 and C_4 plants in the same container (Appendix Figure 15.5).

A large group of samples exhibited negative $\Delta^{13}C$ values, seemingly not from the use of C_3 and C_4 plants in the same pots. A number of ECP and LCP samples at Cueva de los Catalanes showed negative $\Delta^{13}C$, below -2.4‰, likely from camelid fat, while others had values within broader ruminant categories as in Dolbunova et al. (2023) (Figure 5.11). Intermediate APAA-C_{18} E/H values of several of these samples also confirm an origin from animal fat.

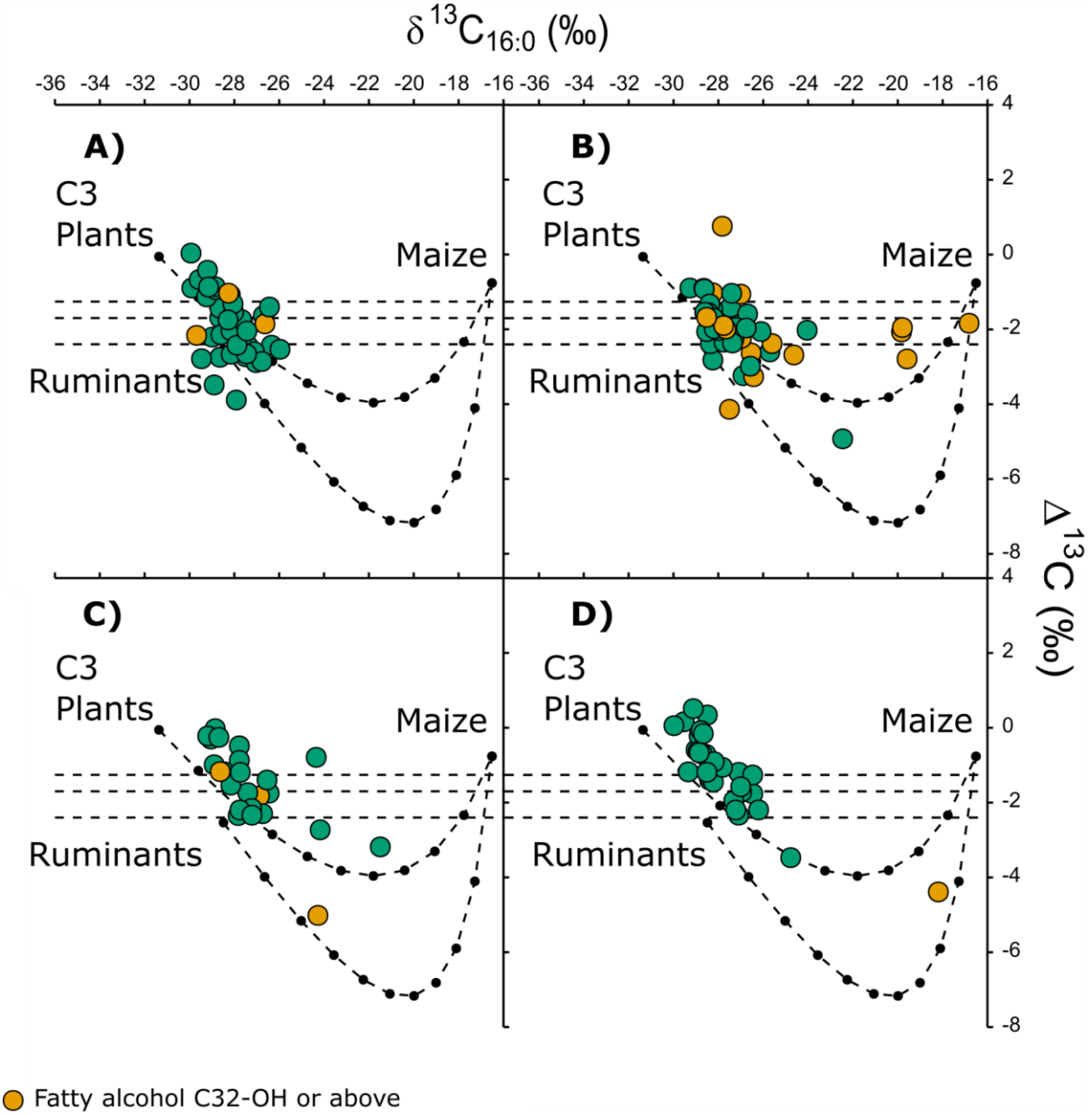


*Figure 5.11: Compound-specific $\delta^{13}C$ analysis of samples from Catalanes Cave and SMR5 showing values for $\delta^{13}C_{16:0}$ versus $\Delta^{13}C_{C18:0\text{-}C16:0}$. Plots show samples from the **A)** ECP and **B)** LCP from Catalanes Cave, and **C)** ECP and **D)** LCP from SMR5. Mixing lines denote a 10% increment between $\delta^{13}C$-averaged endpoints, expressed as % of total fatty acids. Data were gathered from the USDA database. Dashed lines indicate thresholds set by (Dolbunova et al., 2023) at -1.26‰, -1.7‰ for ruminant carcass fat. A third threshold was set at -2.4‰, representing South American camelid carcass fat. Figure created by the author.*

The ratio between phytanic acid 3S,7R,11R,15 (SRR) and 3R,7R,11R,15 (RRR) diastereomers offers a mean to distinguish ruminants (%SRR < 64) from aquatic commodities (i.e., 76% ±16.6) in the residues (Dolbunova et al., 2023; Lucquin et al., 2016b). Several samples showed a phytanic acid-SRR diastereomer contribution of around 64% and a negative $\Delta^{13}C$, consistent with a ruminant origin. Nevertheless, most of the LCP samples at Cueva de los Catalanes showed a high dominance of the SRR-diastereomer, as described for aquatic products. These values could indicate that local ruminants were feeding from C_4 plants. The phytanic acid diastereomer ratio is susceptible to being altered in the rumen, as feeding strategies using C_4 silage seem to impact rumen microbiota, leading to an increase of the SRR-diastereomer in ruminants (Eibler, Kaffarnik and Vetter, 2018; Baars et al., 2012; Schröder and Vetter, 2011). Further experimental work would be needed to support this idea, as well as more $\delta^{13}C$ and $\delta^{15}N$ analysis on local ruminant remains.

At SMR5, the $\Delta^{13}C$ values were generally more positive for both periods than at Cueva de los Catalanes, and did not plot within South American ruminant references ($\Delta^{13}C \leqslant$ -2.4‰). Most samples showing a $\Delta^{13}C \leqslant$ -2.4‰ were related to the use of C_3 and C_4 plants in the pots. Only one of these samples (SMR5.ECP-29) yielded a phytanic acid-SRR diastereomer contribution <64%, possibly reflecting the processing of ruminant carcass fat (Appendix 11). The identification of a relatively ^{13}C enriched $C_{16:0}$, a low $\Delta^{13}C$ offset and the presence of C_{32}-OH in this sample might indicate a small contribution of maize into the pot. At SMR5, there was no definitive evidence indicating the use of LCP pots for processing ruminant fat.

8. Discussion

Through the analysis of organic residues from pottery artefacts from southern Chile it was possible to assess the culinary and economic significance of maize in local foodways. Our results from Cueva de los Catalanes demonstrate that ceramic vessels were mainly used for processing C_3 plants and ruminant carcass fats during the ECP and LCP. This is broadly supported by macrobotanical evidence indicating a significant reliance on wild and domesticated C_3 plants throughout these cultural periods (Roa Solís et al., 2024; Campbell et al. 2018b). Ceramic containers from SMR5 were also primarily used for processing C_3 plants, and much less for animal products. The limited evidence for animal fats in these pots could be attributed to distinctive cooking techniques (e.g. roasting) as attested by historical records (Núñez de Pineda y Bascuñán, 1673, cited in Cayuqueo, 2022, p. 50). From our results, we suggest that in both sites maize did not play a prominent role during the ECP, implying a

limited culinary use by groups from the Pitrén cultural complex. By contrast, the noticeable rise of ^{13}C-enriched compounds in several El Vergel pottery residues suggests an increased use of maize during the LCP, mainly in samples from Cueva de los Catalanes, and less in SMR5. However, the reduced number of pots used in maize cooking possibly indicates its restricted employment in specific culinary preparations.

Furthermore, it is worth noticing that the importance of maize may be underestimated by its inherently lower lipid concentration when mixed with lipid-rich resources such as animal fat (Reber, 2022). However, since most pots were used to process C_3 plants, a C_4 signal would be more visible in the residues in cases where maize was exploited together with C_3 plants. If that were the case, we would expect a progressive ^{13}C enrichment from the residues towards maize references, as attested in Admiraal et al. (2023).

Previous researchers have hypothesised the rapid spread of maize throughout the Americas due to its use in the production and consumption of maize *chicha* (Bonzani and Oyuela-Caycedo, 2006; Staller and Thompson, 2002). Maize adoption in southern Chile might have been related to its use in the preparation of *chicha*, denoting the significance of alcoholic beverages as socio-political mediators during ceremonial activities (Godoy-aguirre, 2018). It is possible that maize first arrived in the Araucanía from central Chile (Roa et al., 2018) as part of networks of reciprocal gift-giving relationships between distant communities to reinforce and negotiate socio-political ties (Logan, Hastorf and Pearsall, 2012; Bonzani and Oyuela-Caycedo, 2006). Maize was already cultivated by Llolleo groups from central Chile (Planella et al., 2014); the similarities between Pitrén and Llolleo pottery traditions denote possible interactions between both groups (Correa Girrulat, 2010; Falabella, Planella and Tykot, 2008). If maize was initially introduced in the form of *chicha* from central Chile during the ECP, it was probably first included as a secondary ingredient of other major preparations, such as C_3 plant-based beverages. The restricted use of maize during the ECP could denote a strong preference for local C_3 plants and initial experimentation on the possible uses and cultivation of this exotic plant in more temperate southern Chile.

Historical documents attest to the consumption of maize-based *chicha* during socio-political and religious gatherings in the Araucanía. *Chicha* was consumed by family groups in residential settings and in collective feastings and celebrations where its intake was considerably larger (Molina 1795, 122). Our results suggest that these practices may have been boosted during the LCP. The intensification of food production by El Vergel farmers could have allowed a constant supply of maize (Campbell and Quiroz, 2015; Dillehay et al., 2007),

likely to be consumed by a growing population in the form of alcoholic beverages during feasts and ceremonies.

Specifically, the proliferation of delimited ceremonial sites during the LCP, such as monumental mounds or *kuel*, natural shrines and ceremonial fields associated with residential settings, denoted socio-political nucleation among local communities (Dillehay, 2014; Dillehay and Saavedra Zapata, 2014b; Dillehay et al., 2007; Dillehay, 2007). Cueva de los Catalanes and SMR5 were possibly areas of local socio-political gatherings where *chicha* was consumed during communal feasts and ceremonies. However, their association with domestic activities cannot be ruled out, as ceremonies and habitational sites have been found to be relatively close in distance, as well as the habitual use of domestic settings for gatherings and feasts (Dillehay and Saavedra Zapata, 2014a; Núñez de Pineda y Bascuñán, 1673, cited in Cayuqueo, 2022, p. 46).

The incorporation of maize into local foodways likely reflects these socio-political and economic changes in southern Chile with the intensification of food production and the emergence of social diversity throughout the region. However, the adoption of maize in the Central Valley of the Araucanía did not overshadow the significance attributed to the region's diverse range of available C_3 plants. These plants might have consisted of local grains, fruits and non-leafy vegetables used in a wide range of culinary preparations consumed at Cueva de los Catalanes and SMR5. We propose that maize was adopted as a secondary ingredient in major culinary preparations, possibly in the production of alcoholic beverages. However, its use as a main ingredient, possibly in the production of *chicha*, occurred only after food production was intensified, and socio-political and economic changes of the LCP created the conditions for its incorporation into local foodways.

9. Conclusion

The significance of maize in local foodways and its potential connection to socio-political and economic events in the Araucanía has tenuously been discussed. The adoption of maize in the Araucanía coincided with a time interval of considerable socio-political and economic changes. Organic residue analysis on Early and Late Ceramic Period potsherds demonstrated that maize became a main component of the local recipes during the Late Ceramic Period, however, it did not show to be more important than C_3 plants and ruminants. The socio-political and economic changes experienced during the transition from the Early to

the Late Ceramic Period created the conditions for its use as a primary component, possibly in the production of *chicha*. The consumption of these beverages during feasts and ceremonies and their use as mediators among participants might have invested maize with socio-political significance. Conversely, maize was not considered a staple.

Pottery was mainly employed for the processing of ruminant carcass fat and C_3 plants. This preferential use demonstrates that, despite the arrival of maize in southern Chile, it did not diminish local groups' reliance on C_3 products. These commodities have been part of local foodways since the Early Ceramic Period. Maize was included in the culinary use of plants, possibly to produce alcoholic beverages; however, the importance of C_3 plants and ruminants in local foodways seems to have been constant throughout the Ceramic Period.

10. Acknowledgements

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Chapter 6: The ceremonial use of maize in the expansion of the Inka state in Tarapacá, Northern Chile

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2. Abstract

During the Inka state expansion maize *chicha* played a prominent role in mediating socio-political negotiations with local communities and establishing reciprocal relations to legitimise the state's authority. However, historical sources have indicated the significance of several other plants in the preparation of alcoholic beverages consumed during feasts and celebrations. In Tarapacá, northern Chile, maize was a staple food. Large quantities of maize remains have been associated with its extensive use in the preparation of *chicha*, while the identification of several local plants has also been associated with the preparation of alcoholic beverages in the region. While maize has been referred to be the main commodity in *chicha* preparation within ceremonial sites, the direct analysis of the content of pottery claimed to be used in storing maize *chicha* has never been assessed before in Tarapacá. Here, we extracted and analysed organic residues from pottery fragments recovered from the Iluga Túmulos site, Tarapacá Region, northern Chile, to assess the significance of maize as a crucial commodity during the celebration of feasts and ceremonies during the Inka state occupation of Tarapacá. Molecular and single-compound carbon stable isotope analysis showed that maize was not

more prominent than C_3 products, possibly associated with a more active role of local communities in the socio-political negotiations with the Inka state.

Keywords: Inka state, Tarapacá, pottery, maize, C_3 products, feasting

3. Introduction

Feasting is a public ritual practice with a strong ideological significance. The communal consumption of food and drinks during feasting disrupts daily social activities while mediating complex socio-political relations among and within groups (Dietler 2001, 66). Concepts such as identity, territoriality, power, and authority are negotiated between the participants through the disposition and consumption of commodities and culinary preparations of ideological prominence (Twiss, 2012, Dietler, 2006, 2001).

Maize held a paramount symbolic significance in consolidating the Inka state or *Tawantinsuyu*, mainly as a fermented beverage called *chicha* (*aqha* in Quechua and *k'usa* in Aymara). Maize *chicha* was the most prestigious and prominent state alcoholic beverage and one of the main drivers of the Inka state expansion (Bray, 2003; Hastorf and Johannessen, 1993). The supply of *chicha* to the occupied provinces and its promotion in feasts and celebrations was considered an act of hospitality to legitimise the Inka state authority and hegemonic control (Bray, 2009; Murra 1980, 95).

In the Atacama Desert, the use of maize as a staple and its ubiquitous finding in Inka archaeological sites indicates that it was likely considered the most prominent product for preparing *chicha* during the Inka state occupation of northern Chile (Santana-Sagredo et al., 2021; Ugalde et al., 2021; Zori 2011, 490-521). However, early Spanish chronicles attest that various other plant products were used in producing alcoholic beverages during the Inka period, in cases showing to be of great significance within different Inka provinces (Cobo, 1979, 28; de Cieza de León 1945, 276). Particularly in the Pampa del Tamarugal, Tarapacá region, large quantities of maize macro remains, as well as *algarrobo* pods (*Prosopis* spp.) and *molle* (*Schinus molle*) have been identified in food-producing areas from Inka archaeological contexts (Palacios Hernández, Vidal Montero and Pellegrino Hurtado, 2022; Uribe, Urbina and Zori, 2012; Zori 2011, 490-521). The identification of Inka and local pottery in these sites has been interpreted as evidence for the preparation and consumption of *chicha*

(Zori 2011, 516 - 519); however, the original content of these pottery containers has never been directly assessed.

Molecular and isotopic analysis of organic residues extracted from ceramic vessels offers an opportunity to explore their actual content during use (Evershed, 2008b), in this case, within ceremonial and feasting activities. Previous studies in South America have successfully identified maize usage in pre-colonial vessels through compound-specific $\delta^{13}C$ and molecular analysis of organic remains trapped in the porous matrix of pottery containers (Montalvo-Cabrera et al., 2024; Admiraal et al., 2023). Through the extraction of organic residues from pottery artefacts from the Iluga Túmulos site, located in the heart of the Pampa del Tamarugal, Tarapacá region, we aim to assess the importance of maize products in feasts celebrated during the Inka occupation of Tarapacá (400-500 cal BP). Given the isotopic evidence and historical importance of maize in the Inka state expansion, we hypothesise that maize was the principal commodity stored in these vessels, especially maize *chicha*.

4. *Chicha* and state power in the Pampa del Tamarugal

The Pampa del Tamarugal is an endorheic basin located in the Tarapacá Region, between the Camiña ravine (19º33' Lat S) and the Loa River (21º25' Lat S), bounded to the east by the Andean foothills and to the west by the Coastal range. Low annual precipitations (<5mm) and high levels of evaporation make the Pampa del Tamarugal an area of extreme hyper-arid conditions and the driest area of the Atacama Desert (McRostie et al., 2022; Uribe et al., 2020a).

The Pampa del Tamarugal served as a nodal point for the articulation between the highlands, lowlands, and the coast, facilitated by the use of complex llama (*Lama glama*) caravan systems and the free movement of people, dating back to pre-Inka times (Núñez and Santana-Sagredo, 2023; De Souza Herreros et al., 2017; Briones, Núñez and Standen, 2005). This characteristic led to the establishment of an Inka administrative centre in the heart of the Pampa del Tamarugal, to control local exploitation and distribution of mining, coastal, and agricultural resources (Santoro and Uribe, 2018; Urbina A. et al., 2018; Uribe et al., 2012). The heterogeneity of local communities and ecological zones led the Inka state to develop diverse domination strategies, enabling the maintenance of socio-political relationships that achieved control over local resources and workforce for their exploitation (Uribe and Sánchez, 2016; Berenguer, Sanhueza and Cáceres, 2011).

The development of public activities leading to social congregation would have been of great importance for the Inka state, with the consumption of alcoholic beverages as a significant mediator for fostering socio-political ties with local authorities and legitimising state control. Particularly maize-based *chicha* has traditionally been referred to as the Inka state's main beverage (Bray, 2009). Its distribution to the provinces ensured reciprocal tributary labour, or *mita,* in several state activities (e.g. construction of roads, agriculture, metallurgy, etc.) led by members of local and distant communities (Murra 1980, 95). Although maize-based alcoholic beverages have been produced since pre-Inka times, the Inka state institutionalised *chicha* production by constructing in every province an *aqllahuasi*, which consisted of an establishment where designated young women known as *aqllakuna* resided to be devoted to textile production and to craft maize *chicha*, ensuring a standardised production and constant stock for all members of the state (Bray, 2003; Guaman Poma de Ayala 1936, 214).

The material culture associated with Inka celebration feasts included a variety of Cuzco polychrome jars and bowls, provincial pottery from the highlands (e.g. Saxamar Inka Pacajes) and local Inka pottery (IKL) specific to Tarapacá (Uribe, Sanhueza and Bahamondes, 2007). Among these utensils, jars with long widening necks and a conical base known as aryballos (Figure 6.1) were the most common containers and the primary vessels used for storing and transporting maize *chicha* in ceremonies (Bray, 2009, 2003). The finding of these pottery assemblages in the Pampa del Tamarugal have been related to the production and consumption of maize *chicha* during important feasts (Zori 2011, 357, 362).

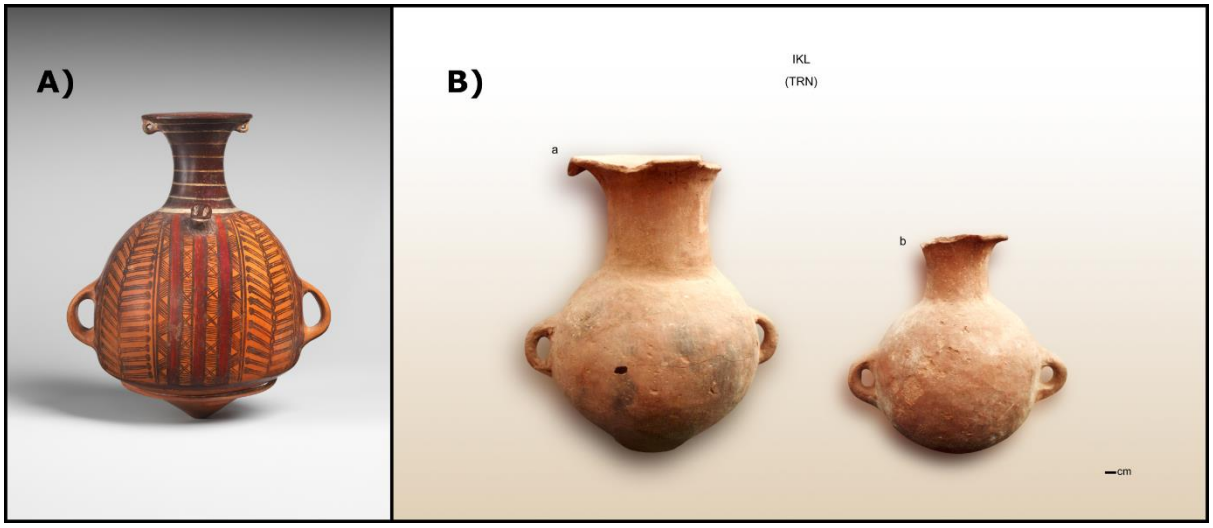


Figure 6.1: A) Characteristic polychrome Inka aryballo. Image was taken from the Metropolitan Museum of Arts collection (Met), New York. The image is available under the Met's open access policy, stablished in February, 2017. B) Local Inka pottery from Tarapacá (IKL). Photograph by Professor Mauricio Uribe from the University of Chile. Figure created by the author.

Stable isotope analysis on human bone remains indicates that maize was already a staple during the Inka expansion into Tarapacá (Santana-Sagredo et al., 2021, 2015). With this, large quantities of maize remains identified in Inka ceremonial areas and administrative centres have been indicated to reflect its use as the main product for *chicha* production in Tarapacá (Santana-Sagredo et al., 2021; Zori 2011, 357, 385). However, the identification of large quantities of *algarrobo* and *molle* has been attributed to their use in producing alcoholic beverages consumed during feasts and celebrations (Zori 2011, 385, 399). Early chronicles have indicated that *molle* was highly important for several Andean communities and that its consumption as an alcoholic beverage was crucial in feasts and celebrations (Valdez, 2012; de Cieza de León 1945, 276). Similarly, the *algarrobo* fruits were ground to prepare the flour and to produce an alcoholic beverage known as *aloja* (Orgaz, 2012, Cobo 1979, 8). The large quantities of *algarrobo* remains found in several Inka sites from Tarapacá have been reported to be even more numerous and represent a higher economic significance than maize (Palacios Hernández et al., 2022; Uribe et al., 2012), thus challenging the general assumption of maize as a prominent resource in ceremonial contexts. This discrepancy and the potential use of other plants as main resources for *chicha* production raises the need to directly assess the content of ceramic containers used in ceremonial contexts from Tarapacá.

5. Iluga Tumulos Site

The Iluga Tumulos site is situated in the Pampa del Tamarugal (Figure 6.2), approximately 6 km south of the "Giant of Tarapacá" geoglyph (Figure 6.3), at the convergence point of the Aroma, Tarapacá, and Quipisca ravines (Uribe et al., 2020b). The site exhibits continuous pre-colonial occupation, spanning from the Formative (ca. 3000 – 1000 cal BP) to the Late Period or Inka occupation of Tarapacá (ca. 500-400 cal BP). The presence of tumuli, public open spaces, rooms, and extensive irrigation systems associated with agricultural production areas (Figures 6.4, 6.5, and 6.6) characterises it as a site of significant productivity and ceremonial activity and a convergent point for a large diversity of local communities from different parts of the region (Palacios Hernández et al., 2022; Uribe et al., 2020b).

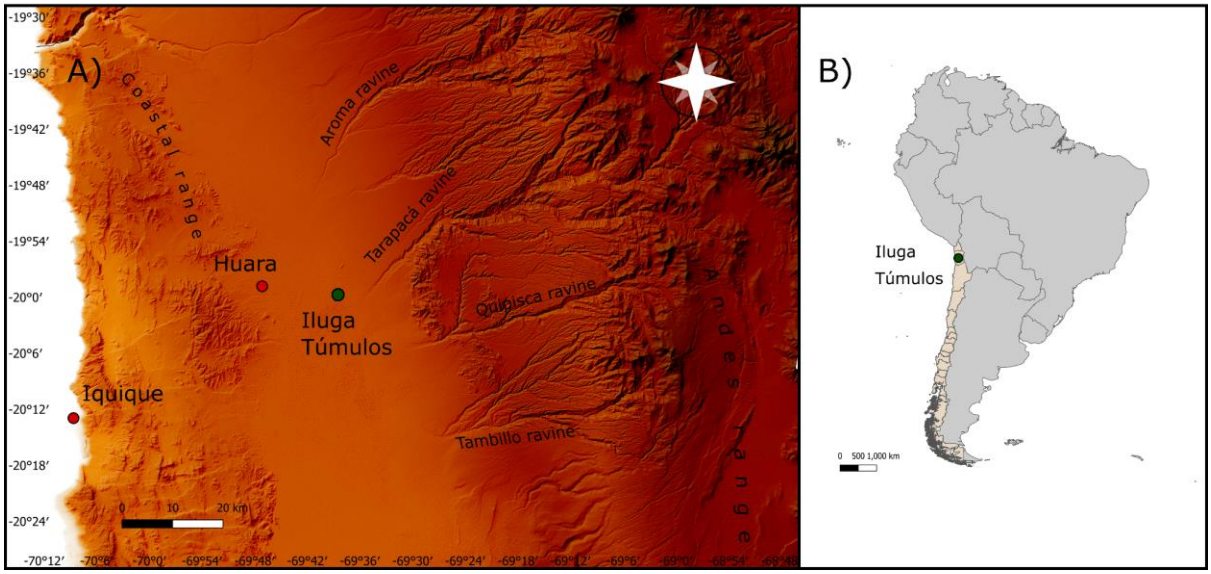


Figure 6.2: ***A)*** *Iluga Túmulos site location in the Pampa del Tamarugal;* ***B)*** *Iluga Túmulos site location in Northern Chile. Figure created by the author.*



Figure 6.3: Photo of the "Gigante de Tarapacá" geoglyph, locatated 6 km from Iluga Túmulos site. The image was obtained from Uribe et al. (2020).

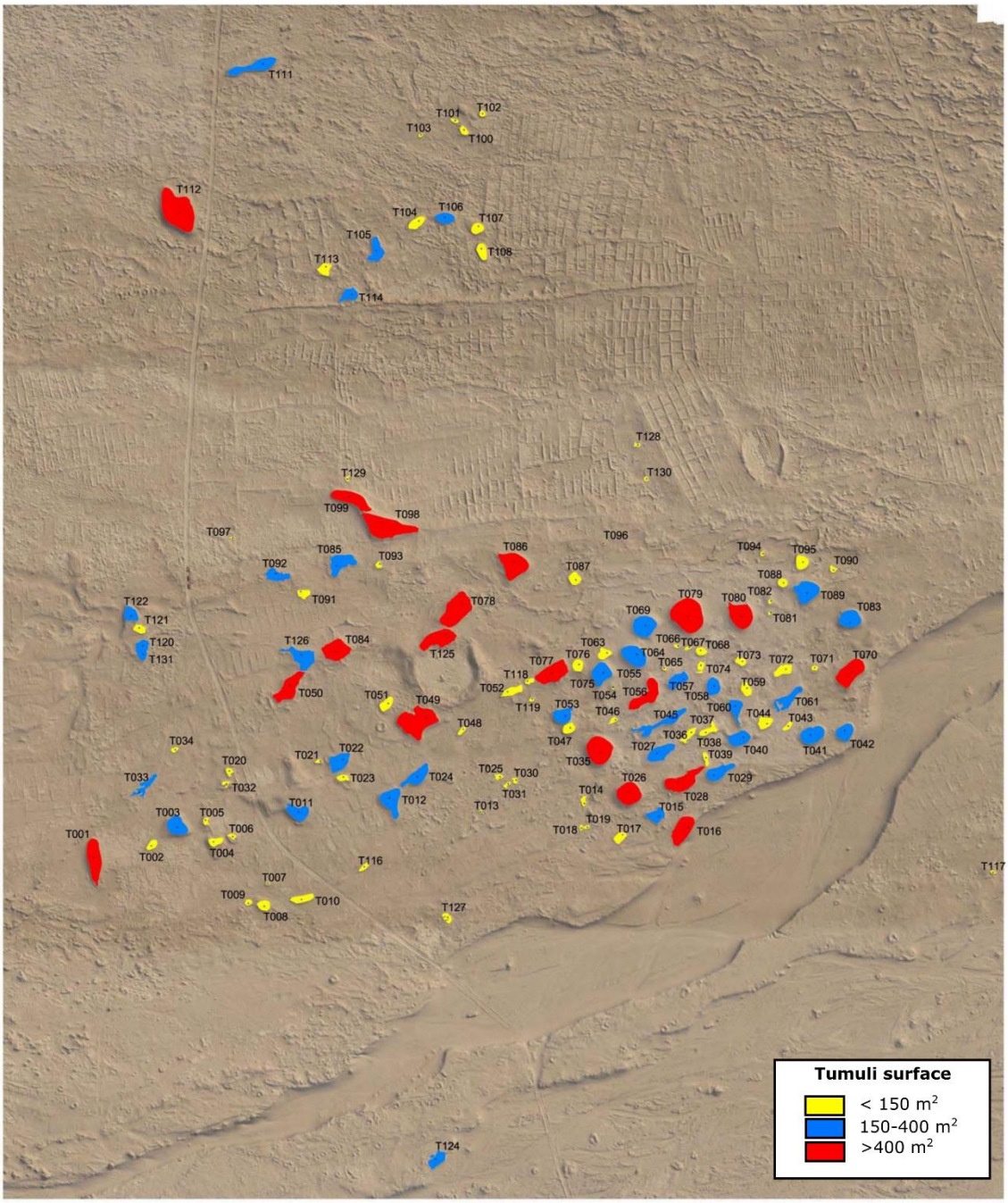


Figure 6.4: Plan of Iluga Túmulos site showing the Tumuli (T). The colours indicate the tumuli's surface area. The plan was obtained and modified from Uribe et al. (2020).

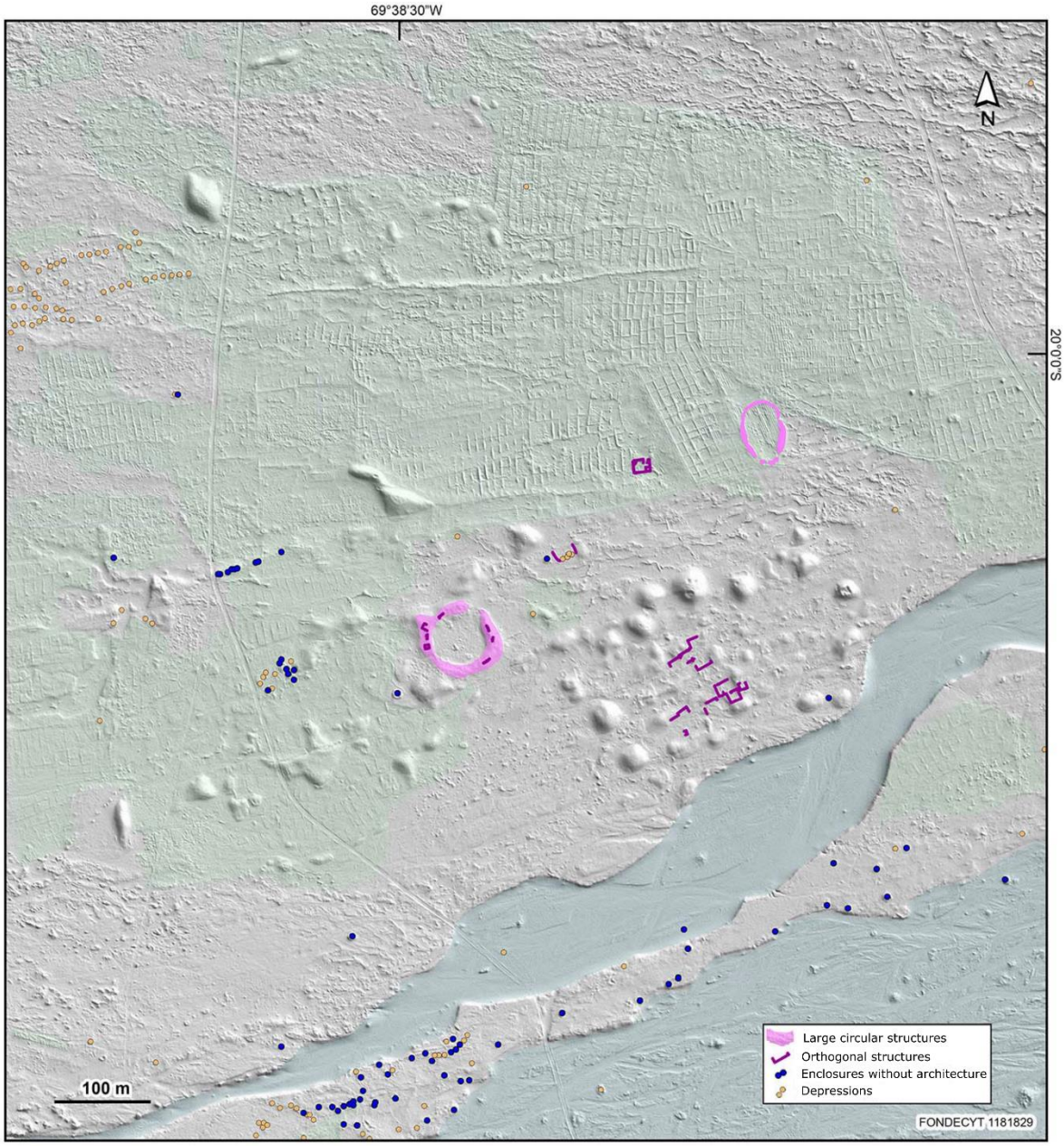


Figure 6.5: Plan of Iluga Túmulos site highlighting different structures scattered throughout the site. The plan was obtained and modified from Uribe et al. (2020).



Figure 6.6: Ground photo of Iluga Túmulos, showing a monolithic structure and a tumuls. Image from Uribe et al. (2020).

The presence of the Inka state in this site is well documented by its proximity to the Inka trail or Qapaq ñan, and the identification of Inka structures and characteristic Late Period pottery such as Inka Cuzco polychrome, provincial Saxzamar Inka Pacajes pottery, and local Inka from Tarapaca (Urrutia Lorenzini, Uribe Rodríguez and González Munita, 2023; Palacios Hernández et al., 2022; Uribe, Torres and Gajardo, 2020c). This assemblage included various bowls and jars, including characteristic Inka aryballos (Uribe et al., 2020c). Apart from these materials, the evidence suggests the presence of ceramics from distant geographical areas (e.g., Atacama, Arica, Isluga) as the result of important exchange and interactions mainly during the Late Intermediate Period (Uribe et al.,2020c). The absence of soot marks in most of the ceramics and the morphofunctional characterization of the containers identified in these ceremonial spaces indicate that, for the most part, ceramic vessels were not used for cooking but rather for storing and transporting food and alcoholic beverages (González, Gómez and Uribe, 2023; Uribe et al., 2020c).

Various ceramic potsherds were found within architectural structures, tumuli, and ceremonial areas (Palacios Hernández et al., 2022). Plant macroremains, primarily *algarrobo*

and secondly maize, were identified in these areas (Figure 6.7 and 6.8), along with marine animals and South American camelid remains (Figure 6.9 and 6.10) (González et al., 2023; González, 2020; Segura and Rivera, 2020).

