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$\delta^{13}C$ and $\delta^{15}N$ in organic residues of Patagonia pottery. Implications for studies of diet and subsistence strategies among late Holocene hunter-gatherers

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ABSTRACT

This paper reports the results of stable isotope studies ($\delta^{13}C$ and $\delta^{15}N$) performed on organic residues in archaeological potsherds recovered from diverse Patagonian environments in Argentina. The objective was to identify the types and provenance of food cooked in ceramic vessels, thus contributing to the study of paleodiets and subsistence strategies among hunter-gatherer groups in Patagonia during the late Holocene. The sample included forty-six potsherds recovered from forest (n = 21), steppe (n = 15) and Atlantic coast (n = 10) sites located in different latitudes of the continental Argentine Patagonia. Our data indicates that the type of food processed in the ceramic vessels may have depended on the availability of resources in each environment. Lower-than-expected $\delta^{13}C$-values likely suggest the cooking of animal fat, while $\delta^{15}N$-values are good indicators of the environmental origin of the cooked resources. This is a reconnaissance study that seeks a broad inter-environmental comparison in order to understand the processing and consumption of food after 1000 years BP, when pottery was adopted by Patagonian hunter-gatherer groups.

1. Introduction

Since the stable isotope composition of animal tissue is determined by their diet (Ambrose et al., 2003), carbon and nitrogen stable isotope studies have been widely used for dietary reconstruction of animals as well as past human populations (Ambrose and DeNiro, 1986; DeNiro and Epstein, 1978; Newsome et al., 2004; Schwarcz, 1991; Tieszen, 1991). Archaeology incorporated these analyses to the study of a variety of issues, such as the introduction of corn, animal and plant domestication processes, social differentiation, breastfeeding and the timing of weaning among human groups, among others (Ambrose et al., 2003; Eerkens et al., 2011; Fogel et al., 1989; Schwarcz et al., 1985).

Hastorf and DeNiro (1985) were the first to apply C and N isotope analyses to the study of organic residues in ceramic vessels and potsherds in order to reconstruct the composition of the food cooked in the vessel, which provides direct evidence of pottery use (Boyd et al., 2008; Heron and Evershed, 1993; Rice, 1996). Whereas the C and N isotope composition of human remains is the result of the average diet of an individual during the last decade of their life —including both raw and cooked food that may or may not have been prepared/cooked in a particular vessel (Beehr and Ambrose, 2007), carbonized organic residues derived from the combustion of food provide direct information on the last foods cooked in the pot (Skibo, 1992). Thus, both types of analyses are complementary and allow expanding knowledge about the diet of past populations.

$\delta^{13}C$-values of carbonized organic residues depend on the biomolecular composition (protein, carbohydrates and lipids) of the resources processed, as well as on the mix of resources cooked, cooking time and the relative contribution of C by each type of food (Hart et al., 2012, 2009, 2007; Lovis et al., 2011). In contrast, $\delta^{15}N$-values derive only from the protein source, and for this reason they constitute a robust indicator of the trophic level of the processed organism (Craig, 2004; Craig et al., 2007).

Ceramic technology was adopted by Patagonian hunter-gatherer groups ca. 1000 years BP, but it constitutes a rare find in Patagonian archaeological sites, especially in high latitudes (Bellelli, 1980;

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Cassiodoro, 2008; Castro et al., 2003; Cordero and March, 2013; Gradin and Aguerre, 1991; Schuster, 2014; Senatore, 1996; Vitores, 2010). Since cooking in ceramic pots requires less attention than using baskets with heated stones, the incorporation of ceramic technology might have reduced the time and work involved in cooking (Eerkens, 2001). This "extra time" could be then used for other tasks, such as childcare and gathering of plants and tubers (Eerkens, 2001). Furthermore, the ability to boil food allowed early Patagonian pottery users to structurally alter animal products, improve their nutritional quality, and efficiently extract bone fat (Lupo and Schmitt, 1997). This strategy might have been important in environments where the main prey species have lean meat, such as guanaco (*Lama guanicoe*) in the steppe and huemul (*Hippocamelus bisulcus*) and pudú (*Pudu puda*) in the Patagonian forest.

There are several hypotheses about the emergence and main uses of pottery, but most of them underline its role in increasing the efficiency of fat extraction in the steppe (Cassiodoro, 2008) and plant processing in the coast (Gómez Otero et al., 2014) in the context of demographic growth (Favier Dubois et al., 2009; Gómez Otero et al., 2014; Goñi, 2010) and social complexity (Gómez Otero et al., 2014).

Stable isotope composition and chemical analyses of organic residues in archaeological Patagonian pottery have shown that these vessels were used to process a wide range of terrestrial and marine animal and vegetable resources (Cassiodoro and Tessone, 2014; Cordero and March, 2013; Gómez Otero, 2007; Gómez Otero et al., 2014; Schuster, 2014; Stoessel et al., 2015). Thus, pottery use may have varied according to the particular situation and habitats occupied by each group. The possibility to determine the specific use given to ceramic vessels offers avenues for exploring different issues in regard to hunter-gatherers and their relationship with resources and environments (Eerkens, 2007; Heron and Evershed, 1993), particularly now that the systematic analyses of modern plants and faunal bone collagen have made available a detailed isotope ecology for Patagonia encompassing all of the biomes in the region (Barberena et al., 2011, 2009; Gómez Otero, 2007; Tessone, 2010; Tessone et al., 2014, among others).

The objective of this paper is to determine the type and environmental origin of resources cooked in ceramic vessels in order to contribute to the discussion about the adoption and use of ceramic technology among Patagonian hunter-gatherers. For this purpose, we analyzed ceramic fragments recovered from the steppe and the forest and coastal sherds available in the "Padre Manuel Jesús Molina" regional museum. We evaluate the variations in δ^{13}C and δ^{15}N-values of organic residues in the light of the available Patagonian isotope ecology and human paleodiet isotope indicators, in order to discuss what was cooked in these vessels. This is the first study to undertake a comparative analysis of potsherd organic residues in a large spatial scale in the region, including the three main environments of Continental Patagonia.

