Camelids on the Coast? Investigating Trade and Early Camelid Herding through Stable Isotope Analysis of Formative to Late Intermediate Period Textiles from the Atacama Desert, Chile

A Thesis Submitted to the Committee of Graduate Studies in Partial Fulfillment of the Requirements for the Degree of Master of Arts in the Faculty of Arts and Science.

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### **ABSTRACT**

<span id="page-1-0"></span>Camelids on the Coast? Investigating Trade and Early Camelid Herding through Stable Isotope Analysis of Formative to Late Intermediate Period Textiles from the Atacama Desert, Chile

Tessa Grogan

This research presents stable carbon and nitrogen isotope data of camelid fiber (n=238) from the coast of Arica, the Lluta Valley, and Azapa Valley – all located in the hyper-arid core of the Atacama Desert in northern Chile. Samples included are attributed to the Formative (1000 BCE– 200 CE), Middle (200 CE – 1000CE), and Late Intermediate (1000 CE – 1450 CE) periods. The principal aim of this research was to determine whether camelid fiber uncovered at archaeological sites on the coast were likely obtained from locally managed camelid herds, or whether fiber was traded from the highlands. The majority of samples are consistent with a highland origin; however, a small group of samples is consistent with having come from animals herded outside the highlands, possibly locally in the coastal river valleys. There was also an increase in fiber  $\delta$ <sup>13</sup>C between the Formative and Late Intermediate Periods, indicating an increased reliance on maize and agricultural intensification between these periods.

**Keywords:** Stable isotope analysis, Carbon, Nitrogen, Keratin, Camelid, Chile, Arica, Agricultural Intensification, Animal Husbandry.

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# <span id="page-7-0"></span>**Chapter 1: Introduction**

The objective of this research is to evaluate the likely geographic origin of camelid (llama [*Lama glama*] or alpaca [*Vicugna pacos*]) fiber used in the manufacture of pre-Hispanic textiles found in the coastal regions of the northern Atacama Desert, Chile. Stable carbon and nitrogen isotope analysis was used to analyze camelid fiber from Atacama textiles to determine whether the lowland occupants of the region were sourcing fiber from local herds or obtaining it from the highlands. Fiber was sampled from textiles originating from archaeological sites in the Lluta and Azapa Valleys in northern Chile as well as from three sites on the coast of Arica. The textiles sampled are attributed to the Formative Period (~900 BCE– 200 BCE) through to the Late Intermediate Period (~1000 CE – 1450 CE).

Fiber was perhaps the most important resource obtained from camelids in the pre-Hispanic Andes – although camelids were also used for their meat (Hesse, 1982), leather (Valenzuela et al., 2015), as beasts of burden in caravans (Tripcevich, 2016; Nielsen, 2001), and their dung was used as fuel in cooking/ceramic production as well as to fertilize crops (Szpak et al., 2012b). Camelid fiber was the material of choice used in the manufacture of textiles which were items of extreme economic and social importance in the Andean region (Dransart, 1991; Costin, 1998).

The Atacama and Peruvian deserts form a 3500 km-long belt of arid land along the west coast of South America and experience some of the lowest precipitation rates in the world (Rundel et al., 1991). The lack of precipitation in the region has allowed for the remarkable preservation of organic materials – such as textiles – which are rare in the archaeological record elsewhere (Boytner, 2004). Stable carbon and nitrogen isotope

analysis were used to analyze camelid fibers from these preserved Atacama textiles to determine whether people sourced fiber locally ̶on the arid coast and western valleys, or whether it was traded in from the highlands. In basic terms, carbon and nitrogen isotopic compositions of the camelid hair are representative of the animal's diet during the period of hair growth – where differences in vegetation consumed at various altitudes will result in different isotope ratios between coastal and highland camelids (Szpak et al., 2015). Using the stable isotope compositions of previously analyzed camelid bones from the highland Pumapunku complex in the Tiwanaku Basin as comparative samples, data from the camelid fiber was used to determine whether each of the textiles demonstrated isotopic compositions which were consistent with being locally sourced or whether the fiber was likely highland in origin. To date, few studies have applied stable isotope analysis to pre-Hispanic Atacama textiles (Szpak and Valenzuela, 2020; Gayo et al., 2020; Santana-Sagredo et al., 2017), though the analysis was limited to samples from the Late Intermediate Period (1000 CE – 1450 CE) and Late Horizon (1450 CE – 1550 CE) which does not give any information about earlier camelid management practices in the region. Similar studies have attempted to source camelid fiber from textiles in regions of northern Peru using a similar approach with stable carbon and nitrogen isotope analyses (Szpak et al. 2015; Szpak et al., 2018).

#### <span id="page-8-0"></span>**1.1 Research Questions**

- 1. Are there textile samples in this dataset that have stable isotope compositions consistent with origins outside the highlands?
- 2. How does the stable carbon and nitrogen isotope data from these textile samples vary between time periods?

3. What can be ascertained about regional and temporal patterns in agricultural intensification and pastoralism in the Arica region from these data?

# <span id="page-9-0"></span>**1.2 Significance**

It has been commonly assumed that camelids – specifically alpacas – were best suited to life in the highlands (Troll, 1958) and that camelid products from archaeological sites at lower altitudes must have been acquired through trade with highland groups (Boytner, 2004; Pollard Rowe, 1980). However, several lines of archaeological evidence have revealed that camelids inhabited the productive coastal river valleys of northern Peru (Bonavia, 2008; Shimada & Shimada, 1985; Szpak et al., 2014a). This therefore opens the possibility that early occupants of the Atacama Desert may not have acquired camelid fiber via networks of trade with the highlands as previously assumed, but instead may have managed their own herds of camelids. One important difference with the Arica region is that the marginal valleys of northern Chile are much smaller and less productive than the valleys of northern Peru. Cultural differences between these two regions aside, the aridity of Arica may not have allowed for any camelid management outside the highland pastures and therefore we cannot assume that the presence of camelid management in northern Peru means that there was camelid management outside the highlands in northern Chile. That being said, the presence of locally managed camelid herds could change our understanding of the relationship between agricultural intensification and pastoralism in the Arica Region. Studying archaeological textiles is a rare opportunity because perishable organic materials usually deteriorate soon after they are deposited. Due to the relative rarity of textiles in the archaeological record, they have often been overlooked in favour of more durable artifacts such as stone tools or

architecture. However, textiles can provide a wealth of information about trade relationships, notions of status, artistic traditions, and resource use – information which is not always available to archaeologists (Boytner, 2004; Rodman, 1992; Millaire, 2009).

# <span id="page-10-0"></span>**1.3 Thesis Summary**

In Chapter 2, I introduce readers to the environmental and historical context of the Arica region. A description of stable isotope analysis will be provided in chapter 3, as well as a review of previous stable isotope studies on camelid tissues. In chapter 4, I describe the archaeological sites from which samples were obtained for this research. The methods used to determine the stable carbon and nitrogen isotope compositions of the Arica textile samples are also detailed. The data are presented in Chapter 5 and discussed at length during Chapter 6. Finally, chapter 7 summarizes the significant findings of this research and briefly outlines future directions in this area.

#### <span id="page-11-0"></span>**Chapter 2: Geography, Climate, and History of the Northern Atacama**

The Atacama and Peruvian deserts form a 3500 km-long belt of arid land along the west coast of South America and experience the lowest precipitation rates in the world (Rundel et al., 1991). Despite the persistent hyper-arid conditions, the pre-Hispanic occupants of the northern Atacama managed to create agricultural settlements in the core of the desert and created complex networks of interaction with surrounding groups. In this chapter, I will discuss the formation and climate of the northern Atacama Desert through time. I will then discuss the cultural history of the northern Atacama from the initial settlement of the desert circa 9000 BCE until the influence of the Inka Empire circa 1450 CE.

# <span id="page-11-1"></span>**2.1 Geography and Terrain**

The geographic area focused on in this thesis includes the northernmost region in Chile in the areas around the modern-day city of Arica (Figure 2.1). The northernmost portion of this region of Chile borders Peru to the north, Bolivia to the east, and the Pacific Ocean to the west. Beginning just south of the Chile-Peru border, exists a series of deep river valleys which originate in the Western Cordillera to the east, and drain into the Pacific Ocean. These occidental valleys, sometimes called western valleys or marginal valleys, are incised 1500 m deep in some locations (Kober et al., 2006). This thesis focuses mainly on archaeological sites in two of the occidental valleys, the Azapa and Lluta river valleys, as well as coastal sites near the modern-day city of Arica, all located in the arid core of the Atacama Desert (Figure 2.2).

Just south of the City of Arica, the Coastal Cordillera rises sharply from the ocean, with cliffsides quickly reaching elevations of 500 masl (Kober et al., 2006). At Arica, the

Coastal Cordillera subducts beneath the Peruvian Coastal Plain, making the coast of Arica closer to sea level than in the Cordillera (Kober et al., 2006). To the east of the Coastal Cordillera begins the Western Escarpment. The Western Escarpment steadily increases in elevation in the eastern direction and the landscape is characterized by a series of undulating hills with small valleys approximately 20 to 100 meters deep (Kober et al., 2006). These rolling hills reach elevations of 3000–3600 masl before meeting with the Western Cordillera (Kober et al., 2006). The Western Escarpment joins with the Western Andean Cordillera which reaches elevations as high as 6000 masl (Kober et al., 2006). On top of the Western Cordillera is a flat highland area called the *Altiplano* or Andean Plateau. This large, highland plateau extends north of Lake Titicaca in Peru, south into Bolivia, and terminates near the Bolivia-Argentina border (Figure 2.1).



<span id="page-12-0"></span>Figure 2.1 Area map of northern Chile. Terrain data from Google Maps (2021a)



<span id="page-13-1"></span>**Figure 2.2** Area map of the Arica region showing the location of the occidental valleys of northern Chile. Terrain data from Google Maps (2021b).

## <span id="page-13-0"></span>**2.2 Formation of the Atacama and Occidental River Valleys**

The arid conditions of the Atacama are thought to have persisted for as long as 200 million years (Clarke, 2006). Several factors have contributed to the antiquity of aridity in the region, including the South American continent maintaining a north-south orientation during this time span and the desert remaining at approximately the same altitude (Clarke, 2006). Due to the positioning of the continent, the Atacama has always been located on the west coast of South America which means it has always been in the subtropical high pressure/low rainfall zone, the prevailing winds have always been dry due to passage of over the continental interior, and due to the continent's configuration it is likely that cold-water currents have always been present along the coast which

decreased rates of evaporation (Clarke, 2006; Hartley, 2003). More recent environmental factors are thought to have intensified the aridity of the Atacama such as uplift of the Andes during the Miocene which increased the intensity of the Andean rain shadow, and changes in oceanic circulation during the Pliocene which increased upwelling of the cold Humboldt current (Hartley, 2003).

The factors discussed above have caused the extremely arid climate of the Atacama Desert. The region itself has no defined precipitation season (McKay et al., 2003). In the modern context, the northernmost city in Chile, Arica, experiences annual precipitation of only 0.5–0.6 mm per year (Clarke, 2006). The mean temperatures during the coldest months are 10–20°C and mean temperatures during the warmest months are between 20–30°C (McKay et al., 2003). The region experiences hyper-aridity in low elevation areas (0-2000 masl), arid climates in the cordillera (2000-4000 masl) and a cold, semi-arid climate in the *altiplano* (>3500 masl) (Santoro et al. 2019). Aridity in the region has been persistent but there is evidence of some climatic fluctuations over time. Most notably, the Andes experienced the retreat of glaciers from the highlands from 10,500 – 9000 BCE (Bonavia, 2008). During this time, arid conditions prevailed outside the highlands in coastal Peru and northern Chile for most of the late Pleistocene (Dillehay et al., 1992). Northern Chile experienced higher precipitation during the Pleistocene-Holocene transition (13,000-9000 cal yr BP) (Santoro et al., 2019) and during these times, vegetation would have been more abundant, which allowed wild camelids and other herbivorous mammals to enter and survive in the region (Craig, 1984). The higher precipitation in the high Andes allowed for excess runoff which produced oases in the low-elevation regions of the desert (Santoro et al. 2019). The more hospitable

environment during this time would have allowed for small groups of hunter-gatherers to settle in the region (Santoro et al. 2019).

Considering the Atacama Desert experiences extremely low rates of erosion due to lack of precipitation (Nishiizumi et al., 1998), the exact mechanism that caused the formation of the deeply incised occidental valleys is subject to debate. Some suggestions include glaciation during the Pleistocene that increased runoff (Wörner et al., 2002), tectonic activity (Mortimer, 1980; Paskoff, 1980), changes in the water cycle driven by the formation of the Andes (Mortimer, 1980; Paskoff 1980), and changes in paleoclimate approximately 3 Ma which increased the rate of incision of the valleys (Kober et al., 2006).

### <span id="page-15-0"></span>**2.3 Suitability of the Northern Atacama for Camelid Husbandry**

It has been commonly assumed by archaeologists that domesticated camelids – especially alpacas – of the pre-Hispanic Andes were ill-suited to life on the coast or environments outside of the highlands. Bonavia (2008) attributes this assumption as stemming originally from early colonial writings in Peru that described camelids as flourishing in cool, high-altitude environments but being generally ill-suited to the coastal heat. The writings of Johan Jacob von Tschudi (1885, 1891, 1918) claimed that camelids sometimes came to the coast in caravans, however they were not acclimated to the coast (Tschudi *in* Bonavia, 2008). Tschudi also claimed that the existence of camelid bones in lower-elevation archaeological tombs was not evidence that camelids lived in the region, but instead only evidence that they were brought from the highlands, dead or alive, and buried in tombs. Troll (1958) reiterated Tschudi's sentiments and argued that domesticated camelids never acclimatized to the coastal Peruvian deserts but were

occasionally brought there as beasts of burden. Other researchers such as Bushnell (1963), Menzel (1977), and Flannery, Marcus & Reynolds (1989) claimed that people on the coast would purchase caravan camelids for food or sacrifice which explains the presence of zooarchaeological remains in coastal sites but stated that no camelids were raised on the Peruvian coast. Not all archaeologists were convinced that camelids were constrained only to highland regions. For example, Horkheimer (1958) noted a considerable amount of camelid bones in cemeteries in the *lomas* (coastal fog zones) and pointed to the fact that camelid herds are maintained in coastal Peru today. Lanning (1967) reiterated that domesticated camelids were born on the coast in modern times, and the amount of camelid wool used on the coast when there was little evidence of trade with the highlands indicates that domesticated camelids must have lived locally. Shimada and Shimada (1985) were instrumental in moving this discussion forward. Until this point, skeptics of coastal camelids could acquiesce that llamas may have been bred in the highlands and sporadically brought to the coast to live for short periods of time, however Shimada and Shimada (1985) used modern ethnographic accounts, ethnohistoric evidence and zooarchaeological data from previously published archaeological excavations to argue for the presence of llamas being bred and maintained on the north coast of Peru as early as 600 C.E. They further suggested that alpaca – that were more prized for their fiber than llamas, but were not well-suited to be pack animals – could have been raised on the coast due to the fact that modern accounts suggested far more climatic and dietary adaptability than had previously been assumed (Shimada & Shimada, 1985). Bonavia (2008) posited that camelids were once evenly distributed throughout the various environmental zones of the Andes and populations only retreated to the highlands after colonization, when they began to be outcompeted for pastureland by Spanish

domesticates like sheep and cattle. Shimada and Shimada (1985) also pointed to the reduced economic significance, number, and distribution of camelids after the conquest when their economic roles were supplanted by European domesticates and their role as transport eventually was replaced by modern forms of transportation. Today llamas mainly range from the central Ecuadorian highlands to northwestern Argentina, with a large concentration 350 km north and south of Lake Titicaca, Peru (Shimada & Shimada, 1985). Distribution of modern alpacas is even more limited, usually within a 50-100 km radius of Lake Titicaca, although the authors note, modern numbers of alpaca are increasing due to them being prized for their fiber (Shimada & Shimada, 1985). A thorn in the side of this area of research is that it is difficult to distinguish llama and alpaca remains osteologically due to overlapping body size and morphology due to the interrelated nature of the two species (Shimada  $\&$  Shimada 1985). This means that assertions about whether llamas or alpacas specifically were maintained on the coast are difficult to distinguish therefore the two domesticates are lumped into the single group 'camelids' in literature. Alpacas are far more prized for their fiber than llamas today, and llamas were more useful as beasts of burden in trade caravans due to their generally larger body size. From this it is accepted that llamas came to the coast in caravans, but the question of fiber-producing alpacas living on the coast has been much more difficult to demonstrate.

Much of the debate surrounding coastal camelid herding has centered around central to northern Peru, with little previous research on the possibility of camelid herding in coastal Chile. The environment of the Arica region is more arid than Peruvian climates and therefore camelid herding would have been comparatively more difficult in northern Chile. This is because the agricultural productivity is far more limited in the northern Chilean valleys than some of the larger northern Peruvian valleys (e.g. Santa, Moche,

Jequetepeque) where most of the previous research on camelid husbandry has been done. Furthermore, most rivers in northern Chile only run seasonally and water volume is far smaller than Peruvian rivers to the north. This is significant because if camelids were eating local crops or grazing on local vegetation, there is less potential to grow enough fodder in northern Chile compared to northern Peru. A large factor driving this low productivity is the small size of rivers in northern Chile relative to those of northern Peru, a pattern that starts in southern Peru where the river valleys are often smaller than in the north. Low amounts of vegetation, intermittently flowing rivers and general water scarcity could have all been limitations on large-scale herding on the north coast of Chile.