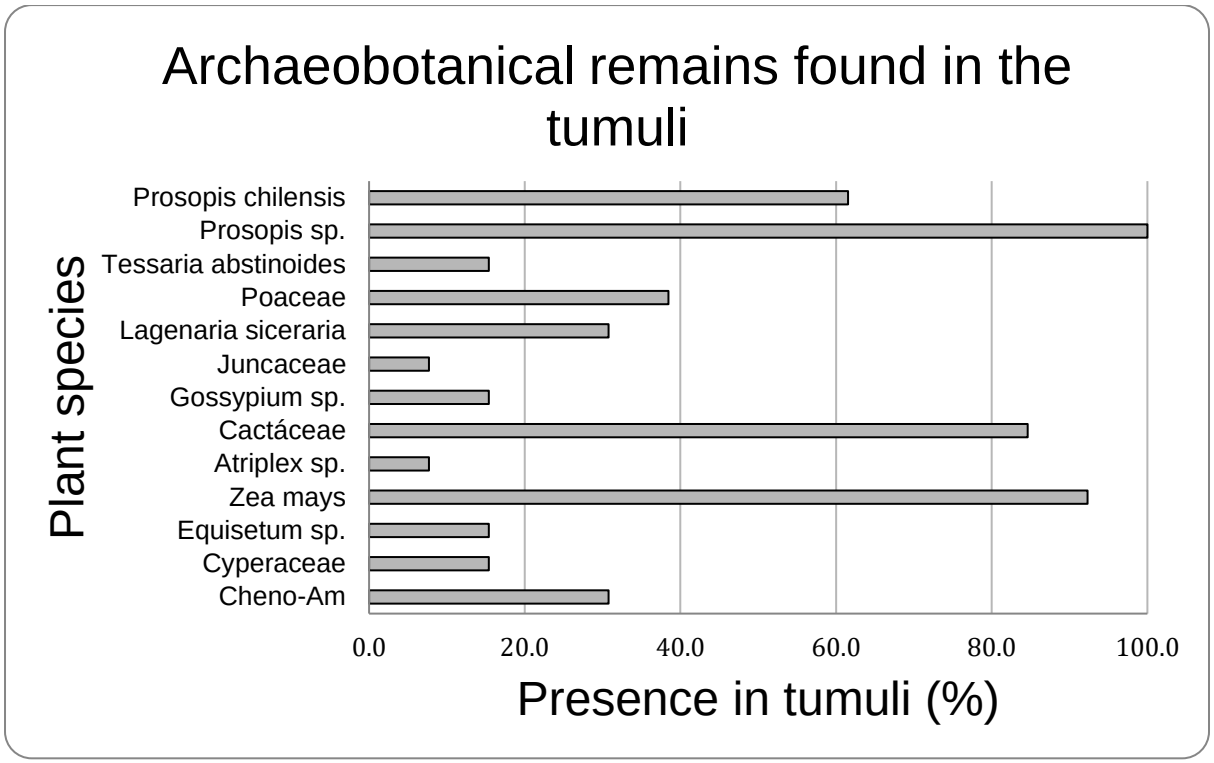


Figure 6.7: Percentage (%) of plant species found in a total of 13 tumuli. Figure created by the author.

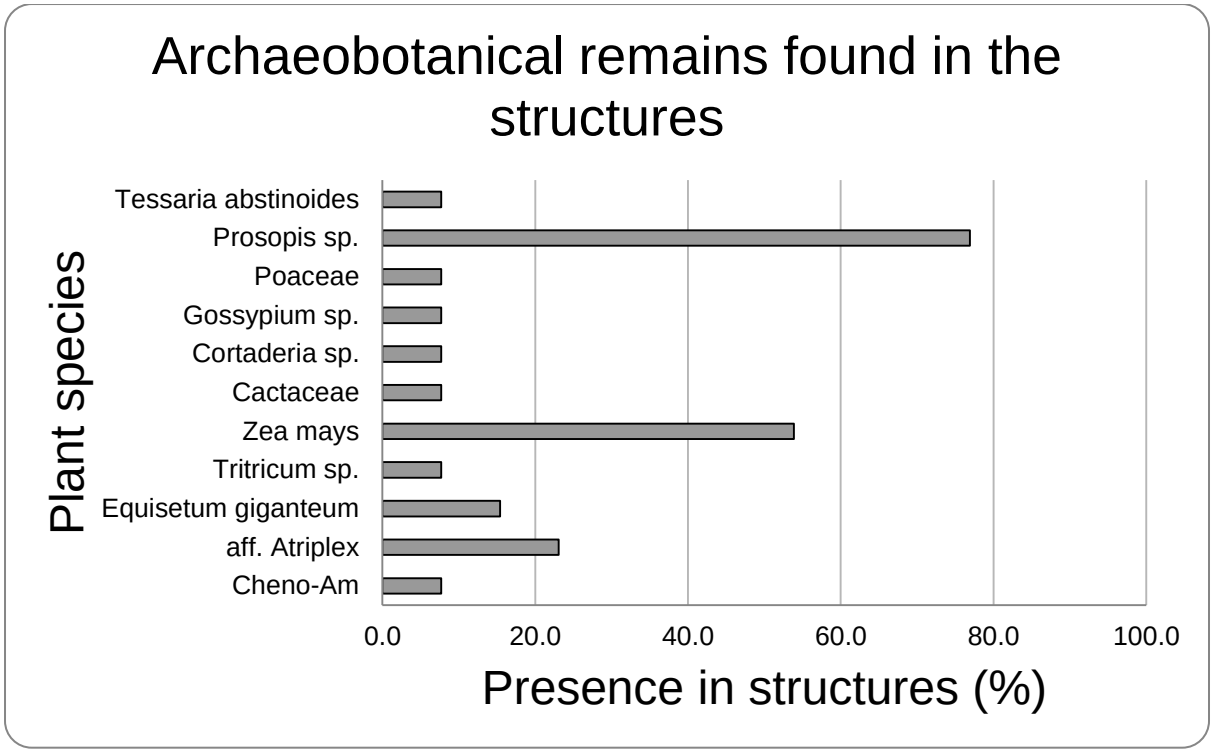


Figure 6.8: Percentage (%) of plant species found in a total of 13 structures. Figure created by the author.

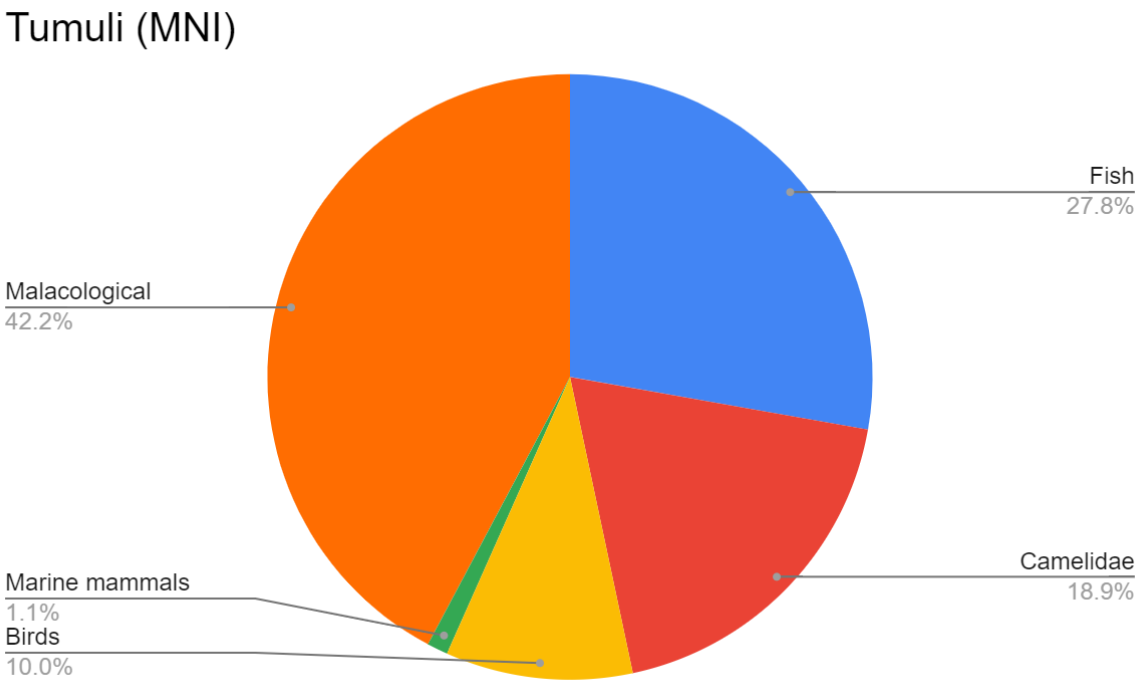


Figure 6.9: Minimal number of individuals (MNI) for zooarchaeological remains found in a total of 13 tumuli from Iluga Túmulos. Figure created by the author.

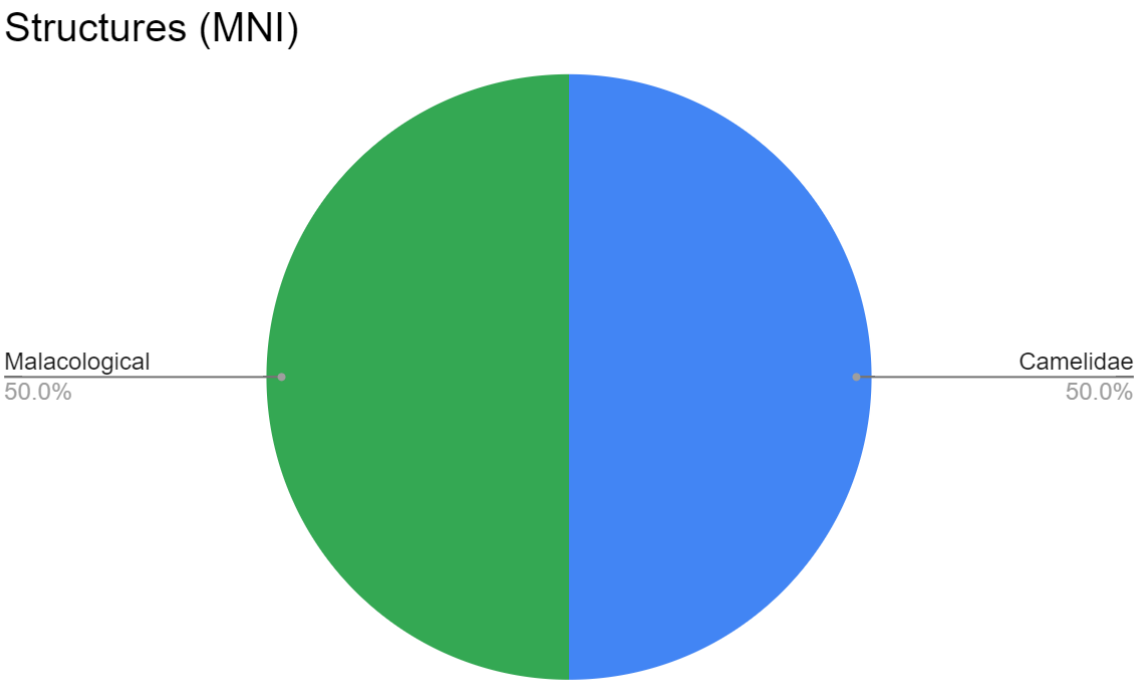


Figure 6.10: Minimal number of individuals (MNI) for zooarchaeological remains found in a total of 13 structures from Iluga Túmulos. Figure created by the author.

6. Materials and Methods

We analysed sixteen potsherds from jars or liquid containers and five bowls associated with different structures and tumuli (Figure 6.11). The majority of the samples consisted of Late Period pottery, mainly Inka Cuzco polychrome, provincial Inka and local Inka vessels (Appendix 12). Pottery from the Formative Period (3000 – 1000 cal BP) and Late Intermediate Period (1000 - 500 cal BP) were included to identify any changes or continuities on their use when compared with the Inka pottery (Figure 6.12). Late Intermediate Period and Formative Period pottery consisted of different styles and traditions from Tarapacá and other regions of the Atacama Desert, which converged in Iluga Túmulos throughout its occupation. All pottery was recovered from the surface of the structures and tumulus; therefore, due to the possible overlap between the Late Intermediate Period and the Late Period, potsherds associated with the former are within the category of Late Intermediate-Late Period (LI-LP) pottery.

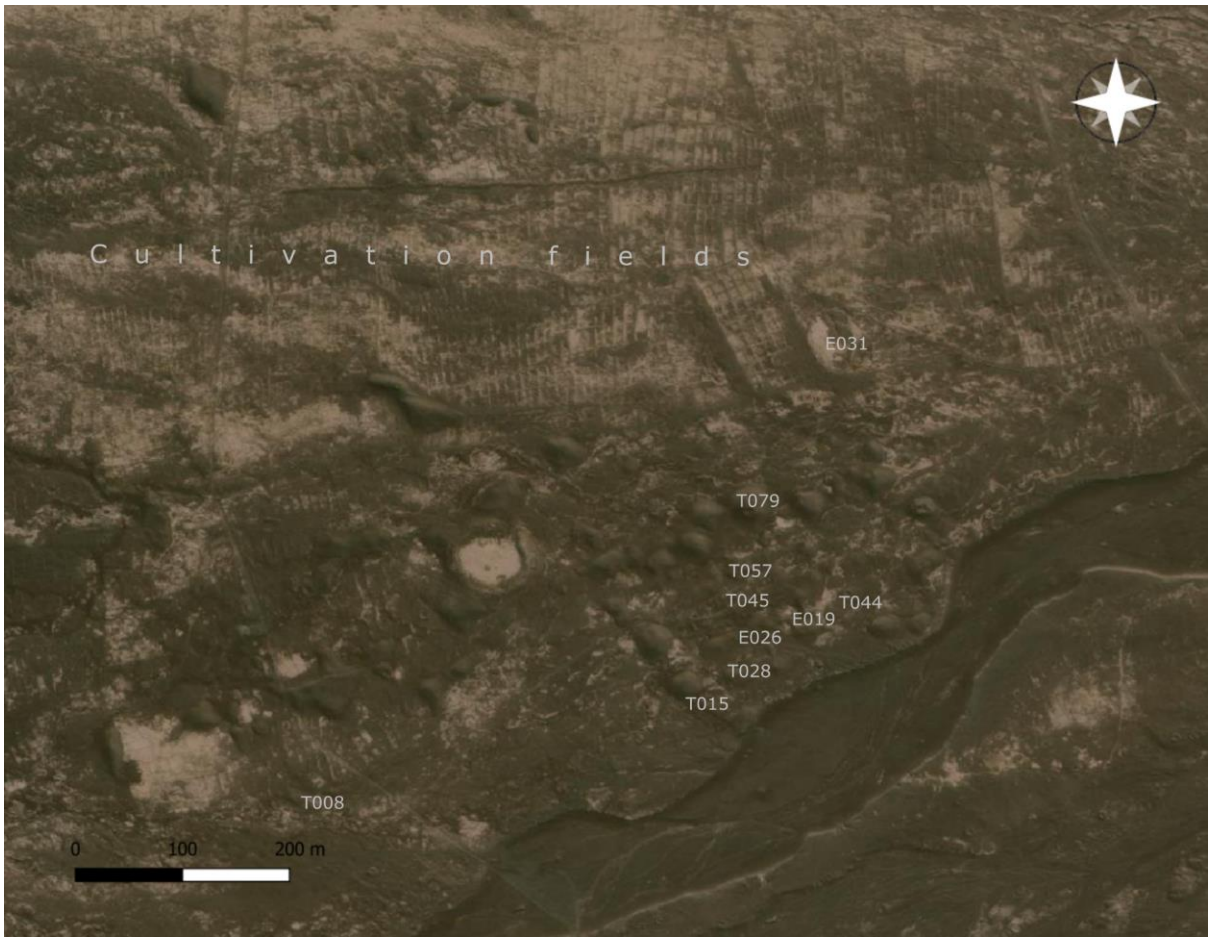


Figure 6.11: View of Iluga Túmulos site. Structures (E) and Tumuli (T) from where the pottery fragments were recovered are labelled. The map also shows a clear image of the cultivation fields. Figure created by the author.

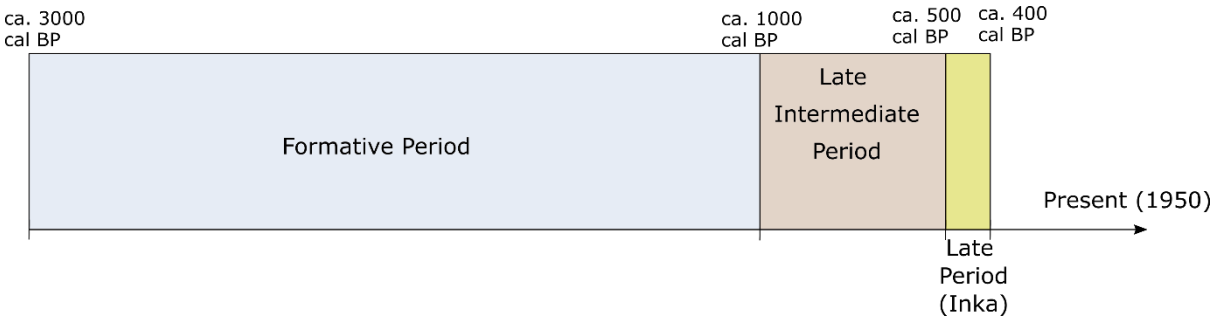


Figure 6.12: Chronological sequence of pre-colonial Tarapaca, northern Chile. Timeframe comprises from the Formative period until the arrival of the European colonisation. Figure created by the author.

All these pots did not show signs of exposure to a heating source (Uribe et al., 2020c). We also included archaeological maize kernels from Iluga Túmulos (Figure 6.13) previously used by Montalvo-Cabrera et al. (2024) for $\delta^{13}C$ and fatty alcohol distribution references.



Figure 6.13: Archaeological maize kernels from Iluga Túmulos site. Photograph by the author. Figure created by the author.

6.1 Organic residue analysis

Prior to the extraction process, around 2 – 3 mm of the inner surface of the pottery fragments was drilled to obtain 1 to 2 g of pottery powder. Samples were stored inside a sealed hatch tube, at room temperature until the next day.

A one-step acidified methanol (AE) protocol was followed for extraction and methylation of the compounds (Craig et al., 2013). Each batch included a blank tube to monitor contamination. To evaluate possible lipid loss throughout the extraction process, an internal standard (n-tetratriacontane (C_{34}): 10 µg) was incorporated in each tube. Around 4 mL of methanol were included in each hatch tube, previous to their sonication for 15 min. Concentrated sulphuric acid (800 µL) were incorporated in each sample, after which they were placed on a heating block at 70ºC for 4 hrs. Three successive extractions were employed using *n*-hexane to separate the lipids from the mixture. Potassium carbonate was employed in neutralising the extracts while being transferred into a new and clean hatch tube. Subsequently, lipids were concentrated under a gentle stream of nitrogen and, resuspended in *n*-hexane at appropriate dilution for gas chromatography-flame ionization detector (GC-FID) and gas chromatography-mass spectrometry (GC-MS) analysis. An internal standard (n-hexatriacontane (C36): 10 µg) was included to infer lipid yield and concentration.

All acid-extracted samples were derivatized with N,O-bis(trimethylsilyl) trifluoroacetamide with 1% trimethylchlorosilane (BSTFA+TMCS, 99:1) to identify long-chain fatty alcohol distributions. Samples were resuspended in 100 µL of n-hexane, from which only half was transferred into a clean hydrolysis vial for further derivatization. Samples were dried completely under a gentle stream of nitrogen and resuspended in 100 µL of n-hexane. Around 6 to 8 drops of BSTFA+TMCS, 99:1 was included before placing the samples on a heating block for 1 hr at 70ºC. Subsequently, lipids were resuspended in half of the dilution employed for GC-c-IRMS injection. Samples were posteriorly transferred into a new GC-vial with a conical insert and analysed within 48 hours through GC-MS.

Around 2 g of pottery powder was drilled from 8 samples for solvent extraction (SE), as in Colonese et al. (2017b). Dichloromethane:methanol (DCM:MeOH) 2/1 v/v was incorporated in each hatch tube before sonicating the samples three times. Each batch included a blank tube to assess contamination, and internal standard (n-tetratriacontane (C34): 10 µg). After being centrifugated, samples were transferred into a clean hatch tube and dried under a gentle stream of nitrogen. Lipids were resuspended in 1 ml of DCM:MeOH 2/1

v/v and transferred to a clean hydrolysis vial. Samples were completely dried and then resuspended in 500 µL of DCM from which a 100 µL aliquot was taken and transferred into a new hydrolysis vial, while the rest was dried and stored under appropriate conditions. Samples were completely dried and resuspended in 100 µL of *n*-hexane. Derivatization of samples was followed by adding six to eight drops of BSTFA+TMCS, 99:1, and placing all samples on a heating block for 1 hr at 70ºC. A gentle stream of nitrogen was used for fully drying the content of each tube. Lipids were resuspended in *n*-hexane at appropriate dilutions, while adding Internal standard C_{36} (10 µg) for lipid yield and concentration. Sample were analysed through GC-FID and GC-MS within 48 hrs.

Lipids were first screened and quantified by employing an Agilent 7890B high-temperature gas chromatograph (Agilent Technologies, Cheadle, Cheshire, UK) coupled with a flame ionization detector (HT-GC-FID). A 100% Dimethylpolysiloxane DB-1 (15 m X 320 µm X 0.1 µm; J&W Scientific Scientific, Folsom, CA, USA) column was used to inject in splitless mode 1µL of acid-extracted samples into the GC at 300°C. A constant flow rate of 2 mL min-1 of helium was used to carry the samples throughout the column. The oven temperature was set at 100°C for 2 minutes, subsequently rising 20°C min -1 up to 325°C, where it set for 3 minutes. Around 1 µL of solvent-extracted samples were injected in splitless mode at 350°C. The oven temperature was set at 50°C, settling for 2 minutes, subsequently rising 10°C min-1 up to 375°C, where it held for 10 minutes.

Samples were analysed by using an Agilent 7890A series chromatograph attached to an Agilent 5975C inert XL mass selective detector with a quadrupole mass analyser (Agilent Technologies, Cheadle, Cheshire, UK), and a Trace Ultra 1310 GC, coupled with an ISQ MS (Thermo Fisher, Bremen, Germany). Fatty alcohols were screened by setting the mass spectrometer in single ion monitoring (SIM) to target specific ions (m/z 71, 75, 299, 327, 355, 383, 411, 439, 467, 495, 523, 551).

Samples were injected (1 µL) in splitless mode into a methylpolysiloxane (5%-phenyl) DB-5ms (30 m X 0.25 mm X 0.25 µm; J&W Scientific, Folsom, CA, USA) column at 300ºC. A constant flow rate of 2 mL min^{-1} of helium was used to carry the samples throughout the column. The temperature of the oven was set at 50ºC for 2 min, then rising 10ºC min^{-1} to 325ºC, holding for 15 min. The ionisation energy of the mass spectrometer was 70 eV, from which a spectrum was obtained by scanning ions between m/z 50 and 800.

The presence of Isoprenoid acids and ω-(o-alkylphenyl) alkanoic acids (APAAs) as biomarkers of several commodities was assessed through GC-MS analysis by employing a

50% cyanopropyl-methylpolysiloxane DB-23 column (60 m x 0.25 mm x 0.25 µm; J&W Scientific, Folsom, CA, USA) as in (Shoda et al., 2017). Samples were injected in splitless mode (1µL) at 300ºC. Helium was used as the carrier gas at a flow rate of 1.5 mL min^{-1}. The temperature of the oven was set at 50ºC for 2 min, increasing 10ºC min-1 to 100ºC, after which it rose 4ºC min^{-1} up to 140ºC, and subsequently, 0.5ºC min^{-1} to 160ºC, to finally rise 20ºC min^{-1}, reaching 250ºC where it settled for 20 min. Target ions of the three main isoprenoid acids (m/z 74, 101, 171, 326 for phytanic acid; m/z 74, 88, 101, and 312 for pristanic acid, m/z 74, 87, 213 and 270 for 4, 8, 12 - trimethyltridecanoic acid (4, 8, 12 - TMTD)), and APAAs of carbon length C_{16} to C_{22} (m/z 74, 105, 262, 290, 318, 346) were screened by setting the mass spectrometer in single ion monitoring (SIM) mode.

GC-MS results were computed by using MSD Chemtation F.01.03.2357 software. Criteria for compound identification were based on retention time, mass spectrum and comparison with the National Institute of Standards and Technology (NIST) library. Integration of peaks was performed using Agilent Mass Hunter Quantitative Analysis software version B.07.01/ Build7.1.524.0 for GC-MS.

6.2. GC-c-IRMS and Single Compound $\delta^{13}C$ Analysis

All samples (n=21) were eligible for single compound $\delta^{13}C$ analysis of $C_{16:0}$ and $C_{18:0}$ alkanoic acids, and meet the minimal alkanoic acid quantity needed for injection. A Delta V Advantage Isotope Ratio Mass Spectrometer (Thermo Fisher Scientific, Bremen, Germany), coupled to a Trace Ultra 1310 Gas Chromatograph (Thermo Fisher) with a GC IsoLink II interface (with a CuO combustion reactor held at 850ºC) was used. Samples were injected (1 µl) into an ultra-inert fused-silica DB-5ms UI column (60 m x 0.25 mm x 0.25 µm; J&W Scientific, Folsom, CA, USA). A flow rate of 2 mL min^{-1} of ultra high-purity grade helium was used to carry the samples throughout the column. Parallel acquisition of molecular data was obtained by deriving a small part of the flow into an ISQ mass spectrometer (Thermo Fisher). The temperature of the oven was set at 50ºC for 0.5 min, rising 25ºC min^{-1} to 175ºC, then 8ºC min^{-1}, reaching 325ºC, settling for 20 min.

Eluted compounds were combusted into CO_2 and ionised in the mass spectrometer through electron impact. Ion intensities of m/z 44, 45 and 46 were recorded for automatic computing of the $^{13}C/^{12}C$ of each peak in the extracts. Softwares Isodat (Thermo Fisher Scientific, Bremen, Germany) and LyticOS (Isoprime, Cheadle, UK) were used for

computation, based on the comparison with repeatedly measured standard reference gas (CO_2) of known isotopic composition. Calculated $\delta^{13}C$ values are presented in parts per mil (‰), relative to the Vienna PeeDee Belemnite (V-PDB) international standard.

Sample batches were calibrated using a linear calibration curve (average $R^2 = 0.994 \pm 0.001$ in one batch) based on expected versus measured $\delta^{13}C$ values of n-alkanes and n-alkanoic acid esters from international standards (Indiana A7 and F8-3 mixtures). The instrument's accuracy was determined on *n*-alkanoic acid methyl esters of known isotopic signal (Indiana F8-3, 6 measurements). The mean and standard deviation of the *n*-alkanoic acids were -30.11 ± 0.14‰ for $C_{16:0}$ (reported mean value vs. V-PDB -29.90 ± 0.03‰) and -23.22 ± 0.18 ‰ for $C_{18:0}$ (reported mean value vs. V-PDB -23.24 ± 0.13‰). Laboratory standard mixtures were regularly injected between samples to monitor the instrument's precision (22 measurements), showing Alkanoic acids mean ± SD values of -30.75 ± 0.18‰ for $C_{16:0}$ methyl ester and -26.40 ± 0.20‰ for $C_{18:0}$ methyl ester. All samples were corrected for the methylation of the alkanoic acids that occurred during acid extractions by using a mass balance formula to compare the carbon isotopic values with a standard mixture of $C_{16:0}$ and $C_{18:0}$ fatty acids of known isotopic composition, previously included in each batch during the acid extractions. To better interpret the archaeological data, the $\delta^{13}C$ values of the modern reference fats were corrected to atmospheric $\delta^{13}C$ variations between the Early Holocene Epoch and the present (Hellevang and Aagaard, 2015).

7. Results

All samples yielded interpretable lipid quantities (>5 μg^{-1}; min: 13.1μg^{-1}; max: 2117.2 μg^{-1}; mean= 192 μg^{-1}) according to standardised criteria (Evershed, 2008a). Extracts exhibited a distribution of aliphatic compounds comprising fatty acids ($C_{8:0}$ - $C_{32:0}$), unsaturated fatty acids ($C_{14:1}$ - $C_{24:1}$), fatty alcohols (C_{20}-C_{32}), and α,ω-dicarboxylic acids (C_5 - C_{22}). Carbon stable isotope ($\delta^{13}C$) analyses of the main alkanoic acids $C_{16:0}$ and $C_{18:0}$ allowed a better discrimination of the residues' origin. The absence of heating biomarkers in most pots confirms previous hypotheses on the unlikely use of these vessels in cooking events, and their main employment for storing, serving, and transporting commodities (Uribe et al., 2020).

7.1 Jars

Most samples showed $\delta^{13}C$ values concordant with an origin from C_3 products or between C_3 products and maize references (Figure 6.14). These pots consisted of Late Formative Period vessels and several Late Intermediate-Late Period and Late Period pots, including two Inka Cuzco polychrome aryballos. Short- and medium-chain diacid distributions were identified in most samples, potentially from the oxidation of unsaturated fatty acids from plant commodities (Regert et al., 1998). Long-chain alkanes and long-chain fatty acids were identified in several samples, indicating the use of leaves and stems (Dove and Mayes, 2006). Plant sterols were present in four samples, while the high palmitic ($C_{16:0}$) over stearic ($C_{18:0}$) fatty acid ratios (P/S) likely denote a predominant origin from plant commodities (Dunne, 2021). Only sample IT_229, a local Inka pot (IKL), yielded symmetrical long mid-chain ketones, which are characteristic compounds found in the epicuticular wax of plant leafs (Drieu et al., 2019; Raven et al., 1997; Evershed et al., 1995).

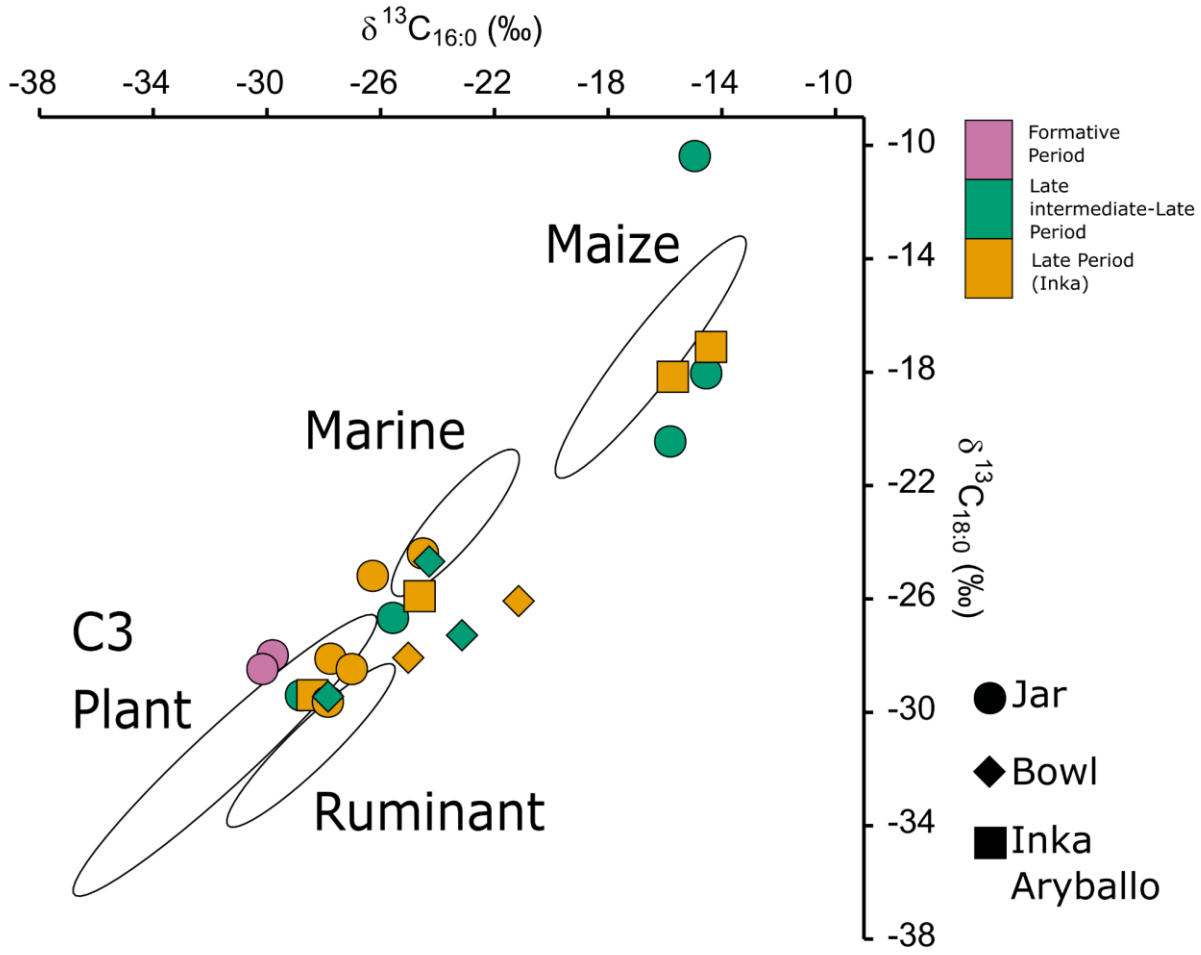


Figure 6.14: Scatterplot for single-compound carbon stable isotope analysis of the main alkanoic acids extracted from pottery from Iluga Túmulos site. Confidence ellipses (P=68%) were built using modern and archaeological references. Figure created by the author.

Three Late Intermediate Period pots and two Inka Cuzco polycrhome aryballos presented more positive $\delta^{13}C$ measurements relative to C_3 plant references, likely reflecting the storage of maize products in these pots (Figure 6.14). Long-chain fatty alcohols up to C_{32} have been associated with the identification of C_4 panicoid grasses like maize (Rommerskirchen et al., 2006; Reber and Evershed, 2004b). Previous studies have shown that C_{32} fatty alcohol is abundant in maize (Bianchi and Avato, 1984), while fatty alcohol distributions up to C_{32} or above have been previously identified in pre-colonial South American pots potentially used to cook and store maize products (Montalvo-Cabrera et al., 2024; Admiraal et al., 2023; Reber and Evershed, 2004a). Fatty alcohols were only detected at trace levels in the archaeological samples and maize kernel. However, by performing spectral subtraction, it was possible to reduce background noise and obtain a characteristic mass

spectrum of the identified peaks, likely reflecting the presence of small traces of even-numbered fatty alcohol distributions up to C_{32}-OH in most samples. It is possible that the high lipid concentration found in every sample masked the low quantity of fatty alcohols, making them almost undetected; this issue requires further analyses and experimentation.

By plotting the offset between $\delta^{13}C_{18:0}$ and $\delta^{13}C_{16:0}$ ($\Delta^{13}C$) against $\delta^{13}C_{16:0}$ (Figure 6.15), it is possible to notice how ^{13}C enriched samples exhibited negative and positive $\Delta^{13}C$ values, different from maize reference $\Delta^{13}C$ offsets. Negative $\Delta^{13}C$ values have been employed as a proxy in identifying ruminant fat and by-products in the residues (Copley et al., 2003; Craig et al., 2012; Dolbunova et al., 2023). Nevertheless, because of the low abundance of $C_{18:0}$ in maize, negative $\Delta^{13}C$ can result from the contribution of C_3 commodities exhibiting a higher abundance of $C_{18:0}$ (Admiraal et al., 2023). Conversely, when C_4 organisms yield a higher contribution of $C_{18:0}$ compared to C_3 commodities, positive $\Delta^{13}C$ values are likely to be expected.