2. Study areas

2.1. Environments

The Continental Argentine Patagonia comprises the provinces of Neuquén, Río Negro, Chubut and Santa Cruz (Fig. 1). From an isotope ecology point of view, the region can be divided into three ecological units, from west to east: the Andes mountain range forest, the steppe and the Atlantic coast. The steppe is the largest of these units, since it occupies the wide expanse of land that lies between the Andes range and the Atlantic coast. (See Fig. 2.)

The forest area included in our study is the mountain sector southwest of Río Negro and northwest of Chubut. This broken landscape is the product of Andean orogeny and glaciations that excavated lake basins and interconnected glacial valleys. The prevailing vegetation is a mixed *Nothofagus* and *Austrocedrus* forest that occupies the band from the 1500 mm isohyet up to the mountain ranges to the east, where rainfall decreases to 500 mm (Fernández and Carballido Calatayud, 2015).

The steppe is represented by the western-central section of Santa Cruz, divided into highland sectors, located above 900 m.a.s.l. (plateaus of Cardiel Chico and Pampa del Asador), and lowland sectors (Lakes Salitroso-Posadas) (Belardi et al., 2013; Goñi, 2010). Highland sectors receive slightly more rainfall (200–400 mm/year) than the lowlands (100–270 mm/year). The steppe is characterized by shrub and grass plant communities (Goñi, 2010).

Finally, the northern coast of Santa Cruz is characterized by an arid to semiarid climate —200 mm/year— with precipitations highly concentrated in the winter. South of the Deseado river estuary there are sand dunes and aeolian mantles on terraces (Hammond, 2014). The vegetation is characterized by shrub steppes composed of grasses and *coirones* (*Stipa humilius* and *S. speciosa*) (Hammond, 2014).

2.2. Isotope ecology

The analysis of C and N stable isotope composition of plants and animal bone collagen has advanced knowledge of the isotope ecology in the Patagonian steppe, Andean forest and Atlantic coast (Barberena et al., 2011, 2009; Fernández and Tessone, 2014; Gómez Otero, 2007; Tessone, 2010; Tessone et al., 2014, among others). C_3 plants dominate all the vegetation communities, including local plant species suitable for human consumption (Gómez Otero, 2007); C_4 vegetation is limited to a few species of little dietary importance for herbivores (Fernández and Panarello, 1991). Accordingly, δ^{13}C-values of herbivores reflect a predominantly C_3-plant diet (Barberena et al., 2009; Gómez Otero, 2007; Tessone et al., 2014). Steppe species such as guanaco and choique (*Pterocnemia pennata*) present higher δ^{13}C and δ^{15}N-values than forest herbivores such as cervids (Barberena et al., 2011; Fernández and Tessone, 2014; Tessone, 2010). In turn, coastal guanacos and choiques present higher δ^{15}N-values than their inland counterparts (Barberena, 2002; Favier Dubois et al., 2009; Gómez Otero, 2007; Martínez et al., 2009). This may be related to ^{15}N-enrichment of plants growing in soils that are more saline or exposed to sea spray (Gómez Otero, 2007). Finally, marine resources such as pinnipeds (*Otaria flavescens*), penguins (*Spheniscus magallanicus*) and cormorant (*Phalacrocorax* spp.) show the highest δ^{13}C and δ^{15}N-values (Gómez Otero, 2007; Gómez Otero et al., 2014).

We present a compilation of δ^{13}C and δ^{15}N-values of Patagonian animal and plant resources representative of the three areas of interest from the perspective of the resource catchment of archaeological sites (Tables 1–3), as a frame of reference for comparison with organic residue stable composition data from ceramic vessels. Therefore, these groups of data are not intended to represent the animal and plant communities of the three environments, but rather groups of resources readily accessible from the archaeological sites in each habitat type. For example, the area with the best representation of samples corresponds to inland of Santa Cruz, presents large amount of sample per species and spatial scale associated with provenance of ceramics (Table 2). On the other hand, forest area has isotopic values of local resources (huemul, Caviidae and *Ctenomys* sp.) but as a reference of the steppe animal prey, we use samples from the inland of Santa Cruz (Table 1). Finally, due to the low number of available samples, in the Atlantic coast we chose to pool coastal data in three broad categories: terrestrial resources, marine fish, and other marine resources, that include pinnipeds and birds. These values come from archaeological sites along the entire Atlantic coast from a larger spatial scale where the ceramics were recovered (Table 3).

2.3. Archaeological background

The Andean forest archaeological sites in our study area are, from north to south: Población Anticura, Paredón Lanfré and Campamento Argentino, all located in the lower Manso River Valley (Province of Río