# <span id="page-18-0"></span>**2.4 Cultural History of the Northern Atacama**



<span id="page-18-1"></span>**Figure 2.3** Representation of the different periods with date ranges for the Arica region of northern Chile accompanied by the cultural groups associated with each period. The length of the boxes associated with each period is not proportional to their actual temporal span for the sake of readability (e.g., the Archaic Period is approximately 80 times as long as the Late Horizon and a proportional representation would not be possible).

#### <span id="page-19-0"></span>**2.4.1 Archaic Period in the Northern Atacama**

Although the Archaic Period is not represented in the sample for this thesis, it is important in understanding the formation of settlements in the northern Atacama. The Archaic Period is generally defined as the period between the initial habitation of the northern Atacama (circa 9000 BCE) until the beginnings of agriculture around 1000 BCE (Muñoz, Agüero & Valenzuela, 2016). The initial peopling of South America is thought to have occurred circa 14,600 cal BP, however due to the extreme conditions of the desert, human occupation in the area did not commence until approximately 9000 B.C.E (Santoro et al., 2011). The initial human presence in the Atacama seems to have been more sporadic instances of occupation (Aldenderfer, 1999; Santoro et al., 2011).

During the Archaic Period, hunting, fishing, and gathering were the main form of subsistence for early occupants of the coastal Atacama (Muñoz, Agüero & Valenzuela, 2016). Around 2000 BCE, new cultural traits began to appear in the archaeological record indicating some form of cultural change occurring in the region which marks the beginning of the Archaic/Formative transition. For example, the practice of artificial mummification practiced by the Chinchorro disappeared and instead bodies began to be deposited into pits, wrapped in textile bundles (Muñoz, Agüero & Valenzuela, 2016). There were also increasing instances of ornamental items such as headdresses and necklaces included in burials which is interpreted to mark the beginnings of social stratification within Archaic Period cultural groups (Muñoz, Agüero & Valenzuela, 2016). The people of the Atacama became more sedentary during the Late Archaic period (Dudognon & Sepúlveda, 2018) and began to domesticate camelids (Wheeler, 1999; Yacobaccio, 2003; Cartajena, Núñez & Grosjean, 2007; Gallardo and Yacobaccio, 2005).

The earliest evidence of agriculture in the region was occurring during the Late Archaic, with the first evidence of corn and cotton in the Lluta valley around 2000 BCE (Muñoz, Agüero & Valenzuela, 2016) and early agricultural practices occurring in wetlands around river mouths including crops like gourds, sweet potato (*Ipomoea batata*), manioc (*Manihot utilissima*), and cotton seeds (*Gossypium sp.*) (Dauelsberg 1974; Muñoz 1982). Beyond the introduction of agriculture, other technological advances began to appear such as cotton and wool fabric, ceramics, and irrigation (Lumbreras, 2006).

The exact reason for the uptake of agriculture at the end of the Archaic Period is debated. Muñoz, Agüero & Valenzuela (2016) argue agriculture may have been initiated by the inhabitants of the Atacama themselves, applying their knowledge of native plants and beginning to experiment or alternatively, crops may have arrived through trade and the local people then initiated agriculture. It has also been argued (Rivera 2008; Rivera 2002) that *Altiplano* populations settled in the occidental valleys and may have driven agricultural intensification in the area by introducing new agricultural practices. Disputes over water resources due to increases in population size may have also encouraged the uptake in agriculture (Muñoz, Agüero & Valenzuela, 2016). The final factor that may have encouraged the adoption of agriculture in the northern Atacama is climate instability during the terminus of the Archaic Period (2000–1500 BCE) (Ramírez, Bryson & Bryson, 2001; Muñoz, 2004). During this time, the flow of the San Jose River experienced disruptions which caused water instability near the mouth of the river (Ramírez, Bryson & Bryson, 2001; Muñoz, 2004). Whatever the reason for agricultural uptake at the end of the Archaic Period, the development of agriculture was coupled with increased social complexity at the onset of the Formative Period (Muñoz, Agüero & Valenzuela, 2016).

# <span id="page-21-0"></span>**2.4.2 Formative Period in the Northern Atacama**

The Formative Period dates between 1000 BCE and 200 CE and is divided into two cultural phases in northern Chile. These groups included agriculturalists of the Faldas del Morro cultural phase on the coast (also called Azapa in the valleys) which existed from 1000 to 500 BCE, and the Alto Ramírez phase between 500 BCE and 200 CE (Muñoz, Agüero & Valenzuela, 2016). This period is marked by increasing agricultural yields and the consolidation of communities into more permanent settlements (Dudognon & Sepúlveda, 2018). Agricultural settlements were centered around the occidental river valleys that flowed from the highlands as well as areas with large numbers of groundwater springs (Muñoz, Agüero & Valenzuela, 2016). Access to water resources, especially groundwater springs, were essential in allowing the local population to settle and produce agricultural settlements. Despite advances in agriculture during the Formative Period, isotopic analyses of human remains suggest that marine resources remained the main component of diet (Andrade et al. 2015; Bonilla et al. 2016). Around 500 BCE, agricultural practices in northern Chile began to become centralized with more stable settlements and evidence of specialized labour (Muñoz, Agüero & Valenzuela, 2016). In the Azapa Valley, settlements were widely dispersed — each settlement containing its own ceremonial center, cemetery, and organizational structure (Muñoz, Agüero & Valenzuela, 2016). During this period, the first evidence of maize appeared in the Azapa Valley circa 500 BCE and other crops like manioc, sweet potato, gourds and tubers continued to be grown (Muñoz, Agüero & Valenzuela, 2016).

A distinctive aspect of Formative Period settlements was the construction of burial mounds. Burial mounds required large amounts of labour which suggests people who had control over water resources could mobilize the economy to complete non-agriculture related tasks (Muñoz, Agüero & Valenzuela, 2016). Instances of burial mounds decreased during the Middle Period when, it has been argued, northern Chile began to be influenced by highland cultures, making burial mounds a fairly distinct feature to the Formative Period (Muñoz, Agüero & Valenzuela, 2016).

The textile tradition of the Formative Period appears to be relatively continuous between the Archaic to Formative. However, in the late Formative Period, textile designs began to feature anthropomorphic, zoomorphic, and geometric motifs (Muñoz, Agüero & Valenzuela, 2016). It was suggested that these motifs were similar to the patterns of the Pucara culture from the Titicaca basin which led Rivera (1976, 1984) to argue that this shift in textile patterns was due to the influence of *Altiplano* cultures. The counterargument is that the Formative Period textile tradition in the occidental valleys had already been established in the Archaic Period, and although some foreign ideas may have been adopted, the textile patterns were organic to the region (Agüero 1995, 2012; Agüero & Cases, 2004; Horta, 2004). There is evidence of trade between highland groups and the northern Chilean coast where cultural ideas may have been exchanged. The presence of camelid caravans in Arica at the end of the Formative Period (circa 200 CE) is evidenced by the presence of highland goods such as felid skins, and dog remains at lower elevation sites (Muñoz, Agüero & Valenzuela, 2016) which indicates expanding networks of connection during this period.

# <span id="page-23-0"></span>**2.4.3 Middle Period in the Northern Atacama**

The Middle Period, which spanned from 500 CE to  $\sim$ 1000 CE, is marked by the influence of highland cultures from the Titicaca Basin on the cultures of Arica. During this period, local groups in the occidental valleys created organized settlements, agriculture further intensified, and evidence of complex networks of interaction began to become apparent in the archaeological record.

 The events of the Middle Period are highly contested with debate over the nature of highland influence in the occidental valleys. It was the dominant idea that the Tiwanaku from the Lake Titicaca Basin established colonies in the Azapa Valley during this time (Berenguer, 1988; Berenguer & Dauelsberg, 1989). This hypothesis aligns with Murra's (1972) model of verticality which posits that *Altiplano* groups would have had a land-use pattern that involved exploiting many different ecological zones and the resources available at each altitude. Common Andean economic activities such as grazing camelids, and growing corn or potatoes would all take place at different altitudes (Maxwell, 2011) and therefore polities would have to control land in a variety of altitudes and ecological niches to have stable access to these resources. In Murra's (1972) model, coastal groups are conceptualized as not being engaged in exploiting ecological zones, or alternatively seen as outposts under the control of the *Altiplano* groups.

According to models that accept the idea that the Tiwanaku established directlycontrolled colonies in northern Chile, the first phase would have been Cabuza and is defined as the period when the *Altiplano* established colonies, and a second phase called Maytas would have correspond to the consolidation of all the colonies started by the

Tiwanaku (Muñoz, Agüero & Valenzuela, 2016). New data suggests that although there were outside influences on the culture of the western valleys at the time, there is no evidence to suggest direct highland colonization. The material culture (including textiles, architecture, and ceramics) all appear to be locally rooted (Agüero 2000; Muñoz 2004; Uribe 1999; Uribe & Agüero 2001; Korpisaari, Oinonen & Chacama, 2014). Furthermore, radiocarbon dating on 16 artifacts from the Azapa Valley attributed to the Middle Horizon due to association with 'Tiwanaku' ceramics (Cabuza style) determined that many of the associated artifacts (save for one sample) were actually dated to the Late Intermediate Period (Korpisaari, Oinonen & Chacama, 2014). The authors argue that this demonstrates the stylistic Tiwanaku influence seen in the Azapa Valley were not the result of colonization during the Middle Period (Korpisaari, Oinonen & Chacama, 2014). Furthering this, the adoption of the Cabuza-style pottery seems to have largely occurred after the collapse of the major Tiwanaku colony in the Moquegua Valley, further arguing against the idea of direct colonization during the Middle Period (Korpisaari, Oinonen  $\&$ Chacama, 2014). The adoption of the Cabuza style could be associated with migrants to the Azapa Valley from the Osmore Valley around the time of the disintegration of the Moquegua Middle Valley Tiwanaku colony in the 10th century C.E. (Korpisaari, Oinonen & Chacama, 2014). Today it is recognized that there were multiple cultural styles existing at the same time and place: first were 'highland influenced' styles known as the Cabuza (500–1200 CE) and Azapa/Charcollo (600–800 CE), and the other from the western valleys is characterized by the Maytas/Chiribaya style (800–1290 CE) (Muñoz, Agüero & Valenzuela, 2016).

The concept of highland colonies in northern Chile has fallen out of favour due to a lack of concrete evidence. For example, Tiwanaku ceramic and textile evidence is

scarce in the western valleys which suggests at most an intermittent Tiwanaku influence (Muñoz, Agüero & Valenzuela, 2016). During this period, there were also no large changes in settlement patterns or agricultural systems and, non-local objects were generally scarce in the western valleys ̶only appearing in some tombs, which is interpreted as highland influence only being felt by certain members of the community and not throughout the entire population (Muñoz, Agüero & Valenzuela, 2016). The Azapa Valley was one of the most intensely agriculturally exploited areas in the region and it is also the region where the most Cabuza ceramics and textiles were found (Korpisaari, Oinonen & Chacama, 2014). With extensive water resources and agricultural land, the occupants of Azapa were likely a part of a caravan traffic network with the surrounding valleys and highlands to move surplus agricultural goods to proximal cultural groups (Muñoz, Agüero & Valenzuela, 2016). Several representations of llama caravans in rock art also possibly correspond to this period (Valenzuela et al., 2015) and reinforce the theory that trade networks were being formed. It therefore seems likely that Tiwanaku cultural influences could have arrived in northern Chile due to trade relationships, rather than colonization.

 Most of the known archaeological sites from the Middle Period in northern Chile come from the Azapa Valley, with the period being scarcely represented in the Lluta Valley (Muñoz, Agüero & Valenzuela, 2016). Settlements in the Middle Period were in similar locations compared to the Formative Period, but settlements began to be built on terraces and hill slopes (Muñoz, Agüero & Valenzuela, 2016). The discovery of weaving and sewing tools indicates that the Middle Horizon populations in the western valleys were producing textiles locally (Muñoz, Agüero & Valenzuela, 2016). Other textile remains include funerary tunics, blankets, and loin cloths accompanied by grave goods

such as bags and small weaving implements like balls of camelid fiber yarn and looms (Muñoz, Agüero & Valenzuela, 2016). Evidence of local textile production demonstrates that camelid fiber was either being obtained through trade networks or potentially, from local camelids.

# **2.4.4 Late Intermediate Period in the Northern Atacama**

The Late Intermediate Period, also sometimes referred to as the Regional Developments, is generally defined as the time between 1000 CE until Inka influence in the Arica region circa 1450 CE. It was a period of strong local culture and increased interaction and mobility throughout the south-central Andes (Muñoz, Agüero & Valenzuela, 2016). The Late Intermediate Period in northern Chile is characterized by agricultural intensification as demonstrated by the construction of canals and irrigation systems as well as the construction of extensive agricultural terraces (Muñoz, Agüero  $\&$ Valenzuela, 2016).

During this period, the local Arica culture flourished along the coast, low valleys, and in the highlands (Muñoz, Agüero & Valenzuela, 2016). On the coast, the subsistence strategy was still based on a mixture of agriculture, wild vegetation, and marine resources (Allison, 1989; Aufderheide & Santoro, 1999). Stable carbon and nitrogen isotope analysis on collagen from 77 Late Intermediate Period humans the from Az-8, CAM-8, and Ll-54 sites, indicated that marine resources remained an important resource during this period as the ocean provided an abundant and predicable food source in the hyperarid core of the Atacama (Alfonso-Durruty et al., 2019). Other stable isotope studies have demonstrated that during the Late Intermediate Period onwards, the consumption of

maize increased to become one of the most important terrestrial resources in human diet in the Atacama (Pestle et al. 2015; Santana-Sagredo, Hubbe & Uribe, 2015; Torres-Rouff, Pestle & Gallardo, 2012). Isotopic analyses by King et al. (2018) demonstrated significant dietary variability between individuals, suggesting that the people of the Atacama maintained diverse diets to buffer against resource instability caused by the harsh environment of the desert.

In the highlands, subsistence was based on agriculture, with complex systems of irrigation and terraces for crops, supplemented by camelid livestock (Muñoz, Agüero & Valenzuela, 2016). Small herds of camelids may have been kept in lowland villages during this time based on the presence of camelid dung (Niemeyer & Schiappacasse, 1981; Santoro, 1995). During this period, the cultures of northern Chile were organized in a complex political structure which involved the exploitation of various ecological zones and trade relationships between the various groups that comprised the Arica culture (Muñoz, Agüero & Valenzuela, 2016). It is generally agreed upon that the Arica culture was made up of independent chiefdoms (Muñoz, 2004; Romero, 2005; Santoro et al., 2004) but the degree of integration of these groups is still subject to debate. Some key indicators of the large networks of interaction during this period is the finding of foreign goods in the Arica area such as minerals, metals, ceramics, obsidian, and high Amazonian bird feathers (Muñoz, Agüero & Valenzuela, 2016; Capriles et al., 2021). Several rock art sites in the mid to lower valleys depict camelid caravans which suggests these locations may have been meeting places for caravan traffic and centres for the transportation of goods (Valenzuela et al. 2015; Valenzuela et al. 2019).

Towards the end of the period (circa 1100–1400 CE), the populations of the Arica

culture established agricultural settlements in the coastal valleys of Lluta, Azapa, and Camarones and then gradually settled in the headwaters of the valleys and mountains of Arica (Muñoz, Agüero & Valenzuela, 2016). These settlements had networks of trails, irrigation, and may have been centers of camelid herding as evidenced by corrals outside of the villages (Muñoz, Agüero & Valenzuela, 2016). After the Arica culture groups moved to the highlands circa 1200 CE, groups from the highlands began to occupy territories in the headwaters of the occidental valleys (Muñoz, Agüero & Valenzuela, 2016). These groups seem to have been offshoots of the Tiwanaku culture and exploited the coastal regions to obtain fruit, corn, legumes, vegetables, marine products/fish (which were dried and taken to the highlands), as well as seabird guano to use as fertilizer (Muñoz, Agüero & Valenzuela, 2016). Unlike local groups, their administrative centers were in the highlands and settlers populated the headwaters and coast in a series of settlements following Murra's (1972) model of the vertical archipelago (Muñoz, Agüero & Valenzuela, 2016). At the end of the Late Intermediate Period, the highlands and headwaters of the occidental valleys were spaces of interaction and agricultural intensification. At the time, a series of trails connected the coast, valleys, headwaters, and highlands which attests to the interconnectedness of the area (Muñoz, Agüero & Valenzuela, 2016). Although groups of the Arica culture continued to be governed locally, between 1450 and 1550 CE, the highlands began to show evidence of Inka influence through the improvement of the road networks throughout the highlands (Muñoz, Agüero & Valenzuela, 2016).