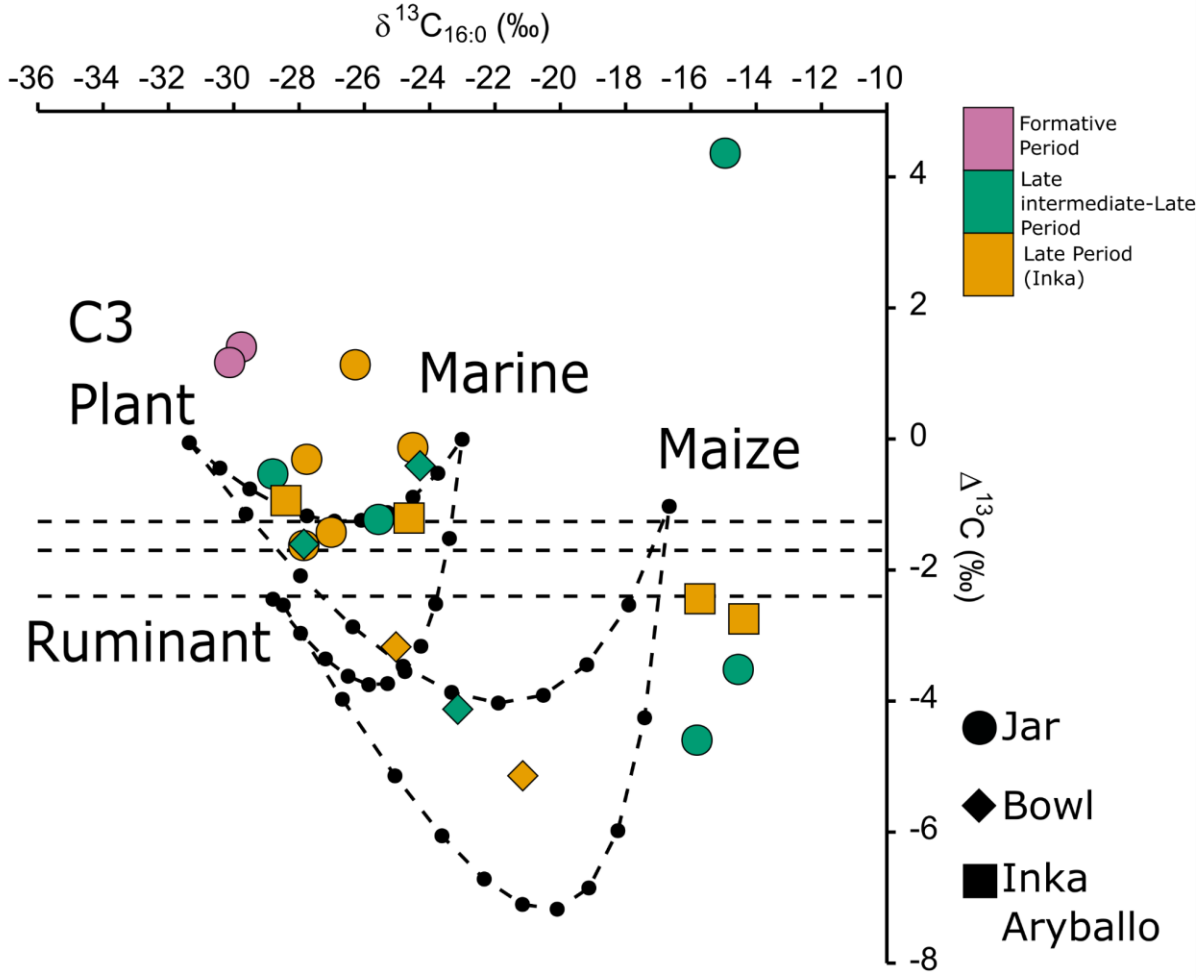


Figure 6.15: Scatterplot for single-compound carbon stable isotope analysis, showing values for $\delta^{13}C_{16:0}$ against $\Delta^{13}C_{(C:18-C:16)}$. Hypothetical mixing lines show a 10% increment between averaged $\delta^{13}C$ endpoints representing commodities found at Iluga Túmulos site. Figure created by the author.

Samples from Iluga Túmulos showed a wide distribution of $\Delta^{13}C$ values, potentially indicating the contribution of $C_{16:0}$ and $C_{18:0}$ fatty acids from C_3 and C_4 products in different proportions. The low $\Delta^{13}C$ values from four samples showing a positive $\delta^{13}C$ signal are likely the result of mixing maize with C_3 products. The identification of plant sterols in these samples strongly supports that C_3 and C_4 plants were likely used in the same container.

Although most samples indicate an origin from plant commodities, the use of animal fat in some containers was also a possibility. Due to the abundance of llama bone remains found at Iluga Túmulos, the possible input of ruminant carcass fat was also assessed. For this,

the relation between both phytanic acid diastereomers 3S,7R,11R,15 (SRR) and 3R,7R,11R,15 (RRR) has been useful in identifying an origin from ruminant fat (>64%) and marine organisms (i.e., 76% ±16.6) in the residues (Dolbunova et al., 2023; Lucquin et al., 2016b). A phytanic acid-SRR diastereomer contribution coincident with a ruminant origin was identified in three jars container samples, including one Late Intermediate-Late Period potsherd associated with the storage of maize products. The latter also exhibited a large $\Delta^{13}C$ offset resembling the use of dairy products (<-3.3‰), which in the context of pre-colonial South America has been associated with the employment of maize and ruminant carcass fat in the same vessel (Admiraal et al., 2023). Previous studies have mentioned the use of ruminant fat to create a waterproofing effect in Andean pottery (Miyano et al., 2017; Lantos et al., 2017); however, further experimentation is needed to evaluate the association of this practice with the presence of phytanic acid in the residues.

7.2. Serving bowls

Residues from the five analysed bowls at Iluga Túmulos showed $\delta^{13}C$ values indicating various origins. Large negative $\Delta^{13}C$ offsets were identified in residues extracted from three bowls: two Inka vessels (IT_145 and IT_7) and one associated with the Late Intermediate-Late Period (IT_1). A negative $\Delta^{13}C$ offsets <-2.4‰ and a phytanic acid-SRR diastereomer contribution below 64% identified in these samples potentially support their origin from South American camelid carcass fat (Chapter 5, Materials and Methods).

Moreover, the relative ^{13}C enrichment in these three samples and their considerably negative $\Delta^{13}C$ values might indicate that maize was incorporated into these containers. Notably, only sample IT_145, an Inka Cuzco polychrome bowl, exhibited a distribution of APAA-C_{18} isomers, suggesting its exposure to a heating source. A series of C_{18}, C_{20} and C_{22} APAAs are formed when unsaturated fatty acids from plants and animal commodities are subjected to high temperatures (>200ºC), during cooking practices (Bondetti et al., 2020; Evershed et al., 2008; Hansel et al., 2004). Particularly, the ratio between APAA-C_{18} E and H isomers (E/H) is an effective proxy to differentiate **1)** cereals, fruits and non-leafy vegetables, **2)** animals, and **3)** leafy plants (Bondetti et al., 2020). The high E/H ratio yielded from sample IT_145 potentially indicates that cereals, fruits and non-leafy vegetables were included inside this container.

Two Late Intermediate-Late Period bowls yielded residues with more positive $\Delta^{13}C$ values. From these pots, lipids recovered from sample IT_73 indicated that it was likely used

to store mainly C_3 products. Notably, sample IT_80 yielded evidence indicating a possible marine origin. Isoprenoid acids pristanic, 4, 8, 12-trimethyltridecanoic and phytanic acids which are highly abundant in aquatic organisms, were identified along with a phytanic acid-SRR diastereomer contribution coincident with an aquatic origin (Lucquin et al., 2016b). A $\delta^{13}C$ signal of -24.3‰ and -24.7‰ for $C_{16:0}$ and $C_{18:0}$, respectively, is concordant with an origin from marine products. The absence of long-chain APAA C_{20} and C_{22} as key biomarkers for the identification of aquatic resources in the residues limits the possibility of suggesting sample IT_80 as a container used to store marine products (Bondetti et al., 2020; Evershed et al., 2008). However, since IT_80 was likely used as a storage vessel and not as a cooking pot, it is possible that it never reached the temperature needed for APAA formation.

8. Discussion

The extraction of organic residues from pottery artefacts allowed us to evaluate the significance that maize had in ceremonies and feasts held at Iluga Tumulos site during the occupation of Tarapacá by the Inka state. Previous stable isotope analyses on human remains have demonstrated that maize was a primary dietary staple during the Late Intermediate and Inka periods in Tarapacá. However, the analysis of organic residues in ceramic fragments indicates that maize was no more prominent than C_3 resources during feasts and ceremonies at Iluga Túmulos. The molecular and isotopic evidence suggests the significance of C_3 products during the Inka state occupation, which seems to have been constant since the Formative Period. Based on our results, we acknowledge the importance of differentiating between diet and foodways, with the latter not necessarily a consequence of the former but rather a reflection of cultural aspects that shaped the social and political life of populations.

The consumption of maize *chicha* during feasts celebrated by the Inka state is argued to have been the most influential socio-political act, for which several jars and containers were employed for storage and drinking (Bray, 2003; Dillehay, 2003). However, molecular and isotopic data from the residues indicate that C_3 plants were likely the main commodity used in these jars. Noticeably, the finding of two Cuzco polycrhome aryballos to store C_3 plant commodities contrasts with general assumptions and traditional views on the function given to aryballos and the significance of maize as the primary commodity used in preparing *chicha*.

Similar results on Inka aryballos from northwest Argentina have been interpreted to reflect the active role of local communities in the celebration of Inka feasts, which also denoted

particular socio-political relations between the Inka state and its southern provinces (Giovannetti, 2021; Lantos et al., 2017, 2015; Orgaz, 2012). In northern Chile, based on the diversity of the material evidence found in Atacama, Uribe (2004) indicates that the Inka state maintained and legitimised its authority by allowing an active role of local leaders and communities in socio-political gatherings and festivities. The employment of Inka pottery, especially Cuzco aryballos, in storing C_3 products might reflect an active role by local communities where other resources were included and permitted by the Inka state during feasts and ceremonies. The large number of *algarrobo* remains found at the Iluga Túmulos site might indicate it as the main resource used in the preparation of alcoholic beverages stored in Inka pottery. *Algarrobo* trees were introduced into Tarapacá from trans-Andean regions during the Formative Period, becoming a crucial food and fuel resource for the communities of the Pampa del Tamarugal, as well as being highly represented in pre-Inka ceremonial sites (McRostie et al., 2017; García et al., 2014; García and Uribe, 2012). Thus, *algarrobo* probably represented a resource with high symbolic significance for local groups. Thus, the Inka state perhaps allowed and supported its consumption during feasts celebrated at Iluga Túmulos to maintain socio-political relations with local leaders and ensure tributary labour or *mita*, in this case, enrolled in agricultural activities.

Although the storage of C_3 plant-based *chicha* is suggested for these containers, signs of fermentation are difficult to spot from organic residue analysis (Craig, 2021). Several studies have indicated the possibility of identifying hopanoid degradation product hopane as a biomarker for the presence of bacteria involved in food fermentation occurring inside archaeological pottery (Rageot et al., 2019; Correa-Ascencio et al., 2014). While this evidence is missing from our data, it is worth noting the potential biases on bacterial hopanoids due to the protagonic role of microorganisms in postdepositional organic diagenesis (Craig, 2021).

Organic residues from several Late Intermediate-Late Period and Inka bowls revealed their potential use for storing South American camelid carcass fat. Large quantities of llama remains have been identified in ceremonial and feast areas in Late Intermediate Period sites, indicating that their consumption was possibly important during socio-political gatherings (García and Uribe, 2012). At the same time, llamas were essential for the Inka state, playing a prominent role in ritual sacrifices and feasts (Valdez, Bettcher and Huamaní, 2020). Parts of llama carcasses were probably included in stews or *locro* served in bowls. *Locro* is part of the Andean cuisine and has also been described for the Inka period (Bray, 2003).

The presence of a Late Intermediate-Late Period bowl (IT_80), possibly used for serving marine products is intriguing. Iluga Túmulos presents evidence for the contact

between the Pampa del Tamarugal and the coast (González et al., 2023; González, 2020). Notably, the identification of marine products at the Inka administrative centre of Tarapacá Viejo indicates that marine resources were likely distributed from there to different points of the region (Uribe et al., 2012). It is interesting to note that marine products were mainly associated with the tumuli, while their identification in architectural structures was relatively limited (Gonález, 2020). Sample IT_80 was found to be associated with tumulus T015, which comprised several fish and mollusc remains, possibly indicating the significance of marine products in ceremonial activities related to the tumuli area. Marine resources might have been a prestigious and essential product as mediators in socio-political gatherings and feasts, possibly to ensure tributary labour through the mita system during the Inka occupation of Tarapacá; however, further research is needed to assess this possibility.

The *Tawantinsuyu* covered a vast territory, comprising various cultural groups. That diversity is reflected in the socio-political relationships between the dominated communities and the Inka state. We cannot assume that maize was the central mediator of socio-political gatherings, as other products, possibly local resources with a solid ideological and symbolic significance, were likely included. Therefore, It becomes imperative to re-evaluate the hegemony of maize as a socio-political mediator in Inka feasts and ceremonies and to acknowledge the cultural and culinary diversity of local communities dominated by the Inka state.

9. Conclusion

As known from multiple historical sources maize had a symbolic and ideological significance for the Inka state, particularly in the form of an intoxicating drink known as *chicha*. The distribution of maize *chicha* and its promotion as a state beverage was a strong driver of the Inka state expansion. However, organic residue analysis on ceramics from the Iluga Túmulos site has shown that maize *chicha* was not the only and primary mediator in socio-political gatherings, as C_3 products held a strong significance for local communities. These products likely consisted of South American camelid carcass fat, probably included in stews, and C_3 plants, possibly used for producing alcoholic beverages.

Archaeobotanical evidence shows that *algarrobo* fruits have largely been exploited and used by local communities in feasts and ceremonies. The fruits of the *algarrobo* were probably an indispensable product to be included during important socio-political gatherings, likely in

the form of alcoholic beverages, which was nonetheless allowed by the Inka state. These considerations reflected the active role of local communities in the decision-making of important Inka celebrations and feasts, possibly due to particular socio-political relations held between local communities and the Inka state.

Our results demonstrate the importance of considering the cultural diversity of local groups dominated by the Inka state, and, therefore, to address their foodways by considering this diversity. We also call for caution when interpreting ceremonial and feasting contexts, and to take into consideration that the products consumed during feasts and ceremonies do not necessarily reflect the population's diet. Foodways are invested with cultural and symbolic meaning and, thus, can reflect the diversity of culinary preparations and their meaning for past societies.

10. Acknowledgements

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Chapter 7: General discussion

1. Introduction

Maize has received significant attention in the literature as a staple food in South American farming societies. Scholars working on the west side of the Andes have referred to its central role in food production and as an essential commodity with strong ideological symbolism when consumed as *chicha* or maize beer during feasts and ceremonies. The main objective of this thesis was to examine the socio-political and economic significance of maize in southwestern South America through organic residue analysis from pre-colonial pottery from southern and northern Chile.

Through a multiproxy analysis of organic residues, which integrated $\delta^{13}C$ analysis of the main alkanoic acids $C_{16:0}$ and $C_{18:0}$ and molecular data extracted from pre-colonial pottery from southern and northern Chile, it was possible to identify the use of ceramic containers to cook and store a variety of resources, including maize. The results from southern Chile indicated that maize did not become a staple food after its adoption and seemed not to have a protagonistic role in socio-political, economic and demographic changes. Organic residues from pottery artefacts from a coastal context indicated that marine and C_3 plants were probably more important than maize in sustaining the permanent settlement of El Vergel farmers on Mocha Island. In the Central Valley, the results from Cueva de los Catalanes and SMR5 sites demonstrated that throughout the Ceramic Period, most pots were used for processing C_3 plants and ruminant carcass fat. Maize was probably constrained to producing specific preparations such as fermented beverages; however, its incorporation into other dishes as a secondary ingredient cannot be ruled out.

The results from southern Chile align with the archaeobotanical and zooarchaeological evidence summarised in Chapters 4 and 5. The analysis of carpological remains identified a diversity of C_3 plants at Mocha Island and Cueva de los Catalanes, while the organic preservation in SMR5 limited further archaeobotanical analyses. From this group, Chenopodium quinoa was the most representative and abundant. Although maize was also identified at these sites, its representation was consistently lower than that of quinoa.

At the same time, camelid remains were also highly abundant in the Central Valley and Mocha Island. While the carbon isotope data on human bone remains from the Central Valley supports the preferential use of C3 products, relative 13C enrichment in human remains from Mocha Island is unlikely to come from maize consumption. Nitrogen stable isotopes on human remains indicated a possible overlap between maize and low-trophic marine products. The limited representation of maize in the organic residues and the identification of marine biomarkers in several pots from Mocha Island further support that δ13C and δ15N values on Mocha Island human remains probably reflected the contribution of marine commodities rather than the intake of C4 plants.

On the other hand, in northern Chile, maize was already a staple since 1000 cal BP (Santana-Sagredo et al., 2021). Maize carpological remains have been identified in large quantities at Iluga Túmulos, potentially aligning with the evidence on $\delta^{13}C$ and $\delta^{15}N$ of human remains (Santana-Sagredo et al., 2021). However, *algarrobo* remains were identified in larger quantities than maize, indicating that a preference for local resources possibly comprised a significant aspect of local culinary practices. The identification of marine and camelid bones in several tumuli and structures also indicated the possible use of these resources during feasts and ceremonies held at Iluga Túmulos.

The results from organic residue analysis from Iluga Túmulos site indicated that during the Inka occupation of Tarapacá around 500 cal BP, maize was not a significant commodity for preparing *chicha* consumed during ceremonies and feasts. Conversely, C3 plants were preferred, while using pottery to store camelid and marine products also denoted that feasting activities were more complex than only relying on the significant role of maize to explain complex socio-political relations in Tarapacá. The access to various products possibly served as a mediator in socio-political gatherings. The results contradict what has generally been assumed for the central role of maize in *chicha* production and consumption during Inka feasts and socio-political ceremonies (Bray, 2009; Dillehay, 2003; Hastorf and Johannessen, 1993; Murra 1980, 95).

Based on these two distant case studies, I suggest that the hegemonic view of maize as the most prominent crop or “the wheat of the Americas” cannot be generalised for southwestern South America. Additionally, I consider the assumption of maize as a primary resource to be undermined by the diversity of plants and animal resources that held culinary, symbolic, and economic significance for local pre-colonial groups. Therefore, more attention is needed to consider the diversity of coastal and terrestrial resources in the region and their

role as potential major drivers to explain western South America's transcendental socio-political and economic events.

The main objective of this thesis was approached through a number of research questions. To better structure the discussion, these research questions are listed below:

1.1 Is it possible to identify the use of maize in archaeological pottery through organic residue analysis?

Chapter 3 highlighted several studies that have indicated the potential use of organic residue analysis to identify the use of pottery containers to process and store maize-based products. However, this possibility had not previously been tested for pottery in western South America. Therefore, the first objective of this research was **to identify maize through a multiproxy approach combining the molecular and carbon isotopic composition of organic residues extracted from pre-colonial pottery from southern and northern Chile.**

Most organic residue studies of archaeological pottery compare their results with a solid molecular and $\delta^{13}C$ baseline. This practice has enabled a more robust identification of organic products from the residues. Maize references for organic residue analysis, as well as for other products from the Americas, are scarce and limited to few case studies (Admiraal et al., 2023; Lantos et al., 2015; March, 2013). Through several cooking experiments using non-tempered replica pots, it was possible to obtain molecular and isotopic references for modern maize, quinoa, Chilean hazelnut, madi, and *Lama glama* meat (Appendix 2, Table 2.2). Lipids were also extracted from archaeological maize kernels to obtain more robust molecular and isotopic references (Appendix 2, Table 2.1). The references presented in this research will help to contribute more reliable baselines for further studies in the Americas.

Due to the high content of C_{18x} unsaturated fatty acid in plant tissues, the exposure of modern plant references to high temperatures during cooking events allowed the formation of ω-(o-alkylphenyl) octadecanoic acid (C_{18}-APAA) isomers. According to Bondetti et al. (2020), a high ratio between the E and H isomers (E/H) can discriminate between **1)** cereals, fruits and non-leafy vegetables from **2)** animals, and **3)** leafy plants. By monitoring the temperatures during cooking experiments, it was possible to reach 200ºC for APAA formation in most plant products. The results showed that maize kernels, as well as seeds, grains and nuts from native

Chilean plant references from this study, exhibit a high E/H value concordant to the category of cereals, fruits and non-leafy vegetables.

Fatty alcohol C_{32} is likely found in panicoid grasses, such as maize (Rommerskirchen et al., 2006; Reber and Evershed, 2004; Bianchi and Avato, 1984). To obtain fatty alcohol distributions, acid-extracted samples from both modern and archaeological maize references were derivatized using BSTFA+TMCS(99:1). To identify fatty alcohol distributions and evaluate the potential use of C_{32}-OH for identifying maize in organic residues from archaeological pottery, the GC-MS was set in selected ion monitoring (SIM) mode to target specific ions (Chapter 5 and 6, Materials and Methods).

Only low amounts of fatty alcohols were detected, and even though a quantification method was employed using Agilent Mass Hunter Quantitative Analysis software, it was not possible to assess the abundance of C_{32}-OH in relation to the whole fatty alcohol distribution from modern and archaeological maize references. The low abundance of long-chain fatty alcohols was likely masked by the high abundance of fatty acids found in fresh modern samples and well-preserved archaeological maize kernels. These archaeological samples showed high lipid yields, which is consistent with the good organic preservation conditions of the Atacama Desert. However, this characteristic might have helped to mask the identification of long-chain fatty alcohols from the residues. This issue needs to be assessed through new extraction methods to allow a better separation of the compounds.

Cooking experiments led by Reber & Evershed (2004) yielded C_{32}-OH; however, most pottery used in these cooking experiments were posteriorly buried for 6 months, where the post-depositional diagenetic factors might have helped to equate the proportion of fatty alcohols and fatty acids found in the pots. At the same time, the authors used a less aggressive extraction method which might have served better for working with fresh plant products.

The extraction of organic residues from pre-colonial pottery from southern and northern Chile and analysis through GC-MS and GC-c-IRMS were successfully achieved in most samples. Notably, despite southern Chile's wet conditions and soil acidity, a considerably good quantity of samples extracted using an acidified methanol protocol yielded interpretable lipids (>5 μg^{-1}). This condition was also replicated for all samples from the Atacama Desert, showing high lipid concentrations. Although samples from northern Chile were considerably fewer than those from southern Chile (Mocha Island and Central Valley sites), the hyper-arid conditions of the Atacama Desert allowed a considerably higher lipid yield compared with southern Chile (Figure 7.1).

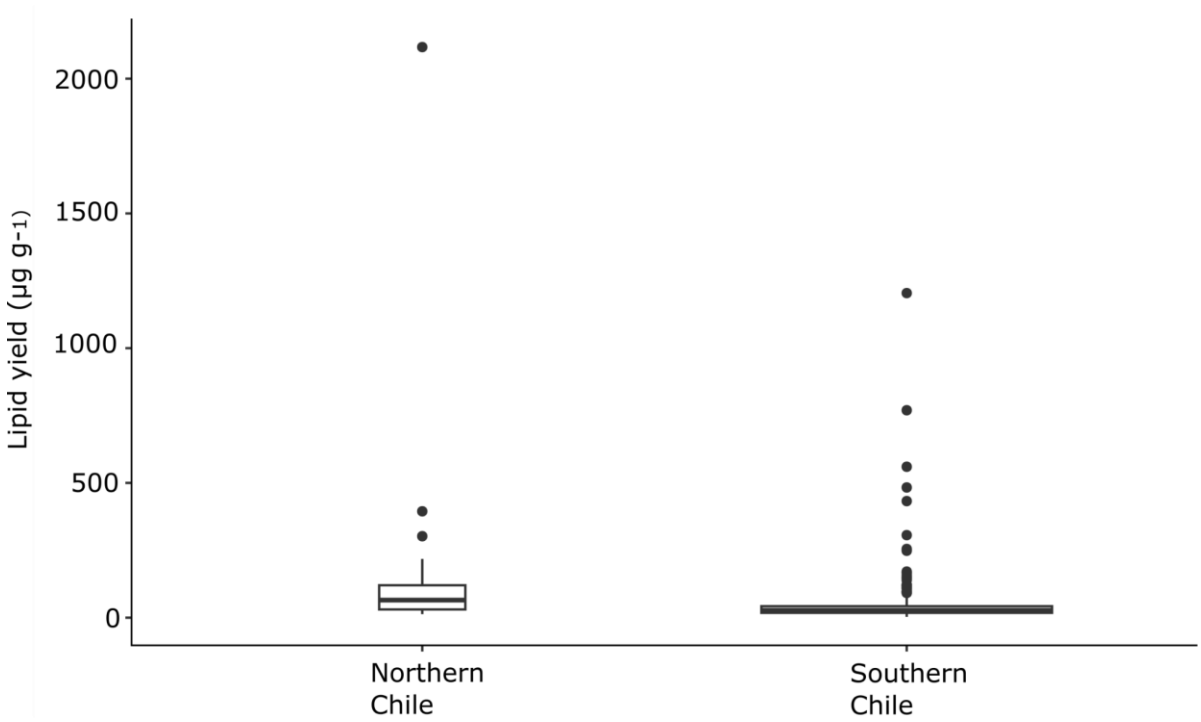


Figure 7.1: Comparison of lipid yields (µg g^{-1}) obtained by employing an extraction method using acidified methanol on samples from northern (n=21) and southern (n=239) Chile. Both areas showed statistically significant differences, with northern Chile showing higher values (Willcox test, W = 1207, p-value =0.000081). Figure created by the author.

After extracting interpretable amounts of lipids, it was possible **to identify maize through a multiproxy approach combining the residues' molecular and carbon isotopic composition.** Using GC-c-IRMS allowed the measurement of the carbon isotopic signal of the residues, allowing for the separation of the samples between C_3 products from those with more positive $\delta^{13}C$ values, likely related to marine products or C_4 plants. Although C_4 plants tend to have more positive $\delta^{13}C$ values than marine organisms, the overlapping between both is likely to happen and has been problematic for interpreting $\delta^{13}C$ analysis in archaeological contexts. By measuring the $\delta^{15}N$ values in human remains, it has been possible to differentiate between the consumption of marine organisms, normally situated in a high trophic level, and C_4 plants, commonly associated with primary producers and hence low trophic position (Santana-Sagredo et al., 2015).

The differentiation between marine and C_4 plants through organic residue analysis is possible by identifying specific biomarkers found in high quantities in aquatic organisms or products derived from them (e.g. oils exposed at high temperatures). These compounds consist of isoprenoid acids and a distribution of long-chain APAAs C_{20} and C_{22} formed from the protracted heating of marine oils (Bondetti et al., 2020; Lucquin et al., 2016; Evershed et al., 2008). At the same time, a high APAA-C_{18} E/H ratio and long-chain fatty alcohol distributions up to C_{32} or above are likely found in C_4 grasses (or derived products) and are not consistent with an aquatic origin. GC-MS was employed for the separation and identification of these compounds. The employment of selected ion monitoring (SIM) mode methods to target specific ions for isoprenoid acids, APAAs and fatty alcohol distributions allowed better precision for the identification of these compounds and to differentiate between marine and C_4 plants from the residues.

In Chapter 4, one of the samples with a marine $\delta^{13}C$ signal did not yield any aquatic biomarker; however, it showed a high APAA-C_{18} E/H ratio and a long-chain fatty alcohol distribution above C_{32}-OH likely to indicate its origin from plant commodities, possibly C_4 plants, likely to be maize. The isotopic ecology of southern Chile is dominated by C_3 plants, making it easier to apply this methodology when identifying maize. This is also exemplified in Chapter 5, where several samples recovered from Cueva de los Catalanes and SMR5 sites exhibited a high APAA-C_{18} E/H ratio and a fatty alcohol distribution above C_{32}-OH, possibly associating them with the use of panicoid grasses in the pots. Southern Chile has around 15 species of panicoid grasses (Finot et al., 2009); however, maize is the only C_4 plant used in the region for culinary purposes; in this case, the use of $\delta^{13}C$ analysis allowed the identification of a C_4 signal for several of these samples showing an APAA and fatty alcohol distribution, leading to conclude the potential use of the pots to process and store maize products.

In Chapter 6, several Inka aryballos, traditionally described for their use in storing maize-based beverages, along with jars associated with the Late Intermediate and Late Period, yielded residues with relatively positive $\delta^{13}C$ values concordant with an origin from maize. However, these samples only showed small traces of C_{32}-OH, possibly due to a high concentration of fatty acids that likely masked the low quantity of fatty alcohols from the residues. APAAs were probably not formed due to the use of the pots for storage and not for cooking maize, which limited the exposure of maize to high temperatures above 200ºC needed for APAA formation.

C_3 plant species dominate the Atacama Desert; however, several CAM and C_4 plants can also be found, although with restricted culinary use (Cadwallader et al., 2012; Ehleringer

et al., 1998). The main issue in Andean archaeology has been the reported consumption of *Amaranthus caudatus* or kiwicha by pre-colonial groups due to its C_4 signal that can be mistaken for maize (Cadwallader et al., 2012). Kiwicha has been identified in several Andean contexts and referred to as an important crop during the Inka period in Perú (McRostie et al., 2019).

Although *Amaranthus* sp. and *Amaranthus caudatus* macro remains have been identified in the Atacama Desert, this evidence is limited and poorly documented (e.g. Santana-Sagredo et al., 2021; McRostie et al., 2019). At Iluga Túmulos site, there was no clear evidence for the finding of *Amaranthus sp* from the archaeobotanical record; conversely, maize remains were extensively found, and the presence of the Inka state at Iluga Túmulos site will likely denote a certain level of maize exploitation and use in the pots.

Fatty alcohol distributions might be a potential biomarker to differentiate maize from *Amaranthus caudatus* or kiwicha in Andean contexts. Since C_{32}-OH mainly represents panicoid grasses, it is more likely to be absent from C_4 non-grasses such as *Amaranthus caudatus*. However, further chemotaxonomic analyses are needed to ensure that kiwicha can be differentiated from maize when using fatty alcohol distributions.

The distribution of fatty alcohols and a high E/H ratio are not exclusive to maize; they are potentially found in other C_4 grasses. Maize cannot be identified without assessing the $\delta^{13}C$ signal of the residues, as previously suggested by Reber & Evershed (2004). So far, there is no specific maize biomarker as seen for other C_4 grasses like millet (Heron et al., 2016; Reber and Evershed, 2004).

Using carbon stable isotope analysis to identify maize from the residues shows a major limitation when considering the potential mixing with lipid-rich resources. Among starchy plants, maize has a high concentration of lipids; hence, when mixing C_3 plants and maize, the input of a C_4 signal will likely be noticed. However, considerably large quantities of maize would be needed to compensate for the overwhelming concentration of lipids from animal fat. This imbalance will likely mask the carbon isotopic signal of maize in the residues (Reber, 2022).

In Chapters 4, 5, and 6, we have applied a simple linear model to assess whether mixing maize with several C_3 commodities can influence the $\delta^{13}C$ signal of the residue when considering the proportion of the main alkanoic acids $C_{16:0}$ and $C_{18:0}$ from each commodity. In the case studies presented above, it was possible to see that most samples showed a $\delta^{13}C$

signal from C_3 products. While several samples showed more positive $\delta^{13}C$ values relative to C_3 products, it was not possible to see a progressive ^{13}C enrichment as evidenced in Admiraal et al. (2023), where the $\delta^{13}C$ data likely represent extensive use of maize in most pots. However, the possible biases towards identifying maize from organic residue analysis need further assessment and more experimentation considering the mixing with different plant and animal products. Therefore, this study shows that maize can potentially be identified from organic residue analysis; however, this assessment needs to consider the possible overlap with other resources, such as marine products. Moreover, using modern references to build a solid baseline is crucial to better support the identification of maize, especially in contexts where other C_4 plants are present, and evidence to differentiate between them is needed.

1.2. Did maize play a prominent socio-political and economic role in southern Chile?

Historical records indicated that maize was already cultivated in the Araucanía by the time of the early contact with the European colonisers. Its identification in the archaeological record revealed its chronological depth and territorial distribution after its early adoption, which coincided with a moment of important socio-political and economic changes that marked the transition from the ECP to the LCP (Roa et al., 2018; Silva, 2014). However, its role in socio-political, economic and demographic processes experienced throughout the Ceramic Period has only been tenuously assessed. Through the two case studies presented for southern Chile, particularly on the coast and the Central Valley, this thesis has successfully addressed this issue for the first time, focusing particularly on the foodways of local populations.

Current data indicates that the arrival of maize into southern Chile coincided with a moment of important socio-political, economic and demographic changes around 950 cal BP, which marked the transition from the Early to the Late Ceramic Period. The identification of plant macro remains of domestic crops showed the first signs of food production by hunter-gatherer groups from the Pitrén cultural complex, along with the proliferation of larger cemeteries as the result of more complex and extensive family units, demographic growth and higher sedentarism (Mera and Munita, 2021; Roa et al., 2018; Ocampo, Mera and Rivas, 2001). These changes were consolidated during the Late Ceramic Period. Food production was intensified by farmers of the El Vergel cultural complex. At the same time, the proliferation of monumental mounds or *kuel*, and the construction of sophisticated raised fields for plant cultivation indicated the emergence of social differentiation. The construction of *kuel* and

extensive irrigation and cultivation systems, and the activities (e.g. farming, ceremonies) associated with them probably demanded the organisation of people as a workforce, possibly coordinated by small group of leaders invested with a certain level of authority and prestige (Campbell, 2020; Campbell and Pfeiffer, 2017; Dillehay and Saavedra Zapata, 2014). Moreover, the extensive coastal occupation and the permanent settlement of these farming groups in the nearshore Islands represented extensive demographic growth during the Late Ceramic Period (Campbell, 2015; Campbell and Quiroz, 2015).

From the molecular and isotopic characterisation of organic residues from pottery artefacts, it was possible **to assess whether maize had a higher competitive advantage compared to marine resources in supporting Island colonisation by pre-colonial farmers from southern Chile**, allowing me to infer that maize was not more important than marine resources and C_3 plants. The late arrival of maize in southern Chile implied a process of adaptation and experimentation by local groups. Thus, the availability of marine resources, local C_3 plants, and the knowledge associated with their exploitation was an advantage over the resently introduced maize. Marine products and C_3 plants were already part of local culinary practices; thus, the reliability of these products by coastal groups was higher than that of maize. Similarly, the employment of a multiproxy approach **to evaluate the significance of maize in fostering socio-political and economic changes throughout the Ceramic Period in the Central Valley of the Auraucanía in southern Chile** also indicated that C_3 plants and wild ruminants were primary products used in pre-colonial pottery from the Central Valley. In contrast, the culinary use of maize was probably more limited to specific culinary preparations. By the time maize arrived in the Central Valley, local groups already exploited local ruminant species and knew how to cultivate quinoa and beans. At the same time, the vast knowledge of the diversity of local wild plants represented an essential part of local culinary practices, into which maize was integrated.

The data presented from these two case studies indicate that maize did not become a staple after its adoption in southern Chile, and it might have not played a prominent role in fostering socio-political, economic and demographic changes throughout the Ceramic Period in the Araucanía. Conversely, its use seems to have been restricted to preparing specific dishes, likely alcoholic beverages or *chicha*. Early Spanish chronicles indicate the large consumption of different types of chicha during feasts and ceremonies (Núñez de Pineda y Bascuñán, 1673, cited in Cayuqueo, 2022, p. 50; Cobo 1979, 28; Rosales, 1674, cited in Barros Arana, 1877, p. 143). Although using maize as a primary resource for *chicha* production likely gave it a certain level of socio-political significance, it might not have been more important than other local plants also employed in producing intoxicating beverages.

Based on these two case studies, it is possible to infer that pottery from southern Chile was mainly employed to process C_3 products in the Central Valley and C_3 plants and marine resources in Mocha Island. The identification of domesticated plants such as quinoa, chilli peppers, beans, and squash remains in the Central Valley, and coast might indicate that several domesticated crops were processed in the pots (Roa Solís et al., 2024; Roa et al., 2021, 2018; Iriarte, 2014). Particularly, quinoa macro remains have been extensively identified in Mocha Island and the Central Valley, possibly denoting its significance as a domestic plant in several contexts (Roa Solís et al., 2024; Roa et al., 2021, 2018).

