Vessels Explored: Applying Archaeometry to South American Ceramics and their Production

Edited by

Emily M. Stovel
Guillermo A. De La Fuente

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Preliminary Study of Stable Carbon Isotopes of Bulk Lipid Residues in Archaeological Ceramics from West Tinogasta, Argentina

Irene Lantos, Norma Ratto, Héctor Panarello, and Marta Maier

Foodways of the pre-Hispanic societies of the West Tinogasta region (Catamarca Province, Argentina) were inferred from stable carbon isotope analysis on bulk lipid residues from eleven archaeological ceramics recovered from sites with occupations ranging from AD 450 - 1550. Nine modern samples were analysed to obtain reference values for typical Andean ingredients. Archaeological maize use patterns can be detected by enriched 13C values typical of C4 plant carbon compounds found in cooking residues. Our preliminary results show a great variability of maize use and consumption practices which can be explained by the multiple recipes and functions a pot had during its use life resulting in organic residue 'palimpsests'. No statistically significant correlation was observed between site chronology and isotopic signals, although we propose differential access to maize resource at the Inca site of Batungasta.

Introduction

Bulk lipid stable carbon isotope analysis is an effective method to discover food use patterns from organic residues absorbed in archaeological ceramics and it can give insight into the cooking practices of West Tinogasta's pre-Hispanic societies (Catamarca province, Argentina). Archaeological maize use patterns can be detected by enriched ^{13}C values typical of C_4 plant carbon compounds found in cooking residues (Hart *et al*., 2009; Hastorf and de Niro 1985; Morton and Schwarcz 2004; Reber and Evershed 2004; Reber *et al.,* 2004; Seinfeld *et al.*, 2009). For this purpose we used an elemental analyser coupled to an isotope ratio mass spectrometer to measure δ13C values in carbon compounds from the bulk lipids extracts of potsherds recovered in sites of the study area with occupations extending from AD 450 to 1550. The samples were selected from expeditions in the late 1970s (Sempé 1976, 1977) and from the continuing research projects that began in the 1990s by Dr. Ratto and her team in the PAChA Project (Proyecto Arqueológico Chaschuil Abaucán).