#### <span id="page-29-0"></span>**Chapter 3: Principles of Stable Isotope Analysis**

This chapter is intended to be a primer on stable isotope analysis and its applications within the field of archaeology. This chapter also touches on stable isotope analysis more broadly but provides more specific information about the two isotope systems used in this research – carbon and nitrogen. The chapter concludes with a discussion of the analysis of keratin in stable isotope studies and previous applications of stable carbon and nitrogen isotope analysis on camelid hair keratin and bone collagen to investigate geographic origin of archaeological samples.

#### <span id="page-29-1"></span>**3.1 What is an Isotope?**

Atoms are composed of subatomic particles called protons, neutrons, and electrons. Protons and neutrons are located within the nucleus of the atom, and electrons orbit the nucleus. An element is defined by the number of protons in the nucleus of the atom. Isotopes of a given element differ from one another by the number of neutrons in the nucleus (Sharp, 2017). For example, a carbon atom will always have 6 protons, however the number of neutrons will vary depending on the isotope (6 neutrons for carbon-12, 7 neutrons for carbon-13, and 8 neutrons for carbon-14). The basic definition of isotopes are atoms of an element with the same number of protons and a variable number of neutrons (Sharp, 2017). However, this definition does not include elements with only one isotope. Most elements have two or more naturally occurring isotopes (either stable or radioactive) but there are 21 elements that are monoisotopic (Sharp, 2017). A more all-encompassing definition for an isotope would be "a particular form of an element defined by a specific number of neutrons" (Sharp, 2017; 2-1).

#### <span id="page-30-0"></span>**3.2 What Makes an Isotope Stable?**

Naturally occurring isotopes can be divided into two groups: stable or unstable (radioactive) (Hoefs, 2009). The difference between these two types of isotopes is that radioactive isotopes undergo radioactive decay whereas stable isotopes do not. Carbon-14 is perhaps the best-known isotope in archaeology as it forms the basis of radiocarbon dating. Carbon-14 is a radioactive isotope that slowly decays with a half-life of approximately 5730 years. The two stable isotopes of carbon (carbon-12 and carbon-13) are unlike carbon-14 in that they do not decay over time and therefore the relative quantities of these isotopes in an archaeological sample are generally representative of the relative quantities of these isotopes during the life of an organism, provided they are not altered in the burial environment. In terms of the total number of nuclides, stable isotopes are less common than unstable isotopes with approximately 300 stable isotopes having been discovered to date and 1200 unstable isotopes having been discovered (Hoefs, 2009). The stability of an isotope is mainly determined by two rules: the "Symmetry Rule" and the "Oddo-Harkins" rule. The rule of symmetry states that in a stable isotope with a low atomic number, the number of protons will be approximately equal to the number of neutrons and in stable nuclei with more than 20 protons or neutrons, the ratio of neutrons to protons will fall between unity and 1.5 (Hoefs, 2009). The "Oddo-Harkins" rule states that stable isotopes are more common when an element has an even atomic number (an even number of protons) as opposed to elements with an odd atomic number (Hoefs, 2009).

#### <span id="page-30-1"></span>**3.3 Fractionation and Isotope Effects**

The atomic mass of an atom is determined by the number of protons and neutrons in the nucleus. Therefore, although the various isotopes of an element can make the same chemical bonds, and the addition of neutrons does not change the charge of an atom, the additional neutron(s) create a mass difference between isotopes of an element (Sharp, 2017). In essence, the more neutrons, the heavier the isotope. Due to slight differences in atomic mass and bond strength, the different isotopes of an element behave in slightly different ways. The two ways that ratios of isotopes in a system can change are through kinetic isotope effects and equilibrium isotope effects. In kinetic isotope effects, the reactions are characterized as being irreversible, and are associated with fast, incomplete, and unidirectional reactions (Sharp, 2017). These effects are characterized by a difference in velocity possessed by the different isotopic forms of molecules as they move from one phase to another (Sharp, 2017). The mass difference between isotopes of an element translates to differences in velocity. Therefore isotopically 'light' molecules will preferentially diffuse out of a system and more of the isotopically heavy molecules will remain in the reservoir (Sharp, 2017). Equilibrium isotope effects are associated with differences in bond strengths with heavier isotopes creating slightly stronger bonds (Sharp, 2017). Both these kinetic and equilibrium isotope effects alter the ratio of heavy and light isotopes in a system. For example, water vapor over a large lake will have lower  $18O/16O$  ratios (more of the lighter oxygen isotope) than the lake water itself (which will retain more of the heavier oxygen isotope) because of the differences in atomic velocity due to the mass differences between the isotopes. These different mechanisms change the natural abundance of certain isotopes in a process called fractionation – and these changes in the ratios of heavy to light isotopes are the basis for stable isotope science. Net

fractionation only occurs in incomplete or open reactions − if a reaction is completed, then the ratio of isotopes in a completed reaction will be the same as the ratio of isotopes at the beginning of the reaction (Sharp, 2017). Considering nearly all reactions in nature can be characterized as occurring in open systems, this allows for a variety of fractionation processes in the natural world.

### <span id="page-32-0"></span>**3.4 Isotope Ratio Mass Spectrometry (IRMS) Analysis**

The ratio of stable isotopes of light elements is typically denoted using delta notation which allows for the distinction of very small differences in the relative proportions of stable isotopes in a given sample. Delta values are unit-less and are written in parts per thousand or per mille (‰). Delta notation is a comparison of the heavy isotope to light isotope within an analyzed sample over the heavy to light isotope ratio of a standard. The formula for delta notation is as follows:

$$
\delta = \left[ \frac{\left(\frac{Amount of heavy isotope in sample}{Amount of light isotope in sample}\right)}{\left(\frac{Amount of heavy isotope in standard}{Amount of light isotope in standard}\right)} - 1 \right]
$$

For isotopic data to be comparable between laboratories, the resulting data are calibrated relative to international standards which have an agreed upon value. For carbon isotope analysis, the agreed-upon reference sample for  $\delta^{3}C$  is the Chicago PDB marine carbonate standard which was created from a Cretaceous-period marine fossil from the Peedee formation in South Carolina. The original fossil sample has been exhausted so a replacement called Vienna-PDB (VPDB) was created by the National Bureau of Standards, the International Atomic Energy Agency and the United States Geological Survey (Craig 1953, 1957; Hut 1987). The agreed upon international standard for  $\delta^{15}N$  is

atmospheric nitrogen (AIR or ambient inhalable reservoir) which by definition has a  $\delta^{5}N$ value of 0 ‰ (Hut, 1987).

Raw isotopic data must be calibrated relative to these international reference standards using calibration (quality assurance) standards with well-established isotopic compositions. It is best practice to do a two-point calibration as opposed to a one-point calibration (Skrzypek, 2013). A two-point calibration will produce a more accurate calibration when the isotopic values of the calibration standards bracket the isotopic values of the sample(s) being analyzed (Szpak, Metcalfe & Macdonald, 2017). Calibration and check standards are analyzed multiple times through-out an IRMS run at the beginning, end, and throughout the run.

Check (quality control) standards are used throughout the analytical session to determine the accuracy of the session and account for any instrumental drift which may have occurred. The  $\delta^{13}C$  and  $\delta^{15}N$  values of a check standard are well-established but are treated as "unknowns" during calibration (Carter & Fry, 2013). The values for the check standards are then compared to the accepted values to determine accuracy of the calibration. Check standards should have  $\delta^{13}$ C and  $\delta^{15}$ N values within the range of values expected for the analyzed samples and should be similar in elemental composition (wt% carbon and wt% nitrogen) to the analyzed sample (Carter  $\&$  Fry, 2013). Calibration standards and check standards must be different. To measure the overall analytical precision of a session, the standard deviation in  $\delta^{13}$ C and  $\delta^{15}$ N values of check standards, calibration standards, and duplicate samples is calculated (Szpak, Metcalfe & Macdonald, 2017).

### <span id="page-34-0"></span>**3.5 Stable Carbon Isotopes in the Biosphere**

The evolution of stable carbon isotope analysis in archaeology stemmed from radiocarbon dating. Bender (1968) realized that animals (including humans) who consumed large quantities of the C<sup>4</sup> plant corn (*Zea mays*) typically had radiocarbon dates that were more recent than accompanying material culture would suggest. Bender was detecting the different ratios of <sup>12</sup>C/<sup>14</sup>C caused by the C<sub>4</sub> photosynthetic pathway – as previous research had focused on specimens from exclusively  $C_3$  environments.  $C_4$  plants (such as maize and many tropical grasses) discriminate less against the heavier isotope of carbon during  $CO_2$  uptake and assimilation relative to  $C_3$  plants (Smith & Epstein 1971).  $C_4$  plants, therefore, will have tissues containing relatively more <sup>14</sup>C and <sup>13</sup>C compared to  $C_3$  plants, causing them to produce unusually young radiocarbon ages and have high  $\delta^{13}C$ values. It wasn't long before it was discovered that differences in  $\delta^{13}$ C values caused by photosynthetic pathway are passed on through the food chain when plants are eaten by consumers. DeNiro and Epstein (1978) were the first to recognize that the stable carbon isotope values from an animal's tissues were derived from the animal's food sources. Therefore, the differences in  $\delta^{13}$ C caused by C<sub>3</sub> versus C<sub>4</sub> photosynthetic fractionation makes stable carbon isotope analysis an ideal method for interpreting differences in plant consumption, especially when  $C_4$  plants are introduced into primarily  $C_3$  environments (van der Merwe & Vogel, 1978), when graze consists entirely of  $C_4$  plants and browse consists entirely of  $C_3$  plants (Ambrose & DeNiro, 1986), or when different geographic regions are dominated by  $C_3$  and  $C_4$  plants, respectively (Szpak et al., 2013).

Photosynthetic pathway is the major driving force that determines the  $\delta^{13}$ C composition of plants. The three pathways  $(C_3, C_4, C_4)$  and CAM plants) have their own

range of  $\delta^{3}$ C values caused by the way each plant-type incorporates carbon. C<sub>3</sub> plants rely on the Calvin cycle for carbon fixation, whereas  $C_4$  plants use the Hatch-Slack cycle to photosynthesize. The differences in plant  $\delta^{3}C$  between C<sub>3</sub> and C<sub>4</sub> plants is caused by the preferential uptake of either <sup>12</sup>C relative to <sup>13</sup>C during the uptake of  $CO_2$  (van der Merwe, 1982; O'Leary, 1988). Most vegetation and trees fall under the  $C_3$  category where the first stable photosynthetic product is a 3-carbon acid.  $C_3$  plants generally have  $\delta^{3}C$ values ranging from −35 ‰ to −20 ‰ with a mean of −27 ‰ due to preferential fixation of  ${}^{12}C$  (Kohn, 2010).  $C_4$  plants (ex. corn, sugarcane, and tropical grasses) have a 4-carbon acid as their first stable photosynthetic product (Hobbie & Werner, 2004; O'Leary, 1988; Tieszen, 1991; van der Merwe, 1982). During the Hatch-Slack cycle, <sup>12</sup>C is discriminated against to a lesser degree, which results in  $\delta^{13}$ C ranging from -15‰ to -7‰ with a mean of −12 ‰ (O'Leary, 1988; Tieszen, 1991; Vogel, 1993). Plants that use the CAM photosynthetic pathway (cacti, succulents, and epiphytes) can switch between these two carbon cycles depending on environmental conditions and therefore have  $\delta^{3}C$  values that overlap with the  $C_3$  and  $C_4$  ranges but are generally closer to the latter. CAM plants have  $\delta^{13}$ C values ranging from -22 ‰ to -10 ‰ (O'Leary, 1988). C<sub>4</sub> plants are more common in hot and arid climates, while  $C_3$  plants thrive in more temperate climates (O'Leary, 1988; van der Merwe, 1982). In the Andean context,  $C_3$  plants dominate the highlands as largescale  $C_4$  maize agriculture mostly took place below 3200 masl for most varieties. Some maize varieties, which were the result of human selection, were able to be grown as high as 3600 masl (Staller, 2016). There is evidence of one highland maize species, *tunqu*, that was grown in the Copacabana Peninsula micro-climate in the Lake Titicaca basin between 3800 – 4100 masl, however its consumption is thought to have been
limited to ceremonial contexts (Staller, 2016).

Other than photosynthetic pathway, there are environmental factors that can also influence plant  $\delta^{13}C$  and be passed on to consumers. For example, the  $\delta^{13}C$  values of  $C_3$ plants are sensitive to water availability, which results in  $C_3$  plants that grow in arid conditions having a higher  $\delta^{13}$ C value than plants growing in places with greater water availability (Stewart et al. 1995). Water availability is not a factor in  $\delta^{13}C$  values of  $C_4$ plants (Swap et al., 2004). Considering the extreme aridity of the Atacama region, all samples in this study were likely influenced by low water availability. Therefore, wild  $C_3$ plants in the region will have higher  $\delta^{3}C$  compositions than  $C_3$  plants from less-arid regions. Due to the distribution of  $C_3$  plants in the highlands which would have been used as graze for camelids, it is expected that camelids grazing in highland environments will have a more  $C_3$ -based diet and therefore lower  $\delta^{13}$ C values than camelids raised outside the highlands. Camelids that lived in the marginal valleys and on the coast are expected have a more C4-based diet due to their proximity to more intensive maize agriculture, and therefore are expected to have higher  $\delta^{3}$ C values compared to highland camelids.

### **3.6 Stable Nitrogen Isotopes in the Biosphere**

Stable nitrogen isotope analysis is a complementary source of information when paired with stable carbon isotope analysis as it can provide information about trophic level and various environmental processes. DeNiro and Epstein (1981) were the first to recognize a trophic level increase of  $\sim$ 3‰ in  $\delta$ <sup>15</sup>N between a consumer and its diet. Nitrogen stable isotope analysis can be used to determine trophic position of animals and humans in the past – however baseline  $\delta^{15}N$  values must be understood to make trophic interpretations. Soil and plant metabolism form the base of the nitrogen cycle, a collection

of biogeochemical processes that transforms nitrogen and is responsible for a wide range of variation in  $\delta^{15}$ N values in plants and animals.

The ultimate source of nitrogen is the main determinant of plant  $\delta^{15}N$ . Transformations of nitrogenous molecules within the soil are a source of fractionation (Craine et al., 2009; Högberg, 1997; Szpak, 2014) and some of these processes, particularly ammonia volatilization and denitrification, are associated with strong discrimination against <sup>15</sup>N, causing some of the largest fractionations observed in nature (Högberg, 1997). Mineralized nitrogen ( $NO<sub>3</sub><sup>-</sup>$  and NH<sub>4</sub><sup>+</sup>) and atmospheric nitrogen (N<sub>2</sub>) are the most common compounds taken up by plants that drive plant  $\delta^{15}N$  (Touraine, 2004; Ramos, 2004). Leguminous plants rely on symbiotic bacteria to convert  $N_2$  to  $NH_3$ (Kohl, 1980). There is very little fractionation associated with this process which results in leguminous plants having  $\delta^{15}N$  values around 0 ‰ (Szpak et al., 2014b). When there is an abundance of labile nitrogen in the soil, such as in the case where fertilizer is applied, legumes will rely on mineralized nitrogen rather than on nitrogen from symbiotic relationships as biological N<sub>2</sub>-fixation is metabolically expensive (Szpak et al., 2014b).

Plants that do not partner with nitrogen-fixing bacteria typically rely on mycorrhizal associations which can also have a strong influence on plant  $\delta^{5}N$  (Hobbie & Högberg, 2012). Mycorrhizae are symbiotic fungi that form associations with nonnitrogen fixing plants to help with the uptake of nutrients in exchange for some of the plant's photosynthates (Gange & Ayres, 1999). The influence of mycorrhizae on plant  $\delta^{15}$ N is based on which of the three types of mycorrhizae are involved in the symbiotic relationship: arbuscular, ectomycorrhiza, or ericoid. Non-mycorrhizal plants and arbuscular mycorrhiza have the highest  $\delta^{5}N$  values and plants with ectomycorrhizal and

ericoid associations have lower  $\delta^{15}N$  values (Craine et al. 2009; Hobbie & Högberg, 2012). The distribution of mycorrhiza varies primarily based on the availability of organic nitrogen and net mineralization rates with each of the mycorrhiza types having a preferred environment. Arbuscular mycorrhizae are dominant in temperate grasslands and savannahs, ectomycorrhizal associations are more common in temperate forests and the taiga, and ericoid mycorrhizae are most common in bog and tundra environments (Read, 1991; Soudzilovskaia, 2017). There is also evidence that mycorrhizal associations vary based on altitude, though this area is not well-researched. The abundance of arbuscular mycorrhiza decreases with increasing altitude, ectomycorrhiza are most common in midrange altitudes (~1000-2500 masl), and ericoid abundance is limited to the distribution of plants in the family Ericaceae which prefer locations with acidic soil and low nitrogen availability such as bogs (Geml, 2017). In the Andes, this pattern would have the effect of plants having lower  $\delta^{15}$ N values with increasing altitude.