The extensive use of C_3 plants in both areas likely included various types of local wild plants. Evidence from the residues indicated a considerable use of grains, fruits and non-leafy vegetables, which could have comprised several grains, nuts and fruits from the mixed forest of the Central Valley and the evergreen forest of Mocha Island. Although the biomarker evidence only reflects these broad plant categories, it is possible that several edible seeds and fruits such as *Madia sativa* seeds, *Gevuina avellana* nuts, *Aristotelia chilensis*, *Fragaria chiloensis*, and *Rubus* sp. fruits, and wild grains such as *Bromus* sp., among other wild edible plants, comprised these broad plant categories. Recent investigations have identified these species, among others, and highlighted the significance of wild plant products in the local foodways of populations inhabiting the coast and Central Valley of the Araucanía (Roa Solís et al., 2024; Mera and Munita, 2021; Roa et al., 2018; Roa et al., 2015). Landscape modifications have been identified on the coast and Central Valley related to vegetation clearing for cultivation and the construction of raised fields and irrigation systems to sustain the agriculture of domestic crops, mainly maize (Gayo et al., 2019; Abarzúa et al., 2014; Lequesne et al.,1999). Roa Solís et al. (2024) suggested that landscape modifications were in play throughout the Ceramic Period and were associated with managing together wild plants and domesticated crops. The finding of wild plant seeds such as *Madia sativa* and remains of maize and quinoa in raised fields and irrigation canals of the area of Puren-Lumaco, Central Valley, indicates that the El Vergel farmers probably practised an intense management of both domesticated and wild plants (Dillehay et al., 2007). Early Spanish chronicles described the cultivation of domesticated crops by local farmers of the Araucanía (Bibar, 1558, cited in Leonard, 1966, p. 153; Mariño de Lobera, 1595, cited in Escobar, 1865, p. 124); however, they also mentioned the importance of wild plants and their extensive use for making bread, oil extraction, and crafting alcoholic beverages, among other culinary preparations (Núñez de Pineda y Bascuñán, 1673, cited in Cayuqueo, 2022, p. 48; Bibar, 1558, cited in Leonard, 1966, p. 153; Molina 1776, 142). The evidence possibly indicates that landscape modifications and

food production were destined for complex polyculture practices that comprised both wild and domesticated plants instead of reflecting an intensification of domestic crop agriculture.

Plant commodities were complemented by exploiting local South American camelids or guanacos. Pottery containers were likely used to process them, as evidenced at Cueva de los Catalanes; however, their exploitation by roasting the meat was likely also used. It is possible that guanacos were hunted during the Early Ceramic Period, although they were likely tamed during the Late Ceramic Period (Westbury et al., 2016; Becker, 1997), potentially leading to a level of dependency towards certain groups that claimed them as their own. Early chronicles indicated that guanacos were possessed by chiefs and wealthy people and consumed only during important feasts and ceremonies (Rosales, 1674, cited in Barros Arana, 1877, p. 142).

The translocation of these animals during the colonisation of Mocha Island likely reflects their significance for local communities, in addition to the necessity of maintaining a constant stock in an isolated and unfamiliar territory. These guanacos were possibly claimed by local leaders from the Island while being consumed only during important occasions and utilised as a source of wool. According to Oliver Van Noort, a Dutch pirate who arrived at Mocha Island in the 16th century AD, guanacos were employed as pack animals; however, they proved difficult to domesticate and did not exhibit docility (Van Noort, 1602, cited in Ijzerman, 1926, p. 56-58).

Therefore, the absence of evidence for the use of pottery for processing guanaco fat on Mocha Island may not only suggest that alternative culinary practices, such as roasting, were in place but also that their translocation served specific purposes that limited their consumption. Meanwhile, coastal resources, such as fish, marine mammals and shellfish, consistently played a significant protein supplement role in the economy of coastal farmers from the Araucanía. A similar situation is possible to identify on the shores of the Araucanía, near Punta Lavapié, where the Arenal-1 site demonstrated extensive use of coastal resources, along with the exploitation of guanacos for both meat and wool (Contreras et al., 2005).

The diversity of products used in the pots from both the coast and Central Valley indicates that socio-political, economic and demographic changes throughout the Ceramic Period cannot be assessed by focusing on the adoption and intensification of maize production only. It is possible that maize had a socio-political significance as a crucial resource used in preparing fermented beverages, as well as several other local plants. However, its impact on the local economy did not surpass other commodities. Moreover, the data in this research

indicates different socio-economic trajectories when comparing the coast with the Central Valley. While the former saw an intense use of plants and marine products to sustain a dense population inhabiting Mocha Island, the Central Valley demonstrated the employment of pottery to process terrestrial resources, including camelids and C_3 plants. In both cases, the processing of maize in the pots was limited.

Distinct socio-economic trajectories and the diversity of resources exploited in the coast and Central Valley limits to specify whether a particular resource or socio-economic strategy was the main driver supporting significant socio-political, economic and demographic changes throughout the Ceramic Period. Although the distribution of ^{14}C dates likely reflects a trend towards population growth in pre-colonial Araucanía, the concentration of dates in certain regions is a potential bias. As evident in Figure 7.2, most radiocarbon dates are concentrated in two main locations: Mocha Island on the coast and the Purén-Lumaco area in the Central Valley. Such radiocarbon dates' uneven distribution undermines further interpretations of the possible factors driving the region's demographic change.

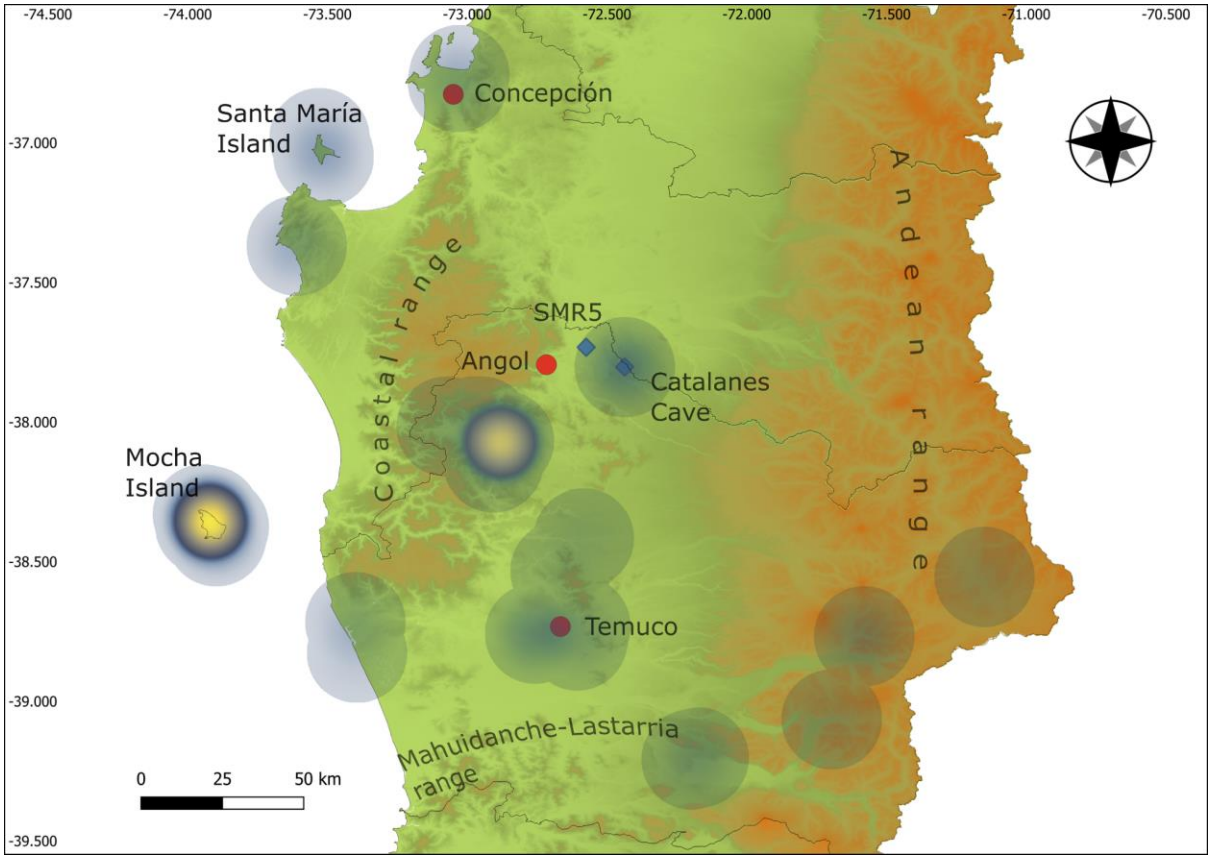


Figure 7.2: Map showing kernel density estimates of ^{14}C date distributions with a radius of 20 km. Data represents ^{14}C dates from the Mid Archaic Period until the start of the colonial Period (8000 - 400 cal BP). Data obtained from Campbell & Quiroz (2015) and Campbell et al.,(2020) (Appendix 14). Figure created by the author.

Ceramic vessels were adopted around 400 years before the earliest evidence of domestic plant exploitation in the Araucanía. Therefore, it is not possible to associate the adoption of this technological innovation with the onset of food production. The adoption of pottery possibly allowed local groups to consume and exploit a large diversity of resources (e.g. terrestrial and aquatic) more efficiently, leading to culinary innovations. The employment of pottery also allowed to provide food for larger groups of people and sustain a growing population. Domesticated crops such as beans, quinoa, and maize were incorporated into local foodways, which helped to broaden the diversity of resources and culinary preparations using ceramic containers. The importance of pottery for the preparation of alcoholic beverages should also be emphasised. The use of pottery to enhance the production of intoxicating drinks likely helped to foster the development of alliances during important feasts and celebrations, contributing to socio-political changes throughout the Ceramic Period.

The employment of pottery for the exploitation of coastal and terrestrial, domesticated and non-domesticated products in the region reflects that several socio-economic strategies such as farming, hunting, gathering, and animal and plant management were practised by local groups at different levels. Therefore, it is possible that a mixed economy, instead of the sole intensification of food production, facilitated sustainable population growth and fostered socio-political and economic changes in the region. Pottery might have played a crucial role in these changes and possibly helped to integrate the different resources exploited and accessed by local groups. However, the importance of other culinary practices cannot be ruled out and needs to be included as part of complex local foodways.

1.3. Did maize hold prominent socio-political importance during feasts celebrated under the Inka occupation of Tarapacá, northern Chile?

While maize was not considered a staple in southern Chile, carbon and nitrogen stable isotope analysis indicated that maize contributed significantly to local diets in northern Chile and in several Andean regions, especially in the Central Andes where important pan-Andean states promoted maize cultivation and consumption throughout their territory (Santana-Sagredo et al., 2021; Goldstein, 2015; Finucane, 2009). During the Late Intermediate Period, populations inhabiting the Pampa del Tamarugal maintained extensive social and political relations with coastal groups to secure access to bird guano (Santana-Sagredo et al., 2021). This resource enabled the cultivation of maize on a large scale in the driest zone of the Atacama Desert, which persisted during the Inka occupation of Tarapacá, where the state ensured and controlled access to bird guano to maintain a constant production of maize (de Cieza de León, 1945; Santana-Sagredo et al., 2021).

Maize held vital importance for the Inka state, not only from an economic standpoint but also in ritual and ideological contexts. As mentioned earlier, maize *chicha* was the principal beverage of the Inka state. Socio-political alliances were formed during celebrations where its consumption was promoted by the Inka state while being the most prominent product consumed in various festivities indicated in the Inka calendar (Guaman Poma de Ayala, 1936).

In Chapter 6, it was demonstrated through organic residue analysis of pottery that, despite the isotopic evidence from human remains indicating an extensive economic and dietary use of maize during the Late Intermediate Period and the Inka occupation of Tarapacá, maize was not more significant than other C_3 plant resources consumed during local feasts

and celebrations. The significance of C_3 resources was also identified in ceramics from the Formative Period, indicating that these were probably well integrated into local foodways before the Inka control in Tarapacá. I propose that while maize was consumed as a basic daily food, C_3 plants, such as *algarrobo* (*Prosopis* spp.), were consumed more extensively than maize only during special occasions such as feasts and celebrations, possibly reflecting their significance in reinforcing socio-political relations between the Inka state and the local groups.

Sporadic consumption of C_3 products, for example, during specific celebrations, would not necessarily register an observable C_3 signal through stable isotope analysis of human remains. This study not only demonstrated the limited use of maize in a ceremonial context at the Iluga Tumulos site but also provided a comprehensive view of the culinary choices made by the populations of Tarapacá, showcasing diversity in decision-making regarding resources consumed in every aspect of both daily and ideological life of these groups.

Therefore, it is worthwhile recognising and differentiating between diet and foodways as two concepts referring to food that needs to be treated separately. While the former indicates nutritional and economic aspects of food in daily life, the latter encompasses nutritional, cultural, political, economic, and ideological ideas related to food, including every step from resource gathering to processing, consumption, and discard (Peres, 2023; Twiss, 2012, 2). Thus, it is crucial to acknowledge that these two concepts must be addressed accordingly.

Moreover, while organic residue analysis can shed light on dietary and economic aspects of food, the diversity of uses that pottery can have must also be considered. These uses can include the employment of pottery as lamps, cooking pots, storage vessels, or funerary urns (Heron et al., 2013; Colombini, Modugno and Ribechini, 2005; Navarro and Aldunate, 2002). Moreover, employing pottery in cooking, storing and serving food represents only a part of broader culinary practices in which resources such as maize might have been involved. Therefore, any associations made regarding the economic and ideological interpretations of the use of pottery through organic residue analysis must consider their context and the potential decisions of those who extensively employed them.

2. Maize as the “wheat of the Americas.”

During the colonisation of the Americas, European chronists referred to maize as “the wheat of the Americas” or “wheat of the Indies” due to its extensive use throughout the continent and its employment in several culinary preparations (Molina, 1776, p.133; de León Pinelo, 1636, p.57). However, this parallelism between maize and Old World crops can be interpreted as reflecting early European biases supported by the familiarisation of maize with European and Asian agricultural economies (Guedes, 2019). Maize was fitted within a traditional discourse that related the development of grain agriculture with major socio-political and economic processes in archaeology, such as state formation and social complexity (Lesure et al., 2014; Finucane, 2009). In western South America maize has been considered to have played a vital role in the formation of pan-Andean states and in supporting socio-political and economic changes towards the intensification of food production (Gayo et al., 2019; Bray, 2009; Finucane, 2009; Hastorf and Johannessen, 1993). Stable isotope analysis has indicated that maize became a staple long after its early adoption (Pezo-Lanfranco and Colonese, 2024; Swift, Bahamodes and Schulting, 2022; Alfonso-Durruty et al., 2017). However, its posterior incorporation into major agricultural developments reflected high levels of maize reliance in different pre-colonial groups, indicating important socio-political and economic transitions (Delgado, 2021; Santana-Sagredo et al., 2021; Lambert et al., 2012).

Stable isotope analysis on human remains has shown variability in maize reliance among and within groups in several contexts from western South America (Falabella et al., 2020; Alfonso-Durruty et al., 2017; Kellner and Schoeninger, 2008). Moreover, further research has also shown evidence for diet diversification where, in some cases, maize did not show to have contributed more to the diet than other products, therefore suggesting the importance of different resources in fostering significant socio-political and economic processes (Pezo-Lanfranco and Colonese, 2024; Delgado, 2021; Washburn et al., 2020). Archaeobotanical remains identified in several contexts from western South America have attested to the potential use of various plant commodities to support these changes as a reflection of complex socioeconomic strategies and technological innovations (Pearsall et al., 2020; Beresford-Jones et al., 2018; McRostie et al., 2017).

Diet diversification and the socio-political and economic aspects behind the use and consumption of various resources contribute to contrast traditional and rigid ideas on the significance of agriculture as the primary model to explain surplus production, sedentism, population growth, and social complexity (Downey et al., 2014; Lesure et al., 2014; Bocquet-Appel, 2011, 2002; Childe, 1936). The idea of agriculture as the main driver for socio-political and economic changes leaves behind a diversity of economically successful groups, generally seen as representing transition states and not as independent economic choices capable of

fostering socio-political and economic processes (Ahedo et al., 2021; Smith, 2001). The data presented in this thesis reflects the choices made by pre-colonial groups from southern and northern Chile regarding the decisions made on which resources were collected, cooked, stored, served, consumed and discarded and which were not. These choices demonstrate the complexity of local pre-colonial foodways.

The data of this thesis adds up to current discussions carried out not only in western South America, but in other South American regions as well. A good example has been the Amazon basin, where landscape modification based on soil nutritional improvement to broaden mixed agroforestry cultivation of non-domesticated and domesticated plants has been an example of local choices and a reflex of complex pre-colonial foodways (Arroyo-Kalin and Riris, 2021; Iriarte et al., 2020; Watling et al., 2018). Here maize was cultivated as part of major polyculture agroforestry systems, reflecting variable levels of reliance among local groups (Iriarte et al., 2020; Hermenegildo et al., 2017; Arroyo-Kalin, 2010). In western Amazonía, extensive forest islands were constructed as inhabitable points within intermittent floodings, while the use of complex fishery systems permitted a surplus of freshwater resources (Lombardo et al., 2020; Blatrix et al., 2018; Lombardo and Prümers, 2010). This region saw the earliest presence of maize, and has been considered an area for domestication improvement (Lombardo et al., 2020; Kistler et al., 2018). While maize likely exhibited high economic significance in pre-colonial lowland Amazonia (Prümers and Betancourt, 2014), the large diversity of local resources and socioeconomic trajectories demonstrate a more complex setting that needs to be considered. Future studies on organic residue analysis from Amazonian pottery artefacts will definitely improve further interpretations of local foodways towards developing enriching discussions on complex socio-political and economic processes.

The large diversity of resources and strategies described for pre-colonial South America indicates that socio-political, economic and demographic changes are likely to be fostered by several factors, which must be assessed within their context and history. Therefore, maize was probably not the “wheat of the Americas,” in certain cases, as several other resources likely held a higher significance for pre-colonial groups in South America. We must pay more attention to the vast universe of resources and their potential relevance in pre-colonial history.

3. Further considerations and methodological improvements

Through the analysis of organic residue from pottery artefacts, it was possible to identify and evaluate the use of maize in several ceramic containers from southern and northern Chile. The results indicated that maize was processed in a limited number of ceramic vessels, while most pottery containers were used for cooking and storing C_3 products. While the identification of maize through organic residue analysis demonstrates a strong contribution to regional archaeology, further attempts should also concentrate on the diversity of C_3 products that comprise most of South American isotopic ecology.

3.1. Potential use of organic residue analysis on C_3 plant identification

The most challenging task is to assess the large diversity of C_3 plants found in South America. As mentioned in Chapter 2, plants have very low lipid concentrations, and the finding of specific biomarkers that can differentiate plants at a high taxonomic resolution is rare. Moreover, systematic studies on the use of organic residue analysis to identify South American C_3 plants are almost nonexistent.

However, studies conducted on plants from the “Old World” can provide insights into how to address this issue. A notable example is the potato. Discussions place the domestication centres of the potato in the central Andes and southern Chile (Hardigan et al., 2017; Spooner et al., 2012, 2005). Nevertheless, archaeological analysis has always been limited apart from scarce archaeobotanical records and chronicles describing its use. However, the identification of α,ω-dicarboxylic acids (diacids) with carbon chain $\geqslant C_{20}$ and a distribution of ω-hydroxy fatty acids of $C_{16:0}$-$C_{26:0}$ are considered principal monomers of suberin (*ca.* 23 - 46 %), a natural polyester mainly found as a component of specialised cell walls of underground storage plant organs such as tubers, roots and rhizoids (Dunne et al., 2022; Pollard et al., 2008). These compounds are readily identified through GC-MS from organic residues trapped in the porous matrix of archaeological pottery (Dunne et al., 2022). However, analysis of the surrounding soil would be needed as these compounds are readily incorporated into the soil organic matter as indicators of roots, tubers and rhizoid degradation from the surrounding vegetation (Mendez-Millan et al., 2010).

The employment of GC-MS and high-performance liquid chromatography (HPLC) has allowed the identification of alkylresorcinols as biomarkers for wheat and rye (Ross, 2012; Ross et al., 2003). However, Ross et al. (2017) employed GC-MS and HPLC to identify alkylrecorsinol distributions in quinoa. The authors indicated that quinoa exhibits even carbon-chain distributions of alkylresorcinol, which are not present in wheat, rye and barley, possibly indicating such alkylresorcinol homologues as potential quinoa biomarkers. Quinoa is one of the most important starchy plants from the Andes and has demonstrated widespread exploitation during pre-colonial times. While alkylresorcinols have been employed to identify major “Old World” grains (Hammann et al., 2022; Hammann and Cramp, 2018; Colonese et al., 2017), it has never been used to identify quinoa from South American pottery. Quinoa exhibits a very low abundance of lipids, even less than maize; therefore, further cooking experiments will be needed to assess the possibility for alkylresorcinols to be transferred from quinoa into the porous matrix of pottery and then assess their possible identification in archaeological pottery from South American contexts. Current studies on alkylresorcinol identification have applied more sensitive methods, such as gas chromatography quadrupole time-of-flight mass spectrometry (GC-Q-ToF-MS), showing great potential in archaeological pottery (Hammann et al., 2022; Hammann and Cramp, 2018). Further studies are needed where GC-Q-ToF-MS can be applied to ensure the identification of alkylresorcinols in archaeological potsherds employed to process quinoa.

3.2. Limitations on identifying fermentation of starchy plants

Early colonial documents attest to the use of several starchy plants in the preparation of fermented beverages or *chicha* in South America, consumed during important feasts and ceremonies. However, direct evidence for fermentation has not been successfully identified in pottery containers through organic residue analysis, and most evidence is associated with the possible presence of bacteria involved in the fermentation process (Craig, 2021; Garnier et al., 2018; Correa-Ascencio et al., 2014).

The identification of specific hopanoids through organic residue analysis of pre-colonial pottery from Mexico has allowed its association with the presence of ethanol-producing bacteria *Zymomonas mobilis*, possibly indicating the fermentation of agave sap to produce pulque alcoholic beverage (Correa-Ascencio et al., 2014). While the identification of hopanoids seems like a promising approach towards identifying the fermentation of plant products in archaeological pottery, questions arise about the microorganisms involved in starchy plant

fermentation for *chicha* production in South America. Several publications have indicated the protagonistic role of lactic acid bacteria in the production of intoxicating beverages based on South American starchy plants (Rebaza-Cardenas et al., 2023a, 2023b; Bassi et al., 2020; Romero-Luna, Hernández-Sánchez and Dávila-Ortiz, 2017; Elizaquível et al., 2015). Kuchta, Kubinec and Farkas (1998) indicated that lactic acid bacteria are unable to produce hopanoids. This limitation might reflect the impossibility of several bacteria to express the enzyme squalene-hopane cyclase (SHC), which synthesises hopanoids from squalene. A similar situation is replicated for yeasts, where hopanoid synthesis is very rare and limited and has not been identified for *Saccharomyces cerevisiae*, the main yeast involved in *chicha* production (Bouwknegt et al., 2021; Vallejo et al., 2013). These limitations undermine the potential use of hopanoids as evidence for plant fermentation in archaeological pottery. Moreover, the identification of biomarkers associated with yeast and bacteria involved in fermentation might also indicate the presence of microorganisms participating in the early diagenesis of organic matter (Craig, 2021). Further exploration is needed to identify possible biomarkers that can lead to the identification of bacteria fermentation occurring in pre-colonial South American pottery.

3.3. The potential use of proteomics in identifying pottery use

The identification of the original content used in archaeological pottery has been attempted through the extraction of proteins (Hendy et al., 2018; Colonese et al., 2017). While lipids denote better preservation compared to proteins, the latter can exhibit more precise taxonomic identification of the residues in cases at a species level. For this, using tandem Mass Spectrometry through LC-MS/MS allows the subsequent mass analysis of a peptide and its constituent fragments (Hendy, 2021). While this approach has allowed the identification of plant and animal commodities from archaeological pottery (Colonese et al., 2017; Solazzo et al., 2008), the susceptibility of proteins to degradation products and potential contamination from laboratory materials can undermine further protein recovery and interpretations (Hendy, 2021; Ramsøe et al., 2021). However, in a study by Hendy et al. (2018), the authors identified calcified deposits covering the surface of archaeological pottery from Çatalhöyük, Turkey, which helped to protect the organic content trapped in the porous matrix of the ceramic vessels, in which case interpretable protein yields were obtained. While this study shows promising results, the particular conditions that allowed protein survival are not always identified in the archaeological contexts. It would be expected that hyper-arid areas, such as

the Pampa del Tamarugal in the Atacama Desert, to show promising conditions for further proteomic analysis. However, this possibility needs to be assessed in future investigations.

4. Final conclusion

By using molecular and isotopic analysis of organic residues extracted from archaeological pottery from southern and northern Chile, it was possible to assess the use of pre-colonial ceramics in the processing and storing of maize in three contexts from southwestern South America. In southern Chile, maize did not become a staple after its adoption. In the Central Valley, local groups strongly relied on C_3 products such as several plants and ruminants. At the same time, on the coast, El Vergel farmers used pottery to exploit C_3 plants and local marine resources to settle on Mocha Island. In northern Chile, organic residue analysis on pottery artefacts indicated that maize was not a major product in culinary preparations consumed in Inka feasts and ceremonies, despite the extensive consumption of maize as a staple. This research contributes to current discussions on re-evaluating the significance of maize in local pre-colonial foodways from western South America, and from there, to question rigid perspectives on socio-political, economic and demographic changes described for pre-colonial South America. Based on these results, this thesis acknowledges the diversity of socio-economic trajectories and socio-political and demographic history of pre-colonial South America. However, it also reflects the need to work on current methodological and research gaps. Further research on organic residue analysis will help us approach the complexity and diversity that shaped South America's pre-colonial and current history.

Appendices

Appendix 1: Organic residue analysis materials and methods

1. Lipid extraction

1.1 Sampling of pottery artefacts

All samples consisted of ceramic potsherds from archaeological sites in northern and southern Chile. The outer layer of all pottery samples was removed by drilling around 1mm of the surface with a low-speed Derma drill. One metal drill bit was used for each sample to avoid contamination. Around 1 - 2 grams of pottery powder was drilled from the inner surface of the potsherds. All pottery powder was stored in aluminium foil, weighted and then transferred to clean hatch tubes for their extraction the following day.

All drilling activities were undertaken in a separate building from the wet chemistry laboratory where lipid extraction occurred.

1.2 Acid extraction

All samples were first extracted using acidified methanol to allow direct methylation of the compounds and high lipid yields (Craig et al., 2013). For this, around 4 mL of methanol and 10 µg of alkane C_{34} standard were included in each hatch tube containing around 1-2 gr of pottery powder prior to ultrasonication. Sulphuric acid (800 µL of H_2SO_4) was added to each sample and heated at 70ºC for 4 hours. Subsequently, the supernatant was transferred into a clean and labelled hatch tube. Lipids were separated from the H_2SO_4 by centrifugation (3000 rpm for 5 min) and by adding 2 mL of *n*-hexane (x3). Samples were concentrated under a gentle stream of nitrogen, and re-suspended in *n*-hexane. 10 µg of alkane C_{36} was added to assess lipid yield. Samples were analysed by using GC-FID, GC-MS and GC-c-IRMS.

1.3 Solvent extraction and trimethylsilyl (TMS) derivatization

Only several selected samples were subjected to solvent extraction, following a published protocol (Colonese et al., 2017b). Around 1-2 g of pottery powder was mixed with 4 mL of a dichloromethane-methanol mixture (DCM:MeOH 2:1 *v*/*v*). Samples were subjected to subsequent sonication (15 min x3) and then centirfuged for 10 min. Supernatan was

transferred into a clean hatchtube. Content was dried under a gentle stream of nitrogen. Lipids were re-suspended in *n*-hexane and followed derivatization by adding around 6 to 8 drops of N,O-bis(trimethylsilyl) trifluoroacetamide with 1% trimethylchlorosilane (BSTFA+TMCS, 99:1) prior to being placed on a heating block at 70ºC for 1 hour. Samples were dried under a gentle stream of nitrogen to get rid of any excess of BSTFA+TMCS, 99:1. Samples wre re-suspended in *n*-hexane and analysed through HT-GC-FID, GC-MS and HT-GC-MS.

1.4 Trimethylsilyl (TMS) derivatization of acid extracts

Derivatization of the acid-extracted samples was undertaken to have a better resolution of fatty alcohols. For this, only half was taken from each sample and transferred into a clean hydrolisis vial. Between 6 to 8 drops were included in each sample before placing them in a heating block for 1 hr at 70ºC. Each sample was dried under a gentle stream of nitrogen and then re-suspended in *n*-hexane. Samples were analysed through GC-MS.

2. Instrumentation setting

2.1 Gas chromatography - Flame ionisation detector (GC-FID)

Lipids were screened and quantified by using an Agilent 7890B high-temperature gas chromatograph (Agilent Technologies, Cheadle, Cheshire, UK) equipped with a flame ionisation detector (HT-GC-FID). A 100% Dimethylpolysiloxane DB-1-High Temperature (HT) (15 m X 320 µm X 0.1 µm; J&W Scientific Scientific, Folsom, CA, USA) column was used with a splitless injector to inject 1µL of sample into the GC at 300°C. Helium was the carrier gas at a constant flow rate of 2 mL min-1. The oven temperature was set at 100°C for 2 minutes, subsequently rising 20°C min -1 up to 325°C, where it set for 3 minutes.

Derivatized solvent-extracted samples used the same settings. However, a splitless injection was carried out at at 350°C. The oven temperature was set at 50°C, settling for 2 minutes, subsequently rising 10°C min-1 up to 375°C, where it held for 10 minutes.

2.2 Gas chromatography - Mass Spectrometry (DB5)

Lipids were identified by using an Agilent 7890A series chromatograph attached to an Agilent 5975C inert XL mass selective detector with a quadrupole mass analyser (Agilent Technologies, Cheadle, Cheshire, UK), and a Trace Ultra 1310 GC, coupled with an ISQ MS (Thermo Fisher, Bremen, Germany). Single ion monitoring (SIM) mode was employed for targeting specific ions (m/z 71, 75, 299, 327, 355, 383, 411, 439, 467, 495, 523, 551) for long-chain fatty alcohol identification.

A methylpolysiloxane (5%-phenyl) DB-5ms (30 m X 0.25 mm X 0.25 µm; J&W Scientific, Folsom, CA, USA) column was used onto which 1 µL of each sample was injected in splitless mode at 300ºC. A constant flow rate of 2 mL min-1 of helium was used as a carrier gas. The temperature of the oven was set at 50ºC for 2 min, subsequently rising 10ºC min-1 to 325ºC, holding for 15 min. The ionisation energy of the mass spectrometer was 70 eV, from which a spectrum by scanning ions between m/z 50 and 800 was obtained.

2.3. Gas chromatography - Mass Spectrometry (DB23)

The identification of Isoprenoid acids and ω-(o-alkylphenyl) alkanoic acids (APAAs), used for assessing the presence of several commodities, was achieved through GC-MS analysis using a 50% cyanopropyl-methylpolysiloxane DB-23 column (60 m x 0.25 mm x 0.25 µm; J&W Scientific, Folsom, CA, USA) as in Shoda and others (2017). Acidified methanol extracts were injected into the GC (1µL) using a splitless injector at 300ºC. The carrier gas was helium at a flow rate of 1.5 mL min-1. The temperature of the oven was set at 50ºC for 2 min, increasing 10ºC min-1 to 100ºC, after which it rose 4ºC min-1 up to 140ºC, and subsequently, 0.5ºC min -1 to 160ºC, to finally rise 20ºC min-1, reaching 250ºC where it settled for 20 min. Single Ion Monitoring (SIM) mode was used to target specific compounds, in which selected ions were used for the identification of the three main isoprenoid acids (m/z 74, 101, 171, 326 for phytanic acid; m/z 74, 88, 101, and 312 for pristanic acid, m/z 74, 87, 213 and 270 for 4, 8, 12 - trimethyltridecanoic acid (4, 8, 12 - TMTD)), and APAAs of carbon length C16 to C22 (m/z 74, 105, 262, 290, 318, 346).

2.4 High-Temperature Gas Chromatography-Mass Spectrometry (HT-GC-MS)

Solvent extracts were analysed using a Perkin Elmer Clarus 690 gas chromatograph coupled to a SQ8-T mass spectrometer. A DB5-HT column (30 m × 0.25 mm × 0.1 µm) was used. Helium was the carrier gas used at a constant flow rate of 1mL min-1. The oven temperature

was set at 50°C for two minutes and then increased to 10°C min-1, reaching a maximum temperature of 375°C held for 15 minutes.

2.5 Sample analysis

All the results obtained from GC-MS analysis were computed by using MSD Chemstation F.01.03.2357 software. Criteria for compound identification were based on retention time, mass spectrum and comparison with the National Institute of Standards and Technology (NIST) library. Integration of peaks was performed using Agilent Mass Hunter Quantitative Analysis software version B.07.01/ Build7.1.524.0 for GC-MS.

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

All samples were analysed based on a minimal alkanoic acid quantity needed for injection. Samples were analysed by using a Delta V Advantage Isotope Ratio Mass Spectrometer (Thermo Fisher Scientific, Bremen, Germany), coupled to a Trace Ultra 1310 Gas Chromatograph (Thermo Fisher) with a GC IsoLink II interface (with a CuO combustion reactor held at 850ºC). 1 µl of sample was injected into an ultra-inert fused-silica DB-5ms UI column (60 m x 0.25 mm x 0.25 µm; J&W Scientific, Folsom, CA, USA). Ultra high-purity grade helium was used as the carrier gas with a flow rate of 2 mL min-1. A small part of the flow was derived to an ISQ mass spectrometer (Thermo Fisher) for parallel acquisition of molecular data. The temperature of the oven was set at 50ºC for 0.5 min, rising 25ºC min-1 to 175ºC, then 8ºC min-1, reaching 325ºC, settling for 20 min.

Eluted compounds were ionised in the mass spectrometer through electron impact. The intensities of ions m/z 44, 45 and 46 were recorded to automatically compute the $^{13}C/^{12}C$ ratios of each peak in the extracts. The software used for the computation were Isodat (Thermo Fisher Scientific, Bremen, Germany) and LyticOS (Isoprime, Cheadle, UK). Computation was based on the comparison with repeatedly measured standard reference gas (CO2). Calculated δ13C values are presented in parts per mil (‰), relative to the Vienna PeeDee Belemnite (V-PDB) international standard.

The accuracy of the instrument was determined on n-alkanoic acid methyl esters of known isotopic composition (Indiana F8-3, 18 measurements). The precision was based on a laboratory standard mixture, regularly injected between samples. Subsequent to the analysis, values were corrected for the methylation of the alkanoic acids that occurred during acid extractions. Corrections were performed using a mass balance formula to compare the values with a standard mixture of $C_{16:0}$ and $C_{18:0}$ fatty acids of known isotopic composition, already included in each batch during the acid extractions. The mass balance formula was the following:

$$\delta^{13}C_{FA} = \frac{(C_{FAME}\ x\ \delta^{13}C_{FAME}) - \delta^{13}C_{MeOH}}{C_{FA}}$$

Modern references were adjusted according to the atmospheric $\delta^{13}C$ variations between the Early Holocene Epoch and the present to better interpret the archaeological data (Hellevang and Aagaard, 2015).

Appendix 2: Archaeological maize and cooking experiments for $\delta^{13}C$ references

Pottery sherds were collected as part of the FONDECYT Project N°11150397: “Trayectoria socio-política y complejización en la Araucanía septentrional: El Complejo El Vergel en la región de Angol (1000-1550 d.C.)” led by the archaeologist Dr. Roberto Campbell. Samples were shipped from Chile under the permit ORD N° 2477 issued by the National Monuments Council of Chile.