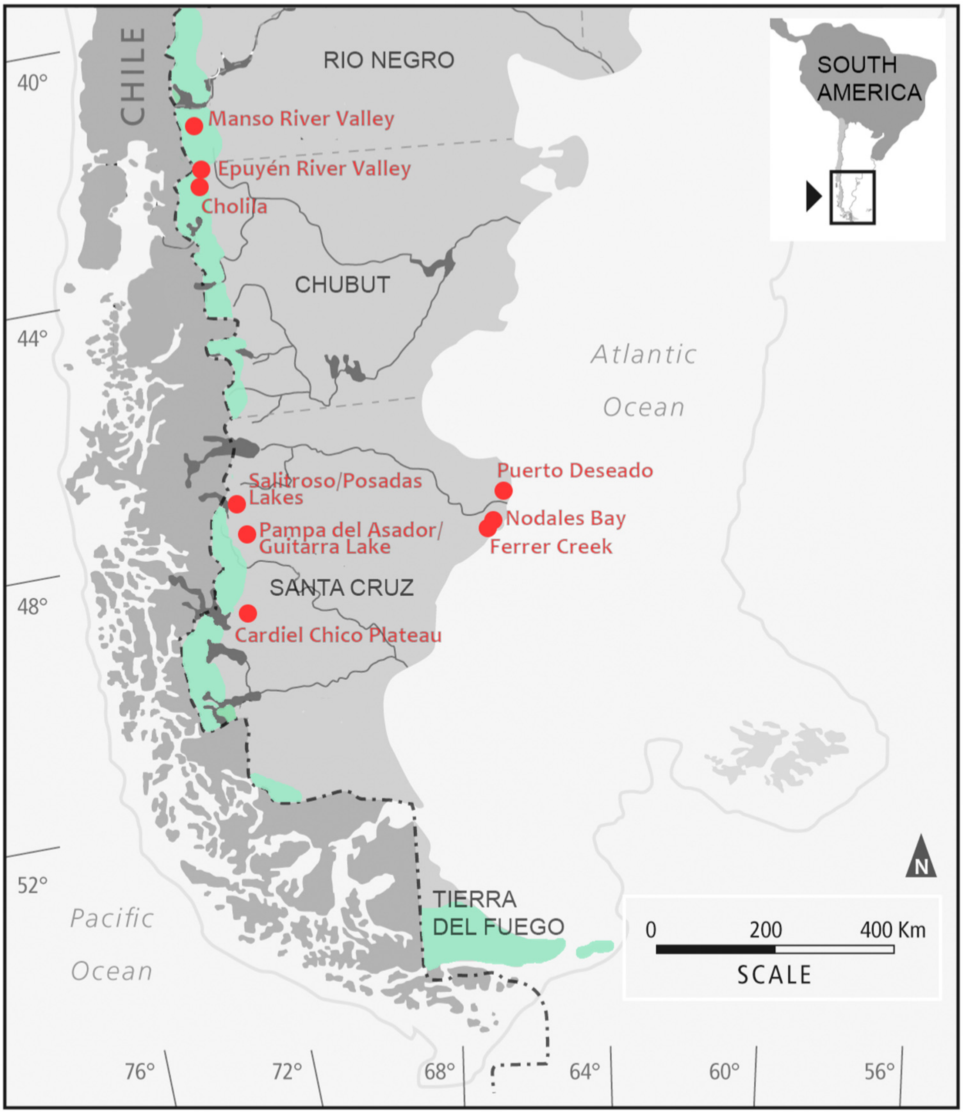

Fig. 1. Location of the analyzed samples in Patagonia, Argentina. In green the area covered by the Patagonian Andean forest. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Negro); Risco de Azócar in the Epuyén River Valley, and Cerro Pintado, in the locality of Cholila (Province of Chubut). Potsherds were recovered in 5 of the 23 sites in the Manso River Valley, 1of 5in El Hoyo, and 1of 14in Cholila. The high state of fragmentation of these ceramic artifacts accounts for the high number of sherds recovered (1167in the three sites of the Manso River Valley, 36 in El Hoyo, and 75 in Cholila). For example, 52.7% of the ceramic sample recovered at Campamento Argentino is made up of fragments under 3 cm^2. No complete ceramic vessels were found in any of the sites, while the small size of most fragments makes it difficult to reconstruct vessel shape. Moreover, there were very few fragments with potential for organic residue analysis. Due to contextual association with dated layers or features, the analyzed fragments were probably deposited during the last 800 years BP (Table 4; Bellelli et al., 2003; Bellelli and Lange, 2014; Fernández et al., 2010; Podestá et al., 2007).

The stable isotope composition of bone collagen in human remains recovered at Chacra de Lobos (698 ± 39 years BP [558 cal BP to 666 cal BP 2σ])[1] and Población Anticura (1550 ± 30 years BP [1313 cal BP to 1476 cal BP 2σ]) is consistent with the consumption of forest resources (Fernández et al., 2010; Fernández and Tessone, 2014). The preservation of organic material is poor in these sites, which could explain the lack of evidence of wild edible plants (Podestá et al., 2007). Ungulates (*Hippocamelus bisulcus*, *Pudu puda* and *Lama guanicoe*) were the most represented taxa in the zooarchaeofaunal record of these forest sites, followed by puma and the rodents *Ctenomys* sp. and *Microcavia* sp. (Fernández et al., 2010; Fernández and Carballido Calatayud, 2015, among others).

In the steppe of inland Santa Cruz, the presence of ceramics is very low in highland sites Cerro Pampa —CP 6 and CP 2 Ojo de Agua— and Lake Guitarra —CG 3— in the Pampa del Asador Plateau and Cardiel Chico Plateau —LCCA. Potsherds have only been recovered at five of the 96 sites in Pampa del Asador/Lake Guitarra. Pottery is also rare in the Cardiel Chico Plateau. Lowland sites are represented here by Sierra Colorada —SAC— in Lake Salitroso and La Costosa dunes in the Lake Posadas. In SAC, ceramics are present in 36.8% of the surface archaeological sites; however, most potsherds are small (1 cm wide by 4 cm long).

Three radiocarbon dates were obtained from organic residue in potsherds: 886 ± 82 years BP (658 cal BP to 924 cal BP 2σ), 373 ± 45 years BP (311 cal BP to 490 cal BP 2σ) and 109 ± 37years

[1] Radiocarbon dates were calibrated using the recent Southern Hemisphere calibration curves published by Hogg et al. (2013) with the radiocarbon calibration program CALIB Rev.7.0.4 (Stuiver et al., 2013).