The addition of nitrogen-rich fertilizers in agricultural areas has the potential to significantly increase plant  $\delta^{5}N$ . Domestic herbivore dung (such as that from camelids) has the potential to slightly increase plant  $\delta^{15}N$  (Szpak et al., 2012a). Seabird guano has the greatest potential to alter plant  $\delta^{15}N$  by as much as 20 ‰ (Szpak et al., 2012a). There is significant evidence that points towards the use of seabird guano to fertilize agricultural crops in the Atacama Desert oases during the Late Intermediate Period and Late Horizon (Santana-Sagredo et al., 2017; Santana-Sagredo et al., 2021). However, there are currently no published isotopic data from macrobotanical remains from the coastal river valleys of northern Chile or the Arica region.

Climatic conditions such as temperature and aridity impact plant  $\delta^{15}N$  which are particularly pertinent in the Atacama region. Plant  $\delta^{5}N$  increases with temperature (Amundson et al., 2003) and increases with decreasing water availability (Austin  $\&$ Vitousek, 1998; Amundson et al., 2003; Murphy & Bowman, 2006). In the highlands of Peru and Chile, the average temperature decreases, and annual precipitation increases when moving from the Pacific coast to the highlands and therefore plants growing in the highlands should have lower  $\delta^{15}N$  than those growing at lower altitudes (Szpak et al., 2013). The highlands of northern Chile are significantly more arid than the Peruvian highlands and therefore water availability may affect plant  $\delta^{15}N$  in both the highlands and the coast.

### **3.7 Keratin**

Keratin is the main protein that comprises tissues such as hair/wool, nails, quills, horns of mammals, and the feathers, claws and beaks of birds (Wang, 2016). Keratinous tissues can be divided into two types ( $\alpha$  and  $\beta$ ) as determined by x-ray diffraction – with mammal tissues like hair and nails being  $\alpha$  -keratin (Wang, 2016). The hydrophobic qualities of  $\alpha$ -keratin make it resistant to deterioration in open environments (Lubec et al. 1987) and therefore, it can preserve in archaeological contexts presumably while maintaining the sample's isotopic integrity. Unlike collagen, which has well-established criteria for quality control (such as C:N ratios, %C and %N), there are no similar criteria for keratin. Based on established C:N ratios from sheep's wool (Boudin et al,. 2016) and modern camelid wool (Szpak et al., 2014) a C:N ratio between 3.0-4.0 are considered acceptable for this thesis. There is an extended discussion of C:N ratios in isotopic studies of keratin in section 4.9.

 The carbon and nitrogen isotope compositions of an animal's tissues reflect the average carbon and nitrogen isotope compositions of the foods consumed during the period of tissue formation (DeNiro & Epstein 1978; 1981). Therefore, the  $\delta^{13}C$  and  $\delta^{15}N$ of a herbivore's tissues are reflective of the  $\delta^{13}$ C and  $\delta^{15}$ N of the plants they consumed (Murphy & Bowman, 2006; Hartman, 2011). The  $\delta^{3}C$  and  $\delta^{15}N$  values of animal keratin are primarily representative of the protein component of the consumer's diet (Tieszen  $\&$ Fagre, 1993).

#### **3.8 Previous Isotope Studies on Camelid Fiber**

To date, only a handful of studies have employed carbon and nitrogen isotope analysis to look at camelid fiber origins in the Atacama region (Santoro et al. 2019; Szpak & Valenzuela, 2020; Gayo et al. 2020). However, using stable carbon and nitrogen isotope analysis as a method to investigate geographic origin has been employed multiple times in other Andean regions and especially in Peru. In this section, I will use previous studies from outside of Chile to outline the assumptions of this method and then discuss camelid fiber isotope studies from Chile to discuss how the Atacama may differ from other Andean environments where previous stable isotope studies have been centered.

Finucane, Arguto & Isbell (2006) conducted one of the early studies on camelid bone collagen as a part of a human diet study at the Wari site of Conchopata in Peru. They realized there was a dietary split in the camelid samples with six being consistent with a  $C_3$ -based diet and eleven being consistent with a diet based heavily on  $C_4$ resources. The authors theorized that the camelids with a C4-based diet were likely foddered with the agricultural by-products of maize whereas the  $C_3$ -based camelids

consumed local grasses in pastures. In Argentina, a 2 ‰ decrease in  $\delta^{13}C$  values was recorded in modern camelids for every 500m in altitude increase (Yacobaccio, Morales & Samec, 2009). Altitude impacts vegetation composition, the main driver being fewer  $C_4$ plants as altitude increases. These altitudinal variations in  $\delta^{13}$ C are then passed onto herbivores that graze on local flora. Although the precise number of 2 ‰ decrease in  $\delta^{13}C$ for every 500 metres altitude increase is specific to the Jujuy Province in Argentina, this study was an early demonstration that isotopic compositions vary with altitude in camelids due to changing vegetation. Thornton et al. (2011) expanded and applied this idea to Middle Horizon camelid remains in the Osmore Valley in southern Peru. Stable carbon and nitrogen isotope ratios were measured on 28 archaeological camelid bone samples from 5 sites spanning from the Middle Horizon to the late pre-Hispanic period. Twenty-three of the camelid samples had  $\delta^{13}$ C and  $\delta^{15}$ N values consistent with modern camelids that lived in highland pastures. Five camelids in the study had higher than expected  $\delta^{13}$ C and  $\delta^{15}$ N which were outside the expected range for camelids pastured in the highlands. The authors attributed these variable isotopic values to different camelid foddering practices, altitudinal herding range, and climate. Dufour et al. (2014) conducted a similar study on Early Intermediate Period (200 BC - 600AD) camelid bone collagen, structural carbonate, and molar tooth enamel to understand camelid life histories along the north coast of Peru. The authors concluded that there were camelid individuals in the sample that likely lived along the coast. From these studies, it was clearly established that camelid  $\delta^{13}$ C and  $\delta^{15}$ N varies based on foddering practices (maize foddering vs. grazing on pastures) as well as by altitude due to limitations on the altitude at which maize can grow.

Szpak et al. (2014) analyzed serially sampled camelid fiber from two Early Intermediate Period (200 BC to 800AD) sites in the Virú Valley, Peru, alongside the analysis of bone collagen. Some of these camelids were sacrificial individuals and serial sampling of their fiber allowed for a history of the period before their death. This study pointed towards the conclusion that small-scale camelid management was occurring in the Virú Valley, but perhaps more significantly, it was the first study to use camelid fiber to apply the idea that  $\delta^{13}$ C and  $\delta^{15}$ N could be used to determine the geographic origins of camelids and their by-products. Szpak et al. (2015) took this idea further by analyzing camelid fiber from textiles to determine whether the fiber recovered from archaeological sites on the north and central coasts of Peru was acquired from the highlands or coastal regions. This study focused on the origins of the fiber itself, and not the whole camelid. Szpak et al. (2015) determined camelid wool was obtained from the highlands for the central coast Chancay culture but also from sites located at lower elevations where C<sup>4</sup> plants made a significant contribution to the camelid's diet for Early Intermediate Period occupations in the Virú Valley on the north coast of Peru. This research was followed by several other studies focusing on analyzing  $\delta^{5}N$  and  $\delta^{13}C$  of camelid bones in Peru (Szpak et al., 2016a; Szpak et al., 2016b; Dufour et al. 2018; Tomczyk et al. 2018; Santana-Sagredo et al. 2018; Szpak et al., 2019b; Vasquez et al., 2020) and Argentina (Grant, 2017). Other studies focused on camelid fiber from Peru (Szpak et al., 2016b; Szpak et al., 2018; Santana-Sagredo et al., 2018; Szpak et al., 2019b). Each of these studies challenged the idea that camelid herding was an exclusively highland activity − each support the idea that camelid herding in South America (particularly in Peru) was occurring in lower altitude valleys as well as along the coast. This body of evidence

opened the possibility that camelid herding may have been occurring at low altitudes and coastal regions in areas outside of Peru and that these activities also included raising animals at low altitudes for fiber production. From these studies it is clearly demonstrated that camelids raised in the highlands have diets mostly composed of  $C_3$  plants as maize generally cannot be cultivated above  $3600$  masl (Staller, 2016) and wild  $C_4$  plants make up a small percentage of plant biomass at high altitudes, while camelids from coastal regions/lowland valleys have diets with a higher  $C_4$  plant composition as a result being foddered with maize or other  $C_4$  plants.

The first application of stable carbon and nitrogen isotopic analysis of camelid fiber in the Atacama Desert, Chile was conducted on a limited number of Late Pleistocene samples from Pampa del Tamarugal (Santoro et al. 2019). All the samples in this study were determined to be of highland origin with  $\delta^{13}$ C values ranging between  $-21.0\%$  and  $-18.2\%$  and  $\delta^{15}N$  values between 6.2‰ and 8.7‰. Gayo et al. (2020) analyzed Archaic to Late Intermediate textile samples from the coastal site of Caleta Vitor in Chile. The authors concluded that none of their fiber samples were imported from highland camelids and instead theorized that fiber was obtained from wild camelids that grazed on vegetation in the *lomas*. However, the ranges of their samples: −21.5‰ to  $-12.0%$  for  $\delta$ <sup>13</sup>C, and between +5.8‰ and +17‰ for  $\delta$ <sup>15</sup>N could indicate that their samples represent mostly highland individuals with some that have more intermediate values. Szpak and Valenzuela (2020) have generated the largest set of camelid fiber data from the Late Intermediate Period and Late Horizon in the Arica region of the Atacama Desert. They concluded that the overwhelming majority of samples were consistent with a highland origin and only a small amount of camelid herding was occurring on the coast

in the Late Intermediate and Late Periods − far less than what was able to occur in the northern valleys in Peru.

# **4. Materials and Methods**

For this study, a total of 272 camelid fiber strands underwent EA-IRMS analysis. These samples came from archaeological sites in the western valleys and coastal regions of the Atacama Desert. Site chronology has been used to attribute each of the textiles to either the Formative Period (1000 BCE – 200 CE), Middle Horizon (200 – 1000 CE), or the Late Intermediate Period (1000 – 1450 CE). Due to the relative rarity of preserved textiles in the archaeological record, the sampling strategy for the present study was constrained to whichever sites had textile materials available. Sections of textile approximately 2.5 cm in length were sampled at the University of Tarapacá in Arica, Chile and sent to Trent University to be prepared for isotopic analysis. Archaeological textile samples were obtained from 9 archaeological sites, including locations on the Pacific coast (PLM-3, PLM-4, PLM-7) as well as the coastal river valleys of Lluta (Ll-38) and Azapa (Az-6, Az-8, Az-11, Az-14, Az-Anm-6). A brief description of each site is provided below.

Due to the impromptu nature of early archaeology in Chile, some archaeological sites have limited published information. Many sites, especially those on the coast, were picked over by looters (and in some cases tourists) before any formal excavation could occur. Formal archaeological work was often done in a rush as salvage expeditions before sites could be developed for industrial and agricultural uses. In this respect many artifacts of significance were separated from their original context. Regarding the early development of archaeology in Chile, one of the pioneering archaeologists in the region, Guillermo Focacci, stated that:

"The decades of the 60s and 70s were crucial for the archaeology of Arica. Many times, we had to rush to rescue and save archaeological specimens that were being destroyed by the construction of canals, irrigation, the expansion of agricultural land, the layout of roads or subdivisions. With little support, and hostility from contractors and operators, we carried out quick salvage operations in which, many times, only scattered material was collected or shattered or, with a bit of good luck, some graves with full contexts or partially destroyed. On other occasions, with some understanding, we were given the necessary time to draw some squares and exhume...funeral bundles and offerings" (1990, 71)

Due to the limitations on early archaeology in Arica discussed by Focacci, the description of some sites discussed in this thesis are brief as detailed information and comprehensive sources are few and far between.

### **4.1. Azapa Valley**

Az-6 is a funerary site located close to the town of San Miguel de Azapa at approximately 300 meters above sea level (masl). The site was excavated in 1973 as a salvage effort before the area was to be turned into a poultry farm – parts of the site had already been destroyed by machinery before excavation could begin (Focacci, 1990). The site contains 206 graves that have been argued to be associated with the Cabuza culture group (Focacci, 1990). Focacci (1990) argues that the ceramic and textile styles at the site indicate that the people either originated from the Lake Titicaca basin, or at least had contact with the Tiwanaku. Middens at the site contained the remains of flora such as corn cobs, pumpkins, and beans, as well as the remains of fauna such as camelids, guinea pigs, fish and shellfish (Focacci, 1990). This assemblage of floral and faunal remains

suggests that the occupants of Az-6 were obtaining resources from both the highlands and the coast (Focacci, 1990). This study incorporates seven samples from this site. Based on the stylistic criteria of ceramics and textiles at Az-6, the site has traditionally been attributed to the Middle Period (Focacci, 1990). The exact chronology of Az-6 has been called into question, however, with some published radiocarbon dates consistent with the Late Intermediate Period (Korpisaari, Oinonen & Chacama, 2014) meaning there is potential that the textile samples from this site are not from the Middle Period. All samples in this thesis were attributed to a time period based on stylistic analysis of artifacts at the site where textile samples were found. Based on this, the Az-6 textile samples in this study had previously been attributed to the Middle Period based on associations with Tiwanaku-style artifacts, however, as demonstrated in Korpisaari, Oinonen & Chacama (2014) there is debate surrounding the occupational history of the Az-6 site. The fact that other samples in this thesis were categorized based on stylistic analysis coupled with the ongoing debate as to the nature of the Middle Period in the northern Atacama, the Middle Period attribution for Az-6 will be retained, keeping in mind that further radiocarbon dating of these fiber strands may turn out to be consistent with the Late Intermediate Period.

The Az-8 site is located about 14 km from the Arica coast, in the Azapa Valley. The site is located close to the town of San Miguel de Azapa (Cornejo & Fernández, 1984). Az-8 was originally excavated in 1966 by archaeologists Oscar Espoueys, Guillermo Focacci, and Percy Dauelsberg (Cornejo & Fernández, 1984). The tombs at this site contained mummified remains which were wrapped in textile bundles (Alfonso-Durruty et al., 2019). Funerary goods included harpoon heads, maize, squash, ceramic

pots, textiles, weaving tools and camelid ears and feet (Alfonso-Durruty et al., 2019). The site is located at approximately 266 masl and consists of 89 excavated tombs (Uribe, 1999). For the present study, 12 samples were collected from funerary contexts. The textiles are associated with the Late Intermediate Period.

Az-11 or the San Lorenzo site is located on the southern slope of the Azapa Valley approximately 12 km from the coast of Arica (Muñoz, 1993). Az-11 is a complex village site that is approximately 1 km long (Muñoz, 1993). Approximately 30 buildings have been excavated (Muñoz, 1993) and are described as rectangular in shape, with thatched walls that would have had poles in the corner of each room to support the ceiling (Muñoz, 1989). The houses were built upon terraces which were constructed with stone. Vegetable fibers were used to level out the terrain (Muñoz, 1989). Small storage pits inside the structures, and larger pits just outside each building contained the remains of beans (*Phaseolus vulgaris*), corn (*Zea mays*), and squash (*Lagenaria* spp.) (Muñoz, 1993). Faunal remains from either camelids, guinea pigs, or dogs (described as 'offerings') were identified in the southwest corner of the structures (Muñoz, 1993). Human burials were identified beside the terrace walls, in the central area of the site where no structures were present, and two cemeteries are located at the edge of the village (Muñoz, 1993). The remains of textiles and tools for textile production have been found at the site. Knitting tools as well as a number of textile pieces that were 'in progress' indicate textiles were being manufactured in the area (Muñoz, 1993). The site is thought to have been an administrative centre due to the complexity of the site, as well as it being situated between the coast and inland area (Muñoz, 1989). This study includes two samples from

one textile that was collected at 281 masl. This textile is associated with the Late Intermediate Period.

Az-14 is a funerary site located in the Azapa Valley at 572 masl. A total of 347 tombs were excavated at the site and it was thought to have been occupied circa 410 BCE until colonization by Europeans (Santoro, 1980). The long occupation period is attributed to the site being located close to both the ocean and the resources of the Azapa Valley (Santoro, 1980). This study includes nine samples which are associated with the Formative Period.

Az-Anm-6, also known as Parcela Las Palmas, is an unexcavated site located on the northern slope of the Azapa Valley (Valenzuela & Casanova, 2014). This study uses four samples collected at Parcela Las Palmas at ~138 masl with the addition of isotopic data from 82 samples which were previously unpublished (discussed further in section 4.7). The site is located on modern-day farmland which has caused a large amount of disturbance to the surface of the site (Valenzuela & Casanova, 2014). Based on the characteristics of the site, such as the style of tombs, rock art, and textiles, it is thought that Parcela Las Palmas dates to the Formative Period (1700/1400 BC - 300/500 AD) (Valenzuela & Casanova, 2014). The site itself is comprised of 6 sectors:

Sector 1 consists of four burial mounds (Valenzuela & Casanova, 2014). The mounds have an exposed profile that demonstrates a construction of alternating layers of stone and vegetation (Valenzuela et al. 2011a). Artifacts of significance can be seen in the exposed profiles such as textiles, ceramics, lithics, vegetable fiber mats, cordage, basketry, botanical, and fragmented human remains (Valenzuela & Casanova, 2014).

Sector 2 is a *cistas* (burial pit) cemetery located east of Sector 1 on a steep slope

of the northern side of the Azapa Valley (Valenzuela & Casanova, 2014). The sector consists of at least 12 *cistas* interspersed with fragments of basketry, textiles, and bioanthropological remains (Valenzuela & Casanova, 2014). The absence of ceramics in this sector and high instances of inhumations in baskets is typical of the late Archaic to Formative Period transition (Valenzuela et al., 2011).