2.1. Archaeological maize references for $\delta^{13}C$

Archaeological maize references were a contribution by the FONDECYT project Nº 1181829 titled *Monumentos arqueológicos y memorias materiales: Historias andinas de larga duración en Pampa Iluga, Tarapacá (900 a.C.-1600 d.C.*), led by Professor Mauricio Uribe. Archaeological maize kernels were recovered from a specific mound from Iluga Túmulos site, Tarapacá Region, Chile. Samples were shipped from Chile under the decree CVE 2292672 issued by the Ministry of Cultures, Arts and Heritage of Chile.

Maize kernels were grouped according to their stratigraphic layer of origin, resulting in 5 groups (Supplemental Table 3). A pestle and mortar was cleaned using dichloromethane before and after crushing each group of maize kernels.

Table Appendix 2.1: Archaeological maize from Iluga Túmulos site

Sample	Mound	Stratigraphic layer	Number of kernels	Weight (mg) after grinding
B-0053	16	I-capa 2	5	622.0
B-0062	16	I-capa 3	6	620.8
B-0074	16	I-capa 4	4	605.5
B-0095	16	I-capa 5	4	622.7
B-0105	16	I-capa 6	4	623.5

Table created by the author.

Lipids were extracted following a one-step acidified methanol protocol as in(Craig et al., 2013; Papakosta et al., 2015) Lipids were diluted and analysed using gas-chromatography-combustion-ratio mass spectrometry to obtain maize $\delta^{13}C$ stable isotope references.

2.2. Cooking experiments

Cooking experiments were conducted at the YEAR Centre, University of York, York, United Kingdom, and in an open area in Concepción, Bío Bío Region, Chile to obtain reference for δ13C stable isotopes and molecular data for ω-(o-alkylphenyl) alkanoic acid and phytanic acid from plant and animal products (Supplemental Table 4).

Replica vessels were used according to Bondetti and others (2020). Pots were rinsed with Dichloromethane (DCM) to eliminate any exogenous organic contamination before each cooking event.

Embers from intensely heated coal and wood were used as a heat source. Vessels were placed on the embers. Animal and plant products were put into a specific pot; no mixing of products was conducted during these experiments. Vessels were filled with deionized water until the products were completely covered, refilling when the water evaporated. Each cooking event lasted *ca.* 30 to 60 min. Cooking events were repeated ca. 7 times in each vessel by adding more product and refilling the pots with water. Temperature was monitored during the cooking events by using a distance thermometer.

Table Appendix 2.2: List of products

Product	Species	Origin	Shop place	
Maize	*Zea mays*	Chile	Local market, Concepción.	Open area, Concepción, Chile.
Quinoa	*Chenopodim quinoa*	Chile	Local market, Villarica	Open area, Concepción, Chile.
Chilean hazelnut	*Gevuina avellana*	Chile	Local market, Talca	Open area, Concepción, Chile.
Llama meat	*Lama glama*	UK	Krezie ltd	YEAR Centre University of York
Llama meat	*Lama glama*	UK	Krezie ltd	YEAR Centre University of York
Madi seeds	*Madia sativa*	Chile	Field	Open area, Concepción, Chile.

Table created by the author.

Lipids from the replica vessels followed a one-step acidified methanol protocol as mentioned in Appendix 1.2.

Appendix 3: $\delta^{13}C$ stable isotope references.

Class	Species	Common name	δ13C16	δ13C18	Provenience	Reference
C4 plant	Zea mays	Maize	-14.7	-15.1	Chile	This work
C4 plant	*Zea mays*	Maize	-15.2	-14.6	Chile	This work
C4 plant	*Zea mays*	Maize	-15	-15.4	Chile	This work
C4 plant	*Zea mays*	Maize	-14.8	-15.3	Chile	This work
C4 plant	*Zea mays*	Maize	-14.2	-13.9	Chile	This work
C4 plant	*Zea mays*	Maize	-18.1	-18.8	Chile	This work
C4 plant	*Zea mays*	Maize	-17.2	-19.2	Argentina	Lantos et al 2015
C4 plant	*Zea mays*	Maize	-19.2	-20.9	Argentina	Lantos et al 2015
C4 plant	*Zea mays*	Maize	-18.1	-21.1	Argentina	Lantos et al 2015
C4 plant	*Zea mays*	Maize	-18.7	-21	Argentina	Lantos et al 2015
C4 plant	*Zea mays*	Maize	-18.5	-18.5	Argentina	Lantos et al 2015

C4 plant	*Zea mays*	Maize	-15.1	-17.8	Argentina	Lantos et al 2015
Ruminant	*Lama glama*	Llama	-23.7	-26.7	Argentina	Lantos et al 2015
Ruminant	*Lama glama*	Llama	-27.5	-30.5	Argentina	Lantos et al 2015
Ruminant	*Lama glama*	Llama	-28.3	-31.3	Argentina	This work
Ruminant	*Lama glama*	Llama	-28.1	-31.5	Argentina	This work
Ruminant	*Lama guanicoe*	Guanaco	-27.8	-30.7	Argentina	Admiraal et al 2023
Ruminant	*Lama guanicoe*	Guanaco	-27.7	-30.9	Argentina	Admiraal et al 2023
Ruminant	*Lama guanicoe*	Guanaco	-26.5	-29	Argentina	Admiraal et al 2023
Ruminant	*Cervus nippon*	Sika deer	-27.8	-29.4	Japan	Lucquin et al. 2016
Ruminant	*Cervus nippon*	Sika deer	-28.1	-31.6	Japan	Lucquin et al. 2016
Ruminant	*Cervus nippon*	Sika deer	-31.9	-35.1	Japan	Lucquin et al. 2016
Ruminant	*Cervus* sp.	Hokkaido deer	-28.1	-28.1	Japan	Lucquin et al. 2016
Ruminant	*Cervus* sp.	Hokkaido deer	-30.4	-32.7	Japan	Lucquin et al. 2016
Ruminant	*Cervus elaphus*	Red deer	-31.5	-33.2	UK	Lucquin et al. 2016
Ruminant	*Cervus elaphus*	Red deer	-31.4	-33.6	UK	Lucquin et al. 2016
C3 plant	*Quercus* sp.	Acorn	-34	-34.4	Japan	Lucquin et al. 2016
C3 plant	*Quercus* sp.	Acorn	-32.7	-33.6	Japan	Lucquin et al. 2016
C3 plant	*Quercus* sp.	Acorn	-35.1	-35.9	Japan	Lucquin et al. 2016
C3 plant	*Quercus* sp.	Acorn	-33.1	-34.6	Japan	Lucquin et al. 2016
C3 plant	*Quercus* sp.	Acorn	-32.1	-34	Japan	Lucquin et al. 2016
C3 plant	*Vicia faba*	Broadbean	-32.4	-31.4	Argentina	March 2013
C3 plant	*Vicia faba*	Broadbean	-32.6	-31.3	Argentina	March 2013
C3 plant	*Manihot esculenta*	Manioc	-37.6	-35.9	Bought in France	March 2013
C3 plant	Avena sativa	Oat	-34.2	-33.7	France	March 2013
C3 plant	Avena sativa	Oat	-33.7	-33.9	France	March 2013
C3 plant	*Solanum tuberosum*	Potato	-34.2	-34.4	Argentina	March 2013
C3 plant	*Chenopodium quinoa*	Quinoa	-31.3	-30.4	Chile	This work
C3 plant	*Gevuina avellana*	Chilean hazelnut	-26.9	-25.6	Chile	This work
C3 plant	*Madia sativa*	Madi	-29.3	-29.8	Chile	This work
C3 plant	*Prosopis alba Girseb.*	Algarrobo	-34	-32.9	Argentina	Lantos et al. 2015
C3 plant	*Phaseolus vulgaris L.*	Alubia bean	-35	-33.8	Argentina	Lantos et al. 2015
C3 plant	*Fabaceae*	Peanut	-26.4	-27.1	China	Guo et al. 2010
C3 plant	*Pinaceae*	Pine nut	-24.9	-26.2	China	Guo et al. 2010
C3 plant	*Curcubitaceae*	Pumpkin	-26.8	-27.5	China	Guo et al. 2010

C3 plant	*Curcubitaceae*	Pumpkin	-27.1	-27.5	China	Guo et al. 2010
C3 plant	*Fabaceae*	Soybean	-28	-28.6	China	Guo et al. 2010
C3 plant	*Juglandaceae*	Walnut	-26.5	-27.1	China	Guo et al. 2010
Marine	*Gadus morhua*	Atlantic cod	-22.3	-24.8	Denmark	Craig et al. 2011
Marine	*Phoca vitulina*	Harbour seal	-18.9	-20.5	Germany	Craig et al. 2011
Marine	*Pogonias cromis*	Black drum	-24.1	-24.7	Brazil	Colonese et al. 2015
Marine	*Cetacea*	Cetacea	-24.2	-25.9	Brazil	Colonese et al. 2015
Marine	*Gymnocranius euanus*	Japanese large-eye bream	-22.4	-22.1	Japan	Craig et al. 2013
Marine	*Gymnocranius euanus*	Japanese large-eye bream	-22.7	-22.5	Japan	Craig et al. 2013
Marine	*Sebastes* sp.	Rockfish	-23.7	-23.1	Japan	Craig et al. 2013
Marine	*Mugil cephalus*	Flathead grey mullet	-21.9	-21.3	Japan	Craig et al. 2013
Marine	*Genyonemus lineatus*	White croaker	-21.8	-21.4	Japan	Craig et al. 2013
Marine	*Anguilla anguilla*	European eel	-18.5	-15.8	UK	Lucquin et al. 2016
Marine	*Gadus morhua*	Atlantic cod	-24.8	-24.4	UK	Lucquin et al. 2016
Marine	*Scomber scombrus*	Atlantic mackerel	-25.4	-25.6	UK	Lucquin et al. 2016
Marine	*Thunnus* sp.	Tuna	-25.3	-24.9	Japan	Lucquin et al. 2016
Marine	*Globicephala macrorhynchus*	Short fin pilot whale	-22.9	-23.3	Japan	Lucquin et al. 2016
Marine	*Babylonia* sp.	Seashell (Babylonia)	-23.6	-23.1	Japan	Lucquin et al. 2016
Marine	*Venerupis philippinarum*	Seashell (Ruditapes)	-25	-24	Japan	Lucquin et al. 2016
Marine	*Globicephala* sp.	Pilot Whale	-23.6	-23.8	Japan	Lucquin et al. 2016

Note: $\delta^{13}C$ stable isotope values of the main alkanoic acids ($C_{16:0}$ and $C_{18:0}$) from modern and archaeological references from this study and published data. Due to variations on the atmospheric $\delta^{13}C$ values after the Industrial revolution (Suess effect), a correction on the $\delta^{13}C$ results of the modern samples was applied (Hellevang and Aagaard, 2015).

Appendix 4: Modern references for the APAA-C18 E/H ratio.

Category	Common name	APAA-C18 E	APAA-C18 H	APAA-C18 E/H	References
Cereal/fruit/non-leafy vegetable	Maize	9569	1368	7	This study
Cereal/fruit/non-leafy vegetable	Maize	1438	217	6.6	This study
Cereal/fruit/non-leafy vegetable	Quinoa	1472	239	6.2	This study
Cereal/fruit/non-leafy vegetable	Quinoa	1011	233	4.3	This study
Cereal/fruit/non-leafy vegetable	Chilean hazelnut	628	73	8.6	This study
Cereal/fruit/non-leafy vegetable	Madi	115395	14212	8.1	This study
Cereal/fruit/non-leafy vegetable	Chestnut Flour	25.6	6.8	3.8	Bondetti et al. 2020
Cereal/fruit/non-leafy vegetable	Hazelnut Oil	25.7	9.2	2.8	Bondetti et al. 2020

Cereal/fruit/non-leafy vegetable	Hemp Oil	28.8	5.1	5.6	Bondetti et al. 2020
Cereal/fruit/non-leafy vegetable	Rice Bran Oil	31.1	5.2	6.0	Bondetti et al. 2020
Cereal/fruit/non-leafy vegetable	Walnut Oil	28.1	7.7	3.6	Bondetti et al. 2020
Cereal/fruit/non-leafy vegetable	Leek white part	28.8	7.5	3.8	Bondetti et al. 2020
Cereal/fruit/non-leafy vegetable	Onion	28.6	6.1	4.7	Bondetti et al. 2020
Cereal/fruit/non-leafy vegetable	Almond	34.9	4.0	8.7	Bondetti et al. 2020
Cereal/fruit/non-leafy vegetable	Walnut	31.6	5.4	5.9	Bondetti et al. 2020
Cereal/fruit/non-leafy vegetable	Wheat	31.9	5.1	6.3	Bondetti et al. 2020
Cereal/fruit/non-leafy vegetable	Barley	32.3	5.5	5.9	Bondetti et al. 2020
Cereal/fruit/non-leafy vegetable	Carrot	28.1	7.4	3.8	Bondetti et al. 2020
Cereal/fruit/non-leafy vegetable	Olive oil	22.1	6.2	3.6	Bondetti et al. 2020
Cereal/fruit/non-leafy vegetable	Pistachio	31.6	4.0	7.9	Bondetti et al. 2020
Cereal/fruit/non-leafy vegetable	Viburnum	29.3	5.6	5.2	Bondetti et al. 2020
Cereal/fruit/non-leafy vegetable	Millet seed	29.9	5.1	5.9	Bondetti et al. 2020
Cereal/fruit/non-leafy vegetable	Quinoa seed	29.3	6.4	4.6	Bondetti et al. 2020
Cereal/fruit/non-leafy vegetable	Rice grain	31.4	4.6	6.8	Bondetti et al. 2020
Cereal/fruit/non-leafy vegetable	Sesame seed	31.6	4.7	6.7	Bondetti et al. 2020
Cereal/fruit/non-leafy vegetable	Acorn seed	26.6	7.5	3.5	Bondetti et al. 2020
Leafy plant	Spinach	11.8	23.5	0.5	Bondetti et al. 2020
Leafy plant	Carrot leave	24.3	12.4	2.0	Bondetti et al. 2020
Leafy plant	Cabbage	15.0	19.0	0.8	Bondetti et al. 2020
Leafy plant	Leek leave	20.7	14.3	1.4	Bondetti et al. 2020
Animal	Red Deer1	27.8	9.3	3.0	Bondetti et al. 2020
Animal	Organic Butter	22.6	7.5	3.0	Bondetti et al. 2020
Animal	Elk	22.6	8.5	2.7	Bondetti et al. 2020
Animal	Red Deer2	28.9	8.1	3.6	Bondetti et al. 2020
Animal	Beaver-6	29.6	6.4	4.6	Bondetti et al. 2020
Animal	Pork	25.1	4.3	5.8	Bondetti et al. 2020
Animal	Cod Liver Oil	22.4	9.5	2.4	Bondetti et al. 2020
Animal	Salmon Fat	26.5	13.6	1.9	Bondetti et al. 2020
Animal	Salmon Oil	28.3	8.7	3.3	Bondetti et al. 2020
Animal	Duck	24.4	12.2	2.0	Bondetti et al. 2020

Animal	Catfish	29.3	7.7	3.8	Bondetti et al. 2020
Animal	Sturgeon	29.4	5.8	5.1	Bondetti et al. 2020
Animal	Unio Shellfish	24.2	10.1	2.4	Bondetti et al. 2020
Animal	Viviparus Shellfish	23.2	13.0	1.8	Bondetti et al. 2020
Animal	Perch	23.1	9.9	2.3	Bondetti et al. 2020

Note: References based on cooking experiments from this study, and Bondetti and others (2020).

Appendix 5: Phytanic acid SRR-isomer references.

Class	Common name	Species	SRR %	Reference
Ruminant	Llama	*Lama glama*	59.4	This study
Ruminant	Llama	*Lama glama*	55.5	This study
Ruminant	Guanaco	*Lama guanicoe*	64.9	Admiraal et al 2023
Ruminant	Guanaco	*Lama guanicoe*	38.8	Admiraal et al 2023
Ruminant	Guanaco	*Lama guanicoe*	60.1	Admiraal et al 2023
Ruminant	Bos taurus	*Cervus elaphus*	59.4	Lucquin et al 2016
Ruminant	Red deer	*Cervus elaphus*	50.0	Lucquin et al 2016
Ruminant	Red deer	*Cervus elaphus*	61.4	Lucquin et al 2016
Ruminant	Red deer	*Cervus elaphus*	49.6	Lucquin et al 2016
Ruminant	Red deer	*Cervus elaphus*	60.0	Lucquin et al 2016
Ruminant	Red deer	*Cervus elaphus*	48.5	Lucquin et al 2016
Ruminant	Red deer	*Cervus elaphus*	51.0	Lucquin et al 2016
Ruminant	Red deer	*Cervus elaphus*	54.5	Lucquin et al 2016
Ruminant	Red deer	*Cervus elaphus*	68.8	Lucquin et al 2016
Ruminant	Serow	*Capricornis crispus*	54.3	Lucquin et al 2016
Ruminant	Sika deer	*Cervus nippon*	74.8	Lucquin et al 2016
Ruminant	Sika deer	*Cervus nippon*	54.3	Lucquin et al 2016
Ruminant	Sika deer	*Cervus nippon*	64.0	Lucquin et al 2016
Aquatic	Marine gastropod	*Babylonia* sp.	94.5	Lucquin et al 2016
Aquatic	Pilot whale	*Globicephala* sp.	69.1	Lucquin et al 2016
Aquatic	Marine bivalve	*Venerupis philippinarum*	36	Lucquin et al 2016
Aquatic	Salmon	*Salmonidae* sp.	80.8	Lucquin et al 2016
Aquatic	Salmon	*Salmonidae* sp.	74.9	Lucquin et al 2016

Aquatic	Salmon	*Salmonidae* sp.	88.5	Lucquin et al 2016

Note: Phytanic acid SRR-isomer contribution indicating a possible aquatic or ruminant origin. References were obtained from (Lucquin et al., 2016b) and data from this study.

Appendix 6: Details of the monochrome pottery from Mocha Island.

Site	Sample	Excavation unit	Level	External surface' treatment	Fragment's shape	Thickness (mm)	Type of pot
P5-1	P5-1.1	05.02.03	0-5cm	Smoothed	Neck-body	10.0	Indet.
P5-1	P5-1.2	05.02.03	10-15cm	Smoothed	Body	15.7	Indet.
P5-1	P5-1.3	05.02.03	25-30cm	Smoothed	Body	10.2	Indet.
P5-1	P5-1.4	05.02.03	25-30cm	Smoothed	Body	9.5	Indet.
P5-1	P5-1.5	05.02.03	30-35cm	Polished	Body	9.5	Indet.
P5-1	P5-1.6	05.02.03	30-35cm	Polished	Base	10.1	Indet.
P5-1	P5-1.7	05.02.03	30-40cm	Smoothed	Body	11.7	Indet.
P5-1	P5-1.8	05.02.03	50-55cm	Polished	Body	9.5	Indet.
P5-1	P5-1.9	05.02.03	50-55cm	Polished	Body	10.7	Indet.
P5-1	P5-1.10	05.02.03	55-60cm	Polished	Body	10.0	Indet.
P5-1	P5-1.11	05.02.03	55-60cm	Polished	Neck-body	8.5	Indet.
P5-1	P5-1.12	05.02.03	60-65cm	Polished	Body	10.7	Indet.
P5-1	P5-1.13	05.02.03	65-70cm	Polished	Neck	14.6	Indet.
P5-1	P5-1.14	05.02.03	80-85cm	Polished	Neck-body	11.1	Indet.
P5-1	P5-1.15	05.02.03	85-90cm	Polished	Body	10.2	Indet.
P5-1	P5-1.16	05.02.03	95-100cm	Polished	Body	11.2	Indet.
P5-1	P5-1.17	05.02.03	95-100cm	Polished	Body	10.0	Indet.
P23-2	P23-2.1	23.01.02	0-5cm	Polished	Base	11.5	Indet.
P23-2	P23-2.2	23.03.01	20-25cm	Polished	Body	11.1	Indet.

P23-2	P23-2.3	23.03.01	20-25cm	Polished	Neck-body	10.0	Indet.
P23-2	P23-2.4	23.03.01	20-25cm	Polished	Rim	9.8	Indet.
P23-2	P23-2.5	23.03.01	25-30cm	Polished	Base	12.5	Indet.
P23-2	P23-2.6	23.03.01	35-40cm	Smoothed	Body	9.8	Indet.
P23-2	P23-2.7	23.03.01	35-40cm	Polished	Neck	10.2	Indet.
P23-2	P23-2.8	23.03.01	40-45cm	Polished	Body	9.7	Indet.
P23-2	P23-2.9	23.03.01	40-45cm	Polished	Body	11.0	Indet.
P23-2	P23-2.10	23.03.01	50-55cm	Polished	Neck-body	11.6	Indet.
P23-2	P23-2.11	23.03.01	60-65cm	Polished	Body	10.8	Indet.
P23-2	P23-2.12	23.03.01	65-70cm	Polished	Body	11.4	Indet.
P23-2	P23-2.13	23.03.01	65-70cm	Polished	Body	12.3	Indet.
P23-2	P23-2.14	23.03.01	100-105cm	Polished	Body	14.5	Indet.
P23-2	P23-2.15	23.03.01	100-105cm	Polished	Body	14.0	Indet.
P25-1	P25-1.1	25.02.02	10-15cm	Polished	Body	12.7	Indet.
P25-1	P25-1.2	25.02.02	20-25cm	Polished	Neck-body	10.4	Indet.
P25-1	P25-1.3	25.02.02	55-60cm	Smoothed	Body	10.8	Indet.
P25-1	P25-1.4	25.02.02	55-60cm	Smoothed	Rim	10.2	Indet.
P25-1	P25-1.5	25.02.02	60-65cm	Smoothed	Rim	12.2	Indet.
P25-1	P25-1.6	25.02.02	60-65cm	Polished	Body	10.1	Indet.
P25-1	P25-1.7	25.02.02	70-75cm	Polished	Rim	14.8	Indet.
P25-1	P25-1.8	25.03.01	95-100cm	Polished	Body	10.1	Indet.
P25-1	P25-1.9	25.03.04	0-5cm	Polished	Body	9.5	Indet.
P25-1	P25-1.10	25.03.04	85-90cm	Polished	Rim	9.5	Indet.
P25-1	P25-1.11	25.03.04	115-120cm	Polished	Body	11.4	Indet.
P25-1	P25-1.12	25.04.02	50-55cm	Polished	Body	14.5	Indet.

P25-1	P25-1.13	25.04.02	75-80cm	Smoothed	Body	10.2	Indet.
P25-1	P25-1.14	25.04.03	50-55cm	Eroded	Base	10.5	Indet.
P25-1	P25-1.15	25.04.03	65-70cm	Polished	Body	15.9	Indet.
P25-1	P25-1.16	25.04.03	65-70cm	Polished	Body	10.3	Indet.
P25-1	P25-1.17	25.04.03	65-70cm	Polished	Body	15.5	Indet.
P25-1	P25-1.18	25.04.03	70-75cm	Smoothed	Body	10.1	Indet.
P25-1	P25-1.10	25.03.04	115-120cm	Polished	Body	10.7	Indet.

Indet: Indeterminate

Appendix 7: Summary of the molecular and isotopic data from Mocha Island

Sample ID	Site	Lipid concentration (µg g-1)	Main compounds detected	P/S	PA SRR%	APAA C18E/H	APAA C20/C18	δ13C 16:0 (‰)	δ13C 18:0 (‰)	Δ13C (‰)	ALK CPI
P5-1.1	P5-1	38	SFA($C_{14:0}$ - $C_{21:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), br, phy, tmtd, pri	1.0	70	N.P	N.P	-27.3	-29	-1.7	N.P
P5-1.2	P5-1	61	SFA ($C_{8:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC (C_8 - C_{11}), ALK (C_{27} - C_{33}), br, tmtd, pri, APAA-C_{18}	1.0	N.P	2.5	N.P	-30.6	-31.3	-0.7	2.2
P5-1.3	P5-1	19	SFA ($C_{10:0}$ - $C_{28:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC (C_8 - C_{10}), br, tmtd, pri	1.0	N.P	N.P	N.P	-22.4	-22.7	0.4	N.P
P5-1.4	P5-1	20	SFA ($C_{8:0}$ - $C_{24:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC (C_8 - C_9), br, pri	1.2	N.P	N.P	N.P	-29.3	-30	-0.7	N.P
P5-1.5	P5-1	13	SFA ($C_{8:0}$ - $C_{24:0}$), UFA ($C_{18:1}$ - $C_{22:1}$), DC (C_9 - C_{10}), pri	1.0	N.P	N.P	N.P	-31.3	-31.6	-0.4	N.P
P5-1.6	P5-1	146	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC (C_9 - C_{10}), ALK (C_{27} - C_{33}), br	0.9	N.P	N.P	N.P	-29.6	-29.1	0.4	0.9
P5-1.7	P5-1	166	SFA ($C_{8:0}$ - $C_{28:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC (C_9 - C_{14}), br, phy, tmtd, pri	1.6	8.4	N.P	N.P	-25.2	-27.3	-2.1	N.P
P5-1.8	P5-1	64	SFA ($C_{12:0}$ - $C_{26:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), tmtd, pri	1.0	N.P	N.P	N.P	-29.3	-29.7	-0.4	N.P
P5-1.9	P5-1	32	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), ALK (C_{27} - C_{31}), br, phy, tmtd, pri, APAA-C_{18}	0.9	77.2	4.6	N.P	-29.1	-29	0.1	5.2

P5-1.10	P5-1	110	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC (C_9 - C_{10}), OH (C_{26} - C_{28}), ALK (C_{27} - C_{31}), br, tmtd, pri	1.4	N.P	N.P	N.P	-29.2	-29.5	-0.3	10.1
P5-1.11	P5-1	39	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{18:1}$ - $C_{24:1}$), VLCOH ($C_{22:0}$ - $C_{30:0}$), ALK (C_{27} - C_{29}),, br, tmtd, APAA-C_{18}	0.8	N.P	1.9	N.P	-29.9	-29.2	0.7	2.8
P5-1.12	P5-1	70	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC (C_9), pri, APAA-C_{18}	1.1	N.P	7.8	N.P	-26.7	-27.6	-0.9	N.P
P5-1.13	P5-1	171	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC (C_9 - C_{10}), br, phy, tmtd, pri, APAAs (C_{18}, C_{20}).	1.2	74.7	3.5	0.3	-21.9	-22.3	-0.3	N.P
P5-1.14	P5-1	70	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), ALK (C_{27} - C_{31}), tmtd, pri, APAA-C_{18}	0.9	N.P	N.P	N.P	-29.4	-29.7	-0.4	47.9
P5-1.15	P5-1	30	SFA ($C_{14:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), ALK (C_{27} - C_{31}), br, tmtd	0.8	N.P	N.P	N.P	-30.1	-29	1.1	1.6
P5-1.16	P5-1	33	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC (C_{11}), ALK (C_{27} - C_{31}), br, phy, tmtd, pri, APAA-C_{18}	1.6	81.7	2.7	N.P	-23.3	-25.9	-2.5	2.6
P5-1.17	P5-1	560	SFA ($C_{12:0}$ - $C_{28:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), DC (C_8 - C_9), br, phy, tmtd, pri	0.5	77.7	N.P	N.P	-29.5	-31.6	-2.1	N.P
P23-2.1	P23-2	29	SFA ($C_{12:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC (C_9), br, tmtd, pri, APAA-C_{18}	1.0	N.P	1.9	N.P	-29.8	-29.4	0.4	N.P
P23-2.2	P23-2	33	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC (C_9), br, tmtd, pri, APAAs (C_{18}, C_{20})	1.5	N.P	5.3	0.3	-29.4	-29.8	-0.4	N.P
P23-2.3	P23-2	8	SFA ($C_{14:0}$ - $C_{22:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), br, tmtd, pri	1.0	N.P	N.P	N.P	-29.7	-29.4	0.3	N.P
P23-2.4	P23-2	69	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC (C_8 - C_{10}), br, tmtd, pri	1.0	N.P	N.P	N.P	-27.5	-28.2	-0.7	N.P
P23-2.5	P23-2	27	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC (C_8 - C_{11}), br, tmtd	1.4	N.P	N.P	N.P	-30.5	-27.8	2.6	N.P
P23-2.6	P23-2	46	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC (C_8 - C_{10}), OH (C_{26} - C_{28}), ALK (C_{27} - C_{33}), br, pri, APAA-C_{18}	1.3	N.P	7.2	N.P	-29.7	-29.7	0.1	16.2
P23-2.7	P23-2	58	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC (C_8 - C_{10}), ALK (C_{27} - C_{31}), br, pri	1.2	N.P	N.P	N.P	-24.7	-27.8	-3.1	5.1
P23-2.8	P23-2	58	SFA ($C_{12:0}$ - $C_{28:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC (C_8), br, tmtd, pri	1.0	N.P	N.P	N.P	-24.8	-24.4	0.06	N.P
P23-2.9	P23-2	12	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC (C_9), br, pri	1.2	N.P	N.P	N.P	-28.9	-27.9	1	N.P
P23-2.10	P23-2	24	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), ALK (C_{27} - C_{33}), br, pri	1.0	N.P	N.P	N.P	-29.6	-29	0.5	1.7

P23-2.11	P23-2	15	SFA ($C_{12:0}$ - $C_{20:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), DC (C_9), br, pri	2.4	N.P	N.P	N.P	-28.9	-28.9	0	N.P
P23-2.12	P23-2	7	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), br, pri	1.1	N.P	N.P	N.P	-29.3	-28.8	0.5	N.P
P23-2.13	P23-2	52	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC (C_9 - C_{11}), ALK (C_{27} - C_{33}), br, pri, APAA-C_{18}	0.9	N.P	2.2	N.P	-29.4	-28.4	1	4.2
P23-2.14	P23-2	16	SFA ($C_{12:0}$ - $C_{26:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), DC (C_8), br, phy, tmtd, pri, APAA-C_{18}	1.1	74.7	2.5	N.P	-31.6	-28.8	2.7	N.P
P23-2.15	P23-2	29	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), ALK (C_{27} - C_{33}), br, pri, APAA-C_{18}	0.9	N.P	1.3	N.P	-28.9	-28.2	0.8	5.2
P25-1.1	P25-1	27	SFA ($C_{12:0}$ - $C_{24:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC (C_9), br, pri	2.9	N.P	N.P	N.P	-30.1	-29.9	0.2	N.P
P25-1.2	P25-1	9	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC (C_9), ALK (C_{27} - C_{33}), br, phy, pri	0.7	27.1	N.P	N.P	-29.9	-31.5	-1.6	4.6
P25-1.3	P25-1	55	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC (C_8 - C_{10}), OH (C_{26} - C_{28}), ALK (C_{27} - C_{33}), br, pri	1.6	N.P	N.P	N.P	-29.8	-29.8	0	3.9
P25-1.4	P25-1	255	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC (C_9 - C_{11}), br, phy, pri	0.7	30.4	N.P	N.P	-26.5	-29.2	-2.7	N.P
P25-1.5	P25-1	60	SFA ($C_{10:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC (C_8 - C_{10}), OH (C_{22} - C_{34}), ALK (C_{27} - C_{31}), br, pri, APAA-C_{18}	2.1	N.P	7.9	N.P	-23.8	-24.6	-0.8	7.7
P25-1.6	P25-1	9	SFA ($C_{12:0}$ - $C_{26:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), br, pri	1.0	N.P	N.P	N.P	-29.1	-28.8	0.3	N.P
P25-1.7	P25-1	34	SFA ($C_{12:0}$ - $C_{26:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), DC (C_8 - C_{10}), OH (C_{22} - C_{28}), br, APAA-C_{18}	2.1	N.P	8.4	N.P	-18.8	-22.6	-3.8	N.P
P25-1.8	P25-1	11	SFA ($C_{12:0}$ - $C_{28:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), ALK (C_{27} - C_{33}), br, pri, APAA-C_{18}	1.0	N.P	4.4	N.P	-29.3	-29.4	-0.1	5.3
P25-1.9	P25-1	58	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC (C_8 - C_{12}), OH (C_{22} - C_{28}), ALK (C_{27} - C_{31}), br, phy, tmtd, pri	1.5	93.9	N.P	N.P	-29.7	-29.4	0.2	0.7
P25-1.10	P25-1	150	SFA ($C_{10:0}$ - $C_{26:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC (C_8 - C_{14}), OH (C_{26}), br, phy, tmtd, pri, APAAs (C_{18}, C_{20}, C_{22})	1.5	93.3	5.1	0.12	-26.3	-27.6	-1.3	N.P
P25-1.11	P25-1	22	SFA ($C_{12:0}$ - $C_{28:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), VLCOH (C_{22} - C_{26}), br, pri, APAA-C_{18}	0.9	N.P	7.3	N.P	-28.8	-28.9	-0.2	N.P
P25-1.12	P25-1	14	SFA ($C_{12:0}$ - $C_{20:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), br, pri, APAA-C_{18}	1.4	N.P	1.8	N.P	-28.9	-29.1	-0.1	N.P

P25-1.13	P25-1	18	SFA ($C_{12:0}$ - $C_{28:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), DC (C_8 - C_{10}), br, phy, tmtd, pri, APAAs (C_{18}, C_{20}, C_{22})	1.0	97.6	4.8	0.06	-28	-28.9	-0.9	N.P
P25-1.14	P25-1	28	SFA ($C_{10:0}$ - $C_{26:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), DC (C_8 - C_{13}), ALK (C_{27} - C_{31}), br, tmtd, pri	1.9	N.P	N.P	N.P	-27	-26.5	0.5	8
P25-1.15	P25-1	7	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), ALK (C_{27} - C_{33}), br	1.7	N.P	N.P	N.P	N.P	N.P	N.P	8.4
P25-1.16	P25-1	11	SFA ($C_{12:0}$ - $C_{20:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), DC (C_9), br	1.2	N.P	N.P	N.P	-29.7	-29.4	0.4	N.P
P25-1.17	P25-1	10	SFA ($C_{12:0}$ - $C_{24:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), br, phy	1.4	50.8	N.P	N.P	-28.7	-28.9	-0.2	N.P
P25-1.18	P25-1	15	SFA ($C_{12:0}$ - $C_{28:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), DC (C_9), ALK (C_{27} - C_{33}), br	1.4	N.P	N.P	N.P	-27.4	-27.7	-0.3	8.8
P25-1.19	P25-1	3			N.P	N.P	N.P	N.P	N.P	N.P	N.P

Note: Data for the recovered residues from the 51 samples analysed in this work. In red is sample P25-1.19 which was removed from this study due to the low lipid yield. SFA=Saturated fatty acids; UFA= Unsaturated fatty acids; DC= Diacids; OH= Fatty alcohols; ALK= Alkanes (odd-numbered); br= Branched fatty acids; phy= Phytanic acid; tmtd = 4, 8, 12-tmtd; pr=Pristanic aids; APAA= ω-(o-alkylphenyl) alkanoic acids. ALK CPI value based on (Diefendorf et al., 2011).

Appendix 8: Pottery from Catalanes Cave, southern Chile

Site	Sample	Excavation unit	Level	External surface' treatment	Fragment's shape	Thickness (mm)	Type of pot
C.Catalanes	C.C.ECP-1	2	16(150-160CM)	Polished	Body	8.9	Indet.
C.Catalanes	C.C.ECP-2	2	16(150-160CM)	Polished	Body	7.2	Indet.
C.Catalanes	C.C.ECP-3	2	15(140-150CM)	Polished	Body	9.8	Indet.
C.Catalanes	C.C.ECP-4	2	15(140-150CM)	Polished	Body	6.8	Indet.
C.Catalanes	C.C.ECP-5	2	15(140-150CM)	Polished	Body	6.8	Indet.
C.Catalanes	C.C.ECP-6	2	15(140-150CM)	Polished	Body	5.6	Indet.