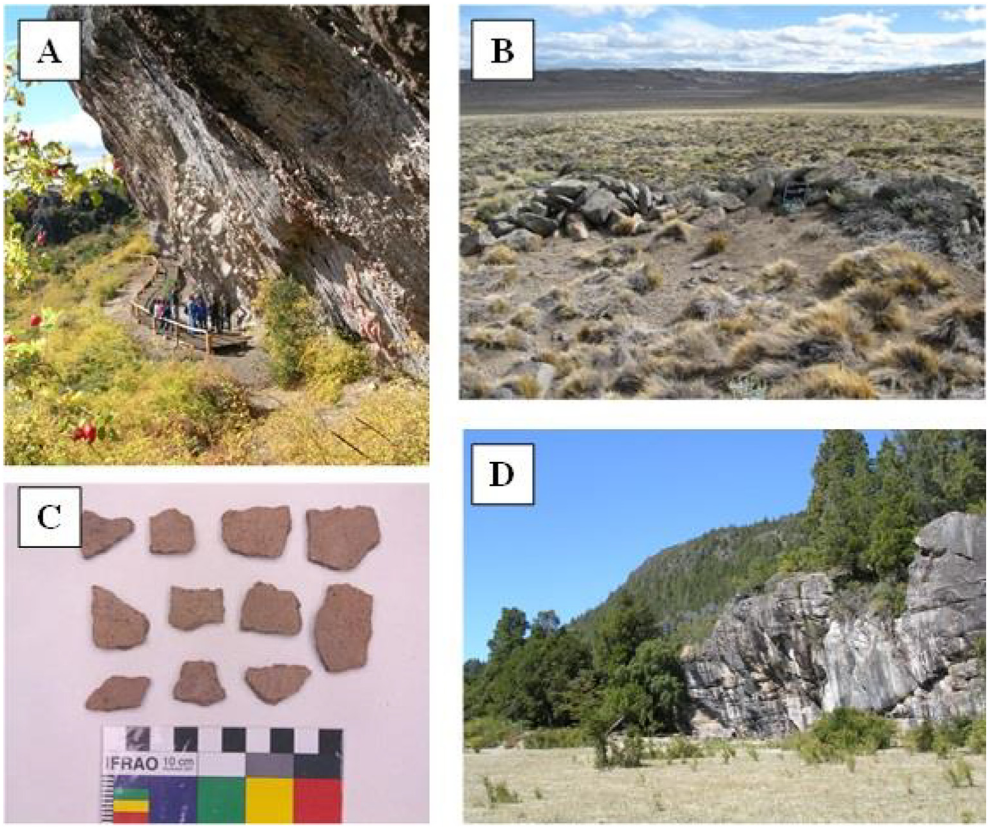

Fig. 2. A. Cerro Pintado (Cholila). B. Cerro Pampa (Santa Cruz). C. Potsherds. D. Lanfré Rock Wall (Lower Manso River).

BP (Cassiodoro and Tessone, 2014). Considering their depositional context, the remaining fragments were probably deposited post-1200 years BP (Table 4; Belardi et al., 2013; Cassiodoro, 2011).

Isotope composition data on human bone collagen recovered from Lake Salitroso are as expected for a diet based on steppe resources (García Guráieb et al., 2015; Tessone, 2010; Tessone et al., 2009). No archaeobotanical analysis of wild edible plants has been undertaken to date, but 19th century chronicles report that human groups gathered roots and bulbs (Musters, 1997 [1871]; Onelli, 1998, among others). Guanaco is the most frequent species in the zooarchaeological record of highland sites, followed by choique (Belardi et al., 2010; Rindel et al., 2007). While the guanaco is the dominant species in the lowland sites as well, there is also evidence of a more generalized consumption of fauna in these sites (Bourlot, 2009), with a higher frequency of choique, dwarf armadillo (*Zaedyus pichiy*), skunk (*Conepatus humboldtii*) and fox (*Pseudalopex culpaeus* and *Pseudalopex griseus*) remains, among others.

Finally, the northern coast of Santa Cruz includes the areas of Ferrer Creek, Puerto Deseado and Nodales Bay. Previous research has described a total of 373 potsherds (Roumec et al., 2017). Although

Table 2
$\delta^{13}C$ and $\delta^{15}N$-values of resources for comparison with organic residue in pottery from steppe sites.

Resources	Environments	n	$\delta^{13}C$		$\delta^{15}N$		References
			Mean	s.d	Mean	s.d.	
Lama guanicoe	Steppe	76	−19.5	0.8	6.3	1.3	Fernández and Tessone, 2014; Tessone, 2010
Pterocnemia pennata	Steppe	35	−20.6	0.8	7.8	1.9	Tessone, 2010
Dasypodidae	Steppe	18	−18.8	1.5	9.4	2.6	Tessone, 2010
Conepatus humboldtii	Steppe	3	−18.0	0.6	7.5	0.1	Tessone, 2010
Hippocamelus bisulcus	Forest	8	−20.8	0.9	1.5	1.1	Tessone et al., 2014
Plant	Steppe	7	−24.3	2.0	3.9	1.3	Tessone, 2010

Table 1
$\delta^{13}C$ and $\delta^{15}N$ values of resources for comparison with organic residue in pottery from forest sites.