Sector 3 is a petroglyph site located to the south of Sector 1 (Valenzuela & Casanova, 2014). It consists of four engraved stone blocks that have been moved from their original context by machinery and placed in alignment with a modern road (Valenzuela & Casanova, 2014). The petroglyphs are eroded which has made identification of many of the motifs difficult – however identified figures include camelids, circles, concentric circles, human figures, and birds (Valenzuela et al., 2011).

Sector 4 (central area of dispersion), Sector 5 (southern area of dispersion), and Sector 6 (eastern area of dispersion) are areas with scattered archaeological and human remains. These areas have the highest amount of disturbance from modern human interventions (Valenzuela & Casanova, 2014).

#### **4.2 Lluta Valley**

Ll-38, also known as Rosario-Petroglifos, is a rock art site located on the southern slope of the Lluta Valley approximately 20 km east of the Pacific coast (Valenzuela, Santoro & Briones, 2011). The site is at approximately 350 masl (Valenzuela, Santoro  $\&$ Briones, 2011). Ll-38 is comprised of a 400 m long volcanic outcrop which has been engraved with 69 panels of petroglyphs (Valenzuela, Santoro & Briones, 2011). Identifiable petroglyphs include depictions of camelids ( $n = 89$ ), anthropomorphic figures  $(n = 79)$ , abstracts  $(n = 68)$ , non-camelid quadrupeds  $(n = 6)$  and birds  $(n = 5)$ 

(Valenzuela, Santoro & Briones, 2011). These petroglyphs predominately date to the Late Intermediate and Late Periods, however a small number of the petroglyphs were created using engraving techniques distinct to the Middle Period (ca. 500 – 1000 CE) (Valenzuela, Santoro & Briones, 2011). Rosario-Petroglifos is referred to as an 'enclave' and it is theorized that the site was formed because it may have been (1) an area at the crossroads of trade routes for regional or interregional travel, (2) an area of high economic interest due to agricultural land, or (3) located close to an area where daily domestic activities were already being carried out (Valenzuela, Briones & Santoro, 2006). Two textile samples were obtained from Ll-38 for the present study and are thought to date to the Late Intermediate Period based on site chronology. Textiles samples from a nearby Late Intermediate/Late Period village site (Ll-36 or Rosario 2) previously underwent carbon and nitrogen isotope analysis and are detailed in Szpak and Valenzuela (2020).

#### **4.3 Southern Coast of Arica**

Playa Miller 3 (PLM-3) is one of a series of sites on the southern coast of Arica located on El Laucho Beach within the city limits of modern-day Arica. The region with the designation PLM was divided into 9 sites (PLM-1 to PLM-9) (Focacci, 1997). The Playa Miller sites were first described in 1876 by John H. Blake for a manuscript published by The American Museum of Natural History (Focacci, 1997), but the site was not formally excavated until the 1940s by Junius Bird (1946) and then again by the Regional Museum of Arica in 1960 (Focacci, 1997). In the years prior to excavation, partially exposed burials and artifacts were collected by beachgoers and looters (Focacci, 1997). Foccaci's team excavated approximately 250 tombs and estimates that there were

likely 1250 individuals buried at the site when accounting for looted graves and graves that were not excavated (Focacci, 1997). It is estimated that the site would have been occupied between 1100 CE to 1450 CE or the Late Intermediate Period (Focacci, 1997). Samples from PLM-3 used in this thesis were collected at 14 masl. This study includes 127 samples from PLM-3.

PLM-4 was a cemetery site located at 47 masl at the southern end of El Laucho beach within the city limits of modern-day Arica (Focacci, 1974). The site has since been destroyed by development of the area, however Guillermo Focacci excavated the site in 1969 and rescued 210 tombs along with their associated mummies and grave goods (Focacci, 1974). Textile samples taken from this site include 18 samples taken from funerary contexts. Chronological dating suggests the sampled textiles are associated with the Late Intermediate Period.

PLM-7, also known as Playa Miller 7, was a cemetery site located on El Laucho beach at 37 masl within the city limits of modern-day Arica (Focacci, 1974). The site was excavated in 1969 by Guillermo Focacci (Focacci, 1974). 12 samples from the PLM-7 site were used in this study. Site chronology dates these textiles to the Formative Period.

Site	Location	Elevation (masl)	Period	$n$ samples
$Az-6$	Azapa	300	Middle	7
$Az-8$	Azapa	266	<b>LIP</b>	12

**Table 4.1** Summary table of archaeological sites included in this thesis with location, elevation, time period data, and number of textil samples included in this study.



### **4.4 Sample Preparation**

Before preparing textile samples for stable carbon and nitrogen isotope analysis, each fiber sample was photographed and assigned a unique lab (TEAL) ID number. Each fiber sample was examined and factors such as colour, state of preservation, and presence of surface dirt were recorded. Aliquots of each fiber sample were weighed to approximately 6 mg and placed into labelled 100 x 13 mm glass culture tubes. Some fiber samples were too small to obtain the desired initial 6 mg sample but were still prepared for analysis so long as there was at least 1 mg of material to sample. A number of samples contained multiple strands of different colours; some samples also included intact warp and weft segments. In such cases, the warp and weft were divided and counted as separate samples, fibers of different colours were also separated and counted as separate samples, as there is a chance that these portions of the textile may contain fiber from distinct camelid individuals. To avoid contamination, tweezers, scissors, and the work area were

cleaned between each sample with acetone and compressed air.

Fiber samples underwent a chemical preparation before IRMS analysis following the hair keratin preparation procedures outlined in Hyland, Millaire & Szpak (2021). Briefly, surface contaminants were first removed by placing 10 mL of Type I water in each culture tube and placing samples in an ultrasonic bath for 30 minutes. Fiber samples were then placed in a centrifuge at 2500 rpm for 20 minutes and the Type 1 water was then removed using a pasteur pipette. Samples were treated with 8 mL of 2:1 chloroformmethanol  $(v/v)$  and placed in an ultrasonic bath for 30 minutes in order to remove any lipids present in the hair. Samples were then centrifuged at 2500 rpm for 20 minutes and the chloroform:methanol was removed from each tube with a glass pasteur pipette. The samples then received a second rinse in 2:1 chloroform-methanol  $(v/v)$  following the same procedures outlined above for a total of 2 rinses in chloroform:methanol. Samples were then rinsed in 10 mL of Type I water and placed in an ultrasonic bath for 30 minutes to remove any remaining solvent. Samples were centrifuged at 2500 rpm for 20 minutes and waste water was decanted into a waste beaker. Samples received a second rinse in Type I water following the same procedures outlined above for a total of 2 rinses in Type I water. Following chemical preparation, the fiber samples were left in a fume hood for approximately 36 hours to dry.

Once dry, each sample was homogenized by chopping the strand into small pieces with a razor blade. Because spun textile strands may have fiber from multiple individuals, homogenizing the sample is meant to ensure all fibers in the spun strand will be more equally sampled. The razor blade and working area was cleaned with acetone and compressed air between each sample to avoid cross contamination. Homogenized samples were placed into tin capsules in aliquots weighing between 420 - 480 μg.

#### **4.5 Isotope Ratio Mass Spectrometry**

Carbon and nitrogen isotopic compositions of keratin samples were determined at the Laboratory for Stable Isotope Science at the University of Western Ontario, using a DELTA V (Thermo Scientific) Isotope Ratio Mass Spectrometer coupled to a Costech 4010 Elemental Analyzer. Stable carbon and nitrogen isotopic compositions were calibrated relative to the international reference scales VPDB and AIR using a two-point calibration with the international standards USGS40 ( $\delta^{13}$ C = -26.39 ± 0.04 ‰,  $\delta^{15}$ N = - $4.52 \pm 0.06$  ‰; Qi et al., 2003) and USGS41a ( $\delta^{13}$ C= +36.55  $\pm$  0.08 ‰,  $\delta^{15}$ N = +47.55  $\pm$ 0.15 ‰; Qi et al., 2016) weighed between 570 - 640 μg. Measurement uncertainty was monitored using the in-house standards SRM-1 (caribou bone collagen;  $\delta^{13}C = -19.39 \pm$ 0.09 ‰,  $\delta^{15}N = +1.84 \pm 0.12$  ‰) and SRM-14 (polar bear bone collagen;  $\delta^{13}C = -13.67$  $\pm 0.08$  ‰,  $\delta^{15}N = +21.62 \pm 0.18$ ‰) weighed between 420 - 480 μg. Approximately 10% of samples were analyzed in duplicate in order to gauge analytical uncertainty.

### **4.6 Analytical Uncertainty**

Calculations of analytical uncertainty for the fiber samples followed the methods and calculations outlined in Szpak et al., 2017. Precision  $(u(Rw))$  was determined to be  $\pm$ 0.10 ‰ and  $\pm$  0.20 ‰ for  $\delta$ <sup>13</sup>C and  $\delta$ <sup>15</sup>N respectively, based on repeated measurements of calibration standards, check standards, and sample replicates. Systematic error (*u(bias)*) was determined to be  $\pm$  0.09 ‰ for  $\delta^{13}$ C and  $\pm$  0.25 ‰ for  $\delta^{15}$ N based on the difference between observed and known values of the check standards. The total analytical uncertainty was estimated to be  $\pm$  0.13 ‰ for  $\delta^{13}$ C and  $\pm$  0.32 ‰ for  $\delta^{15}$ N.

# **4.7 Additional Data Set**

This research includes an additional unpublished data set consisting of 82 Formative Period textile samples from Az-Anm-6. Textile samples were prepared in 2018 by Paul Szpak following the methods outlined in Szpak and Valenzuela (2020). Briefly, the textile samples were cleaned of any visible surface dirt using a dental pick. Samples were sonicated for 60 minutes in Type I water, centrifuged, and the type I water was drained. The samples were then treated with 2:1 chloroform-methanol (*v/v*) for 60 minutes and remaining chloroform-methanol was drained. Samples were then air-dried. The carbon and nitrogen isotopic compositions were determined using a Nu Horizon continuous flow isotope ratio mass spectrometer coupled with a EuroEA 3000 elemental analyzer located at the Trent University Water Quality Centre. Samples were calibrated using USGS40 ( $\delta^{13}$ C = –26.39 ± 0.04 ‰,  $\delta^{15}$ N = –4.52 ± 0.06 ‰; Qi et al., 2003) and USGS41a ( $\delta^{13}$ C= +36.55 ± 0.08 ‰,  $\delta^{15}$ N = +47.55 ± 0.15 ‰; Qi et al., 2016) or USGS66  $(\delta^{13}C = -0.67 \pm 0.04 \%, \delta^{15}N = +40.83 \pm 0.06 \%,$  Schimmelmann et al. 2016).

### **4.8 Comparative Data**

To better contextualize the textile data produced for this thesis,  $\delta^{13}C$  and  $\delta^{15}N$ measurements of bone collagen from 8 archaeological highland camelids were included. These samples were from the Pumapunku complex located in the Lake Titicaca basin, approximately 350 kilometers northeast of the modern city of Arica. The camelid bones date to c. 1400-1532 CE. These samples have previously been described in Knudson, Gardella & Yaeger (2012) where <sup>87</sup>Sr/<sup>86</sup>Sr as well as carbonate  $\delta^{13}$ C and  $\delta^{18}$ O have been measured. In order to make the collagen data directly comparable to the hair keratin data from the textiles, the collagen data was adjusted by  $-1.40$  ‰ for  $\delta^{13}$ C and  $-0.86$  ‰ for

 $\delta^{15}$ N (O'Connell et al., 2001).

<b>Sample</b>	$\delta^{13}C$	$\delta^{15}N$	<b>Adjuste</b> d $\delta^{13}C$	<b>Adjuste</b> d $\delta^{15}N$	$\%C$	$\%N$	C: N
PUMA C2552	$-17.50$	8.69	$-18.90$	7.83	38.49	15.21	2.95
<b>PUMA C3515</b>	$-17.60$	8.72	$-19.00$	7.86	39.32	15.63	2.94
PUMA C3704	$-19.33$	6.92	$-20.73$	6.06	40.47	15.96	2.96
PUMA C4247	$-18.47$	6.40	$-19.87$	5.54	35.55	14.00	2.96
PUMA C4607	$-18.55$	8.30	$-19.95$	7.44	35.33	13.90	2.96
PUMA C4715	$-19.67$	6.21	$-21.07$	5.35	38.59	15.28	2.95
<b>PUMA C4819</b>	$-16.88$	8.34	$-18.28$	7.48	41.42	16.55	2.92
PUMA C6208	$-18.00$	6.56	$-19.40$	5.70	38.88	15.35	2.95

**Table 4.2** Highland Pumapunku complex camelid bone collagen  $\delta^{13}$ C and  $\delta^{15}$ N isotopic compositions

### **4.9 Data Treatment**

To assess the presence of different isotopic groupings in the textile data set, an unweighted pair group method with arithmetic mean (UPGMA) cluster analysis was conducted using Paleontological Statistics (PAST 4.06) (Hammer, Harper & Ryan, 2001). The cluster analysis resulted in a dendrogram that grouped similar data into clusters on the basis of Euclidean distance. The clusters created can potentially point towards groups of camelids that were from distinct environments. This method gives a more statistically robust delineation of isotopic groupings than a strictly qualitative interpretation ('calibrated eyeball' approach) of a standard  $\delta^{13}$ C vs.  $\delta^{15}$ N scatterplot.

Quality control criteria are not as clearly established for hair keratin as they are for bone collagen. Atomic C:N ratios, such as the range of 2.9-3.6 for bone collagen outlined by DeNiro (1985), are a common method for determining sample integrity. However, there are no quality control parameters for hair keratin that have been experimentally validated and those that do exist are rough generalizations that have not been explicitly shown to correlate with unusual or modified isotopic compositions. As hair keratin is a relatively pure protein, the presence of hair in archaeological context suggests relatively unaltered stable isotope ratios as any diagenetic processes would likely quickly lead to the complete degradation of the hair strand. Nonetheless, C:N ratios are currently the most used quality control parameter for hair keratin. Sheep wool that has an atomic C:N ratio between 3.4 to 3.8 is considered acceptable in some radiocarbon studies (Boudin et al., 2016). However, it has been demonstrated that modern camelid fiber has C:N ratios between 3.10 and 3.45 (Szpak et al., 2014) and it is therefore unreasonable to eliminate camelid fiber samples based on a C:N ratio of another species. Szpak and Valenzuela (2020) suggest eliminating camelid fiber samples with a C:N ratio greater than 4.0. Based on 452 previously-published archaeological camelid fiber samples from Peru and Chile, it was determined that samples with a C:N ratio over 4.0 had consistently low  $\delta^{13}$ C and high  $\delta$ <sup>15</sup>N values (Szpak & Valenzuela, 2020). Drawing from these sources, I have excluded camelid fiber samples with C:N ratios above 4.0 from my interpretations.

### **Chapter 5: Results**

# **5.1 Sample Integrity**

Due to the concerns of high C:N ratios brought up in Szpak and Valenzuela (2020), I eliminated samples with a C:N ratio over 4.0 as their isotopic compositions may have been altered in the burial environment. In total, 11 samples were eliminated from this thesis dataset. An additional 23 samples were eliminated for the same reason from the existing Formative Period data set from Az-Amn-006 that has been included in this study (Figure 5.1). These eliminated data are presented in Appendix A. Based on the comparison of the elemental and isotopic compositions presented in Figure 5.1, the samples with C:N ratios over 4.0 did not generally possess especially high or low  $\delta^{13}$ C or  $\delta$ <sup>15</sup>N values, suggesting that they may not have been altered in the burial environment and the quality control criterion suggested by Szpak and Valenzuela (2020) may be too conservative for camelid keratin.



**Figure 5.1** Stable carbon and nitrogen isotope compositions of camelid fiber samples included in this study classified according to their atomic C:N ratios. Samples with a C:N ratio over 4.0 were eliminated from this study.

# **5.2 Camelid Fiber Stable Isotope Compositions**

Plotting the textile isotopic data suggested the presence of some groups (Figure 5.2). Within this dataset, there is a clear group of textiles with high  $\delta^{13}C$  and high  $\delta^{15}N$ whereas the majority of samples fell into a group with relatively low  $\delta^{13}$ C and low  $\delta^{15}$ N. Between these two groups there are a number of samples with more intermediate  $\delta^{13}C$ and  $\delta^{15}$ N values. Finally, there is a small group of samples with low  $\delta^{15}$ N but moderate to high  $\delta^{13}$ C values. Individual sample  $\delta^{13}$ C and  $\delta^{15}$ N compositions from this thesis are presented in supplementary materials (Appendix A).



**Figure 5.2**  $\delta^{13}$ C and  $\delta^{15}$ N values for all textile fibers included in this study

In order to better define these groups, the samples in this thesis were categorized with a cluster analysis (Appendix B). The cluster analysis confirmed and defined the rough groupings from Figure 5.2 – Group A (n=5) with comparatively high  $\delta^{13}C$  and  $\delta^{15}N$ values, Group B (n=15) with intermediate  $\delta^{13}$ C and  $\delta^{15}$ N values, Group C (n=215) with low  $\delta^{13}$ C and  $\delta^{15}$ N, and the small Group D (n=3) with high  $\delta^{13}$ C and low  $\delta^{15}$ N (Figure 5.3).