C.Catalanes	C.C.ECP-7	2	15(140-150CM)	Polished	Body	7.3	Indet.
C.Catalanes	C.C.ECP-8	2	15(140-150CM)	Polished	Body	7.6	Indet.
C.Catalanes	C.C.ECP-9	2	16(150-160CM)	Polished	Body	9.0	Indet.
C.Catalanes	C.C.ECP-10	2	16(150-160CM)	Polished	Body	9.3	Indet.
C.Catalanes	C.C.ECP-11	2	16(150-160CM)	Polished	Body	8.5	Indet.
C.Catalanes	C.C.ECP-12	2	16(150-160CM)	Polished	Neck-body	10.1	Indet.
C.Catalanes	C.C.ECP-13	2	16(150-160CM)	Polished	Rim	9.5	Indet.
C.Catalanes	C.C.ECP-14	2	17(160-170CM)	Polished	Neck-body	6.5	Indet.
C.Catalanes	C.C.ECP-15	2	15(140-150CM)	Polished	Base	7.5	Indet.
C.Catalanes	C.C.ECP-16	2	15(140-150CM)	Polished	Rim-neck	7.5	Indet.
C.Catalanes	C.C.ECP-17	2	15(140-150CM)	Smoothed	Body	11.2	Indet.
C.Catalanes	C.C.ECP-18	2	14(130-140CM)	Polished	Neck-body	6.0	Indet.
C.Catalanes	C.C.ECP-19	2	14(130-140CM)	Polished	Body	7.5	Indet.
C.Catalanes	C.C.ECP-20	2	14(130-140CM)	Polished	Body	8.5	Indet.
C.Catalanes	C.C.ECP-21	2	14(130-140CM)	Polished	Body	6.0	Indet.
C.Catalanes	C.C.ECP-22	2	14(130-140CM)	Polished	Body	7.2	Indet.
C.Catalanes	C.C.ECP-23	2	14(130-140CM)	Polished	Body	7.2	Indet.
C.Catalanes	C.C.ECP-24	2	13(120-130CM)	Polished	Body	11.2	Indet.
C.Catalanes	C.C.ECP-25	2	13(120-130CM)	Polished	Body	7.4	Indet.
C.Catalanes	C.C.ECP-26	2	13(120-130CM)	Polished	Base	7.3	Indet.
C.Catalanes	C.C.ECP-27	2	15(140-150CM)	Polished	Neck-body	7.0	Indet.

C.Catalanes	C.C.ECP-28	2	13(120-130CM)	Polished	Body	10.0	Indet.
C.Catalanes	C.C.ECP-29	2	14(130-140CM)	Smoothed	Body	9.5	Indet.
C.Catalanes	C.C.ECP-30	2	17(160-170CM)	Polished	Body	7.5	Indet.
C.Catalanes	C.C.ECP-31	2	17(160-170CM)	Polished	Body	7.3	Indet.
C.Catalanes	C.C.ECP-32	2	17(160-170CM)	Polished	Body	8.6	Indet.
C.Catalanes	C.C.ECP-33	2	14(130-140CM)	Polished	Body	10.0	Indet.
C.Catalanes	C.C.ECP-34	1	16(150-160CM)	Polished	Base	6.7	Indet.
C.Catalanes	C.C.ECP-35	1	17(160-170CM)	Polished	Rim	6.5	Indet.
C.Catalanes	C.C.ECP-36	1	18(170-180CM)	Polished	Body	7.0	Indet.
C.Catalanes	C.C.ECP-37	1	18(170-180CM)	Polished	Body	7.8	Indet.
C.Catalanes	C.C.ECP-38	1	18(170-180CM)	Polished	Body	6.3	Indet.
C.Catalanes	C.C.ECP-39	1	18(170-180CM)	Polished	Body	6.8	Indet.
C.Catalanes	C.C.ECP-40	1	19(180-190CM)	Polished	Rim-neck	6.0	Indet.
C.Catalanes	C.C.ECP-41	1	15(140-150CM)	Polished	Body	9.1	Indet.
C.Catalanes	C.C.ECP-42	1	15(140-150CM)	Polished	Body	9.0	Indet.
C.Catalanes	C.C.ECP-43	1	17(160-170CM)	Polished	Base	7.6	Indet.
C.Catalanes	C.C.ECP-44	1	18(170-180CM)	Polished	Neck-body	8.2	Indet.
C.Catalanes	C.C.ECP-45	1	18(170-180CM)	Polished	Neck-body	6.9	Indet.
C.Catalanes	C.C.ECP-46	1	17(160-170CM)	Polished	Body	5.6	Indet.
C.Catalanes	C.C.ECP-47	1	17(160-170CM)	Polished	Body	5.6	Indet.
C.Catalanes	C.C.ECP-48	1	17(160-170CM)	Polished	Body	5.9	Indet.

C.Catalanes	C.C.ECP-49	1	18(170-180CM)	Polished	Body	6.0	Indet.
C.Catalanes	C.C.ECP-50	1	18(170-180CM)	Polished	Body	6.1	Indet.
C.Catalanes	C.C.ECP-51	1	18(170-180CM)	Polished	Body	5.6	Indet.
C.Catalanes	C.C.ECP-52	1	19(180-190CM)	Polished	Body	6.3	Indet.
C.Catalanes	C.C.ECP-53	1	19(180-190CM)	Smoothed	Body	5.8	Indet.
C.Catalanes	C.C.ECP-54	1	18(170-180CM)	Polished	Neck-body	6.8	Indet.
C.Catalanes	C.C.ECP-55	1	15(140-150CM)	Polished	Body	7.7	Indet.
C.Catalanes	C.C.ECP-56	1	19(180-190CM)	Polished	Body	5.8	Indet.
C.Catalanes	C.C.ECP-57	1	18(170-180CM)	Polished	Body	7.1	Indet.
C.Catalanes	C.C.ECP-58	1	16(150-160CM)	Polished	Body	7.4	Indet.
C.Catalanes	C.C.ECP-59	1	16(150-160CM)	Smoothed	Body	6.3	Indet.
C.Catalanes	C.C.ECP-60	1	16(150-160CM)	Polished	Body	7.4	Indet.
C.Catalanes	C.C.ECP-61	2	15(140-150CM)	Polished	Body	6.5	Indet.
C.Catalanes	C.C.ECP-62	2	15(140-150CM)	Polished	Body	8.0	Indet.
C.Catalanes	C.C.ECP-63	2	15(140-150CM)	Polished	Body	6.6	Indet.
C.Catalanes	C.C.ECP-64	2	16(150-160CM)	Polished	Body	6.2	Indet.
C.Catalanes	C.C.LCP-1	1	14(130-140cm)	Polished	Body	9.0	Indet.
C.Catalanes	C.C.LCP-2	1	14(130-140cm)	Polished	Body	6.0	Indet.
C.Catalanes	C.C.LCP-3	1	14(130-140cm)	Polished	Base	7.6	Indet.
C.Catalanes	C.C.LCP-4	1	14(130-140cm)	Polished	Base	6.8	Indet.
C.Catalanes	C.C.LCP-5	1	Section cleaning	Polished	Rim	5.2	Indet.

			(80-100)				
C.Catalanes	C.C.LCP-6	1	14(130-140cm)	Smoothed	Body	9.1	Indet.
C.Catalanes	C.C.LCP-7	1	Section cleaning (70-150cm)	Smoothed	Rim	6.9	Indet.
C.Catalanes	C.C.LCP-8	1	Section cleaning (70-150cm)	Polished	Body	6.2	Indet.
C.Catalanes	C.C.LCP-9	1	Section cleaning (70-150cm)	Polished	Body	6.5	Indet.
C.Catalanes	C.C.LCP-10	1	12(110-120cm)	Smoothed	Body	10.8	Indet.
C.Catalanes	C.C.LCP-11	1	12(110-120cm)	Polished	Body	7.7	Indet.
C.Catalanes	C.C.LCP-12	1	12(110-120cm)	Polished	Body	7.3	Indet.
C.Catalanes	C.C.LCP-13	2	13(120-130cm)	Smoothed	Body	6.6	Indet.
C.Catalanes	C.C.LCP-14	2	13(120-130cm)	Polished	Body	7.3	Indet.
C.Catalanes	C.C.LCP-15	2	13(120-130cm)	Polished	Body	5.8	Indet.
C.Catalanes	C.C.LCP-16	2	13(120-130cm)	Polished	Body	6.2	Indet.
C.Catalanes	C.C.LCP-17	2	10(90-100cm)	Polished	Body	6.0	Indet.
C.Catalanes	C.C.LCP-18	2	11(100-110cm)	Polished	Body	6.9	Indet.
C.Catalanes	C.C.LCP-19	2	11(100-110cm)	Polished	Body	7.7	Indet.
C.Catalanes	C.C.LCP-20	2	11(100-110cm)	Polished	Body	5.5	Indet.
C.Catalanes	C.C.LCP-21	2	11(100-110cm)	Smoothed	Neck-body	6.3	Indet.
C.Catalanes	C.C.LCP-22	2	11(100-110cm)	Smoothed	Neck	7.0	Indet.
C.Catalanes	C.C.LCP-23	2	11(100-110cm)	Polished	Neck	6.4	Indet.

C.Catalanes	C.C.LCP-24	2	5(40-50cm)	Polished	Body	7.2	Indet.
C.Catalanes	C.C.LCP-25	2	3(20-30cm)	Smoothed	Body	6.6	Indet.
C.Catalanes	C.C.LCP-26	1	12(110-120cm)	Polished	Neck-body	6.5	Indet.
C.Catalanes	C.C.LCP-27	1	9(80-90cm)	Smoothed	Body	7.6	Indet.
C.Catalanes	C.C.LCP-28	1	14(130-140cm)	Polished	Base	6.0	Indet.
C.Catalanes	C.C.LCP-29	1	12(110-120cm)	Polished	Neck	8.0	Indet.
C.Catalanes	C.C.LCP-30	2	1(0-10cm)	Smoothed	Rim	7.2	Indet.
C.Catalanes	C.C.LCP-31	2	9(80-90cm)	Polished	Body	7.8	Indet.
C.Catalanes	C.C.LCP-32	2	1(0-10cm)	Polished	Rim	7.3	Indet.
C.Catalanes	C.C.LCP-33	2	12(110-120cm)	Smoothed	Body	8.0	Indet.
C.Catalanes	C.C.LCP-34	2	12(110-120cm)	Polished	Body	7.8	Indet.
C.Catalanes	C.C.LCP-35	2	4(30-40cm)	Smoothed	Rim	7.9	Indet.
C.Catalanes	C.C.LCP-36	2	12(110-120cm)	Polished	Neck	7.6	Indet.
C.Catalanes	C.C.LCP-37	2	4(30-40cm)	Polished	Body	6.4	Indet.
C.Catalanes	C.C.LCP-38	2	4(30-40cm)	Polished	Body	5.2	Indet.
C.Catalanes	C.C.LCP-39	2	9(80-90cm)	Smoothed	Base	10.0	Indet.
C.Catalanes	C.C.LCP-40	2	2(10-20cm)	Polished	Base	7.1	Indet.
C.Catalanes	C.C.LCP-41	2	3(20-30cm)	Polished	Body	8.2	Indet.
C.Catalanes	C.C.LCP-42	2	3(20-30cm)	Polished	Body	7.0	Indet.
C.Catalanes	C.C.LCP-43	2	3(20-30cm)	Smoothed	Body	6.8	Indet.
C.Catalanes	C.C.LCP-44	2	3(20-30cm)	Polished	Body	8.0	Indet.

C.Catalanes	C.C.LCP-45	2	3(20-30cm)	Smoothed	Body	7.5	Indet.
C.Catalanes	C.C.LCP-46	2	7(60-70cm)	Polished	Body	7.9	Indet.
C.Catalanes	C.C.LCP-47	1	13(120-130cm)	Smoothed	Body	9.5	Indet.
C.Catalanes	C.C.LCP-48	2	6(50-60cm)	Smoothed	Neck	10.0	Indet.
C.Catalanes	C.C.LCP-49	2	12(110-120cm)	Polished	Neck-body	5.7	Indet.
C.Catalanes	C.C.LCP-50	2	1(0-10cm)	Polished	Body	6.1	Indet.
C.Catalanes	C.C.LCP-51	2	7(60-70cm)	Polished	Body	7.5	Indet.
C.Catalanes	C.C.LCP-52	2	1(0-10cm)	Polished	Base	8.2	Indet.
C.Catalanes	C.C.LCP-53	2	12(110-120cm)	Polished	Neck	5.5	Indet.
C.Catalanes	C.C.LCP-54	2	12(110-120cm)	Polished	Neck	5.8	Indet.
C.Catalanes	C.C.LCP-55	2	7(60-70cm)	Polished	Body	7.6	Indet.
C.Catalanes	C.C.LCP-56	1	11(100-110cm)	Smoothed	Neck-body	5.9	Indet.
C.Catalanes	C.C.LCP-57	1	11(100-110cm)	Polished	Body	5.6	Indet.
C.Catalanes	C.C.LCP-58	1	10(90-100cm)	Polished	Body	5.4	Indet.
C.Catalanes	C.C.LCP-59	1	13(120-130cm)	Polished	Body	5.8	Indet.
C.Catalanes	C.C.LCP-60	1	13(120-130cm)	Polished	Body	5.0	Indet.
C.Catalanes	C.C.LCP-61	1	13(120-130cm)	Polished	Body	5.9	Indet.
C.Catalanes	C.C.LCP-62	2	3(20-30cm)	Smoothed	Body	7.4	Indet.
C.Catalanes	C.C.LCP-63	2	4(30-40cm)	Polished	Body	8.0	Indet.
C.Catalanes	C.C.LCP-64	2	4(30-40cm)	Smoothed	Body	7.4	Indet.

Indet: Indeterminate

Appendix 9: Molecular and isotopic data from Catalanes Cave, southern Chile

Sample ID	Site	Lipid conc. (µg g-1)	Main compounds detected	P/S	SRR%	APAA C18E/H	APAA C20/C18	δ13C16:0 (‰)	δ13C 18:0 (‰)	Δ13C (‰)	ALK CPI
C.C.ECP-1	C.Catalanes	23.7	SFA($C_{9:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_8-C_{19}), OH (C_{16}-C_{28}), ALK (C_{23} - C_{35}), pri, APAA-C_{18}	1.9	n.p	1.9	n.p	-28.2	-29.3	-1.1	16.5
C.C.ECP-2	C.Catalanes	30.6	SFA ($C_{12:0}$ - $C_{226:0}$), UFA ($C_{16:1}$ - $C_{22:1}$),OH (C_{16}-C_{22}), ALK (C_{23} - C_{31}),pri	1.2	n.p	n.p	n.p	-29.3	-30.0	-0.7	1.3
C.C.ECP-3	C.Catalanes	6.6	SFA ($C_{12:0}$ - $C_{22:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), ALK(C_{23}-C_{31}), pri	1.7	n.p	5.0	n.p	n.p	n.p	n.p	1.6
C.C.ECP-4	C.Catalanes	14.5	SFA ($C_{12:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), OH (C_{16}-C_{22}), pri	1.7	n.p	n.p	n.p	-28.3	-30.1	-1.8	0.0
C.C.ECP-5	C.Catalanes	18.9	SFA ($C_{12:0}$ - $C_{26:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC (C_9), OH (C_{16}-C_{20}), pri	1.4	n.p	n.p	n.p	-28.7	-30.3	-1.7	0.0
C.C.ECP-6	C.Catalanes	11.9	SFA ($C_{12:0}$ - $C_{26:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), ALK (C_{23} - C_{35}), OH(C_{16}-C_{26}), phy, pr, APAA-C_{18}	1.1	n.p	3.0	n.p	-29.0	-29.9	-0.9	3.0
C.C.ECP-7	C.Catalanes	8.4	SFA ($C_{12:0}$ - $C_{24:0}$), UFA ($C_{14:1}$ - $C_{22:1}$),OH(C_{16}-C_{20}), pri	1.2	n.p	n.p	n.p	-29.7	-30.6	-0.9	0.0
C.C.ECP-8	C.Catalanes	14.2	SFA ($C_{12:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_9-C_{10}), OH(C_{16}-C_{18}), pri, APAA-C_{18}	1.2	n.p	7.0	n.p	-29.6	-30.5	-0.9	0.0
C.C.ECP-9	C.Catalanes	248.3	SFA ($C_{8:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), DC(C_8-C_{19}), ALK (C_{27} - C_{35}), OH(C_{26}-C_{28}), phy, APAAs(C_{18}-C_{20})	4.2	n.p	3.6	0.17	-26.3	-28.8	-2.4	9.2
C.C.ECP-10	C.Catalanes	122.4	SFA ($C_{8:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC (C_6 - C_{19}), ALK (C_{24} - C_{35}), phy, APAAs(C_{18}-C_{20})	3.3	n.p	3.3	0.14	-26.7	-28.4	-1.6	8.4
C.C.ECP-11	C.Catalanes	11.8	SFA ($C_{9:0}$ - $C_{22:0}$), UFA ($C_{16:1}$ - $C_{21:1}$),, , ALK (C_{23} - C_{35}),pri	1.3	n.p	n.p	n.p	-29.9	-30.8	-0.9	4.1
C.C.ECP-12	C.Catalanes	46.5	SFA ($C_{8:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC (C_6-C_{13}), ALK(C_{23}-C_{35}), OH(C_{16}-C_{30}), phy, pri,	1.8	n.p	n.p	n.p	-28.6	-30.1	-1.4	37.1
C.C.ECP-13	C.Catalanes	57.9	SFA ($C_{8:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), , DC (C_6 - C_{13}), Alk(C_{24}-C_{35}), OH(C_{16}-C_{30}), pri, APAAs (C_{18}, C_{20}).	2.8	n.p	3.5	n.p	-28.0	-29.7	-1.7	8.7
C.C.ECP-14	C.Catalanes	24.8	SFA ($C_{9:0}$ - $C_{24:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), , OH(C_{16}-C_{20}), pri	2.0	n.p	n.p	n.p	-29.0	-31.2	-2.2	0.0

C.C.ECP-15	C.Catalanes	34.7	SFA ($C_{9:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), ALK (C_{23} - C_{35}), OH(C_{16}-C_{28}), pri,	1.7	n.p	n.p	n.p	-28.6	-30.7	-2.1	3.5
C.C.ECP-16	C.Catalanes	25.6	SFA ($C_{9:0}$ - $C_{32:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), DC (C_8-C_{19}), ALK (C_{23} - C_{35}), OH(C_{16}-C_{32}), phy, pri, APAA-C_{18}	2.5	n.p	5.5	n.p	-27.6	-29.4	-1.8	9.5
C.C.ECP-17	C.Catalanes	10.4	SFA ($C_{11:0}$ - $C_{18:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), pri	2.0	n.p	n.p	n.p	n.p	n.p	n.p	0.0
C.C.ECP-18	C.Catalanes	114.0	SFA ($C_{8:0}$ - $C_{32:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC (C_7-C_{19}), Alk (C_{24}-C_{35}), OH(C_{20}-C_{32}), phy, tmtd, pri, APAA-C_{18}	0.6	78.1	4.2	n.p	-29.7	-31.8	-2.2	19.1
C.C.ECP-19	C.Catalanes	24.1	SFA ($C_{11:0}$ - $C_{28:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), Alk (C_{23}-C_{35}), OH(C_{16}-C_{30}), pri, APAAs-C_{18}	1.6	n.p	3.7	n.p	-28.6	-31.4	-2.7	4.9
C.C.ECP-20	C.Catalanes	19.5	SFA ($C_{8:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{22:1}$)	1.4	n.p	n.p	n.p	-29.2	-30.4	-1.1	0.0
C.C.ECP-21	C.Catalanes	11.2	SFA ($C_{11:0}$ - $C_{26:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), Alk (C_{23} - C_{35}) pri	1.3	n.p	n.p	n.p	-29.6	-30.2	-0.7	8.2
C.C.ECP-22	C.Catalanes	21.5	SFA ($C_{9:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC (C_9), Alk(C_{23}-C_{35}),pri, APAA-C_{18}	1.8	n.p	4.7	n.p	n.p	n.p	n.p	12.2
C.C.ECP-23	C.Catalanes	29.3	SFA ($C_{8:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC (C_8 - C_9), ALK (C_{23} - C_{35}), pri	2.7	n.p	n.p	n.p	n.p	n.p	n.p	9.2
C.C.ECP-24	C.Catalanes	14.0	SFA ($C_{12:0}$ - $C_{26:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC (C_{10}), ALK (C_{23} - C_{35}), pri	2.0	n.p	n.p	n.p	n.p	n.p	n.p	4.5
C.C.ECP-25	C.Catalanes	9.7	SFA ($C_{12:0}$ - $C_{24:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), pri	2.5	n.p	n.p	n.p	n.p	n.p	n.p	0.0
C.C.ECP-26	C.Catalanes	27.4	SFA ($C_{12:0}$ - $C_{26:0}$), UFA ($C_{14:1}$ - $C_{22:1}$)	2.5	n.p	n.p	n.p	n.p	n.p	n.p	0.0
C.C.ECP-27	C.Catalanes	50.6	SFA ($C_{10:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_8, C_9), ALK (C_{23} - C_{35}), OH(C_{18}-C_{22}), pri	3.5	n.p	n.p	n.p	-26.0	-28.5	-2.5	4.1
C.C.ECP-28	C.Catalanes	22.1	SFA ($C_{8:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC (C_8-C_{22}), Alk(C_{24}-C_{33}), OH(C_{16}-C_{30}), Lup, Amy, phy, pri	5.3	n.p	n.p	n.p	-27.5	-30.2	-2.7	29.9
C.C.ECP-29	C.Catalanes	16.3	SFA ($C_{12:0}$ - $C_{24:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), pri	2.3	n.p	n.p	n.p	-27.4	-29.8	-2.5	0.0
C.C.ECP-30	C.Catalanes	14.6	SFA ($C_{9:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC (C_8 - C_9), ALK (C_{23} - C_{35}), pri	2.0	n.p	n.p	n.p	n.p	n.p	n.p	15.9
C.C.ECP-31	C.Catalanes	37.0	SFA ($C_{10:0}$ - $C_{32:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), Alk(C_{23}-C_{35}), OH(C_{16}-C_{30}), K_{29}, phy, pri, APAA-C_{18}	1.3	72.4	4.7	n.p	-28.1	-30.2	-2.0	2.5
C.C.ECP-32	C.Catalanes	10.4	SFA ($C_{12:0}$ - $C_{26:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), pri	1.6	n.p	n.p	n.p	n.p	n.p	n.p	0.0
C.C.ECP-33	C.Catalanes	7.9	SFA ($C_{12:0}$ - $C_{26:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC (C_9), pri, APAA-C_{18}	2.5	n.p	4.4	n.p	n.p	n.p	n.p	0.0
C.C.ECP-34	C.Catalanes	18.7	SFA ($C_{12:0}$ - $C_{24:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), pri	1.6	n.p	n.p	n.p	n.p	n.p	n.p	0.8

C.C.ECP-35	C.Catalanes	12.3	SFA ($C_{12:0}$ - $C_{24:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), pri	2.6	n.p	n.p	n.p	n.p	n.p	n.p	0.0
C.C.ECP-36	C.Catalanes	8.9	SFA ($C_{12:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC (C_9), pri	3.0	n.p	n.p	n.p	n.p	n.p	n.p	0.0
C.C.ECP-37	C.Catalanes	15.5	SFA ($C_{12:0}$ - $C_{28:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), ALK (C_{23} - C_{33}), pri	2.5	n.p	n.p	n.p	n.p	n.p	n.p	0.8
C.C.ECP-38	C.Catalanes	34.2	SFA ($C_{10:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_9), Alk(C_{23}-C_{33}), OH(C_{16}-C_{30}), pri	3.4	n.p	n.p	n.p	-27.1	-29.7	-2.6	0.8
C.C.ECP-39	C.Catalanes	24.0	SFA ($C_{12:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC (C_9 - C_{10}), Alk (C_{23} - C_{33}), OH(C_{16}-C_{20}), pri	3.3	n.p	n.p	n.p	-27.0	-29.9	-2.9	0.7
C.C.ECP-40	C.Catalanes	37.9	SFA ($C_{11:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_9-C_{19}), ALK (C_{24} - C_{35}), OH(C_{16}-C_{32}), K_{31}, phy, pri, APAA-C_{18}	2.4	92.1	8.6	n.p	-26.6	-28.5	-1.9	4.2
C.C.ECP-41	C.Catalanes	27.9	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), ALK (C_{23} - C_{33}), pri	2.0	n.p	n.p	n.p	-27.7	-29.4	-1.7	0.7
C.C.ECP-42	C.Catalanes	21.8	SFA ($C_{12:0}$ - $C_{26:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC (C_9), Alk (C_{23}-C_{33}), pri,	1.5	n.p	n.p	n.p	-28.8	-29.8	-0.9	0.7
C.C.ECP-43	C.Catalanes	24.5	SFA ($C_{8:0}$ - $C_{26:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC (C_9), Alk(C_{23}-C_{33}), pri	1.4	n.p	n.p	n.p	-29.2	-29.6	-0.4	0.6
C.C.ECP-44	C.Catalanes	31.9	SFA ($C_{12:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_8-C_{18}), Alk(C_{23}-C_{33}), OH(C_{20}-C_{30}), phy, pri, APAAs(C_{18}-C_{20})	2.4	63.2	6.6	0.06	-26.4	-27.8	-1.4	0.7
C.C.ECP-45	C.Catalanes	120.6	SFA ($C_{8:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC (C_8 - C_{26}), Alk(C_{23}-C_{33}), phy, pri, APAAs (C_{18}- C_{20}).	0.5	43.9	5.6	0.06	-29.5	-32.3	-2.8	1.5
C.C.ECP-46	C.Catalanes	7.3	SFA ($C_{12:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), ALK (C_{23} - C_{33}), pri	1.9	n.p	n.p	n.p	n.p	n.p	n.p	0.7
C.C.ECP-47	C.Catalanes	16.7	SFA ($C_{12:0}$ - $C_{26:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), ALK (C_{23} - C_{33}), pri	1.9	n.p	n.p	n.p	-28.0	-29.6	-1.5	0.7
C.C.ECP-48	C.Catalanes	19.6	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), Alk (C_{23}-C_{33}), pri	2.6	n.p	n.p	n.p	-26.8	-29.6	-2.9	0.7
C.C.ECP-49	C.Catalanes	35.5	SFA ($C_{8:0}$ - $C_{26:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), Alk(C_{23}-C_{33}), pri	1.5	n.p	n.p	n.p	-28.9	-29.7	-0.9	0.7
C.C.ECP-50	C.Catalanes	32.0	SFA ($C_{9:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), ALK (C_{24} - C_{35}), pri, tmtd, APAA-C_{18}	1.8	n.p	1.7	n.p	-28.1	-29.4	-1.3	2.0
C.C.ECP-51	C.Catalanes	68.5	SFA ($C_{9:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_7-C_{19}), ALK (C_{24} - C_{35}), phy, pri, tmtd, APAA-C_{18}	0.7	34.8	2.5	n.p	-27.9	-31.8	-3.9	2.4
C.C.ECP-52	C.Catalanes	27.4	SFA ($C_{8:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_9-C_{24}), ALK (C_{24} - C_{35}), OH(C_{16}-C_{32}), pri, tmtd, APAA-C_{18}	2.6	n.p	n.p	n.p	-28.3	-29.3	-1.0	10.1
C.C.ECP-53	C.Catalanes	38.4	SFA ($C_{9:0}$ - $C_{28:0}$), UFA ($C_{16:1}$ - $C_{24:1}$),, ALK (C_{24} - C_{35}), pri, tmtd	2.1	n.p	n.p	n.p	-28.1	-30.8	-2.7	1.9

C.C.EC P-54	C.Catalanes	31.1	SFA ($C_{9:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_8-C_{13}), ALK (C_{24} - C_{35}), pri, tmtd	2.0	n.p	n.p	n.p	-27.5	-30.1	-2.7	2.0
C.C.EC P-55	C.Catalanes	23.4	SFA ($C_{10:0}$ - $C_{26:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), ALK (C_{23} - C_{33}), pri, tmtd	2.1	n.p	n.p	n.p	n.p	n.p	n.p	0.7
C.C.EC P-56	C.Catalanes	1204.4	SFA ($C_{8:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_6-C_{18}), ALK (C_{24} - C_{35}), phy, pri, tmtd, APAA-C_{18}	0.7	28.5	2.8	n.p	-28.9	-32.4	-3.5	1.6
C.C.EC P-57	C.Catalanes	21.7	SFA ($C_{9:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_8-C_{19}), ALK (C_{24} - C_{33}), pri, tmtd	4.1	n.p	n.p	n.p	n.p	n.p	n.p	1.1
C.C.EC P-58	C.Catalanes	11.2	SFA ($C_{11:0}$ - $C_{26:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_9), ALK (C_{23} - C_{33}), pri, tmtd	1.6	n.p	n.p	n.p	-29.2	-30.0	-0.9	0.7
C.C.EC P-59	C.Catalanes	22.1	SFA ($C_{8:0}$ - $C_{24:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_6), ALK (C_{23} - C_{33}), pri, tmtd	1.4	n.p	n.p	n.p	-29.9	-29.9	0.0	0.7
C.C.EC P-60	C.Catalanes	30.3	SFA ($C_{9:0}$ - $C_{26:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), pri	2.1	n.p	n.p	n.p	-27.9	-30.3	-2.4	0.0
C.C.EC P-61	C.Catalanes	15.0	SFA ($C_{11:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), ALK (C_{24} - C_{35}), OH(C_{16}-C_{30}), pri, tmtd	1.7	n.p	n.p	n.p	-27.4	-29.5	-2.0	4.6
C.C.EC P-62	C.Catalanes	19.3	SFA ($C_{12:0}$ - $C_{32:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), ALK (C_{24} - C_{35}), pri	2.0	n.p	n.p	n.p	n.p	n.p	n.p	5.3
C.C.EC P-63	C.Catalanes	14.9	SFA ($C_{12:0}$ - $C_{32:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), ALK (C_{23} - C_{35}), pri	1.9	n.p	n.p	n.p	n.p	n.p	n.p	9.0
C.C.EC P-64	C.Catalanes	22.4	SFA ($C_{11:0}$ - $C_{26:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), Alk(C_{23}-C_{33}), pri, tmtd	2.1	n.p	n.p	n.p	-28.3	-30.0	-1.7	0.9
C.C.LC P-1	C.Catalanes	34.7	SFA ($C_{10:0}$ - $C_{28:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC(C_8-C_9), OH(C_{16}-C_{18}), pri, APAA-C_{18}	2.7	n.p	3.3	n.p	-28.2	-31.1	-2.8	0.0
C.C.LC P-2	C.Catalanes	18.7	SFA ($C_{12:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_9), Alk(C_{23}-C_{35}), OH(C_{16}-C_{18}, C_{28}), pri	2.0	n.p	n.p	n.p	-28.1	-29.7	-1.6	3.4
C.C.LC P-3	C.Catalanes	56.2	SFA ($C_{10:0}$ - $C_{26:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_9), Alk(C_{23}-C_{35}), OH(C_{16}-C_{22}), pri	3.0	n.p	n.p	n.p	-26.9	-28.7	-1.7	1.6
C.C.LC P-4	C.Catalanes	91.8	SFA ($C_{8:0}$ - $C_{26:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC(C_7-C_{18}), Alk(C_{24}-C_{35}), OH(C_{16}-$C_{20,}$ C_{28}, C_{30}), phy, tmtd, APAAs(C_{18}-C_{20})	2.1	n.p	5.2	0.01	-29.3	-30.2	-0.9	10.3
C.C.LC P-5	C.Catalanes	27.1	SFA ($C_{12:0}$ - $C_{32:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC(C_9, C_{10}), ALK (C_{23} - C_{35}), OH(C_{16}-C_{32}), pri, APAA-C_{18}	2.1	n.p	4.8	n.p	-27.8	-29.7	-1.9	11.6
C.C.LC P-6	C.Catalanes	28.7	SFA ($C_{8:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_9), ALK (C_{24} - C_{35}), OH(C_{16}-C_{22}), pri, APAA-C_{18}	2.9	n.p	3.1	n.p	-28.7	-29.6	-0.9	0.9
C.C.LC P-7	C.Catalanes	14.5	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_9), ALK (C_{23} - C_{35}), OH(C_{16}-C_{22}), pri, APAA-C_{18}	1.9	n.p	1.9	n.p	-28.4	-30.7	-2.4	2.2

C.C.LCP-8	C.Catalanes	20.6	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), ALK (C_{23} - C_{35}), OH(C_{16}-C_{32}), phy, pri, APAA-C_{18}	1.1	82.9	2.3	n.p	-27.5	-31.6	-4.1	10.4
C.C.LCP-9	C.Catalanes	73.9	SFA ($C_{12:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_9), ALK (C_{24} - C_{35}), OH(C_{16}-C_{28}), phy, pri, tmtd, APAA-C_{18}	1.8	94.5	2.1	n.p	-24.0	-26.1	-2.0	6.6
C.C.LCP-10	C.Catalanes	9.1	SFA ($C_{12:0}$ - $C_{24:0}$), UFA ($C_{16:1}$ - $C_{18:1}$), pri	1.3	n.p	n.p	n.p	n.p	n.p	n.p	0.0
C.C.LCP-11	C.Catalanes	14.4	SFA ($C_{12:0}$ - $C_{32:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), DC(C_9), ALK (C_{23} - C_{35}), OH(C_{16}-C_{32}), pri, APAA-C_{18}	1.8	n.p	6.0	n.p	-28.2	-29.3	-1.0	14.6
C.C.LCP-12	C.Catalanes	13.8	SFA ($C_{12:0}$ - $C_{28:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), ALK (C_{24} - C_{35}), pri, APAA-C_{18}	2.0	n.p	4.4	n.p	n.p	n.p	n.p	2.4
C.C.LCP-13	C.Catalanes	9.7	SFA ($C_{12:0}$ - $C_{28:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), DC(C_{13}), ALK (C_{23} - C_{35}), pri	1.4	n.p	n.p	n.p	n.p	n.p	n.p	1.7
C.C.LCP-14	C.Catalanes	22.6	SFA ($C_{12:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), ALK (C_{23} - C_{35}), OH(C_{16}-C_{32}), pri, tmtd	2.1	n.p	n.p	n.p	-19.6	-22.4	-2.8	7.4
C.C.LCP-15	C.Catalanes	18.3	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), ALK (C_{23} - C_{35}), pri, tmtd	1.9	n.p	n.p	n.p	n.p	n.p	n.p	2.9
C.C.LCP-16	C.Catalanes	11.1	SFA ($C_{12:0}$ - $C_{28:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), ALK (C_{23} - C_{35}), pri, APAA-C_{18}	1.5	n.p	2.8	0.05	n.p	n.p	n.p	1.3
C.C.LCP-17	C.Catalanes	432.6	SFA ($C_{8:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), DC(C_7-C_{18}), ALK (C_{24} - C_{35}), OH(C_{26}-C_{32}), phy, pri, APAAs(C_{18}-C_{20})	1.9	81.7	8.6	0.04	-27.8	-27.1	0.8	10.0
C.C.LCP-18	C.Catalanes	11.1	SFA ($C_{12:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_9-C_{24}), ALK (C_{24} - C_{35}), pri, APAAs-C_{18}	1.6	n.p	7.5	n.p	n.p	n.p	n.p	2.7
C.C.LCP-19	C.Catalanes	13.2	SFA ($C_{12:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), ALK (C_{23} - C_{33}), pri	1.9	n.p	n.p	n.p	n.p	n.p	n.p	1.3
C.C.LCP-20	C.Catalanes	10.7	SFA ($C_{12:0}$ - $C_{28:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), ALK (C_{24} - C_{33}), OH(C_{16}-C_{20}), pri	1.1	n.p	n.p	n.p	-27.5	-28.9	-1.4	2.9
C.C.LCP-21	C.Catalanes	25.4	SFA ($C_{10:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), ALK (C_{23} - C_{33}), OH(C_{16}-C_{20}), pri	1.7	n.p	n.p	n.p	-27.7	-30.0	-2.3	0.9
C.C.LCP-22	C.Catalanes	44.1	SFA ($C_{9:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_9), ALK (C_{24} - C_{35}), OH(C_{16}-C_{34}), pri, APAAs-C_{18}	2.2	n.p	6.8	n.p	-19.8	-21.8	-1.9	30.6
C.C.LCP-23	C.Catalanes	99.1	SFA ($C_{9:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_6-C_9), ALK (C_{24} - C_{35}), OH(C_{16}-C_{32}), APAAs-C_{18}	2.8	n.p	5.5	0.25	-16.8	-18.7	-1.8	6.8
C.C.LCP-24	C.Catalanes	26.2	SFA ($C_{10:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_6-C_{26}), ALK (C_{24} - C_{35}), OH(C_{16}-C_{18}), pri	1.9	n.p	n.p	n.p	-28.5	-30.6	-2.1	63.5