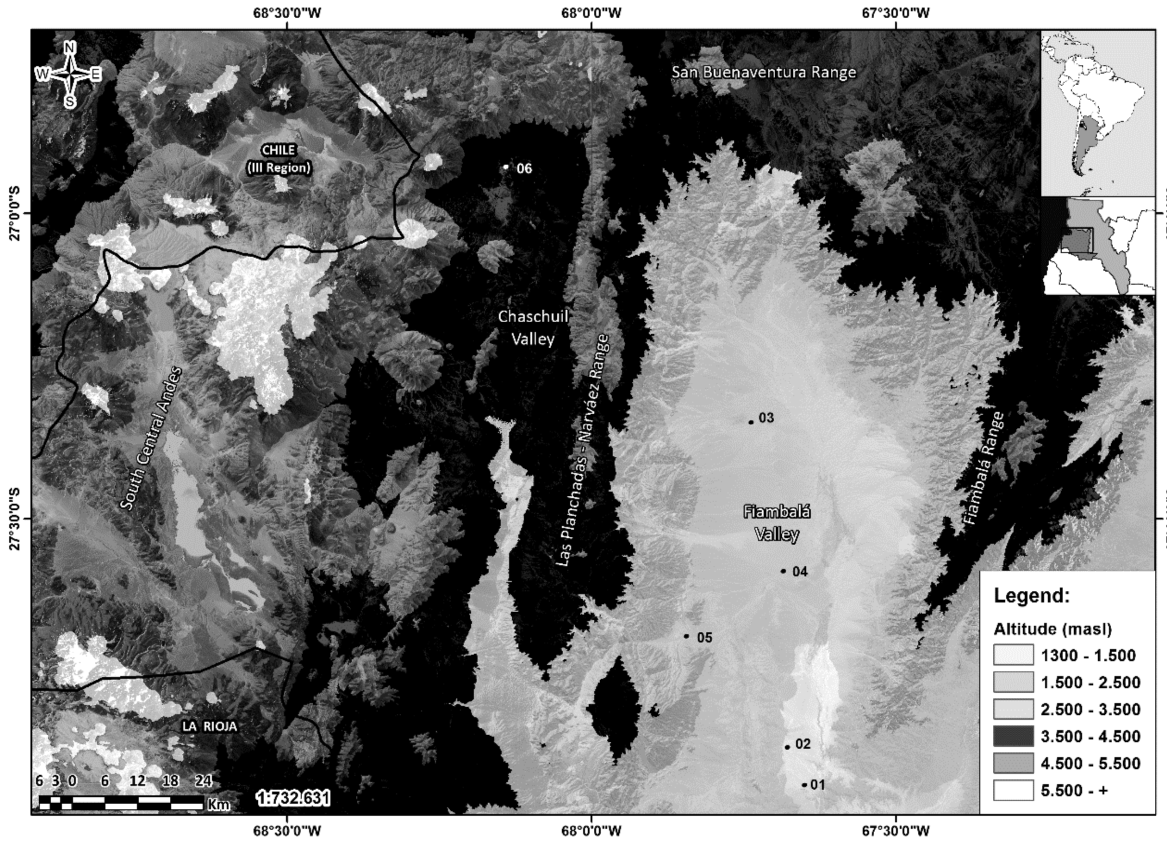

Figure 16. Location of sites in the West Tinogasta region, Catamarca province, Argentina. Sites in the Fiambalá mesothermal valley: (1) La Troya LTV50, (2) Batungasta, (3) Palo Blanco NH3, (4) Mishma 7 and (5) Punta Colorada. Site in the transitional Chaschuil puna: (6) San Francisco.

Carbon stable isotope analysis measures the $^{13}C/^{12}C$ ratio expressed in $\delta^{13}C$ values. C_3 plants and C4 plants have different photosynthetic pathways leading to distinct isotopic $^{13}C/^{12}C$ ratios (Deines 1980; O'Leary 1993; Panarello and Sánchez 1985; Tykot 2006). C_3 plants use the Calvin-Benson cycle for CO_2 fixation and include most South American fruits, vegetables and cool season grasses. Their δ ^{13}C values fall into the range -35‰ to -22‰. On the other hand, C_4 plants use the Hatch-Slack cycle and are adapted to hot and arid environments. They include maize, sugar cane and warm season grasses, and their δ ^{13}C values range from -16‰ to -9‰. Maize is unique because it is a C_4 plant widely cultivated as a staple food and it contains more lipids than other edible seeds (Reber and Evershed 2004). However, fractionation is greater in lipids than in other metabolites such as carbohydrates or proteins, resulting in depleted δ ^{13}C values (Brugnoli and Farquhar 2004; Post *et al.,* 2007; Samec *et al.,* 2010). Therefore, the C4 detection values should be brought down approximately -6‰ or -8‰ for lipids (de Niro and Epstein 1977).

Stable carbon isotope analysis on lipid extracts from the ceramic matrix, rather than charred foodstuff adhered to the inner surface of a vessel, has important benefits. Ceramic matrixes are considered 'clean slates' after firing, given that any lipids contained in clay are