Resources	Environments	n	$\delta^{13}C$		$\delta^{15}N$		References
			Mean	s.d	Mean	s.d.	
Lama guanicoe	Steppe	76	−19.5	0.8	6.3	1.3	Fernández and Tessone, 2014; Tessone, 2010
Pterocnemia pennata	Steppe	35	−20.6	0.8	7.8	1.9	Tessone, 2010
Dasypodidae	Steppe	18	−18.8	1.5	9.4	2.6	Tessone, 2010
Conepatus humboldtii	Steppe	3	−18.0	0.6	7.5	0.1	Tessone, 2010
Hippocamelus bisulcus	Forest	4	−21.2	1.2	2.2	1.3	Fernández and Tessone, 2014;
Ctenomys sp.	Forest	3	−21.5	0.1	1.4	0.7	Fernández and Tessone, 2014
Caviidae	Forest	4	−22.3	1.0	1.7	0.6	Fernández and Tessone, 2014
Plant	Forest	38	−27.8	1	−1.2	3	Fernández and Tessone, 2014

Table 3
$\delta^{13}C$ and $\delta^{15}N$-values of resources for comparison with organic residue in pottery from coastal sites.

Resources	Environments	n	$\delta^{13}C$		$\delta^{15}N$		References
			Mean	s.d	Mean	s.d.	
Terrestrial resources	Coast	5	−20.3	0.9	9.4	1.4	Barberena, 2002; Favier Dubois et al., 2009; Gómez Otero, 2007; Gómez Otero et al., 2014
Marine fish	Coast	3	−18.9	1.7	15.5	1.6	Favier Dubois et al., 2009; Gómez Otero, 2007; Gómez Otero et al., 2014
Other marine resources	Coast	6	−13.0	1.8	20.2	2.6	Barberena, 2002; Favier Dubois et al., 2009; Gómez Otero, 2007; Gómez Otero et al., 2014

Table 4
Location and chronology of analyzed archaeological sample.

Environment	Area	Site	n	^{14}C BP	Cal BP (2σ)	Reference
Forest	Manso River Valley	Población Anticura	2	Last 700 years		Fernández et al., 2010
		Paredón Lanfré	9	Last 500 years		Bellelli et al., 2007
		Campamento Argentino	1	560 ± 60 (dated layer)	472–574	Fernández et al., 2010
	Epuyén River Valley	Risco de Azócar	2	820 ± 60 (dated layer)	633–800	Podestá et al., 2007
	Locality of Cholila	Cerro Pintado	7	680 ± 60 (dated layer)	536–676	Bellelli et al., 2003
Steppe	Pampa del Asador/ Guitarra	CP 6	4	373 ± 45[a] and 886 ± 82[a]	311–490	Cassiodoro and Tessone, 2014
		CP 2 Ojo de Agua	2	1582 ± 46 and 1411 ± 45 (date of the site)	1344–1533 and 1256–1351	Rindel, 2009; Dellepiane, 2014
		CG 3	1	1217 ± 36 (date of the site)	980–1179	Cassiodoro et al. 2013
	Lake Posadas	La Costosa, Site 2	1	Undated		Cassiodoro, 2011
		Fortuni collection	1	Undated		Cassiodoro, 2011
	Lake Salitroso	SAC 29	1	Undated		Cassiodoro, 2011
		SAC 6	1	Undated		Cassiodoro, 2011
		SAC 3	1	960 ± 125 (date of the site)	642–1068	Goñi, 2000–2002
		SAC 24	1	Undated		Cassiodoro, 2011
		SAC 11	1	750 ± 60 BP (date of the site)	558–730	Goñi, 2000–2002
	Cardiel Chico	LCCA 2	1	109 ± 37[a]		Cassiodoro and Tessone, 2014
Coast	Ferrer Creek	No Data	1	Undated		This paper (Padre Manuel Jesús Molina)
	Puerto Deseado	No Data	2	Undated		This paper (Padre Manuel Jesús Molina)
	Nodales Bay	No Data	7	Undated		This paper (Padre Manuel Jesús Molina)

[a] AMS radiocarbon dates obtained from organic residues attached to their inner walls.

absolute dates are not available for this sample, its estimated minimum age is 1000 years BP (Castro et al., 2003; Roumec et al., 2017).

Studies on technology, faunal remains and stable isotope composition of human bone tissue suggest that marine resources played an important role during the late Holocene (Castro et al., 2003; Hammond, 2014; Zilio, 2017; Zubimendi, 2015). In turn, archaeobotanical analyses have identified remains of *Schinus* sp. fruit in hearths and aff. *Prosopis* sp. in a grindstone recovered near Puerto Deseado (Ciampagna, 2015). The most common type of archaeological site in this area is the shell midden (Hammond, 2014); the most abundant taxa in coastal zooarcheological record are pinnipeds and mollusks, followed by sea birds and guanaco (Hammond, 2014; Moreno et al., 2011; Zubimendi, 2015).

3. Materials

Here we present an analysis of 46 potsherds with carbonized residues attached to their interior walls (Table 4). The sample includes ceramic fragments recovered from the steppe and the forest by three different research teams, and coastal sherds available in the "Padre Manuel Jesús Molina" regional museum (Río Gallegos, Santa Cruz). The objective was to achieve a balanced representation of pottery from the three main environmental units as defined by the isotope ecology of Continental Patagonia. In the case of the steppe, only one potsherd was selected from each concentration of potsherds, in order to avoid overrepresentation of the same container. Given the small sample size of forest and coastal ceramics, we selected the potsherds with the largest amount of attached organic residues. Thus, it is possible that we overrepresented the signal obtained from the same container in the forest and the coast, but we believe that it is a valid strategy given the exploratory nature of this research.