C.C.LCP-25	C.Catalanes	12.4	SFA ($C_{12:0}$ - $C_{26:0}$), UFA ($C_{14:1}$ - $C_{18:1}$), DC(C_9-C_2), ALK (C_{26} - C_{33}), pri	3.2	n.p	n.p	n.p	n.p	n.p	n.p	1.6
C.C.LCP-26	C.Catalanes	28.8	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), ALK (C_{23} - C_{35}), OH(C_{16}-C_{30}), phy, pri, APAA-C_{18}	1.5	n.p	3.0	n.p	-28.3	-29.7	-1.5	4.0
C.C.LCP-27	C.Catalanes	29.0	SFA ($C_{10:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), DC(C_7-C_{19}), ALK (C_{24} - C_{35}), OH(C_{16}-C_{30}), pri, APAA-C_{18}	1.7	n.p	8.2	n.p	-28.6	-29.6	-0.9	7.6
C.C.LCP-28	C.Catalanes	34.1	SFA ($C_{10:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_6-C_{13}), ALK (C_{23} - C_{35}), OH(C_{16}-C_{22}), pri	2.7	n.p	n.p	n.p	-25.7	-28.3	-2.6	1.8
C.C.LCP-29	C.Catalanes	9.6	SFA ($C_{12:0}$ - $C_{24:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), ALK (C_{23} - C_{33}), pri	2.5	n.p	n.p	n.p	n.p	n.p	n.p	0.8
C.C.LCP-30	C.Catalanes	118.7	SFA ($C_{10:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_7-C_{28}), ALK (C_{26} - C_{35}), OH(C_{16}-C_{30}), phy, pri, tmtd, APAA-C_{18}	1.0	62.2	2.1	n.p	-26.9	-30.1	-3.2	10.0
C.C.LCP-31	C.Catalanes	20.7	SFA ($C_{11:0}$ - $C_{26:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), DC(C_7-C_{28}), ALK (C_{26} - C_{35}), pri, tmtd	2.9	n.p	n.p	n.p	n.p	n.p	n.p	4.5
C.C.LCP-32	C.Catalanes	50.2	SFA ($C_{10:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{18:1}$), DC(C_6-C_{13}), ALK (C_{26} - C_{35}), OH(C_{16}-C_{32}), pri, tmtd	4.2	n.p	n.p	n.p	-27.0	-28.1	-1.1	1.1
C.C.LCP-33	C.Catalanes	33.4	SFA ($C_{10:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_9), ALK (C_{23} - C_{35}), OH(C_{16}-C_{32}), pri, tmtd	3.2	n.p	n.p	n.p	-26.5	-29.2	-2.6	7.4
C.C.LCP-34	C.Catalanes	44.2	SFA ($C_{10:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_9), ALK (C_{24} - C_{35}), OH(C_{16}-C_{34}), pl_st, pri, tmtd	2.0	n.p	n.p	n.p	-19.8	-21.7	-2.0	25.3
C.C.LCP-35	C.Catalanes	31.1	SFA ($C_{11:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_9-C_{21}), ALK (C_{24} - C_{35}), OH(C_{16}-C_{26}), pri, tmtd	3.3	n.p	n.p	n.p	-26.7	-28.3	-1.6	1.5
C.C.LCP-36	C.Catalanes	28.8	SFA ($C_{11:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_8-C_9), ALK (C_{23} - C_{33}), OH(C_{16}-C_{22}), pri,APAA-C_{18}	1.8	n.p	3.1	n.p	-28.4	-29.7	-1.3	1.2
C.C.LCP-37	C.Catalanes	23.5	SFA ($C_{11:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_9-C_{26}), ALK (C_{23} - C_{35}), OH(C_{16}-C_{32}), pri, tmtd	2.2	n.p	n.p	n.p	-27.7	-29.7	-2.0	2.2
C.C.LCP-38	C.Catalanes	24.9	SFA ($C_{11:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_8-C_{28}), ALK (C_{23} - C_{35}), OH(C_{16}-C_{32}), pri	1.5	n.p	n.p	n.p	-28.5	-30.1	-1.6	2.1
C.C.LCP-39	C.Catalanes	13.4	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_9,C_{13}), ALK (C_{23} - C_{35}), pri, tmtd	3.1	n.p	n.p	n.p	n.p	n.p	n.p	5.3
C.C.LCP-40	C.Catalanes	31.7	SFA ($C_{12:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_8-C_{24}), ALK (C_{26} - C_{35}), phy, pri	2.5	n.p	n.p	n.p	n.p	n.p	n.p	8.1

C.C.LCP-41	C.Catalanes	61.8	SFA ($C_{8:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_6-C_{24}), ALK (C_{26} - C_{35}), OH(C_{16}-C_{26}), K_{29}, pri, tmtd	2.5	n.p	n.p	n.p	-27.2	-29.2	-1.9	44.6
C.C.LCP-42	C.Catalanes	65.7	SFA ($C_{8:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_6-C_{20}), ALK (C_{23} - C_{35}), OH(C_{16}-C_{26}), pri, tmtd	1.8	n.p	n.p	n.p	-28.4	-30.0	-1.6	31.5
C.C.LCP-43	C.Catalanes	18.0	SFA ($C_{11:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_7-C_{26}), ALK (C_{26} - C_{35}), OH(C_{16}-C_{32}), pri, tmtd	2.3	n.p	n.p	n.p	-27.0	-29.2	-2.2	6.4
C.C.LCP-44	C.Catalanes	14.4	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), pri, tmtd	2.8	n.p	n.p	n.p	n.p	n.p	n.p	0.0
C.C.LCP-45	C.Catalanes	7.4	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{18:1}$), DC(C_9-C_{22}), Alk(C_{24}-C_{33}), phy, pri, tmtd	1.6	n.p	n.p	n.p	n.p	n.p	n.p	2.7
C.C.LCP-46	C.Catalanes	22.2	SFA ($C_{13:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_{22}-C_{28}), ALK (C_{24} - C_{35}), OH(C_{16}-C_{32}), phy, pri, tmtd	2.5	95.0	n.p	n.p	-26.4	-29.7	-3.3	4.1
C.C.LCP-47	C.Catalanes	21.1	SFA ($C_{12:0}$ - $C_{32:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), DC(C_{16}-C_{28}), ALK (C_{23} - C_{35}), amy, pri, tmtd	1.7	n.p	n.p	n.p	n.p	n.p	n.p	3.5
C.C.LCP-48	C.Catalanes	70.7	SFA ($C_{13:0}$ - $C_{32:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), DC(C_{22}-C_{28}), ALK (C_{23} - C_{35}), OH(C_{16}-C_{32}), phy, pri, tmtd, APAA-C_{18}	1.6	81.5	5.6	n.p	-27.4	-29.6	-2.3	11.3
C.C.LCP-49	C.Catalanes	21.4	SFA ($C_{12:0}$ - $C_{28:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), ALK (C_{24} - C_{33}), OH(C_{16}-C_{18}), pri, tmtd	1.4	n.p	n.p	n.p	-27.8	-30.1	-2.3	1.4
C.C.LCP-50	C.Catalanes	11.3	SFA ($C_{12:0}$ - $C_{32:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), DC(C_9-C_{14}), ALK (C_{24} - C_{35}), OH(C_{16}-C_{18}), phy, pri, tmtd	1.1	89.8	n.p	n.p	-28.6	-30.2	-1.5	4.7
C.C.LCP-51	C.Catalanes	30.1	SFA ($C_{12:0}$ - $C_{32:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), DC(C_9-C_{25}), ALK (C_{24} - C_{35}), OH(C_{16}-C_{32}), pl_st, phy, pri, tmtd	1.5	92.5	n.p	n.p	-25.6	-28.0	-2.4	4.4
C.C.LCP-52	C.Catalanes	769.7	SFA ($C_{8:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{20:1}$), DC(C_6-C_{14}), ALK (C_{26} - C_{35}), phy, pri, tmtd, APAA-C_{18}	1.4	58.8	1.6	n.p	-26.1	-28.2	-2.1	6.4
C.C.LCP-53	C.Catalanes	26.6	SFA ($C_{14:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), ALK (C_{23} - C_{33}), OH(C_{16}-C_{22}), pri, tmtd	3.5	n.p	n.p	n.p	-26.6	-29.6	-3.0	1.0
C.C.LCP-54	C.Catalanes	22.3	SFA ($C_{14:0}$ - $C_{32:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), DC(C_8-C_{22}), phy, pri, tmtd, APAAs (C_{18}-C_{20})	1.4	60.5	5.2	0.04	-27.3	-29.6	-2.3	0.0
C.C.LCP-55	C.Catalanes	29.3	SFA ($C_{12:0}$ - $C_{32:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), DC(C_9-C_{28}), Alk(C_{23}-C_{35}), OH(C_{16}-C_{32}), phy, pri, tmtd, APAA-C_{18}	1.3	93.2	0.8	n.p	-28.5	-30.2	-1.7	4.6
C.C.LCP-56	C.Catalanes	46.6	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_9,C_{24}-C_{27}), Alk(C_{24}-C_{33}), OH(C_{16}-C_{22}) pri, APAA-C_{18}	2.2	n.p	1.6	n.p	-28.2	-29.9	-1.7	0.8

C.C.LCP-57	C.Catalanes	47.7	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), Alk(C_{23}-C_{35}), OH(C_{16}-C_{22}) pri, APAAs(C_{18}-C_{20})	1.9	n.p	4.6	0.03	-27.4	-29.7	-2.4	2.7
C.C.LCP-58	C.Catalanes	31.0	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_9), Alk(C_{23}-C_{33}), OH(C_{16}-C_{20}) phy, pri, APAAs(C_{18}-C_{20})	2.7	n.p	4.6	0.03	-28.0	-30.0	-2.0	0.9
C.C.LCP-59	C.Catalanes	47.5	SFA ($C_{11:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), Alk(C_{23}-C_{35}), OH(C_{16}-C_{24}) pri	2.8	n.p	n.p	n.p	-26.8	-28.7	-2.0	2.8
C.C.LCP-60	C.Catalanes	96.1	SFA ($C_{8:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_8-C_9, C_{20}-C_{26}), Alk(C_{24}-C_{35}), OH(C_{16}-C_{32}) pl_st, pri, APAAs(C_{18}-C_{20})	1.5	n.p	6.3	0.08	-24.6	-27.3	-2.7	29.3
C.C.LCP-61	C.Catalanes	36.0	SFA ($C_{8:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_9, C_{19}-C_{26}), Alk(C_{24}-C_{35}), OH(C_{16}-C_{22}) pri, APAAs(C_{18}-C_{20})	1.6	n.p	2.5	0.14	-27.4	-28.4	-1.0	2.0
C.C.LCP-62	C.Catalanes	16.6	SFA ($C_{10:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_9-C_{10}, C_{16}-C_{24}), Alk(C_{24}-C_{35}), OH(C_{16}-C_{30}) pri, tmtd	2.1	n.p	n.p	n.p	-28.2	-30.2	-2.0	1.9
C.C.LCP-63	C.Catalanes	29.5	SFA ($C_{8:0}$ - $C_{32:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), DC(C_7-C_{27}), Alk(C_{23}-C_{35}), OH(C_{16}-C_{30}) phy, pri, tmtd, APAA-C_{18}	1.9	86.0	1.1	n.p	-22.5	-27.4	-4.9	3.1
C.C.LCP-64	C.Catalanes	12.0	SFA ($C_{8:0}$ - $C_{26:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), DC(C_9,C_{16}-C_{18}), Alk(C_{24}-C_{33}) phy, pri, tmtd	1.7	29.8	n.p	n.p	n.p	n.p	n.p	1.0

Appendix 10: Pottery from San Miguel de Renaico 5 (SMR5), southern Chile

Site	Sample	Excavation unit	Level	External surface' treatment	Fragment's shape	Thickness (mm)	Type of pot
SMR5	SMR5.ECP-1	35	5(40-50CM)	Polished	Rim-neck	7.0	Indet.
SMR5	SMR5.ECP-2	32	5(40-50CM)	Polished	Body	7.0	Indet.
SMR5	SMR5.ECP-3	24	6(50-60CM)	Polished	Rim	6.2	Indet.
SMR5	SMR5.ECP-4	24	8(70-80CM)	Polished	Body	8.0	Indet.
SMR5	SMR5.ECP-5	24	6(50-60CM)	Smoothed	Body	7.6	Indet.
SMR5	SMR5.ECP-6	24	6(50-60CM)	Smoothed	Body	8.1	Indet.

SMR5	SMR5.ECP-7	24	6(50-60CM)	Polished	Body	5.6	Indet.
SMR5	SMR5.ECP-8	24	7(60-70CM)	Polished	Body	10.9	Indet.
SMR5	SMR5.ECP-9	31	6(50-60CM)	Polished	Body	7.2	Indet.
SMR5	SMR5.ECP10	17	9(80-90CM)	Polished	Body	6.3	Indet.
SMR5	SMR5.ECP-11	35	5(40-50CM)	Smoothed	Neck	8.3	Indet.
SMR5	SMR5.ECP-12	32	6(50-60CM)	Polished	Rim	5	Indet.
SMR5	SMR5.ECP-13	29	5(40-50CM)	Polished	Body	8.3	Indet.
SMR5	SMR5.ECP-14	28	6(50-60CM)	Polished	Rim	6.6	Indet.
SMR5	SMR5.ECP-15	28	5(40-50CM)	Polished	Body	9.6	Indet.
SMR5	SMR5.ECP-16	24	6(50-60CM)	Polished	Rim	7.1	Indet.
SMR5	SMR5.ECP-17	24	6(50-60CM)	Polished	Body	9.6	Indet.
SMR5	SMR5.ECP-18	31	5(40-50CM)	Polished	Rim	7.4	Indet.
SMR5	SMR5.ECP-19	17	7(60-70CM)	Polished	Rim	7.6	Indet.
SMR5	SMR5.ECP-20	17	5(40-50CM)	Polished	Rim	7.9	Indet.
SMR5	SMR5.ECP-21	17	5(40-50CM)	Polished	Rim	7.5	Indet.
SMR5	SMR5.ECP-22	17	6(50-60CM)	Smoothed	Neck	8.7	Indet.
SMR5	SMR5.ECP-23	31	5(40-50CM)	Smoothed	Rim	8.8	Indet.
SMR5	SMR5.ECP-24	24	7(60-70CM)	Smoothed	Rim	6.8	Indet.
SMR5	SMR5.ECP-25	29	6(50-60CM)	Polished	Neck	6.1	Indet.
SMR5	SMR5.ECP-26	24	6(50-60CM)	Smoothed	Rim	6.7	Indet.
SMR5	SMR5.ECP-27	32	5(40-50CM)	Polished	Rim	5.5	Indet.
SMR5	SMR5.ECP-28	24	7(60-70CM)	Polished	Body	8.7	Indet.
SMR5	SMR5.ECP-29	35	6(50-60CM)	Polished	Rim	6.6	Indet.
SMR5	SMR5.ECP-30	24	7(60-70CM)	Polished	Body	8.5	Indet.
SMR5	SMR5.LCP-1	35	4(30-40CM)	Polished	Rim-neck	6.7	Indet.
SMR5	SMR5.LCP-2	35	1(0-10CM)	Polished	Body	7.6	Indet.

SMR5	SMR5.LCP-3	35	4(30-40CM)	Polished	Rim	7.8	Indet.
SMR5	SMR5.LCP-4	32	3(20-30CM)	Polished	Body	7	Indet.
SMR5	SMR5.LCP-5	32	4(30-40CM)	Smoothed	Body	9.3	Indet.
SMR5	SMR5.LCP-6	32	3(20-30CM)	Smoothed	Body	9	Indet.
SMR5	SMR5.LCP-7	33	1(0-10CM)	Polished	Body	7.5	Indet.
SMR5	SMR5.LCP-8	33	4(30-40CM)	Polished	Body	7.3	Indet.
SMR5	SMR5.LCP-9	33	4(30-40CM)	Polished	Body	7.8	Indet.
SMR5	SMR5.LCP-10	34	3(20-30CM)	Polished	Body	9.9	Indet.
SMR5	SMR5.LCP-11	35	3(20-30CM)	Smoothed	Neck	8.5	Indet.
SMR5	SMR5.LCP-12	35	2(10-20CM)	Polished	Rim	8.3	Indet.
SMR5	SMR5.LCP-13	35	4(30-40CM)	Polished	Body	7.8	Indet.
SMR5	SMR5.LCP-14	35	4(30-40CM)	Polished	Rim	8	Indet.
SMR5	SMR5.LCP-15	35	4(30-40CM)	Polished	Body	8.7	Indet.
SMR5	SMR5.LCP-16	32	2(10-20CM)	Polished	Rim	6.7	Indet.
SMR5	SMR5.LCP-17	32	3(20-30CM)	Polished	Body	8.2	Indet.
SMR5	SMR5.LCP-18	33	3(20-30CM)	Polished	Rim	8.5	Indet.
SMR5	SMR5.LCP-19	33	3(20-30CM)	Polished	Rim	8.1	Indet.
SMR5	SMR5.LCP-20	34	1(0-10CM)	Polished	Rim	6.9	Indet.
SMR5	SMR5.LCP-21	35	4(30-40CM)	Polished	Rim	7.8	Indet.
SMR5	SMR5.LCP-22	32	3(20-30CM)	Smoothed	Neck	6.2	Indet.
SMR5	SMR5.LCP-23	32	4(30-40CM)	Smoothed	Rim	6.7	Indet.
SMR5	SMR5.LCP-24	32	4(30-40CM)	Smoothed	Rim	7.4	Indet.
SMR5	SMR5.LCP-25	32	4(30-40CM)	Polished	Neck	6.5	Indet.
SMR5	SMR5.LCP-26	32	3(20-30CM)	Smoothed	Rim	6.7	Indet.
SMR5	SMR5.LCP-27	33	3(20-30CM)	Polished	Rim	6.6	Indet.

SMR5	SMR5.LCP-28	33	4(30-40CM)	Polished	Body	8.3	Indet.
SMR5	SMR5.LCP-29	33	4(30-40CM)	Polished	Rim	8	Indet.
SMR5	SMR5.LCP-30	34	3(20-30CM)	Polished	Body	8.1	Indet.

Indet: Indeterminate

Appendix 11: Molecular and isotopic data from SMR5, southern Chile

Sample ID	Site	Lipid concentration (µg g-1)	Main compounds detected	P/S	PA SRR%	APAA C18E/H	APAA C20/C18	δ13C 16:0 (‰)	δ13C 18:0 (‰)	Δ13C (‰)	ALK CPI
SMR5.ECP-1	SMR5	93.8	SFA ($C_{12:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{18:1}$), DC(C_8-C_{19}), Alk(C_{23}-C_{35}) pri, tmtd, APAA-C_{18}	3.3	N.P	N.P	N.P	-26.5	-28.2	-1.7	4.9
SMR5.ECP-2	SMR5	157.2	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{18:1}$), DC(C_9-C_{10}), OH(C_{26}-C_{28}) pri, APAA-C_{18}	3.1	N.P	N.P	N.P	-21.5	-24.7	-3.2	0.0
SMR5.ECP-3	SMR5	64.9	SFA ($C_{11:0}$ - $C_{34:0}$), UFA ($C_{14:1}$ - $C_{20:1}$), DC(C_8-C_{24}), Alk(C_{24}-C_{35}), OH(C_{16}-C_{32}), pri, tmtd	3.3	N.P	N.P	N.P	-26.9	-28.7	-1.8	24.2
SMR5.ECP-4	SMR5	46.3	SFA ($C_{11:0}$ - $C_{34:0}$), UFA ($C_{14:1}$ - $C_{20:1}$), DC(C_9-C_{26}), Alk(C_{24}-C_{35}), OH(C_{16}-C_{30}), pri, APAA-C_{18}	2.3	N.P	1.6	N.P	-28.5	-29.6	-1.2	18.9
SMR5.ECP-5	SMR5	25.6	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{18:1}$), DC(C_{14}), Alk(C_{24}-C_{35}), pri, tmtd	1.5	N.P	N.P	N.P	-29.0	-29.3	-0.3	4.3
SMR5.ECP-6	SMR5	138.9	SFA ($C_{10:0}$ - $C_{32:0}$), UFA ($C_{16:1}$ - $C_{20:1}$), DC(C_9-C_{18}), Alk(C_{23}-C_{35}), OH(C_{20}-C_{30}), phy, pri, tmtd, APAAs(C_{18}-C_{20}).	1.7	81.2	6.5	0.04	-26.5	-27.9	-1.4	10.4
SMR5.ECP-7	SMR5	28.0	SFA ($C_{12:0}$ - $C_{32:0}$), UFA ($C_{16:1}$ - $C_{20:1}$), DC(C_9-C_{19}), Alk(C_{24}-C_{35}), OH(C_{16}-C_{18}), pri, tmtd	1.6	N.P	N.P	N.P	-27.8	-28.3	-0.5	3.3
SMR5.ECP-8	SMR5	20.3	SFA ($C_{12:0}$ - $C_{32:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC(C_{17}-C_{26}), Alk(C_{24}-C_{35}), pri	2.1	N.P	N.P	N.P	-28.9	-28.9	0.0	6.2
SMR5.ECP-9	SMR5	48.2	SFA ($C_{12:0}$ - $C_{28:0}$), UFA ($C_{16:1}$ - $C_{20:1}$), DC(C_9-C_{14}, C_{24}-C_{26}), Alk(C_{24}-C_{35}), pri, tmtd	1.6	N.P	N.P	N.P	-29.2	-29.4	-0.2	4.0
SMR5.ECP10	SMR5	35.0	SFA ($C_{12:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{18:1}$), DC(C_9), Alk(C_{23}-C_{35}), pri, tmtd, APAA-C_{18}	1.6	N.P	7.1	N.P	-28.7	-28.9	-0.3	2.3
SMR5.ECP-11	SMR5	26.3	SFA ($C_{10:0}$ - $C_{28:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC(C_8-C_{13}), Alk(C_{24}-C_{35}), OH(C_{16}-C_{18}), pri, tmtd	4.3	N.P	N.P	N.P	-26.7	-29.0	-2.3	5.1

SMR5.ECP-12	SMR5	21.7	SFA ($C_{10:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{20:1}$), DC(C_7-C_{13}, C_{19}), Alk(C_{23}-C_{35}), OH(C_{16}-C_{30}), pl_st, pri, tmtd	1.9	N.P	N.P	N.P	-24.2	-26.9	-2.7	6.7
SMR5.ECP-13	SMR5	17.4	SFA ($C_{9:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_8-C_9,, C_{17}- C_{18}), Alk(C_{23}-C_{35}), pl_st, pri, tmtd	3.0	N.P	N.P	N.P				2.2
SMR5.ECP-14	SMR5	20.9	SFA ($C_{10:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{20:1}$), DC(C_7-C_{10}), Alk(C_{23}-C_{35}), pl_st, pri	3.2	N.P	N.P	N.P				0.0
SMR5.ECP-15	SMR5	25.3	SFA ($C_{10:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_8-C_9), Alk(C_{24}-C_{35}), phy, pri, tmtd	1.2	N.P	N.P	N.P	-28.9	-29.9	-1.0	3.5
SMR5.ECP-16	SMR5	15.7	SFA ($C_{10:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_7-C_9), Alk(C_{24}-C_{35}), OH(C_{18}) pri, tmtd	2.1	N.P	N.P	N.P	-27.4	-29.1	-1.7	0.0
SMR5.ECP-17	SMR5	26.1	SFA ($C_{9:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{18:1}$), DC(C_8-C_9), Alk(C_{23}-C_{35}), pri	3.1	N.P	N.P	N.P				0.0
SMR5.ECP-18	SMR5	23.7	SFA ($C_{10:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_8-C_9), Alk(C_{23}-C_{35}), pri, APAA-C_{18}	3.1	N.P	3.0	N.P	-27.8	-28.6	-0.9	2.3
SMR5.ECP-19	SMR5	32.8	SFA ($C_{10:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{20:1}$), DC(C_8-C_{11}), Alk(C_{27}-C_{35}), pri	2.0	N.P	N.P	N.P	-27.8	-30.2	-2.3	3.4
SMR5.ECP-20	SMR5	27.6	SFA ($C_{10:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_8-C_{26}), Alk(C_{27}-C_{35}), pl_st, pri, tmtd	2.3	N.P	N.P	N.P	-27.2	-29.4	-2.2	4.1
SMR5.ECP-21	SMR5	24.8	SFA ($C_{9:0}$ - $C_{28:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC(C_7-C_{17}), pri, tmtd	2.3	N.P	N.P	N.P	-28.2	-29.7	-1.5	0.0
SMR5.ECP-22	SMR5	27.7	SFA ($C_{10:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{18:1}$), DC(C_8-C_{14}), Alk(C_{24}-C_{35}), OH(C_{16}-C_{30}), pri, tmtd	2.6	N.P	N.P	N.P	-27.7	-28.9	-1.2	10.5
SMR5.ECP-23	SMR5	45.5	SFA ($C_{9:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{20:1}$), DC(C_9-C_{18}), Alk(C_{23}-C_{35}), OH(C_{16}-C_{32}), pri, tmtd	1.5	N.P	N.P	N.P	-28.6	-29.8	-1.2	15.5
SMR5.ECP-24	SMR5	18.7	SFA ($C_{12:0}$ - $C_{26:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_9-C_{12}), Alk(C_{27}-C_{35}), pl_st, pri, tmtd	6.9	N.P	N.P	N.P				6.2
SMR5.ECP-25	SMR5	24.5	SFA ($C_{11:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_9,C_{13}), Alk(C_{24}-C_{35}), OH(C_{16}-C_{18}), pri, tmtd	3.8	N.P	N.P	N.P	-27.8	-30.0	-2.2	4.2
SMR5.ECP-26	SMR5	16.7	SFA ($C_{11:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_9,C_{13}), Alk(C_{24}-C_{35}), OH(C_{16}-C_{18}), pri	2.6	N.P	N.P	N.P	-27.2	-29.6	-2.3	1.0
SMR5.ECP-27	SMR5	19.5	SFA ($C_{10:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_8-C_{11}, C_{19}), Alk(C_{24}-C_{35}), OH(C_{16}-C_{30}), pl_st, pri, tmtd	2.4	N.P	N.P	N.P	-24.3	-25.1	-0.8	21.2
SMR5.ECP-28	SMR5	32.6	SFA ($C_{10:0}$ - $C_{34:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_8-C_{11}, C_{19}-C_{26}), Alk(C_{24}-C_{35}), pri	4.5	N.P	N.P	N.P				22.0
SMR5.ECP-29	SMR5	32.9	SFA ($C_{10:0}$ - $C_{34:0}$), UFA ($C_{14:1}$ - $C_{20:1}$), DC(C_8-C_{13}), Alk(C_{23}-C_{35}), OH(C_{16}-C_{34}), phy, pri, tmtd	1.3	43.3	N.P	N.P	-24.3	-29.3	-5.0	14.2
SMR5.ECP-30	SMR5	9.3	SFA ($C_{12:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_9), Alk(C_{26}-C_{35}), pri	3.3	N.P	N.P	N.P				3.1

SMR5.LCP-1	SMR5	40.8	SFA ($C_{8:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC(C_8-C_{26}), Alk(C_{23}-C_{35}), OH(C_{16}), pri, tmtd	1.4	N.P	N.P	N.P	-29.0	-29.6	-0.6	3.5
SMR5.LCP-2	SMR5	34.4	SFA ($C_{8:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_8-C_{26}), Alk(C_{24}-C_{35}), pri, tmtd	2.1	N.P	N.P	N.P	-28.4	-29.8	-1.4	1.7
SMR5.LCP-3	SMR5	46.4	SFA ($C_{8:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_7-C_{28}), Alk(C_{24}-C_{35}), pri, tmtd, APAAs(C_{18}-C_{20})	2.7	N.P	7.2	0.01	-28.5	-29.3	-0.7	2.4
SMR5.LCP-4	SMR5	24.8	SFA ($C_{8:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_8-C_{26}), Alk(C_{23}-C_{35}), pri, tmtd	2.0	N.P	N.P	N.P	-28.9	-29.1	-0.2	0.9
SMR5.LCP-5	SMR5	43.3	SFA ($C_{8:0}$ - $C_{32:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC(C_8-C_{26}), Alk(C_{23}-C_{35}), OH(C_{16}-C_{30}), pri, tmtd	3.1	N.P	N.P	N.P	-29.5	-29.4	0.2	6.6
SMR5.LCP-6	SMR5	36.6	SFA ($C_{8:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_8-C_{28}), Alk(C_{23}-C_{35}), OH(C_{16}-C_{30}), pri, tmtd	1.7	N.P	N.P	N.P	-28.4	-29.7	-1.3	7.3
SMR5.LCP-7	SMR5	40.7	SFA ($C_{8:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{18:1}$), DC(C_7-C_{26}), Alk(C_{24}-C_{35}), pri, tmtd, APAAs(C_{18}-C_{20})	3.2	N.P	7.1	0.08	-28.5	-28.1	0.3	2.8
SMR5.LCP-8	SMR5	42.8	SFA ($C_{8:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_8-C_{28}), Alk(C_{23}-C_{35}), pri, tmtd	2.4	N.P	N.P	N.P	-29.3	-30.5	-1.2	0.8
SMR5.LCP-9	SMR5	26.4	SFA ($C_{10:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_8-C_{28}), Alk(C_{23}-C_{35}), OH(C_{16}-C_{18}), pri, tmtd	1.8	N.P	N.P	N.P	-28.2	-29.7	-1.4	0.9
SMR5.LCP-10	SMR5	53.9	SFA ($C_{8:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC(C_7-C_{28}), Alk(C_{24}-C_{35}), OH(C_{16}-C_{18}), pri, tmtd	4.1	N.P	N.P	N.P	-26.5	-28.2	-1.8	3.1
SMR5.LCP-11	SMR5	41.1	SFA ($C_{8:0}$ - $C_{28:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC(C_9-C_{28}), Alk(C_{23}-C_{33}), OH(C_{16}-C_{18}), pri, tmtd	3.1	N.P	N.P	N.P	-27.1	-28.3	-1.2	1.7
SMR5.LCP-12	SMR5	29.3	SFA ($C_{8:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_9-C_{26}), Alk(C_{23}-C_{33}), OH(C_{16}-C_{18}), pri, tmtd	1.8	N.P	N.P	N.P	-28.9	-29.5	-0.6	6.2
SMR5.LCP-13	SMR5	23.2	SFA ($C_{8:0}$ - $C_{27:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC(C_9-C_{26}), Alk(C_{24}-C_{31}), pri, tmtd	1.9	N.P	N.P	N.P	-29.1	-28.6	0.5	1.4
SMR5.LCP-14	SMR5	49.8	SFA ($C_{8:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_8-C_{28}), Alk(C_{24}-C_{33}), pri, tmtd, APAAs (C_{18}-C_{20})	2.5	44.7	6.1	0.01	-27.8	-28.9	-1.1	4.4
SMR5.LCP-15	SMR5	18.5	SFA ($C_{8:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC(C_9-C_{28}), Alk(C_{23}-C_{33}), pri, tmtd, APAAs (C_{18}-C_{20})	1.9	N.P	N.P	N.P	-28.7	-29.4	-0.7	3.8
SMR5.LCP-16	SMR5	306.3	SFA ($C_{8:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{18:1}$), DC(C_7-C_{22}), Alk(C_{24}-C_{33}), OH(C_{16}-C_{32}), pri, tmtd	4.5	69.3	N.P	N.P	-18.2	-22.6	-4.4	16.6
SMR5.LCP-17	SMR5	38.1	SFA ($C_{8:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_8-C_{22}), Alk(C_{24}-C_{33}), OH(C_{16}-C_{32}), pri, tmtd	1.6	N.P	N.P	N.P	-28.8	-28.9	-0.1	1.8
SMR5.LCP-18	SMR5	35.1	SFA ($C_{8:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC(C_8-C_{28}), Alk(C_{23}-C_{33}), OH(C_{16}-C_{18}), pri, tmtd	2.8	N.P	N.P	N.P	-28.7	-28.8	-0.2	31.2
SMR5.LCP-19	SMR5	42.4	SFA ($C_{8:0}$ - $C_{28:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC(C_9-C_{26}), Alk(C_{23}-C_{33}), OH(C_{16}-C_{18}), pri, tmtd	3.2	N.P	N.P	N.P	-30.0	-29.9	0.1	3.2
SMR5.LCP-20	SMR5	33.9	SFA ($C_{8:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{18:1}$), DC(C_8-C_{28}), Alk(C_{23}-C_{33}), OH(C_{16}-C_{28}), pri, tmtd	3.3	N.P	N.P	N.P	-27.0	-28.7	-1.7	8.6
SMR5.LCP-21	SMR5	25.1	SFA ($C_{8:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_8-C_{26}), Alk(C_{23}-C_{31}), OH(C_{16}-C_{28}), pri, tmtd	2.1	N.P	N.P	N.P	-27.3	-29.3	-1.9	1.3

SMR5.LCP-22	SMR5	29.1	SFA ($C_{8:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_8-C_{26}), Alk(C_{23}-C_{31}), OH(C_{16}-C_{28}), pri, tmtd	1.9	N.P	N.P	N.P	-27.1	-29.4	-2.3	4.8
SMR5.LCP-23	SMR5	483.1	SFA ($C_{8:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_6-C_{23}), Alk(C_{23}-C_{31}), OH(C_{16}-C_{28}), pri, tmtd	1.9	N.P	N.P	N.P	-24.8	-28.2	-3.5	
SMR5.LCP-24	SMR5	25.0	SFA ($C_{8:0}$ - $C_{26:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC(C_7-C_{22}), Alk(C_{24}-C_{29}), pri, tmtd	3.0	N.P	N.P	N.P	-28.2	-29.1	-0.9	1.0
SMR5.LCP-25	SMR5	24.5	SFA ($C_{8:0}$ - $C_{28:0}$), UFA ($C_{16:1}$ - $C_{24:1}$), DC(C_8-C_{22}), Alk(C_{23}-C_{33}), OH(C_{16}-C_{18}), pri, tmtd	1.7	N.P	N.P	N.P	-26.2	-28.4	-2.2	3.1
SMR5.LCP-26	SMR5	28.0	SFA ($C_{8:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_8-C_{26}), Alk(C_{23}-C_{33}), OH(C_{16}-C_{18}), pri, tmtd	1.8	N.P	N.P	N.P	-28.5	-29.7	-1.2	2.4
SMR5.LCP-27	SMR5	24.8	SFA ($C_{8:0}$ - $C_{26:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_8-C_{19}), Alk(C_{23}-C_{33}), OH(C_{16}-C_{20}), pri, tmtd	3.1	N.P	N.P	N.P	-27.2	-29.4	-2.2	2.1
SMR5.LCP-28	SMR5	13.6	SFA ($C_{8:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{18:1}$), DC(C_8-C_{26}), Alk(C_{23}-C_{33}), OH(C_{16}-C_{20}), pri, tmtd	1.4	N.P	N.P	N.P	-28.9	-29.5	-0.7	3.2
SMR5.LCP-29	SMR5	107.9	SFA ($C_{8:0}$ - $C_{26:0}$), UFA ($C_{16:1}$ - $C_{18:1}$), DC(C_7-C_{24}), Alk(C_{24}-C_{27}), pri	3.8	N.P	N.P	N.P	-26.5	-27.7	-1.3	0
SMR5.LCP-30	SMR5	24.2	SFA ($C_{8:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_7-C_{26}), Alk(C_{24}-C_{33}), OH(C_{16}-C_{20}), pri, tmtd	2.6	N.P	N.P	N.P	-27.0	-28.6	-1.6	3.8