	#	Sample name	Site	Calibrated dates (yrs AD)	Ecozone	Altitude (m.a.s.l.)	Vessel Section
REFERENCE SAMPLES	1	Pisingallo maize	n/a	n/a	Humahuaca Gorge, Jujuy	1750-2400	n/a
	2	Chullpi maize	n/a	n/a			n/a
	3	Dentado Blanco maize	n/a	n/a			n/a
	4	Capia maize	n/a	n/a			n/a
	5	Bovine fat I	n/a	n/a	Cruz Alta, Tucumán	1300	n/a
	6	Bovine fat II	n/a	n/a	San Antonio de Areco, Buenos Aires	100	n/a
	7	Llama charqui fat	n/a	n/a	La Candelaria, Jujuy	4000	n/a
	8	Green pepper	n/a	n/a	Fiambala valley, Catamarca	1300	n/a
	9	Alubia beans	n/a	n/a		1300	n/a
ARCHAEOLOGICAL SAMPLES	11	PB-NH3-228	Palo Blanco NH3	458-535 596-639	Fiambala valley, Catamarca	1900	body
	12	PB-NH3-153-09					neck
	14	LT-V50-BATH-35	La Troya	641-719	Fiambala valley, Catamarca	1380	lip-body
	15	PC-2b-B	Punta Colorada	661-694 972-1020	Fiambala valley, Catamarca	2290	body-base
	18	M7-H1	Mishma 7	1414-1439 1405 - 1435 1522 - 1573	Fiambala valley, Catamarca	1760	base
	19	M7-HA-urna					neck-body
	20	SF-R15-17	San Francisco	1400 - 1500	Chaschuil Puna	4000	body
	21	SF-R12					body
	22	VBAT-035-02	Batungasta	1445-1522 1524-1558	Fiambala valley, Catamarca	1480	body
	23	VBAT-032					body
	24	VBAT-020-05					body-base

Figure 17. Description of the reference and archaeological samples studied in this paper. Calibrated dates were taken from Ratto (2013).

completely combusted during pottery firing (Eerkens 2005). Therefore, any lipids recovered from the ceramic matrixes are absorbed residues of the foodstuffs cooked and/or stored in the vessels. Also, given the hydrophobic characteristics of lipids and the protective effect of the ceramic porous matrix, lipid residues are relatively well preserved. Potsherds that do not have apparent residues adhered to their inner surface can be good candidates for analysis if they have absorbed residues invisible to the naked eye (Evershed 2008). Hence, bulk isotopic analysis determines the presence of C^4 carbon compounds even in samples that have undergone post-depositional processes, as it measures the $\delta\ ^{13}C$ value in the mixture of the intact lipids and their degradation products (Seinfeld *et al.,* 2009).

Maize Use in the West Tinogasta Region

The West Tinogasta Region is set in the south-western tip of Catamarca province in northwestern Argentina, and is part of the South Central Andes (Figure 16). West Tinogasta is a vast area comprising two longitudinal valleys named Fiambalá and Chaschuil, separated by the Narváez and Las Planchadas ranges. Both valleys have diverse and contrasting eco-zones which include the mesothermal valley (1400-2400masl), the foothills (2400-3500masl), the transitional puna (3500-4000masl) and the Andes mountain range (4000-6700masl). The geographical limits of this extended area are the humid valleys to the east, the southern puna highlands to the north, and Chile to the west.

The cultural landscape of pre-Hispanic West Tinogasta was characterized by discontinuous settlement of human populations in response to the Mid-Holocene environmental variations associated with large-scale changes in climate, explosive volcanism, and recurrent seismic activity that shaped the topography and determined the habitability of the area (Ratto *et al.,* 2013). Throughout the 1st millennium AD, communities populated the region and developed herding and agricultural economies while still maintaining hunting and gathering practices. Settlements were distributed sparsely at different altitudinal levels and eco-zones taking advantage of the different local resources (Ratto 2013). Recent research shows that between the 10th and 13th centuries AD the unstable environmental conditions combined with catastrophic volcanic events triggered population movements and site abandonments in search of eco-refuges in higher valleys where they continued to carry out their traditional ways of life (Ratto *et al.,* 2013). The area was most probably repopulated when conditions improved in the mid-13th century AD (Ratto 2013). This also occurred during the Inka expansion between the 14th and 16th centuries AD, which promoted the movement of people with new cultural characteristics from other areas as part of a territorial domination strategy. During the 17th century AD, the Spanish colonial administration created new politically unstable conditions and caused further community relocation and new de-population (Ratto and Boxaidós 2012).