In forest sites the ceramic fragments were recovered from the bottom of rock walls and shelters located in the lower Manso River Valley (n = 12), the Epuyén River Valley (n = 2), and the locality of Cholila (n = 7) (Bellelli et al., 2003; Bellelli et al., 2007; Bellelli and Lange, 2014; Fernández et al., 2010; Podestá et al., 2007). In the case of the inland steppe most of the potsherds were recovered in surface concentrations (n = 8), but also in sites associated to basalt rock walls (n = 2), hunting blinds (n = 4) and a private collection of archaeological artifacts (n = 1) (Cassiodoro, 2011; Cassiodoro et al., 2013; Cassiodoro and Tessone, 2014; Dellepiane, 2014; Goñi, 2000–2002; Rindel, 2009). Finally, the coastal sample consists of 10 potsherds from the "Padre Manuel Jesús Molina" museum collection. It was included to evaluate the feasibility of identifying $\delta^{13}C$ and $\delta^{15}N$-values associated to the use of marine resources in Patagonia. This sample was recovered from the areas of Ferrer Creek (n = 1), Puerto Deseado locality (n = 2) and Nodales Bay (n = 7).

4. Methods

Stable isotope analysis of the organic residues was performed at the Instituto de Geocronología y Geología Isotópica (INGEIS, UBA-CONICET) with a Carlo Erba Elemental Analyzer (CHONS) connected to a continuous flow Thermo Scientific Delta V Advantage mass spectrometer through a Thermo Scientific ConFlo IV interface. Stable isotopes results are reported as ratios ($^{13}C/^{12}C$ y $^{15}N/^{14}N$) and expressed as δ-values in parts per thousand (‰) relative to internationally accepted standards: V-PDB for carbon and AIR for nitrogen. Analytical uncertainty is ± 0.3‰ for both C and N.

Samples were prepared following Beehr and Ambrose (2007). A scalpel was used to remove the residues attached to the interior portion of each sherd. Uttermost caution was used to avoid damaging the surface, so as to avoid sample contamination with organic substances used as temper, such as shells and plants.

In order to determine whether there were alterations due to contaminating processes, a sub-group of the total sample (n = 10) was pretreated with a weak (0.1 N) sodium hydroxide (NaOH) solution during 24 h at room temperature. Then, the sub-group was rinsed and centrifuged three times with distilled water, and oven-dried at < 40 °C during 24 h (Beehr and Ambrose, 2007). Only the samples with enough amounts of N and C to generate a major ion beam of at least 500 MV were selected to guarantee measurement reproducibility (Beehr and Ambrose, 2007).

The δ^{13}C and δ^{15}N-values on the organic residues of potsherds were compared to the isotope ecology of each of the Patagonian environments described above (Tables 1–3). To that end, we used the discrimination factor of −2‰ for C and +2‰ for N cited by Fernandes (2016). Two-σ ranges are considered to illustrate the distribution of resources (Hastorf and DeNiro, 1985; Yoshida et al., 2013). Finally, values measured in organic residues were compared to those of human bone collagen samples associated to the sites where the potsherds were recovered (Fernández and Tessone, 2014; Gómez Otero et al., 2014; Gordón et al., 2015; Tessone, 2010; Zilio, 2017).

5. Results

Previous results indicate that alterations in the isotope ratio were not significant and that the pre-treatment was not necessary (Chaile, 2015) because δ^{13}C y δ^{15}N values of treated potsherds were similar to those of untreated sherds (Table 5). These results are in accordance to other previous studies (Beehr and Ambrose, 2007; Morton and Schwarcz, 2004). In addition, eight sherds were discarded from the original set of 46 potsherds due to insufficient amount of carbon and/or nitrogen to generate replicable results. The rest of the set was grouped according to the different environments in which they were recovered.

Forest site potsherds (n = 18) present a mean δ^{13}C of −25.7 ‰ ± 1.0 ‰ and δ^{15}N 6.0‰ ± 1.5 ‰. δ^{13}C-values vary between −27.4 ‰ and − 23.8 ‰ while δ^{15}N-values ranged from 3.1 ‰ to 9.3 ‰. Coastal site potsherds (n = 9) showed a mean δ^{13}C of −25.2 ± 1.7‰, ranging from −26.6‰ to −21.7‰; mean δ^{15}N was 9.4‰ ± 2.7‰, and ranged from 7.5‰ to 16.4‰. Finally, the sample from the Santa Cruz steppe (n = 11) presented a mean δ^{13}C of −25.7‰ ± 1.5‰, while mean δ^{15}N was 7.7‰ ± 1.6‰, ranging from 4.5‰ to 11.0‰, and − 27.9‰ to −23.0‰, respectively (Table 6).

6. Discussion

Despite the differences observed in mean C isotope composition of resources from different environmental settings (Tables 1–3), there is a remarkable consistency among mean δ^{13}C-values obtained from organic residue in potsherds recovered in forest, steppe and coastal archaeological sites. In contrast, δ^{15}N-values vary more noticeably, in accordance with the site environment. Forest site potsherds present the lowest δ^{15}N means, while the mean δ^{15}N of organic residues in coastal potsherds is the highest. These values are as expected from the isotope ecology of each environment.

Figs. 3, 4 and 5 plot both C and N stable isotope compositions of organic residues in potsherds and the main resources typical of forest, coast and steppe environments. Most potsherds —66.6%— recovered in forest sites presented organic residue stable isotope compositions (δ^{13}C-values vary between −26.6‰ and − 24.8‰ while δ^{15}N-values ranged from 3.1‰ to 7.1‰) compatible with those of typical forest resources (Fig. 3). The most represented resource had isotope composition values comparable to those of huemul, followed in frequency by rodents (Caviidae) and plants. Only one potsherd found in the Risco de Azócar site presented values (δ^{13}C = −23.8‰, δ^{15}N = 9.3‰) comparable to steppe resources, i.e. choique and Dasypodidae. The lowest δ^{13}C-values in forest sites are from the Cerro Pintado site, while four potsherds from Paredón Lanfré have the lowest δ^{15}N-values. The zooarcheological record of Paredon Lanfré has low resolution due to its location in a flood plain and the frequent natural fires recorded along the sequence, which contributed to the poor preservation of the bone sample. However, remains of typical forest prey —huemul and pudú— have been found at this site (Fernández and Carballido Calatayud, 2015), providing support to our results. There is also a correspondence between human paleodiet reconstructions for forest sites (Fernández and Tessone, 2014) and our conclusions regarding the processing of food in ceramic vessels.