Appendix 12: Pottery from Iluga Túmulos, northern Chile

Site	Sample	Associated structure (E)/Tumuli (T)	Level	Type of container	Subtype	Period
I.Túmulos	IT_66	T-015	Superficial	Jar	San Miguel	Late-Intermediate-Late
I.Túmulos	IT_111	T-079	Superficial	Jar	San Miguel	Late-Intermediate-Late
I.Túmulos	IT_38	T-028	Superficial	Jar	Pocoma y Gentilar	Late-Intermediate-Late
I.Túmulos	IT_27	T-045	Superficial	Jar	Isluga black over red	Late-Intermediate-Late
I.Túmulos	IT_123	T-057	Superficial	Jar	Isluga black over red	Late-Intermediate-Late
I.Túmulos	IT_25	T-045	Superficial	Aryballo	Inka polychrome	Late/Inka
I.Túmulos	IT_53	E-031	Superficial	Jar	Inka polychrome	Late/Inka
I.Túmulos	IT_85	E-019	Superficial	Aryballo	Inka polychrome	Late/Inka
I.Túmulos	IT_214	E:19	Superficial	Aryballo	Inka polychrome	Late/Inka
I.Túmulos	IT_225	E:26	Superficial	Jar	Inka polychrome	Late/Inka
I.Túmulos	IT_6	T-057	Superficial	Jar	Inka Altiplánico	Late/Inka
I.Túmulos	IT_233	T-044	Superficial	Jar	Inka Altiplánico	Late/Inka
I.Túmulos	IT_229	T-079	Superficial	Jar	Inka Altiplánico	Late/Inka

I.Túmulos	IT_224	E-026	Superficial	Aryballo	Inka polychrome	Late/Inka
I.Túmulos	IT_7	T-057	Superficial	Bowl	Saxamar Inka Pacajes	Late/Inka
I.Túmulos	IT_145	T:44	Superficial	Bowl	Inka polychrome	Late/Inka
I.Túmulos	IT_1	T-079	Superficial	Bowl	Chilpe	Late-Intermediate-Late
I.Túmulos	IT_73	T-015	Superficial	Bowl	Chilpe	Late-Intermediate-Late
I.Túmulos	IT_80	T-015	Superficial	Bowl	Aiquina	Late-Intermediate-Late
I.Túmulos	IT_202	T:8	Superficial	Mug	Quillagua red polished	Late Formative
I.Túmulos	IT_204	T:8	Superficial	Mug	Caserones black polished	Late Formative

Appendix 13: Molecular and isotopic data from Iluga Túmulos, northern Chile

Sample ID	Site	Exc. unit	Exc. Level	Lipid concentration (µg g-1)	Main compounds detected	P/S	PA SRR %	APAA C18E/H	δ13C 16:0 (‰)	δ13C 18:0 (‰)	Δ13C (‰)	ALK CPI
IT_66	I.Túmulos	T-015	Superficial	42.8	SFA($C_{8:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_6-C_{18}, Akl(C_{23}-C_{35}), OH(C_{16}-C_{32}), pri, tmtd	1.2	N.P	N.P	-28.8	-29.4	-0.5	4.7
IT_111	I.Túmulos	T-079	Superficial	69.3	SFA($C_{12:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_7-C_{22}), Akl(C_{26}-C_{35}), OH(C_{16}-C_{32}), pri, tmtd	1.3	N.P	N.P	-15.0	-10.4	4.4	7.0
IT_38	I.Túmulos	T-028	Superficial	218.0	SFA($C_{8:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_6-C_{22}), Akl(C_{26}-C_{27}), OH(C_{16}-C_{32}), pl_st, pri, tmtd	3.3	N.P	N.P	-14.6	-18.0	-3.5	1.0
IT_27	I.Túmulos	T-045	Superficial	394.7	SFA($C_{11:0}$ - $C_{30:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), DC(C_7-C_{19}), Akl(C_{27}-C_{33}), OH(C_{16}-C_{32}), pl_st, phy, pri, tmtd	3.5	55.0	N.P	-15.8	-20.4	-4.6	18.7
IT_123	I.Túmulos	T-057	Superficial	65.4	SFA($C_{12:0}$ - $C_{32:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), DC(C_8-C_{16}), Akl(C_{24}-C_{35}), OH(C_{16}-C_{32}), pri, tmtd	3.5	N.P	N.P	-25.6	-26.7	-1.2	7.7
IT_25	I.Túmulos	T-045	Superficial	39.0	SFA($C_{8:0}$ - $C_{32:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_8-C_{16}), Akl(C_{24}-C_{35}), OH(C_{16}-C_{32})	1.5	N.P	N.P	-28.4	-29.4	-0.9	2.2

IT_53	I.Túmulos	E-031	Superficial	29.7	SFA($C_{12:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_9-C_{18}), Akl(C_{24}-C_{35}), OH(C_{16}-C_{32}), pri	2.2	N.P	N.P	-27.9	-29.6	-1.6	0.9
IT_85	I.Túmulos	E-019	Superficial	302.3	SFA($C_{8:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_6-C_{21}), Akl(C_{27}-C_{33}), OH(C_{16}-C_{32}), phy, pri, tmtd	1.6	50.9	N.P	-24.6	-25.9	-1.2	7.9
IT_214	I.Túmulos	E:19	Superficial	2117.2	SFA($C_{8:0}$ - $C_{28:0}$), UFA ($C_{16:1}$ - $C_{20:1}$), DC(C_5-C_{20}), OH(C_{16}-C_{32}), pl_st, pri	3.7	N.P	N.P	-14.4	-17.1	-2.7	0.0
IT_225	I.Túmulos	E:26	Superficial	13.1	SFA($C_{11:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_8-C_{18}), Alk (C_{26}-C_{33}), OH(C_{16}-C_{32}), pri, tmtd	1.4	N.P	N.P	-27.8	-28.1	-0.3	0.7
IT_6	I.Túmulos	T-057	Superficial	64.5	SFA($C_{11:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_7-C_{22}), Alk (C_{24}-C_{33}), OH(C_{16}-C_{32}), pri	2.0	N.P	N.P	-24.5	-24.4	-0.1	3.6
IT_233	I.Túmulos	T-044	Superficial	30.3	SFA($C_{8:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_6-C_{22}), Alk (C_{26}-C_{35}), OH(C_{16}-C_{32}), phy, pri, tmtd	1.5	59.1	N.P	-26.3	-25.2	1.1	10.6
IT_229	I.Túmulos	T-079	Superficial	20.1	SFA($C_{11:0}$ - $C_{28:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), DC(C_9-C_{13}), Alk (C_{23}-C_{35}), OH(C_{16}-C_{32}), K_{31}, K_{35}, pri	1.7	N.P	N.P	-27.0	-28.5	-1.4	2.0
IT_224	I.Túmulos	E-026	Superficial	50.7	SFA($C_{11:0}$ - $C_{28:0}$), UFA ($C_{18:1}$ - $C_{22:1}$), DC(C_7-C_{18}), Alk (C_{26}-C_{31}), OH(C_{16}-C_{32}), pl_st, pri, tmtd	6.3	N.P	N.P	-15.7	-18.2	-2.4	0.8
IT_7	I.Túmulos	T-057	Superficial	114.0	SFA($C_{12:0}$ - $C_{28:0}$), UFA ($C_{16:1}$ - $C_{22:1}$), DC(C_7-C_{18}), Alk (C_{24}-C_{35}), OH(C_{16}-C_{32}), phy, pri, tmtd	1.2	47.9	N.P	-25.0	-28.1	-3.2	4.1
IT_145	I.Túmulos	T:44	Superficial	128.0	SFA($C_{8:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_8-C_{19}), Alk (C_{27}-C_{33}), OH(C_{16}-C_{32}), phy, pri, tmtd, APAA-C_{18}	1.8	58.1	6.3	-21.1	-26.1	-5.1	17.7
IT_1	I.Túmulos	T-079	Superficial	120.3	SFA($C_{10:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_7-C_{20}), Alk (C_{24}-C_{33}), OH(C_{16}-C_{32}), phy, pri	0.7	57.3	N.P	-23.1	-27.3	-4.1	1.5
IT_73	I.Túmulos	T-015	Superficial	30.5	SFA($C_{11:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_8-C_{16}), Alk (C_{24}-C_{30}), OH(C_{16}-C_{32})	1.8	N.P	N.P	-27.9	-29.4	-1.6	1.2
IT_80	I.Túmulos	T-015	Superficial	97.3	SFA($C_{10:0}$ - $C_{30:0}$), UFA ($C_{14:1}$ - $C_{22:1}$), DC(C_8-C_{18}), Alk (C_{24}-C_{35}), OH(C_{16}-C_{32}), phy, pri, tmtd	3.0	83.7	N.P	-24.3	-24.7	-0.4	13.8

IT_202	I.Túmulos	T:8	Superficial	19.6	SFA($C_{12:0}$ - $C_{26:0}$), UFA ($C_{16:1}$ - $C_{20:1}$), DC(C_8-C_{16}), Alk (C_{26}-C_{35}), OH(C_{16}-C_{28}), pri, tmtd	1.2	N.P	N.P	-30.0	-28.8	1.0	1.0
IT_204	I.Túmulos	T:8	Superficial	65.2	SFA($C_{11:0}$ - $C_{28:0}$), UFA ($C_{14:1}$ - $C_{24:1}$), DC(C_8-C_{17}), Alk (C_{23}-C_{35}), OH(C_{16}-C_{32}), pri	3.1	N.P	N.P	-29.6	-28.3	1.3	6.5

Appendix 14: Radiocarbon dates from the Araucanía between the Middle Archaic Period and the beginning of the Colonial Period (8000 – 400 cal BP)

Zone	Locality	Period	Laboratory code	Method	Material	Year of analysis	14C date	Sigma	Median Probability cal yrs BP	One sigma (σ) BP	Two sigma (σ) BP
Valley	Puren-Lumaco	Mid.Archaic	Beta 191662	14C	Charcoal	2007	6140	40	6976	7152 to 7127	7157 to 7092
Coast	Concepción	Mid.Archaic	Beta 193345	14C AMS	Charcoal	2004	4180	40	4684	4816 to 4779	4827 to 4567
Coast	Concepción	Mid.Archaic	IVIC 844	14C Conv.	Charcoal	1973	3870	80	4239	4403 to 4368	4505 to 4493
Coast	Concepción	Late.Archaic	IVIC 845	14C Conv.	Charcoal	1973	3330	80	3524	3618 to 3601	3817 to 3799
Coast	Isla Mocha	Late.Archaic	Gd 4885	14C Conv.	Charcoal	1993	3310	90	3503	3614 to 3608	3817 to 3798
Coast	Isla Mocha	Late.Archaic	Beta 57810 / CAMS 5348	14C AMS	Charcoal	1993	3280	60	3459	3558 to 3529	3634 to 3601
Coast	Isla Mocha	Late.Archaic	Beta 71647 / CAMS 13062	14C AMS	Charcoal	1996	3220	50	3398	3452 to 3357	3557 to 3555
Valley	Puren-Lumaco	Late.Archaic	AA 64648	14C AMS	Charcoal	2007	2735	55	2814	2849 to 2756	2948 to 2736
Valley	Puren-Lumaco	Late.Archaic	AA 64650	14C AMS	Charcoal	2007	2720	40	2797	2846 to 2815	2874 to 2738
Valley	Puren-Lumaco	Late.Archaic	AA 64658	14C AMS	Charcoal	2007	2530	40	2576	2717 to 2673	2727 to 2409
Valley	Puren-Lumaco	Late.Archaic	Beta 167557	14C	Charcoal	2007	2520	40	2569	2712 to 2666	2723 to 2407

Valley	Puren-Lumaco	Late.Archaic	AA 64649	14C AMS	Charcoal	2007	2200	40	2137	2300 to 2255	2318 to 2223
Cordillera	Galletué-Liucura	Late.Archaic	Beta 273150	14C	-	2011	2190	40	2124	2297 to 2263	2312 to 2227
Valley	Puren-Lumaco	Late.Archaic	AA 64656	14C AMS	Charcoal	2007	2175	40	2106	2289 to 2273	2305 to 2234
Cordillera	Villarrica	Late.Archaic	Beta 253960	14C AMS	Charcoal	2009	2110	40	2038	2091 to 1999	2284 to 2280
Valley	Puren-Lumaco	Late.Archaic	AA 64978	14C AMS	Charcoal	2007	2080	55	1999	2083 to 2072	2131 to 1876
Cordillera	Villarrica	Late.Archaic	Beta 273148	14C	-	2011	2080	40	1999	2053 to 2025	2095 to 1918
Valley	Galvarino-Lautaro	Late.Archaic	Beta 4709	14C Conv.	Charcoal	1985	2030	70	1943	2012 to 1868	2103 to 1807
Valley	Puren-Lumaco	Late.Archaic	AA 64646	14C AMS	Charcoal	2007	2010	40	1922	1994 to 1952	2004 to 1831
Coast	Puerto Saavedra	Late.Archaic	GrN 16322	14C	Charcoal	1989	2000	80	1907	2003 to 1826	2093 to 1722
Valley	Puren-Lumaco	Late.Archaic	AA 64641	14C AMS	Charcoal	2007	1930	40	1826	1883 to 1811	1925 to 1724
Coast	Puerto Saavedra	Late.Archaic	GrN 16323	14C	Charcoal	1989	1900	70	1791	1876 to 1848	1991 to 1967
Valley	Puren-Lumaco	Late.Archaic	AA 64977	14C AMS	Charcoal	2007	1850	35	1735	1814 to 1770	1831 to 1693
Valley	Puren-Lumaco	Late.Archaic	AA 64652	14C AMS	Charcoal	2007	1845	40	1729	1813 to 1770	1829 to 1689
Cordillera	Melipeuco	Late.Archaic	Beta 253959	14C AMS	Charcoal	2009	1830	40	1708	1808 to 1800	1824 to 1764
Valley	Puren-Lumaco	Late.Archaic	AA 13780	14C AMS	Charcoal	2007	1810	90	1685	1815 to 1769	1920 to 1913
Valley	Puren-Lumaco	Late.Archaic	AA 64647	14C AMS	Charcoal	2007	1800	40	1659	1707 to 1610	1810 to 1797
Valley	Puren-Lumaco	Late.Archaic	AA 64642	14C AMS	Charcoal	2007	1780	35	1648	1699 to 1645	1724 to 1564
Valley	Puren-Lumaco	Late.Archaic	AA 64979	14C AMS	Charcoal	2007	1780	40	1648	1700 to 1610	1731 to 1559
Coast	Isla Mocha	Late.Archaic	Beta 62523	14C	Charcoal	1999	1760	80	1631	1721 to 1716	1824 to 1764
Valley	Puren-Lumaco	Late.Archaic	AA 64655	14C AMS	Charcoal	2007	1740	35	1606	1694 to 1659	1699 to 1643
Valley	Puren-Lumaco	Late.Archaic	AA 64645	14C AMS	Charcoal	2007	1735	40	1601	1693 to 1660	1702 to 1528
Valley	Puren-Lumaco	Late.Archaic	AA 64653	14C AMS	Charcoal	2007	1730	40	1595	1692 to 1677	1702 to 1520
Coast	Isla Santa María	Late.Archaic	Beta 269489	14C AMS	Charcoal	2009	1730	40	1595	1692 to 1677	1702 to 1520

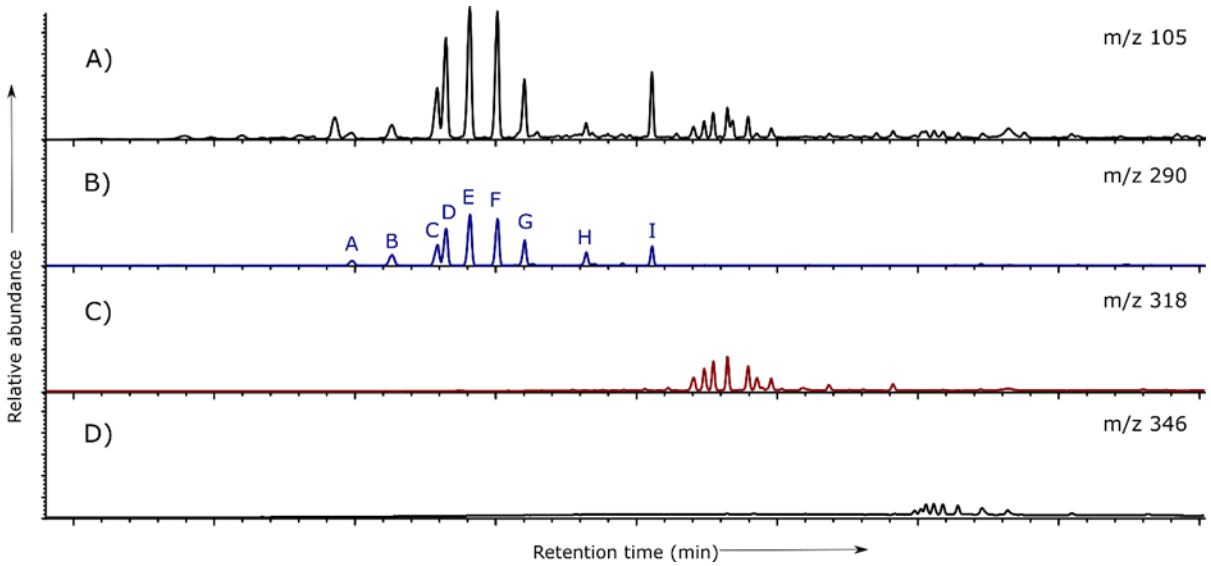
Valley	Puren-Lumaco	Late.Archaic	AA 64980	14C AMS	Charcoal	2007	1715	60	1578	1697 to 1651	1706 to 1418
Valley	Puren-Lumaco	ECP	AA 64651	14C AMS	Charcoal	2007	1680	40	1535	1586 to 1509	1692 to 1679
Valley	Puren-Lumaco	ECP	AA 64657	14C AMS	Charcoal	2007	1615	40	1463	1519 to 1419	1558 to 1553
Valley	Puren-Lumaco	ECP	AA 13772	14C AMS	Charcoal	2007	1315	50	1201	1273 to 1177	1286 to 1175
Valley	Galvarino-Lautaro	ECP	Gif 4984	14C Conv.	Charcoal	1985	1290	80	1154	1268 to 1204	1299 to 1046
Valley	Temuco	ECP	A 12240	14C Conv.	-	2003	1110	60	975	1055 to 1013	1175 to 1166
Valley	Puren-Lumaco	ECP	Beta 168999	14C	Charcoal	2007	1100	80	963	1059 to 905	1176 to 1134
Valley	Puren-Lumaco	ECP	Beta 68999	14C	Charcoal	2007	1100	80	963	1059 to 905	1176 to 1134
Coast	Isla Mocha	ECP	AA 89420	14C AMS	Charcoal	2011	1105	36	962	1046 to 1033	1056 to 921
Cordillera	Melipeuco	ECP	Beta 241264	14C AMS	Charcoal	2009	1100	40	959	1046 to 1032	1059 to 906
Coast	Isla Mocha	ECP	AA 89415	14C AMS	Charcoal	2011	1096	37	954	1045 to 1033	1057 to 906
Cordillera	Villarrica	ECP	Beta 273151	14C	Maize seed	2011	1080	40	943	1046 to 1032	1053 to 1018
Cordillera	Colico-Caburgua	ECP	Beta 227246	14C AMS	Charcoal	2009	1070	40	934	960 to 905	1051 to 1024
Valley	Temuco	ECP	Beta 241265	14C AMS	Charcoal	2011	1060	40	924	958 to 905	1046 to 1031
Coast	Isla Mocha	LCP	AA 89419	14C AMS	Charcoal	2011	964	36	841	904 to 862	921 to 764
Coast	Isla Mocha	LCP	Beta 69935	14C Conv.	Charcoal	1997	910	70	783	901 to 866	918 to 677
Coast	Isla Mocha	LCP	Beta 114462	14C Conv.	Charcoal	2004	900	80	777	900 to 868	924 to 666
Coast	Isla Santa María	LCP	Beta 237530	14C AMS	Charcoal	2008	910	40	771	894 to 893	904 to 861
Coast	Isla Mocha	LCP	Beta 181243	14C Conv.	Charcoal	2005	900	60	770	898 to 889	905 to 675
Coast	Isla Mocha	LCP	Beta 62819 / CAMS 14037	14C AMS	Charcoal	1997	890	70	765	895 to 892	914 to 669
Valley	Temuco	LCP	Beta 296450	14C	Charcoal	2012	900	30	759	790 to 779	898 to 888
Coast	Isla Mocha	LCP	AA 89416	14C AMS	Charcoal	2011	895	38	757	794 to 725	900 to 868

Coast	Isla Mocha	LCP	Beta 137970	14C Conv.	Charcoal	2004	880	70	756	876 to 874	914 to 665
Coast	Isla Mocha	LCP	Beta 162421	14C Conv.	Charcoal	2005	870	60	742	789 to 781	904 to 861
Coast	Isla Mocha	LCP	Gd 6428	14C Conv.	Charcoal	1993	840	70	721	786 to 784	903 to 864
Coast	Isla Mocha	LCP	AA 89417	14C AMS	Charcoal	2011	825	36	704	724 to 678	764 to 750
Coast	Isla Mocha	LCP	AA 89424	14C AMS	Charcoal	2011	826	27	703	724 to 696	734 to 668
Coast	Isla Mocha	LCP	AA 89418	14C AMS	Charcoal	2011	759	38	661	714 to 708	724 to 694
Coast	Isla Mocha	LCP	Beta 132089	14C Conv.	Charcoal	2004	720	80	631	675 to 624	735 to 530
Coast	Isla Mocha	LCP	Gd 5902	14C Conv.	Charcoal	1993	710	50	617	664 to 630	712 to 710
Coast	Isla Mocha	LCP	Beta 95086	14C Conv.	Charcoal	2004	700	50	611	660 to 629	674 to 550
Coast	Isla Mocha	LCP	Beta 79918	14C Conv.	Charcoal	1995	680	80	608	656 to 557	722 to 700
Valley	Temuco	LCP	Gif 3841	14C Conv.	Charcoal	1978	670	80	605	654 to 554	719 to 704
Valley	Puren-Lumaco	LCP	AA 64654	14C AMS	Charcoal	2007	660	40	604	646 to 642	657 to 546
Coast	Puerto Saavedra	LCP	Beta 167596	14C	Organic Sediment	2007	670	40	605	649 to 623	659 to 550
Cordillera	Melipeuco	LCP	Beta 241263	14C AMS	Charcoal	2009	670	40	605	649 to 623	659 to 550
Coast	Isla Mocha	LCP	Beta 75239	14C Conv.	Charcoal	2005	640	50	600	636 to 590	657 to 528
Coast	Quidico-Yani	LCP	NZA 26115	14C AMS	Gallus gallus bone	2007	622	35	600	628 to 600	648 to 588
Coast	Isla Mocha	LCP	Gd 6431	14C Conv.	Charcoal	1993	640	90	595	653 to 582	722 to 700
Coast	Isla Mocha	LCP	Beta 132088	14C Conv.	Charcoal	2004	620	60	589	633 to 593	655 to 511
Valley	Puren-Lumaco	LCP	Beta 191668	14C	Charcoal	2007	600	20	550	621 to 616	626 to 603
Coast	Isla Santa María	LCP	Beta 237532	14C AMS	Chenopodium quinoa seed	2008	570	40	537	552 to 513	627 to 602

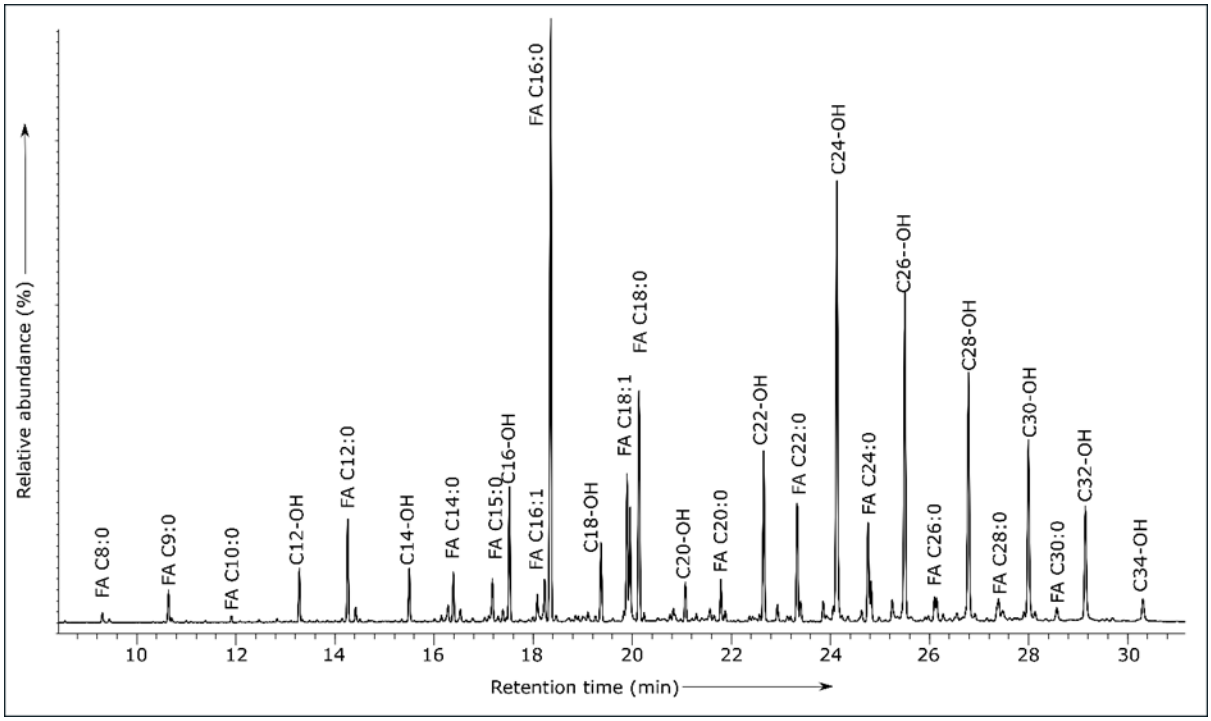
Valley	Puren-Lumaco	LCP	Beta 169771	14C	Charcoal	2007	560	50	535	553 to 506	631 to 596
Coast	Isla Mocha	LCP	Gd 5901	14C Conv.	Charcoal	1993	560	40	533	548 to 512	625 to 605
Coast	Isla Mocha	LCP	Beta 79917	14C Conv.	Charcoal	1995	550	70	532	624 to 608	653 to 582
Valley	Puren-Lumaco	LCP	Beta 167559_Rapahuekuel	14C	Charcoal	2007	550	40	529	544 to 510	623 to 610
Coast	Isla Mocha	LCP	Gd 7144	14C Conv.	Charcoal	1994	530	60	521	552 to 494	631 to 595
Coast	Isla Mocha	LCP	Gd 6429	14C Conv.	Charcoal	1993	530	80	517	626 to 604	651 to 585
Coast	Isla Mocha	LCP	AA 89422	14C AMS	Charcoal	2011	519	37	517	530 to 500	552 to 490
Coast	Quidico-Yani	LCP	NZA 28271	14C AMS	Gallus gallus bone	2007	510	30	513	523 to 499	541 to 493
Coast	Quidico-Yani	LCP	NZA 28272	14C AMS	Gallus gallus bone	2007	506	30	511	521 to 498	542 to 491
Coast	Isla Mocha	LCP	Beta 95085	14C Conv.	Charcoal	2004	510	60	509	549 to 488	628 to 601
Coast	Isla Mocha	LCP	Gd 7174	14C Conv.	Charcoal	1994	500	40	508	529 to 494	547 to 452
Coast	Isla Mocha	LCP	Beta 57811	14C Conv.	Charcoal	1994	500	50	506	540 to 489	620 to 619
Valley	Temuco	LCP	NO_NAME_8	14C	Charcoal	2005	505	65	504	549 to 465	627 to 601
Coast	Isla Santa María	LCP	Beta 237529	14C AMS	Charcoal	2008	480	40	497	525 to 487	541 to 446
Coast	Isla Mocha	LCP	Gd 7152	14C Conv.	Charcoal	1994	450	50	462	509 to 446	530 to 434
Valley	Puren-Lumaco	LCP	AA 203868	14C AMS	Zea mays	2007	430	40	453	498 to 446	510 to 435
Coast	Isla Mocha	LCP	AA 89421	14C AMS	Charcoal	2011	408	37	422	493 to 442	498 to 433
Coast	Isla Mocha	LCP	Beta 75240	14C Conv.	Charcoal	1997	420	80	415	501 to 438	540 to 288
Valley	Angol	LCP	UGAMS 27385	14C AMS	Zea mays	2016	660	20	606	632 to 622	650 to 587
Valley	Angol	ECP	UGAMS 27386	14C AMS	Phaseolus vulgars	2016	1300	25	1214	1263 to 1209	1268 to 1201
Valley	Angol	ECP	UGAMS 27387	14C AMS	Camelid	2016	1410	25	1288	1303 to 1276	1310 to 1267

Valley	Angol	ECP	AA 110999	14C AMS	Human bone	2018	972	23	853	904 to 863	919 to 791
Valley	Angol	ECP	AA 111538	14C AMS	Camelid bone	2018	1527	24	1360	1402 to 1392	1408 to 1313
Valley	Angol	ECP	AA 111539	14C AMS	Canid bone	2018	1547	24	1380	1409 to 1356	1467 to 1467
Valley	Temuco	ECP	UGAMS 23784	14C AMS	Human tooth	2020	930	25	783	878 to 872	902 to 865
Valley	Temuco	ECP	UGAMS 23782	14C AMS	Human tooth	2020	920	25	773	794 to 762	900 to 867
Valley	Temuco	ECP	UGAMS 23783	14C AMS	Humanto oth	2020	910	25	765	791 to 779	898 to 888

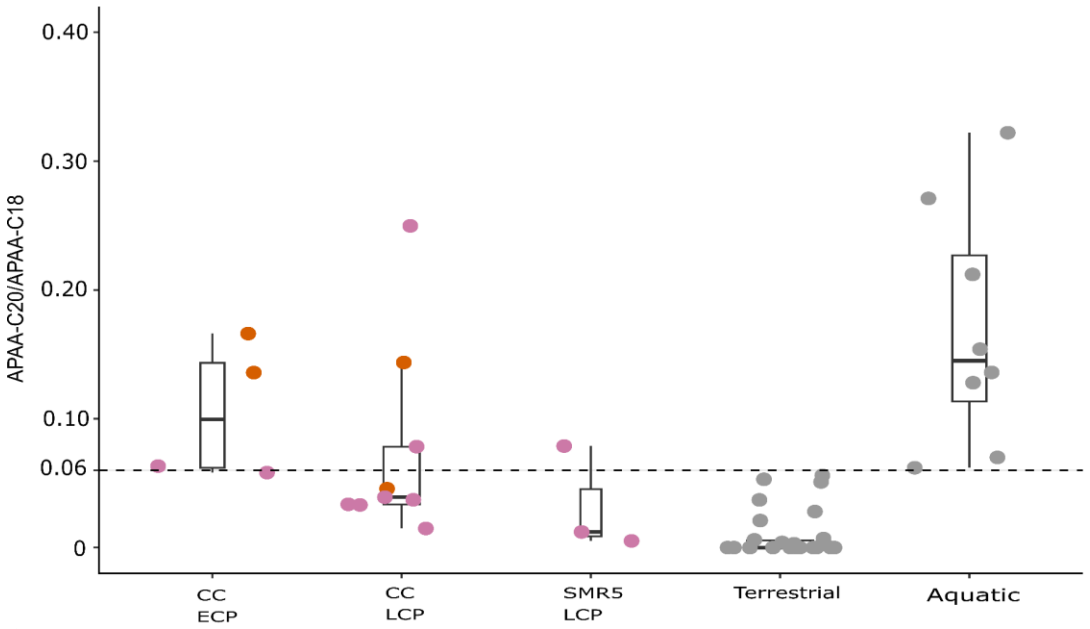
Appendix 15: Figures



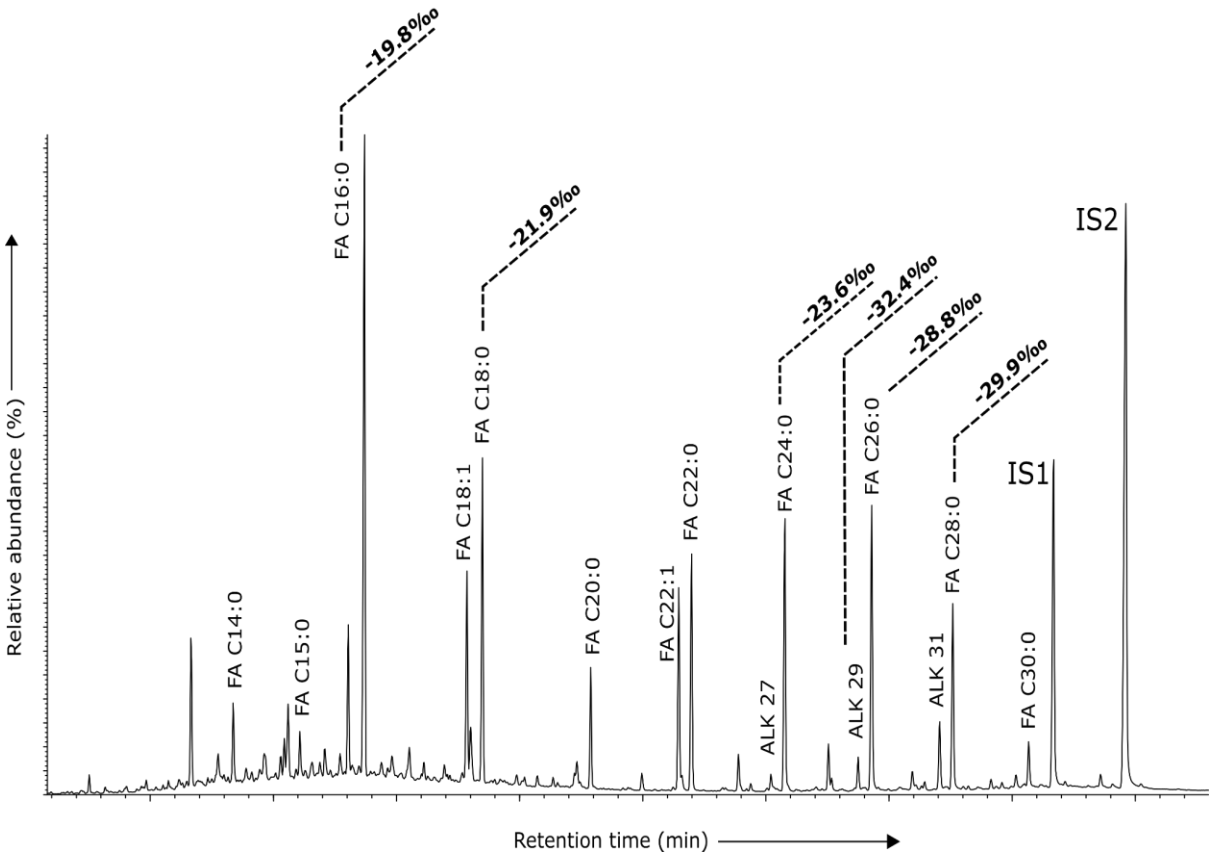
Appendix Figure 15.1: Partial chromatogram of sample P25-1.10 showing APAAs C_{18}, C_{20}, C_{22}. APAA-C_{18} isomers are labelled A-I. Figure by the author.



Appendix Figure 15.2: Partial ion chromatogram (*m/z* 75) of the total lipid extract (solvent extract) of sample P25-1.5 exhibiting a distribution of fatty alcohols and fatty acids. Figure by the author.



Appendix Figure 15.3: APAA C_{20}/C_{18} from several samples from Cueva de losCatalanes and SMR5 sites. The dashed line indicates a set threshold to differentiate between terrestrial (<0.06) and aquatic (>0.06) organisms (Bondetti et al., 2020). Figure by the author.



Appendix Figure 15.4: Chromatogram of sample C.C.LCP-22 from Cueva de los Catalanes showing the carbon isotope values of the main alkanoic acids and several very long chain fatty acids and alkanes. The data indicates different carbon isotopic origin for several compounds from the same sample, possibly indicating the mixing of different products with different carbon isotope origin. Figure by the author.

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