The archaeological evidence of maize cultivation and consumption in West Tinogasta illustrates the importance of this staple grain in local foodways. For example, archaeobotanical remains of maize cobs and kernels were found in Fiambalá mesothermal valley in Punta Colorada (*c.* 650-1050 AD), Batungasta (1450-1550 AD), and the nearby site of Lorohuasi (*c.* 1400-1600 AD). Morphological analysis carried out by Dr. Cámara Hernández identified the local landraces Pisingallo-Capia, Morocho-Chaucha, Rosita-Colorado, and Capia-Pisingallo. Ancient DNA analysis on nine specimens determined strong relationships with three complexes: Andean, South American, and those derived from the introduction of modern varieties (Lia *et al.*, 2007). Continuity between archaeological and modern landraces is proposed for varieties such as Amarillo Chico, Amarillo Grande, Blanco, and Altiplano, all within the Andean complex (Cámara Hernández and Arancibia de Cabezas 2007).

In addition, local maize cultivation can be inferred from the extensive agricultural installations at different altitude levels in the mesothermal valley. These locations were intended for food production throughout the first millennium AD. During the mid-thirteenth to sixteenth centuries, the cultivated land for food production was expanded by the Inka administration in order to increase food production (Orgaz and Ratto 2013). The agricultural expansion took place in a context of interaction between local socio-political entities and foreign populations that were moved and established in the area by the Inka empire (Ratto and Boxaidós 2012; Orgaz and Ratto 2013). This particular situation was materialized with the presence of certain symbolic items such as tombs, rock art manifestations, and offerings to the sacred mountains that were displayed in the productive landscape, together with numerous lithic milling artefacts which were prevalent in these sites.

Isotopic studies on bioarchaeological remains of individuals from the Fiambalá Valley suggest differences in diet through time (Aranda *et al.*, 2014). One case of a lactating individual from the first millennium AD indicated that the mother's diet was based on C_4 plants, most probably maize.

On the other hand, the samples from the Inka period had a wide range of values, but the general tendency suggested that during Inka state presence in the region (14th to 16th century AD), there was a mixed diet with an important C_4 component, a minor contribution of C_3 plants, and limited access to animal protein.

Materials and Methods

Samples

Carbon stable isotope analysis was carried out on the bulk lipid extracts from absorbed residues of eleven archaeological potsherds. Nine modern reference samples of traditional ingredients in Andean cookery were also extracted for lipids.

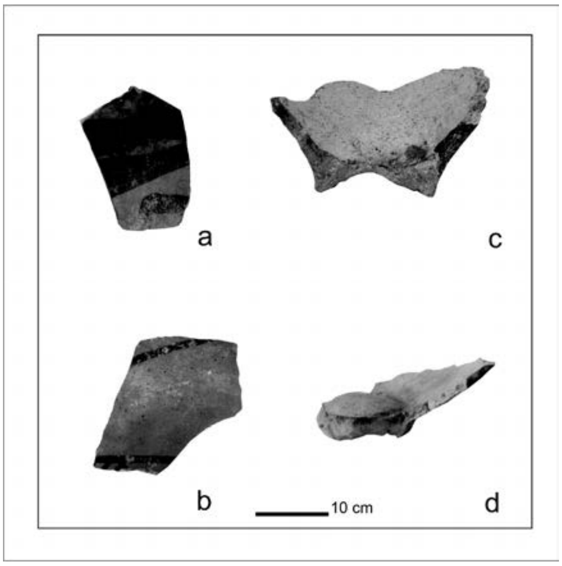

Figure 18. Examples of archaeological potsherds selected for analysis. (a) Belén urn neck-body fragment; (b) aryballos vessel body fragment; (c) and (d) cooking pot base-body fragments.

The archaeological ceramic samples were recovered from sites that illustrate the different chronological moments of the cultural development from 450 to 1550 AD. They were recovered from different altitude levels of the mesothermal valley and the transitional puna.