In the case of coastal sample, only one sherd (δ^{13}C = −21.7‰, δ^{15}N = 16.4‰) presents values consistent with the processing of marine fish and another (δ^{13}C = −22.6‰, δ^{15}N = 8.7‰) is compatible with local terrestrial resources. Most of the sample (78%) yielded δ^{15}N-values typical of local terrestrial animal protein but their δ^{13}C-values are lower than expected for such resources. Bone collagen data from human remains recovered from the north coast of Santa Cruz suggest a mixed marine/terrestrial diet during the last 1000 years (Zilio, 2017), which is not consistent with the isotope composition of the organic residues measured here. However, in other coastal areas located further north, stable isotope evidence points to a mixed diet with a greater emphasis on terrestrial animals, especially after European contact (Favier Dubois et al., 2009; Gómez Otero, 2007; Gordón et al., 2015). Thus, direct indicators show that the diets of coastal hunter-gatherer groups included at least some terrestrial resources by the end of the late Holocene, which is also reflected in the C and N isotope composition of organic residues in potsherds. Likewise, organic residues in Chubut pottery present δ^{13}C-values compatible with the processing of C_3 plants and/or animal protein (Gómez Otero et al., 2014). In addition, analysis of fatty acids by gas chromatography mass spectrometry provides evidence for the processing of vegetable oil mixed with terrestrial and marine animal fat (Gómez Otero et al., 2014).

For pottery recovered in steppe sites, only three out of the 11 potsherds yielded values consistent with the processing of steppe resources. Two of these (δ^{13}C = −23.0‰; δ^{15}N = 8.7‰ and δ^{13}C = 23.5‰, δ^{15}N = 7.4‰) are compatible with guanaco or choique (Bourlot, 2009; Rindel, 2009). The third sherd, belonging to the Fortuni collection (δ^{13}C = −27.3‰, δ^{15}N = 4.5‰) produced isotope composition values matching those of the steppe vegetation. Thus, the association between the C and N isotope compositions of carbonized residues and paleodiet indicators is not straightforward. The latter suggests a diet based on steppe animal protein (Tessone, 2010; Tessone et al., 2015), as do the δ^{15}N-values of organic residues presented here. However, this is not the case for eight of the 11 organic residue δ^{13}C-values, which are lower than expected for the processing of these types of resources. The same pattern is present in most of the coastal potsherds (78%) and a few from forest environments (28%).

As mentioned above, the C stable isotope composition of carbonized organic remains depend on a variety of factors (Hart et al., 2012, 2009, 2007; Lovis et al., 2011); therefore, bulk δ^{13}C-values in residue should not be treated as straightforward indicators of the types of food cooked

Table 5
Treated vs untreated δ^{13}C and δ^{15}N values of organic residues.

	Untreated δ^{13}C	Treated δ^{13}C	Untreated δ^{15}N	Treated δ^{15}N
N	10	10	10	10
Mean	−25.9	−25.8	5.8	5.9
Stand. dev.	0.9	0.9	1.6	1.5
Min	−27.4	−27.3	3.1	2.9
Max	−24.8	−24.6	8.1	8

Table 6
Summary statistics for C and N stable isotope composition of organic residue in Patagonian potsherds.

		δ^{13}C (‰)			δ^{15}N (‰)		
Site environment	Sherds (N)	Mean ± s.d.	Min	Max	Mean ± s.d.	Min	Max
Forest	18	−25.7 ± 1.0	−27.4	−23.8	6.0 ± 1.5	3.1	9.3
Coast	9	−25.2 ± 1.7	−26.6	−21.7	9.4 ± 2.7	7.5	16.4
Steppe	11	−25.7 ± 1.5	−27.9	−23.0	7.7 ± 1.6	4.5	11.0

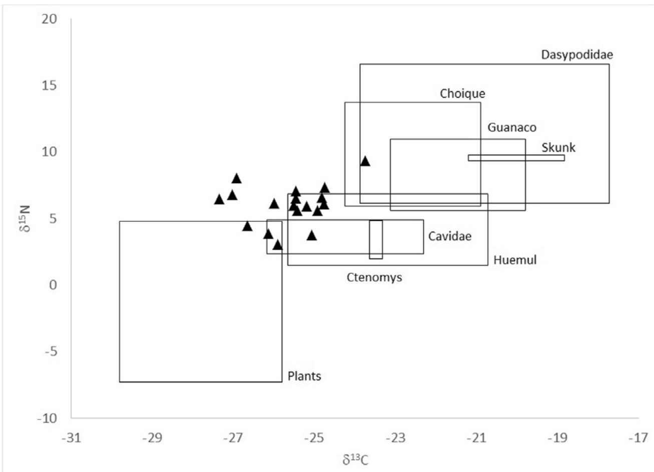

Fig. 3. δ^{13}C and δ^{15}N of modern resources and carbonized residues in the forest.