**Figure 5.3** Amended scatter plot with convex polygons surrounding data points of a grouping as defined by the cluster analysis.

# **5.3 Comparison to Previously Published Data**

Textile isotope data from this thesis was plotted alongside previously published textile stable isotope data from Szpak and Valenzuela (2020), as well as the adjusted highland camelid collagen data from Pumapunku. The camelid collagen data from Pumapunku was adjusted by  $-1.4\%$  for  $\delta^{13}$ C and  $-0.86\%$  for  $\delta^{15}$ N to make it comparable to keratinous tissues (O'Connell et al., 2001).



**Figure 5.4** All camelid fiber  $\delta^{13}C$  and  $\delta^{15}N$  compositions from this study (blue dots), with camelid fiber  $\delta^{13}C$  and  $\delta^{15}N$  from Szpak and Valenzuela, 2020 (yellow squares) and adjusted highland camelid bone collagen  $\delta^{13}C$  and  $\delta^{15}N$  from the Pumapunku complex (red triangles).

Figure 5.4 demonstrates that the highland camelid isotope compositions from Pumapunku plot in the same region as Group C defined by the cluster analysis. The majority of data points from Szpak and Valenzuela 2020 also plot within the range of Group C. The remaining 10 data points from Szpak and Valenzuela (2020) which were interpreted by the original authors as originating outside the highlands (high  $\delta^{13}$ C and  $\delta^{15}$ N), plot within the range of group B though they seem to have somewhat higher  $\delta^{13}$ C values. Samples from Group A were all obtained through the data produced in this study and have the highest  $\delta^{15}$ N values of previously published archaeological camelid fiber in the Arica region.

# **5.4 Isotope Compositions over Time**



**Figure 5.5** Textile  $\delta^{13}$ C and  $\delta^{15}$ N compositions compared to the time period attributed to each sample

Samples from Group A are entirely attributed to the Late Intermediate Period. Group B is composed of a mixture of Late Intermediate and Formative Period samples. Group C, which accounts for the majority of samples, has samples attributed to all periods represented in this study. Notably, all samples from the Middle Horizon (n=7) are contained within Group C. All samples from Group D are attributed to the Formative Period.

Box and whisker plots were generated for both the  $\delta^{15}N$  and  $\delta^{13}C$  data in this thesis to visually represent any change in isotope compositions over time. Figure 5.6 appears to demonstrate an incremental positive increase in mean  $\delta^{13}$ C values from the Formative Period through to the Late Intermediate Period. Figure 5.7 appears to demonstrate slight changes in mean  $\delta^{15}N$  over time, however mean  $\delta^{15}N$  appears to remain relatively constant through-out each time period.



**Figure 5.6** Box plots of  $\delta^{13}C$  values for the time periods represented in this thesis



**Figure 5.7** Box plots of  $\delta^{15}N$  values for the time periods represented in this thesis

Further statistical tests were conducted in order to better understand any potential differences in isotope compositions between time periods. The results of a Shapiro-Wilk test on  $\delta^{13}$ C of each category indicate that the data from each time period group are not normally distributed (Table 5.1). A Shapiro-Wilk test for  $\delta^{15}N$  data of each time period category indicates the Formative and Late Intermediate Period data are not normally distributed (Table 5.1), however the data for the Middle Period are normally distributed  $(W = 0.9383, p = 0.6234)$ . The results from a series of Mann-Whitney U tests indicate that the only time periods with a statistically significant difference in  $\delta^{13}$ C was the Formative and Late Intermediate Periods ( $U = 4551.5$ ,  $p = 0.001698$ ). Mann-Whitney U tests indicated there were no statistically significant changes in  $\delta^{15}N$  between any of the time periods included in this thesis.



**Table 5.1** Shapiro-Wilk test statistics for Formative, Middle and Late Intermediate Period data presented in this thesis

**Table 5.2** Mann-Whitney U test statistic comparisons for  $\delta^{13}$ C and  $\delta^{15}$ N data for Formative, Middle and Late Intermediate Periods. U-statistics are presented in the lower left boxes and pvalues are presented in the upper right boxes.



Table 5.3 indicates the relative proportion of samples in each time period that fall into each of the cluster analysis groupings. From these data it appears that there is an approximately equal proportion of samples of non-highland origin (i.e., those falling outside of Group C) in the Formative and LIP; there are too few samples from the Middle Period to make any substantive statements about these data.

<b>Period</b>	<b>Total</b>	<b>Group A</b>		<b>Group B</b>		<b>Group C</b>		<b>Group D</b>	
	N	$(n=5)$		$(n=15)$		$(n=215)$		$(n=3)$	
		N	$\%$	N	$\%$	N	$\%$	N	$\%$
<b>Formative</b>	75	0	$\theta$	4	5.3	68	90.7	3	4.0
<b>Middle</b>	⇁	$\overline{0}$	$\overline{0}$	$\overline{0}$		$\mathbf{r}$	100	$\overline{0}$	$\theta$
<b>LIP</b>	156		3.2	11	7.1	140	89.7	$\overline{0}$	

**Table 5.3** Percentage of samples from a given period that fall into each cluster analysis group

### **5.5 Site Location**

In order to understand spatial distribution of stable isotope compositions from site to site, Figures 5.8 was generated to compare archaeological site and site type to the resulting  $\delta^{13}$ C and  $\delta^{15}$ N compositions. The samples in Group A were all obtained from coastal sites (PLM-3, PLM-4, and PLM-7). Samples from Group B and Group C were from a variety of archaeological sites located on the coast and in the western Valleys. Finally, all samples in Group D were obtained from the valley site of Az-Anm-6.



**Figure 5.8** Simplified plot denoting whether the sample was discovered on a coastal beach site or from a site in the marginal valleys

A Shapiro-Wilk test was conducted to determine whether valley and coastal site isotopic data were normally distributed. For both coastal and valley sites, the  $\delta^{15}N$ (W=0.90, p<0.001 for valley sites; W=0.87, p<0.001 for coastal sites) and  $\delta^{13}C$  (W=0.78,  $p<0.001$  for valley sites; W=0.72,  $p<0.001$  for coastal sites) data was not normally distributed and therefore a Mann-Whitney U test was conducted to determine whether there were statistically significance differences between isotopic data from valley and coastal sites. There were no statistically significant differences in  $\delta^{13}$ C for valley and coastal sites (U=5838, p=0.15), and there were no statistically significant differences in  $\delta$ <sup>15</sup>N for valley and coastal sites (U=5977.5, p=0.25).

#### **6. Discussion**

#### **6.1 Quality Control**

Of the 11 samples that were eliminated from the analysis due to having C:N ratios over 4.0, 8 had C:N ratios that were unusually high for animal fiber (Table 6.1). Animal fiber is composed primarily of proteinaceous materials, so it is expected to have a low C:N ratio since most amino acids themselves have atomic C:N ratios under 5. This indicates that the camelid fibers may have been mixed with plant fiber (potentially cotton) or the entire strand itself was mis-identified as camelid fiber. Plant fibers have a higher C:N ratio than animal fiber which is driven by the low abundance of nitrogen in plant materials. Szpak and Chiou (2020) found an atomic C:N ratio of 17.7 for charred cotton seed and fluff from the archaeological site of San Jose de Moro on the north coast of Peru. For eight desiccated cotton fiber samples from Cerro de Oro on the south coast of Peru, Gonzalez (2022) found an atomic C:N ratio of 95±23. The low, but highly variable nitrogen content in plant fiber compared to relatively invariant carbon content causes a large array of C:N ratios so there is no fixed C:N ratio to reference for plant materials (Gonzalez, 2022). Beyond the C:N ratios indicating these fibers are not (entirely) composed of animal fiber, the isotopic data are meaningless due to the unknown mixture of materials as we are unable to tease apart whether the plant or animal components are driving the isotope values. Despite not being able to interpret the isotopic data, the C:N ratios indicate that small numbers of textiles from the Formative and Late Intermediate Periods may contain mixed materials.

From these data, it is difficult to interpret why the people of the Arica region may have been mixing fibers. The mixing of plant and camelid fibers could be a tactic to make smaller amounts of highly-valuable camelid fiber 'go further' when spinning yarn –

therefore getting more textile material using less camelid fiber. This could potentially be due to certain weavers having minimal access to camelid fiber or to keep costs lower when creating new textiles.

<b>Sample</b>	<b>Site</b>	<b>Period</b>	<b>Composition</b> estimate	C: N
11139	PLM-3	Late Intermediate	Plant	245.81
11479	PLM-3	Late Intermediate	Plant	202.89
11240	PLM-3	Late Intermediate	Plant	287.18
11499	$Az-Anm-6$	Formative	Plant	158.56
11228	$Az-14$	Formative	Plant	88.48
11141	PLM-3	Late Intermediate	Potentially mixed	62.48
11223	$Az-14$	Formative	Potentially mixed	24.73
11227	$Az-14$	Formative	Potentially mixed	42.40

**Table 6.1** Fiber Samples that may be partially or entirely plant material based on C:N ratios

# **6.2 Likely Geographic Origins of Camelid Fiber**

The resulting isotopic data and cluster analysis indicated the presence of four groupings of textiles based on the isotopic data (Figure 5.3), the likely geographic origins of which are discussed below.

Group A is a cluster of points (n=5) with especially high  $\delta^{15}N$  and  $\delta^{13}C$  values compared to the other groupings (Figure 5.3). The high  $\delta^{15}$ N and  $\delta^{13}$ C values are consistent with camelids that lived outside of the highlands during the period of hair growth as highland environments in the Andes tend to be less dry relative to lower altitudes and have few  $C_4$  plants (Szpak et al., 2013). The high  $\delta^{13}C$  values are consistent with consumption of  $C_4$  grasses and the high  $\delta^{15}N$  values are likely the result of the
camelids grazing in an arid environment and/or the consumption of fertilized plants. However, the  $\delta^{15}N$  values of Group A are even higher than expected values for nonhighland camelids based on previous data from the northern Chilean valleys. Szpak and Valenzuela (2020) observed comparably high  $\delta^{13}$ C values for a small percentage of Late Intermediate Period and Late Horizon camelid fiber textiles from the Lluta and Camarones Valleys, but the  $\delta^{15}N$  values observed in this study were much higher. The most parsimonious explanation for these high stable isotope values would be that domestic camelids were foddered with crops that had been fertilized with seabird guano – an explanation that has been used to account for high crop and consumer  $\delta^{15}N$  in the past (Szpak et al., 2012a; Szpak et al., 2012b; Santana-Sagredo et al., 2021; Pinder et al., 2019). The application of manure to crops can lead to markedly high  $\delta^{15}N$  (Bogaard et al., 2007; Bogaard et al., 2013). Growth chamber experiments on crop plants demonstrated that the application of seabird guano can increase plant  $\delta^{15}$ N by as much as 20–40‰ (Szpak et al. 2012a) and further field experiments in Peru demonstrated llama dung fertilizer only increases maize  $\delta^{15}N$  values by 1.8–4.2 ‰, whereas seabird guano can increase plant  $\delta^{15}N$  by as much as 11.3–20 ‰ (Szpak et al., 2012b). From this research it is evident that guano fertilizer can cause markedly high  $\delta^{15}N$  values, like those observed in Group A. If further analysis were to be done, high  $\delta^4$ S values in these textiles would also be consistent with guano fertilization as this practice increases the  $\delta^{4}S$  values of crops (Szpak et al., 2019a) and would in turn result in high  $\delta^{13}C$ ,  $\delta^{15}N$ , and  $\delta^{4}S$  values in animals consuming those crops.

An alternate explanation for high camelid  $\delta^{15}$ N compositions which is sometimes cited in the literature is the idea that camelids may have been foddered with seaweed.

This explanation is attributed to DeNiro (1988), and finds some ethnographic support as discussed in Masuda (1985). This notion of seaweed-eating camelids was likely also influenced by the long-held practice of using seaweed as fodder for sheep on the island of North Ronaldsay in the Orkney Islands, which has received an extensive examination through isotopic analysis of modern and archaeological sheep remains (Balasse et al. 2005, 2019; Balasse, Tresset &Ambrose, 2006; Balasse, Mainland & Richards, 2009; Schulting et al. 2017; Blanz et al., 2020; Guiry & Szpak, 2020). However, there is simply no concrete evidence that foddering with seaweed occurred in the pre-Columbian Andes. Moreover, the  $\delta^{15}$ N values of marine plants in this region are *lower* than those of plants living at low altitudes and coastal locations (Szpak et al., 2013). Although outside the scope of this research,  $\delta^{34}$ S compositions of camelid tissues could be collected to further pursue this avenue of inquiry and perhaps put it to rest. Another potential method to investigate marine contribution to camelid diet would be paired radiocarbon dates with camelid  $\delta^{15}$ N and  $\delta^{13}$ C to investigate offsets and therefore marine contribution to diet (Santana-Sagredo et al., 2017). Although there is a dearth of evidence to support foddering camelids with seaweed, there is an abundant and growing body of evidence to support the use of seabird guano fertilizer in the Andean region. Ethno-historic accounts (De Bibar & Leonard, 1966; De San Miguel, 1966; De la Vega, 1970; Frézier, 2014) as well as archaeological evidence (Szpak et al., 2012b; Pinder et al., 2019; Santana-Sagredo et al., 2021) indicating that the use of seabird guano is currently the strongest explanation for especially high camelid  $\delta^{15}$ N in the Andean region.

The samples in Group B have relatively high  $\delta^{15}N$  and  $\delta^{13}C$ , however they are not as high as the extremely high values demonstrated in Group A (Figure 5.3). The samples

in Group B have similar values as those identified as originating outside the highlands in Szpak and Valenzuela (2020) (Figure 5.4). The Group B samples may also have been from camelids that were foddered with maize products and raised outside the highlands. However, the lower  $\delta^{15}N$  values of Group B compared to Group A indicate that the fodder given to these camelids was likely not fertilized with seabird guano but instead may have been fertilized with less <sup>15</sup>N-enriched fertilizers such as camelid dung. In the case of Group B, the high  $\delta^{15}N$  values are likely to have been driven by the aridity of the region (Amundson et al., 2003; Murphy & Bowman, 2006) rather than fertilizer.

In the case of Group B, it should also be noted that it cannot be guaranteed that each fiber sample is composed of fiber from one camelid individual. That is to say, the samples in Group B may be a mixture of fiber from different individuals. If this is the case, the more 'intermediate' values of Group B can also be explained by a mixture of fiber from high  $\delta^{15}N$  and  $\delta^{13}C$  camelids (similar to Group A) with fiber from camelids with comparatively low  $\delta^{15}N$  and  $\delta^{13}C$  (similar to group C). A mixture of fiber from these two groups could potentially 'average out' resulting in the intermediate values observed in Group B.

The overwhelming majority of samples included in this thesis fall into Group C, which has relatively low  $\delta^{13}$ C and  $\delta^{15}$ N values when compared to Groups A and B (Figure 5.3). The lower  $\delta^{13}C$  of Group C are consistent with camelids that consumed C<sub>3</sub> grasses, and the lower  $\delta^{15}$ N values indicate these camelids lived in a less arid region during the period of fiber growth. The comparative samples from the highland Pumapunku complex also plot within the range of Group C samples (Figure 5.4). Group C, when compared to previous studies and highland bone collagen, is consistent with camelids of highland

origin. The majority of samples in this study fall into the highland group, which indicates that although there was some small-scale camelid herding occurring outside of the highlands, people outside the highlands continued to engage in networks of exchange with the highlands through the time periods included in this thesis.

The final group, Group D (n= 3), has low  $\delta^{15}N$  (6.30% to 8.32%) but high  $\delta^{13}C$  (-14.35‰ to –15.89‰). It was a somewhat unexpected group and a dietary regime that has not been identified previously in camelids in the Arica region. Group D samples have  $\delta$ <sup>13</sup>C compositions consistent with animals that grazed on C<sub>4</sub> plants (i.e., they were potentially foddered with maize products), however, the lower  $\delta^{15}N$  compositions point to the idea that these individuals may have lived in a less arid region during the period of hair growth. These camelids are unusual in that maize agriculture is not usually possible in the highlands due to the altitude, and therefore these camelids would likely have been from a region that is less arid than the Atacama, but also at a low enough altitude to support maize agriculture. Although it is not impossible this camelid fiber was from a distant less-arid region, camelid caravans could only travel at a rate of about approximately 25 km per day, with long breaks needed for longer treks, for example a 400 km trek would have taken 2-3 months (Browman 1990; Nielsen 2001; Tripcevich, 2007). Another compounding factor against this interpretation is that all individuals in Group D are attributed to the Formative Period – a period before agricultural intensification occurred in the region – making a heavily maize-based diet less likely.

An alternative explanation for the unique isotopic compositions of Group D could be that the low  $\delta^{15}N$  values were caused by camelids that grazed on the near-shore  $C_4$ grass *Distichlis spicata* at low altitudes. *D. spicata* (commonly called *gramma salada*) is

recorded to have low  $\delta^{15}$ N values even in arid regions (Szpak & Valenzuela, 2020). Szpak et al. (2013) measured and averaged the  $\delta^{13}$ C and  $\delta^{15}$ N values of five *gramma salada* plant collected at 10 masl in northern Peru. The plant had a  $\delta^{13}$ C value of -14.9 ‰, and a  $\delta$ <sup>15</sup>N value of –3.2 ‰. Arriaza et al. (2001) mentions the presence of large amounts of *gramma salada* in an area of the Arica coast called the *Chimbas* (meadows), which is a 10 kilometer long strip along the coast, positioned near the mouth of the Lluta River. Santoro et al. (2019) mentioned the presence of *gramma salada* along the banks of rivers along the Pampa del Tamarugal transect to the south of this area of study as well as near salt flats. The geographic range of *gramma salada* mentioned in these studies is based on where the plant has been discovered in the modern context and as such it is possible that the areas where *D. Spicata* grow may have shifted over time – however it is clear that the species grows along the coast, in riparian environments, as well as desert salt flats. If this interpretation is correct is suggests a range of foddering strategies for camelids in this region, including provisioning them with fertilized maize in the lower valleys and allowing them to graze on fields of *gramma salada* along the coast. Additional data from other isotope systems (e.g.,  $\delta^{4}S$  or  ${}^{87}Sr/{}^{86}Sr$ ) (*see* Frei et al., 2009) combined with  $\delta^{13}C$ and  $\delta^{15}$ N data would help in clarifying the dietary regime of Group D camelids.

# **6.3 Isotopic Groups and Time Period**

When discussing the temporal patterns it is first important to recognize that the large number of samples in Group C, which are attributed to all time periods in this study, demonstrate that throughout all periods, camelid fiber in the Arica region was consistently being obtained from the highlands. Group C represents 90% of the Formative Period samples, 100% of the Middle Period samples, and 90% of the Late Intermediate

Period samples. This indicates that the two regions were engaged in either direct trade or at least in networks of interaction throughout the ~2500 year span represented by samples in this thesis.

The samples in Group B (n=15) are attributed both to the Formative Period and Late Intermediate Period. This indicates that some camelid management was occurring outside the highlands during these periods – though the Late Intermediate Period is better represented in Group B. This could indicate that it was easier for the people in the Arica area to manage camelids during the Late Intermediate Period compared to earlier periods. It should be noted that any differences in relative quantities of non-highland samples in different time periods could be a product of sample size from each period and the relative rarity of camelids falling outside of Group C (low  $\delta^{13}$ C and  $\delta^{15}$ N) in general. There were no Middle Period samples in Group B, but this could also be due to a sampling issue as only seven samples attributed to this period were available for analysis.

Group A, with comparatively high  $\delta^{13}$ C and very high  $\delta^{15}$ N values, is composed completely of samples attributed to the Late Intermediate Period. The interpretation that high sample  $\delta^{15}$ N is due to the application of seabird guano to fodder crops makes sense during this period. In more southern archaeological sites, the practice of using seabird guano fertilizer seems to have taken off during the Late Intermediate Period in most locations (Santana-Sagredo et al., 2021; Pinder et al. 2019), with some evidence of guano fertilizer use as early as the Middle Period (Santana-Sagredo et al., 2021). Herding camelids outside the highlands during the Late Intermediate Period may have been more practical due to agricultural intensification occurring during the time.

Group D (n=3) is composed completely of samples attributed to the Formative Period. It is difficult to discuss temporal trends with this group as it is so small but at this point it appears to be a practice that was limited to the Formative Period. This is an interesting isotopic pattern which perhaps could represent a regional form of camelid management that occurred at a small scale in this region during the Formative Period. In this vein, Group D could indicate small-scale camelid herding with very specific regional, village, or even family-specific, camelid husbandry practices.

Only about 10% of samples in each period appear to be sourced from camelids attributed to having lived outside the highlands (Table 5.3). Previous stable isotope analysis of Arica region samples had 12.7% of Late Intermediate Period samples being non-highland and 12.9% of samples from the Late Period being non-highland in origin (Szpak & Valenzuela, 2020). From these data is seems that during any given period, occupants of the Atacama were only able to acquire 10-15% of their fiber from nonhighland (possibly local) sources. There appear to be small increases in the percent of fiber used from non-highland camelids over time, but this may be due to sample size. Nonetheless, the idea that the percentage of non-highland fiber is relatively stable over time is intriguing. This data, however does not speak to overall quantity of fiber, only proportion.

Broadly speaking, these data exemplify consistent trade or at least a network of interaction between the Arica region and the highlands. That being said, Group A and B are evidence for camelid fiber being produced outside of the highlands during the Formative Period and Late Intermediate Period. It is interesting that there appears to be camelid herding outside the highlands as early as the Formative Period (as indicated by

Group B and D), however compared to later data, the non-highland samples from the Formative Period indicate that seabird guano was not yet used in agriculture in the Arica Region. The high  $\delta^{15}N$  values of some samples in the Late Intermediate Period not only point to camelids being reared outside the highlands, but also demonstrates innovations in agriculture such as the adoption of seabird guano fertilizer. These 'ultra-high'  $\delta^{15}N$ samples from the Late Intermediate Period coincide with and support previously proposed theories that some level of agricultural intensification was occurring during the Late Intermediate Period in northern Chile. While this has previously been demonstrated for the desert oases further to the south in the Atacama (Santana-Sagredo et al., 2021), this is the first evidence that suggests the use of seabird guano in the northern valleys of Chile.

The results of Mann-Whitney U tests showed a statistically significant difference in  $\delta^{13}$ C values between the Formative and Late Intermediate Periods (Table 5.2) and Figure 5.6 clearly demonstrates a steady increase in  $\delta^{13}$ C values between these two periods. An increase in camelid  $\delta^{13}$ C values indicates a shift towards a more heavily C<sub>4</sub>based diet between the Formative and Late Intermediate Periods. What would have allowed for this dietary shift would be an increase in maize production in the Arica region, and therefore an increase in maize consumption by camelids. This points towards the previously-theorized agricultural intensification during the Late Intermediate Period to the south also occurring in the Arica region – and that the increase in camelid consumption of maize products may have dovetailed with an increase in both production and consumption of maize among humans in the Arica region. Due to there only being 7 samples from the Middle Period included in this study, it is likely that the lack of statistically significant differences in  $\delta^{13}$ C values between the Formative and Middle

Periods and the Middle and Late Intermediate Periods is due to small sample size. Further studies with a larger sample size of Middle Period textile samples would further elucidate whether statistically significant shifts in  $\delta$ <sup>13</sup>C occurred between the Formative and Middle Periods, therefore indicating agricultural intensification after the Formative, or whether shifts in  $\delta^{13}$ C, and therefore agricultural intensification, did not occur until the Late Intermediate Period in the Arica region.

The results of Mann-Whitney U tests demonstrated that there were no statistically significant differences in  $\delta^{15}N$  values between any of the time periods represented in this study (Table 5.2) with Figure 5.7 also demonstrating relatively stable  $\delta^{5}N$  values between periods. This is somewhat surprising as Group A samples all appear to have especially high  $\delta^{15}N$  values which would be congruent with increased use of seabird guano fertilizer during the Late Intermediate Period. Furthermore, previous studies on human stable isotope values from Tarapacá (to the south of Arica) indicated an increase in  $\delta^{15}$ N values in inland human remains, potentially linked to guano use, as early as the Middle Period (Santana-Sagredo et al., 2021). In contrast, coastal individuals in the same study did not have the same increase in  $\delta^{5}N$  values when compared to Formative Period individuals (Santana-Sagredo et al., 2021). Other research has observed <sup>15</sup>N-enrichment in plants during the Late Intermediate Period (Pinder et al, 2019). The lack of any statistically significant differences in  $\delta^{15}N$  values between periods in this study, yet the presence of the 5 samples in Group A with comparatively high  $\delta^{15}N$  values, potentially indicates that while there was seabird guano use in the Late Intermediate Period, the practice was constrained such that it was not common enough to cause demonstrable shifts in  $\delta^{15}$ N values. There is evidence that seabird guano was not evenly distributed

throughout agricultural areas within a community, and it is theorized that the practice was socially constrained with only some members of a community having access to the resource (Santana-Sagredo et al. 2021). This social-constraint and limited use of seabird guano may explain why dramatic shifts in  $\delta^{5}N$  values are not observed across time periods in this study.

# **6.4 Location**

The high  $\delta^{13}C$  and  $\delta^{15}N$  Group (Group A) were all uncovered at sites located in close proximity to the coast (Figure 5.8). Group A consists of the samples for which it is suspected that camelids were foddered with maize products that had been fertilized with seabird guano. This suggests that sites directly on the coast could have had more steady access to seabird guano fertilizer during the Late Intermediate Period relative to sites located slightly further inland. Groups directly on the coast may have controlled this valuable resource, possibly prohibiting certain people/families/villages from gaining access to the islands where seabird guano accumulated. It is notable that all samples tied to guano-use were acquired right on the coast, however more evidence is required to make assertions about the level of control coastal groups exerted over seabird guano as a resource.

Group B contains samples both from valley and coastal sites which indicates that camelid herding outside of the highlands was possible and occurring throughout the Arica region – both on the coast and in the valleys (Figure 5.8). Group C samples were also recovered from a mix of coastal and valley sites which again indicates that both these zones were connected continuously to the highlands through trade networks. The fact that the people who lived in the marginal valleys and on the coast continued to rely on the

highlands for their camelid products indicates that they were likely limited in terms of producing enough fiber for textiles. In this vein it may be that connections with the highlands were a necessity to obtain enough goods to sustain life in the Atacama. Alternatively, the continued trade relationships between the coast, valley, and highlands may represent an interregional exchange system with larger social and cultural importance (discussed further in section 6.4).

All samples in Group D were recovered from the valley site Az-Anm-6 (Figure 5.8). This may represent a regional and time-specific form of foddering where camelids could graze on *gramma salada* along the coast and along riverbanks. However, if the alternate theory is true, that the camelids were from a distant and less arid region, uncovering the fiber in a valley site would merely indicate the area being in a network of trade with other areas. Thornton et al. (2011) observed similar  $\delta^{13}$ C and  $\delta^{15}$ N values to the Group D camelids at Torata Alta, a Late Horizon archaeological site located at 2,500 masl in the low sierra of the Osmore Drainage region of southern Peru. This is not to imply that this region is the geographic origin of the Group D fibers, but it does demonstrate that these isotopic compositions have been observed in camelids recovered at archaeological sites in relatively close geographic proximity.

The results of a Mann-Whitney U test comparing  $\delta^{13}$ C values of fiber found at coastal sites versus valley sites indicated that there were no statistically significant differences (Table 5.4). Likewise, the Mann-Whitney U test comparing  $\delta^{15}N$  values of fiber found at coastal versus valley sites also indicated there were no statistically significant differences (Table 5.4). This suggests that there were no large differences in the ways in which people directly on the coast, and those in the valleys obtained their

camelid fiber. Throughout time people in the valleys and on the coast were likely involved in similar networks of trade with the highlands and may in fact be indicative that people living directly on the coast and in the western valleys represent a single societal group.

# **6.5 Notions of Status**

It is, in some ways, difficult to determine whether the presence of camelid herding on the coast would have been a marker of status or rather an attempt by lower status individuals to gain limited access to a resource reserved for mostly wealthy individuals. As a resource, camelids are costly to maintain in the Atacama where there is little natural vegetation for grazing and water is scarce, however it also requires substantial resources to transport camelid fiber from the highlands to the coast.

The trade in other resources – specifically tropical Amazonian birds and their feathers – can potentially give some insight in this area. Capriles et al. (2021) sampled the mummified remains of 15 tropical parrots which were included as grave goods from various sites in the Atacama Desert – namely Pica 8, Camarones 8, and a site included in this thesis, Az-6. Tropical bird feathers were symbols of prestige and status in the pre-Columbian Atacama and were often used to make elaborate headdresses (Capriles et al., 2021). Tropical feathers became increasingly more common in the Atacama Desert during the Late Intermediate Period which is thought to be related to the increase in movement caused by llama caravans that connected communities along the coastal Atacama and the marginal valleys (Capriles et al., 2021). The authors aimed to determine whether parrot feathers were brought from the forests east of the Andes, or whether

tropical birds were brought across the Andes while still alive and reared locally. Physical evidence signaled these birds may have been kept domestically on the coast during their lives – such as two birds with leather straps on their legs, three birds that had the tip of their beaks cut off, evidence of clipped or plucked wing feathers, and overgrown beaks and claws (Capriles et al., 2021).

Through stable isotope analysis the authors identified three distinct dietary regimes for the birds. Most notable was the group with comparatively high carbon and nitrogen isotope values (analogous to the Group A camelid fibers in this study) which points to the birds consuming maize that was likely fertilized with seabird guano (Capriles et al., 2021). These results indicate not only that there were complex trade routes between the Atacama, highlands, and Amazon but also that tropical birds were moved hundreds of kilometers from the Amazon and raised domestically in the Atacama during the Late Intermediate Period as evidenced by the dietary stable isotope evidence.

This research, in combination with camelid fiber data indicates that at least in the Late Intermediate Period, there was a desire to maintain high-status resources outside the highland regions of the Atacama. This also coincides with previously noted agricultural intensification during the Late Intermediate Period which is linked to the usage of seabird guano fertilizers (Santana-Sagredo et al. 2021). Seabird guano as a resource was also not evenly distributed throughout agricultural areas within a community and it is theorized that it was an item of prestige as only some members of a community had access to the resource (Santana-Sagredo et al. 2021). The linkage of camelid fiber in Group A to seabird guano use indicates that the same individuals who had access to guano during the Late Intermediate Period also had the resources to maintain camelids in marginal and arid

environments. This indicates that community members with access to seabird guano were able to maintain a variety of prestige resources – such as the aforementioned tropical parrots, as well as camelids. From this evidence it seems likely that the ability to maintain camelids in the Arica area was in fact an indication of prestige, although links to the highlands appear to still be significant during the Late Intermediate Period. The continual coast-highland trade in camelid goods also parallels the Amazonian parrots - as there was seemingly a continual supply of new parrots entering the Atacama from the Amazon. This suggests that local husbandry needed to be supplemented with new birds from the tropical rainforests, which implies the people of the Atacama may not have been able to breed the parrots very successfully even if husbandry activities were occurring. This mirrors the camelids in that the continual supply of camelid fiber arriving from the highlands may be indicative that it was difficult to successfully breed large numbers of camelids on the coast even if people had the capability to maintain them.

Perhaps beyond the scope of what isotopic evidence can tell us, but worth exploring, is the idea that the constant exchange of goods between the coast, valleys, highlands, and even the Amazon rainforest were exchange networks with greater social and cultural importance than the mere exchange of goods. Marcel Mauss (1957) theorized that in economic systems, the exchange of goods is rarely just an exchange between individuals, but rather there are hierarchies and collectives that "impose obligations of exchange and contract upon each other (1957; pg. 7) and economic transaction is only one element of exchange. That is to say that networks of exchange are not only predicated on consolidating wealth through exchange but also work to build alliances and social solidarity between groups. Although Mauss' analysis was not focused on the Andean

region, it is worth considering whether the networks of exchange in northern Chile represent more than mere trade, but perhaps demonstrate the political and social relationship between trading groups. The presence of Amazonian parrots in the Atacama demonstrates the dynamic nature of the region and the complex trade relationships between different ecological areas. From this we begin to see a picture of a dynamic region with seabird guano fertilizer being acquired from the coast, camelids and their products traded from the highlands, and parrots brought from the tropical forests. These data demonstrate that the people of the Atacama were exploiting resources from all over the region in such a way to increase agropastoral production and bolster prestige in marginal environments.

# **6.6. Contributions to Knowledge, Limitations, and Future Directions**

This study has expanded the data set of stable carbon and nitrogen isotope measurements of camelid fiber in the Arica region to time periods that had not been studied. Previously, Szpak and Valenzuela (2020) conducted isotopic analysis on Arica camelid fiber samples from the Late Intermediate and Late Periods. This study has expanded the time periods investigated with samples from the Middle Period and Formative Period and the addition of more samples from the Late Intermediate Period. This research suggests that camelid management outside the highlands appears to have been occurring in the Arica region as early as the Formative Period. Further research into the antiquity of camelid management in Arica is merited and it would be interesting to investigate whether there is evidence to support camelid management (or at least the extraction of camelid resources) outside the highlands during the even-earlier Archaic Period.

The findings of this study further corroborate the conclusion that despite the aridity of the Arica region, people were still able to maintain small herds of camelids outside the highlands. The findings of this thesis also support the notion that camelid herding, although occurring outside the highlands, was a small-scale activity and that networks of interaction between coastal and marginal valley communities and the highlands remained important throughout all time periods in this study. Lastly, my findings further corroborate the importance of seabird guano as a fertilizer, as well as it being a practice that seems to start during the Late Intermediate Period which coincides with previously proposed agricultural intensification thought to be occurring during the time further to the south in the desert oases. The data suggests that seabird guano, although important for the acceleration of agriculture during the Late Intermediate Period, was not necessarily widely-used which suggests the potential for some kind of social constraint over access to the resource. Alternatively, crops fertilized with guano may have been mostly used for human consumption and not directed towards supporting camelid herds.