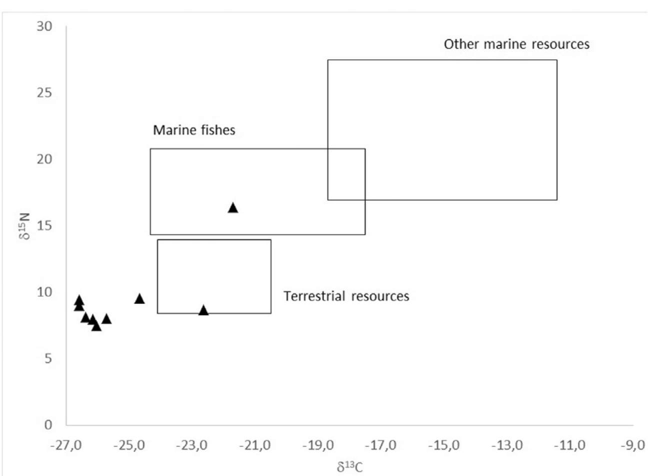

Fig. 4. δ^{13}C and δ^{15}N of modern resources and organic residues in the coast. The other marine resources square comprise *Otaria flavescens*, *Spheniscus magallanicus* and *Phalacrocorax* spp.

in ceramic vessels. There are several, non-mutually exclusive hypotheses to explain why δ^{13}C-values in organic residue are lower than expected from isotope ecology models. First, proteins and carbohydrates are more susceptible to degradation than lipids (Heron and Evershed, 1993). Cooking or degradation during burial might have led to the destruction of these macro-molecules of food while lipids were preserved, resulting in low bulk δ^{13}C-values (Craig, 2004). Second, experimental research carried out by Lovis et al. (2011) demonstrated that bulk δ^{13}C-values can be affected by the nixtamalization process, although there is no evidence that hardwood ash has been added to pottery by hunter-gatherer groups of Patagonia. Finally, another possible explanation is that human groups used pottery to extract animal fat. This use of pottery vessels has been recorded in ethnographic

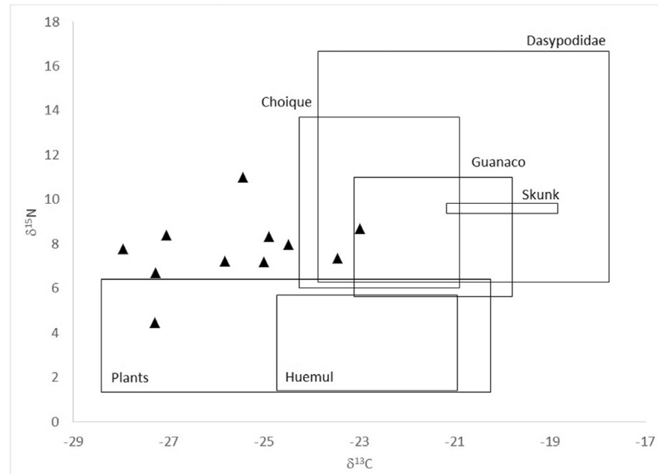

Fig. 5. δ^{13}C and δ^{15}N of modern resources and organic residues in the steppe.

studies (Bormida and Casamiquela, 1958–1959; Casamiquela, 1987, among others) and in narratives by travelers who came into contact with different hunter-gatherer groups. They witnessed the extraction of animal fat for consumption, mainly during winter when fat is scarce, as well as to use in paint preparation (Bourne, 1998; Claraz, 1988; Musters, 1997 [1871]; Onelli, 1998). Therefore, we suggest that the low δ^{13}C-values of organic residues in Patagonian potsherds are due to the significant contribution of lipids, which are depleted in ^{13}C by 6‰ compared to protein.

In short, the study of C and N stable isotope composition of carbonized organic residue in potsherds has allowed us to confirm that pottery was used among late Holocene hunter-gatherer groups in Patagonia to process forest, steppe and marine resources. A large part of the pottery recovered at forest sites show isotope compositions compatible with huemul and/or rodents. The coast sample results suggest that terrestrial resources had a dominant role; only one potsherd yielded values consistent with marine fish. Finally, resources processed in steppe sites pottery were mainly steppe taxa like guanaco, choique and Dasypodidae, while the stable isotope composition of plants was reflected in only one sherd. However, the δ^{13}C-values recorded in most of the steppe and coastal potsherd samples, as well as three from forest sites, were lower than expected from the isotope ecology of these environments. A higher contribution of fats (vs protein and/or carbohydrates) to the carbonized residues might be a better explanation for this pattern, since lipids have lower δ^{13}C-values than protein. This suggests that ceramic vessels were being used to process animal fat, a strategy that might have been important in environments where the main prey species have lean meat (Speth and Spielmann, 1983). These data provide support to the notion that the adoption of pottery was linked to a more efficient extraction of steppe guanaco fat (Cassiodoro, 2008; Gradin, 1997) or to extract the fat and the bone marrow from choiques and use them for cooking, as recorded by Musters (1997 [1871]).

7. Conclusion

The analysis of C and N stable isotope composition of organic residues in ceramic potsherds recovered in different environments in Argentine Patagonia constitutes a valuable tool to explore past pottery use. Moreover, this research is complementary to zooarchaeological data and the study of stable isotope composition of human remains, given that organic residues in pottery are not a direct reflection of past diets. This study was carried out from a regional perspective with a large spatial scale, which adds valuable information to discuss the use of ceramic technology and human paleodiets among Patagonian hunter-gatherer groups during the late Holocene (see Sturm et al., 2016).

We are aware of the limitations of this study, such as the probable overrepresentation of single ceramic vessels in forest and coastal samples. However, we consider these results to be valuable as a first step in developing a comparative analysis of organic residues in pottery in a large spatial scale, including the three main environments of Continental Patagonia. One of the main achievements of this paper is the generation of raw isotope composition data on organic residues, which are still scarce for Patagonian ceramics.

On one hand, δ^{15}N-values of organic residues allowed us to differentiate the environmental origin of resources cooked in ceramic vessels; on the other, δ^{13}C-values suggest that hunter-gatherer groups used these vessels to process animal fat. However, the interpretation of isotope values presents equifinality problems; to address them, future research efforts should be directed at increasing sample size, analyzing fatty acids obtained from absorbed organic residues, and conducting experimental cooking studies with local resources, in order to obtain a modern stable isotope composition data set for comparison with archaeological data.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jasrep.2018.04.011.

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