There are some interpretive limitations in this study that are worth mentioning. First, the use of seabird guano fertilizer is only evident because of its remarkable effects on  $\delta^{15}$ N values and from this we can assume that the resource would have been a prestige item. That being said, there is potential that there were other more significant markers of status that do not have any effect on isotopic ratios, and therefore our understanding of status is limited in this study to practices that impact isotopic compositions. This is, however, a larger problem with archaeological research employing stable isotope analysis and is not limited to this study.

Another significant limitation is that samples are currently only attributed to

broader time periods and further dating work is required to understand the temporal aspect of camelid husbandry in more detail. There may have been a more specific temporal context to certain sample groups (for example Group A) that may have been occurring in a constrained time but it is difficult to tell with classifications based on larger time periods alone.

A factor that limited interpretations in this study is that spun textile fibers are essentially bulk samples where the fiber of many camelid individuals – and their distinct isotopic compositions – could be mixed together into a single sample. It is therefore difficult to make claims about specific camelid management practices that may have been occurring, especially in Group B where it may represent non-highland individuals that were fed non-guano crops or alternatively could merely represent a mixture of fiber from camelids belonging to Groups A and C.

The final limitation worth stating is the dearth of samples from the Middle Period. A lack of samples from the Middle Period makes it difficult to ascertain whether the temporal trends observed in this study were consistent through-out time periods or perhaps if camelid management outside the highlands was a more discontinuous practice. For example, studies of human stable isotope values from Tarapacá indicated an increase in  $\delta^{15}$ N values in inland human remains, potentially linked to guano use, as early as the Middle Period (Santana-Sagredo et al., 2021). Coastal individuals in the same study did not have the same increase in  $\delta^{15}$ N values when compared to Formative Period individuals (Santana-Sagredo et al., 2021). Contrasting research has observed no significant shifts in <sup>15</sup>N-enrichment in plants until the Late Intermediate Period (Pinder et al, 2019). Although these studies focused on archaeological sites to the south of the Arica

area, it is clear that the Middle Period is important for understanding the possible origins of seabird guano use in the Arica area which hinges on whether samples belonging to Group A would also occur in the Middle Period if more samples were analyzed.

#### **7. Conclusions**

This research sought to provide a better understanding of camelid fiber acquisition in the Arica region of the Atacama Desert from the Formative to Late Intermediate periods (circa 1000 BCE – 1450 CE). Carbon and nitrogen isotopic analysis of fiber segments from archaeological textiles resulted in a dataset which further elucidated the question of whether camelid fiber was being acquired locally in the occidental valleys and coast of northern Chile or whether fiber was traded into the area from the highlands.

This research resulted in the identification of four broad groupings of camelid fiber based on the  $\delta^{13}$ C and  $\delta^{15}$ N data. Two of these groups, Groups A (n=5) and B (n=15), are consistent with animals that were raised outside the highlands. The high  $\delta^{15}N$ values in these groups were likely driven by the hyper-arid environment of the Atacama outside the highlands. Furthermore, the most likely explanation for the especially high  $\delta$ <sup>15</sup>N values demonstrated by Group A is the application of seabird guano fertilizer to crops that were used to fodder local camelids. Groups A and B also had high  $\delta^{13}$ C values compared to the other groups which suggests they were being foddered with maize. Maize agriculture is difficult in highland environments and therefore a heavy maize diet indicates these camelids were likely raised outside the highlands in environments better suited to maize agriculture. Just over 90% of samples included in this thesis fell into Group C ( $n=215$ ). Group C is consistent with camelids that were raised in highland environments – with comparatively lower  $\delta^{13}$ C and  $\delta^{15}$ N values. Group C camelids also plotted in the same area as comparative camelid collagen samples from the highland Pumapunku complex, further confirming these samples were from camelids originating in the highlands. The final grouping, Group  $D(n=3)$ , was a group of Formative Period

camelids with low  $\delta^{15}N$  (6.30‰ to 8.32‰) but high  $\delta^{13}C$  (-14.35‰ to -15.89‰). These camelids appeared to have isotopic compositions consistent with animals that were foddered with  $C_4$  plants but lived in a region that was not arid enough to cause high  $\delta^{15}N$ values. Due to the difficulties in growing maize in the less-arid highlands, an alternate theory for Group D's isotopic values is that they were raised near the coast and grazed on the wild C<sup>4</sup> plant *D. spicata*. This is perhaps a more likely explanation than Formative Period camelids consuming maize as agricultural intensification in the Arica region is thought to have occurred closer to the Late Intermediate Period. From this analysis it is clear that small amounts of camelid fiber  $(\sim 10\%)$  in the Arica region were being sourced from locally raised camelids, however, the highlands remained an important location for obtaining fiber through-out all periods represented in this study. The data from this thesis suggest that although there was a small number of fiber-producing animals in the northern Chilean valleys, these regions were not able to support the same level of camelid husbandry as the more productive valleys in northern Peru.

Of the non-highland samples in Group A and B ( $n=20$ ), 80% were associated with the Late Intermediate Period and 20% were associated with the Formative Period. Most notably, all of the samples in Group A were associated with the Late Intermediate Period. The onset of use of seabird guano fertilizer is thought to be one of the driving forces behind agricultural intensification in the Arica region and it therefore makes sense that all the samples with especially high  $\delta^{15}N$  values, thought to associated with the use of seabird guano fertilizer, are associated with the Late Intermediate Period when agricultural intensification has been theorized to have been occurring. Furthering the idea of agricultural intensification, the data in this thesis demonstrated a statistically

significant increase in  $\delta^{13}$ C values between the Formative and Late Intermediate Periods, which points to an increase in maize consumption by camelids between these two periods. There were no statistically significant differences in  $\delta^{15}$ N compositions between time periods in this thesis which suggests that, although seabird guano may have been used on crops in the Late Intermediate Period, the use of guano was not widespread enough to cause an overall increase in  $\delta^{15}N$  values through time. This idea tracks with previous studies that have suggested the harvest and use of seabird guano in the Arica region had restrictions on who had access to the resource. This study was only able to include 7 samples from the Middle Period and therefore much is still unknown about whether seabird guano was in use during this period. Further research on Middle Period samples may elucidate whether seabird guano use and agricultural intensification in the Arica region occurred before the onset of the Late Intermediate Period.

Previous studies on the presence of Amazonian parrots and their feathers in the Arica region points to complex networks of trade which allowed these prestige items to be available far outside the natural range of the parrots. Isotopic evidence suggests that not only were parrot feathers traded into the Atacama Desert, but eventually live parrots were raised in captivity in the Arica region – as evidenced by them having a maize-based diet with  $\delta^{15}$ N values that pointed towards the use of seabird guano to fertilize the maize used to feed the birds. In this scenario, the presence of Amazonian parrots in the Arica region are essentially analogous to camelids raised outside the highlands – whereby, especially in the Late Intermediate Period, people of the marginal valleys and coast were able to maintain small amounts of prestige items in their local environs. This suggests that the agricultural boom caused by the uptake in use of seabird guano fertilizer was the

linchpin which allowed for parallel increases in camelid herding outside the highlands as well as the maintenance of prestige items such as Amazonian parrots. From this data it appears that the Late Intermediate Period represents the confluence of guano use, agricultural intensification, camelid husbandry, and vast networks of interregional trade.

The C:N ratios of 11 fiber samples were higher than the values expected for degraded keratin which indicated some textiles were composed entirely of plant fiber or in some cases potentially a mixture of plant and animal fiber. Interpretations in this realm are limited due to the lack of established quality control criteria for keratin samples. This area of inquiry is further obfuscated by the fact that there are no studies that link slightly high keratin C:N ratios (e.g., around 4.0) with altered  $\delta^{15}$ N and  $\delta^{13}$ C values and we therefore cannot be certain that these higher keratin C:N ratios have any bearing on whether samples have experienced diagenetic alteration of their isotopic compositions.

Conducting stable isotope analysis on camelid fiber has strengthened the argument that small herds of camelids were maintained outside the highlands in the marginal regions of the Atacama Desert. Despite this, it is evident that people living on the coast of Arica and in the marginal valleys were almost certainly engaged in trade with groups living at higher altitudes. Throughout the time periods discussed in this thesis, camelid fiber was one product involved in these highland-coast trade relationships. Although there are significant ecological differences between the coast and highlands, trade in resources between these two zones would have been relatively easy due to the proximity of the locations. Further isotopic analysis of camelid fiber and bone collagen samples from the northern Atacama would further illuminate the extent of camelid herding outside the highlands in the Arica region. These additional data may overturn

some of the interpretations presented in this thesis, as there is currently a dearth of comparative stable isotope samples from camelids that lived in the Arica highlands. Despite this, this research demonstrates the extreme adaptability of camelids in how they were able to live in such a diverse range of environments prior to the arrival of the European colonizers.

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## **Appendix A: Stable isotopic and elemental data**



Table A1: Summary table of all stable isotopic and elemental (C:N ratio) data used in this thesis.











<b>Sample</b>	<b>Textile</b>	<b>Site</b>	Period	$\delta^{13}C_{VPDB}$	$\delta^{15}N_{\rm Air}$	% C	% N	$C:$ Natomic
436	367	Az-Anm-6	Formative	$-21.24$	9.37	37.40	10.80	4.04
441	372	Az-Anm-6	Formative	$-19.27$	12.12	38.50	9.60	4.68
442	373	Az-Anm-6	Formative	$-19.14$	9.88	40.40	10.40	4.53
443	374	Az-Anm-6	Formative	$-18.81$	10.10	39.70	11.00	4.21
477	408	Az-Anm-6	Formative	$-18.34$	11.86	39.70	11.30	4.10
478	409	Az-Anm-6	Formative	$-17.92$	12.64	44.10	11.80	4.36
479	410	Az-Anm-6	Formative	$-17.68$	12.14	45.70	12.00	4.44
480	411	Az-Anm-6	Formative	$-19.94$	9.17	44.20	11.80	4.37
481	412	Az-Anm-6	Formative	$-18.92$	8.44	43.20	11.50	4.38
482	413	Az-Anm-6	Formative	$-20.10$	10.56	45.40	10.90	4.86
483	414	Az-Anm-6	Formative	$-19.78$	8.89	42.50	11.50	4.31
484	415	Az-Anm-6	Formative	$-18.87$	9.57	42.10	11.70	4.20
485	416	Az-Anm-6	Formative	$-18.56$	10.65	38.40	10.10	4.43
486	417	Az-Anm-6	Formative	$-19.45$	10.71	38.20	10.50	4.24
487	418	Az-Anm-6	Formative	$-18.93$	9.72	40.10	11.40	4.10
488	419	Az-Anm-6	Formative	$-18.92$	9.10	41.40	11.70	4.13
490	421	Az-Anm-6	Formative	$-18.92$	10.15	40.00	11.10	4.20
491	422	Az-Anm-6	Formative	$-19.08$	10.30	41.80	11.30	4.31
492	423	Az-Anm-6	Formative	$-19.03$	10.76	40.40	11.10	4.24
504	435	Az-Anm-6	Formative	$-20.68$	8.69	31.60	9.20	4.01
510	441	Az-Anm-6	Formative	$-20.66$	8.77	27.20	7.70	4.12
511	442	Az-Anm-6	Formative	$-20.46$	11.92	44.90	10.30	5.08
513	444	Az-Anm-6	Formative	$-20.80$	9.28	39.30	11.40	4.02
11139	106.4	PLM-003	LIP	$-22.32$	$-2.01$	42.21	0.20	245.81
11141	108.4	PLM-003	LIP	$-22.73$	7.32	42.46	0.79	62.48
11165	137.3	PLM-003	LIP	$-20.97$	8.01	89.85	15.22	6.89
11223	191.2	$Az-014$	Formative	$-22.74$	21.32	41.55	1.96	24.73
11225	194.3	AZ-014	Formative	$-18.78$	9.20	45.58	13.17	4.04
11227	196.2	$Az-014$	Formative	$-22.06$	18.70	42.47	1.17	42.40
11228	196.3	$Az-014$	Formative	$-20.71$	4.23	42.50	0.56	88.48
11240	570	PLM-003	LIP	$-22.41$	$-1.04$	43.21	0.18	287.18
11479	106.4	PLM-003	LIP.	$-22.63$	3.75	40.83	0.23	202.89
11497	644.2	Az-Anm-006	Formative	$-20.80$	11.94	21.40	5.50	4.54
11499	645.2	Az-Anm-006	Formative	$-24.98$	6.90	41.28	0.30	158.56

A2: Summary of textile data eliminated from this thesis due to having C:N ratios over 4







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3913 ORD.:

CON ... MEXO

REF.: Carta de 01.09.2018 con solicitud de salida de muestras arqueológicas (Ingreso CMN Nº 5418 de 10.08.2018).

MAT.: Autoriza salida del país de 444 muestras arqueológicas provenientes de colecciones depositadas en la Universidad de Tarapacá, Región de Arica y Parinacota.

SANTIAGO, 25 SEP 2018

## SRA. DANIELA VALENZUELA REYES A: ARQUEÓLOGA

## DE: SRA. SUSANA SIMONETTI DE GROOTE SECRETARIA (S) DEL CONSEJO DE MONUMENTOS NACIONALES

Junto con saludarla, damos respuesta a su carta citada en la referencia, que solicita autorizar el traslado fuera del país, para la realización de análisis de radiocarbono AMS (Accelerator Mass Spectrometry) y de isótopos estables de 15N y 13C, de un conjunto de 444 muestras arqueológicas provenientes de colecciones arqueológicas depositadas en la Universidad de Tarapacá, Región de Arica y Parinacota.

Las muestras provienen de 22 sitios arqueológicos localizados en el valle de Azapa (Azapa 11, Azapa 14, Azapa 6, Azapa 70, Azapa 8 y Las Ánimas-6), valle de Lluta (Lluta 19, Lluta 21, Lluta 36, Lluta 38, Lluta 43, Lluta 54 y Lluta 93), valle de Camarones (Poblado Huancarane 1 y Conanoxa Este 7), costa de Arica (Morro 2, Playa Miller 3, Playa Miller 4, Playa Miller 7 y Quiani 7) y costa de Vítor (Caleta Vitor 6), en la Región de Arica y Parinacota, y del sitio Quebrada Maní-32 de la Región de Tarapacá.

Algunas de las muestras provienen de sitios que fueron excavados mediante salvataje e investigaciones sistemáticas entre las décadas de 1960 y 1990 por investigadores del Museo Arqueológico San Miguel de Azapa de la Universidad de Tarapacá. Otras muestras provienen de sitios arqueológicos

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que fueron recolectados y excavados con autorización de este Consejo, entre los años 2009 y 2012 en el marco de proyectos Fondecyt y Conicyt.

Los análisis de radiocarbono AMS serán efectuados en el Laboratorio de la empresa DirecAMS, en Bothell, Washington, Estados Unidos y en el Laboratorio NSF Arizona AMS Facility, de la Universidad de Arizona, Estados Unidos. Los análisis isotópicos se efectuarán en el Water Quality Center en la Universidad de Trent, Peterborough, Ontario Canadá.

Estos análisis se realizarán en el marco del proyecto Fondecyt Nº 1151046 "Consumo integral de animales en la prehistoria del extremo norte de Chile: Una perspectiva interdisciplinaria a través de diferentes líneas de evidencias (ca. 1000 a 1400 d.C.)" del cual usted es investigadora responsable.

En consideración a que los restos que serán sometidos a análisis destructivos corresponden a mínimas cantidades, menores a 10 gramos, de elementos de origen animal, de origen vegetal, y fragmentos óseos humanos descontextualizados, este Consejo autoriza la salida del país de las muestras detalladas a continuación:



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FV: Fibra Vegetal (cestería); FL Fibra de Lana (hilados de textiles); FA: Fibra Algodón; VE: Vegetal (madera, maíz, calabaza); CA: Carbón; MA: Malacológico; ZO: Zooarqueológico o fauna (Óseo, Plumas, cuero); HU: Fragmentos de huesos humanos descontextualizados.

Las muestras serán trasladadas mediante un servicio de correo internacional, adecuadamente embaladas en tubos y bolsas debidamente identificados, acompañados con la tabla resumen y la copia del presente documento.

Una vez se hayan realizado los análisis correspondientes, se deberá remitir a este Consejo el o los informes resultantes de las investigaciones a las que pudiesen haber sido sometidas las muestras en cuestión, en un plazo no superior a dos años de emitido el presente documento.

Saluda atentamente a usted, NUMENT SUSANA SIMON DE GRÓOTE SECRETARIA (S) DEL DE MONUMENTOS NACIONALES  $CC.$ **CHILE** × Sr. Héctor González C., Director Departamento de Antropología, UTA. Sr. Álvaro Romero G., Encargado (s) OTR de Arica y Parinacota, CMN.  $\mathcal{L}$ Archivo CMN Adjuntos: -GD con fotografías de las muestras. **LUXEG/ARG/SRA** CMN-ARQUEO N°665/2018 Consejo de Monumentos Nacionales<br>Tel: +56 2 2726 1400<br>Vicuña Mackenna 84 Providencia, Chile

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