Sites settled during the 5th to 11th centuries AD include Palo Blanco NH3, La Troya V50 and Punta Colorada, and sites settled during the 14th to 16th centuries AD during the Inka domination of the region include Mishma 7, Batungasta and San Francisco. None of the archaeological potsherds selected for analysis had visible adhered or charred residues in their inner surface, but they had a dark and oily appearance typical of absorbed organic residues. The samples were taken from the section of the vessel with most signs of absorbed residue and they were about 4x4 centimetres in size and weighed between 20 and 30 grams. In Figure 17 the geographical, chronological and morphological details are given for each sample. Photographs of some ceramic samples are shown in Figure 18.

The nine modern reference samples included C_3 and C_4 plants, and animals fed mostly on C_3 or C_4 plants (Figure 2). Four landraces of maize were chosen to obtain C_4 plant values. Bovine fat was selected from NW Argentina and the Central Argentine Pampas as references of animals fed on mostly C_4 or C_3 plants, respectively. Also, a sample of llama jerky was included from the puna area of Jujuy province in NW Argentina. Green pepper and kidney beans were selected for C_3 plant references.

Sample Preparation

Lipid extraction was carried out on the archaeological potsherd samples and the reference samples. Preparation of dry reference samples was included grinding them in a coffee mill, which was carefully cleaned with solvent before each use. Preparation of humid or fresh reference samples was done by grinding them in a porcelain mortar with a pestle. Archaeological potsherds were cleaned by rinsing both surfaces with solvent. They were then broken into small fragments with a hammer and ground to dust in a clean porcelain mortar with a pestle.

Organic extraction was carried out with a 2:1 mixture of chloroform and methanol, solvents were pre-distilled and of chromatographic quality. The samples were ultrasonificated twice for 5 minutes, and then filtered with 16 ml of distilled water. Samples were then centrifuged for 3 minutes and the organic phase was separated, this was done twice to ensure no water remained. The extracted solvents were evaporated under nitrogen current and stored in 2 ml glass vials at -20°C.

Elemental Analysis Coupled to Isotope Ratio Mass Spectrometry (EA-IRMS)

Samples were weighted, loaded in tin capsules and combusted in an elemental analyser (EA) Carlo Erba coupled via a CONFLO IV interface to a Thermo Delta-V Advantage isotope ratio mass spectrometer (IRMS). Helium was used as the carrier gas. A standard of pure CO_2 was measured prior to each sample. Three internal

	#	Sample name	$\delta^{13}C$ (‰)	C_4 fraction (%)
REFERENCE SAMPLES	1	Pisingallo maize	-14,8	100,0
	2	Chullpi maize	-15,5	96,5
	3	Dentado Blanco maize	-15,6	96,0
	4	Capia maize	-15,9	94,5
	5	Bovine fat I	-19,8	75,1
	6	Bovine fat II	-20,8	70,1
	7	Llama jerkey fat	-28,6	31,3
	8	Green pepper	-32,0	14,4
	9	Alubia beans	-34,9	0,0
ARCHAEOLOGICAL SAMPLES	10	Palo Blanco NH3 cooking pot	-30,7	20,9
	11	Palo Blanco NH3 cooking pot	-30,8	20,4
	12	La Troya V50 cooking pot	-25,9	44,8
	13	Punta Colorada cooking pot	-26,3	42,8
	14	Mishma 7 cooking pot	-27,5	36,8
	15	Mishma 7 Belén vessel	-26,1	43,8
	16	San Francisco aribaloid vessel	-27,8	35,3
	17	San Francisco aribalo vessel	-27,1	38,8
	18	Batungasta cooking pot	-23,2	58,2
	19	Batungasta cooking pot	-27,4	37,3
	20	Batungasta cooking pot	-26,3	42,8

Figure 19. Isotopic values and percent C_4 estimates of bulk total lipid extracts.

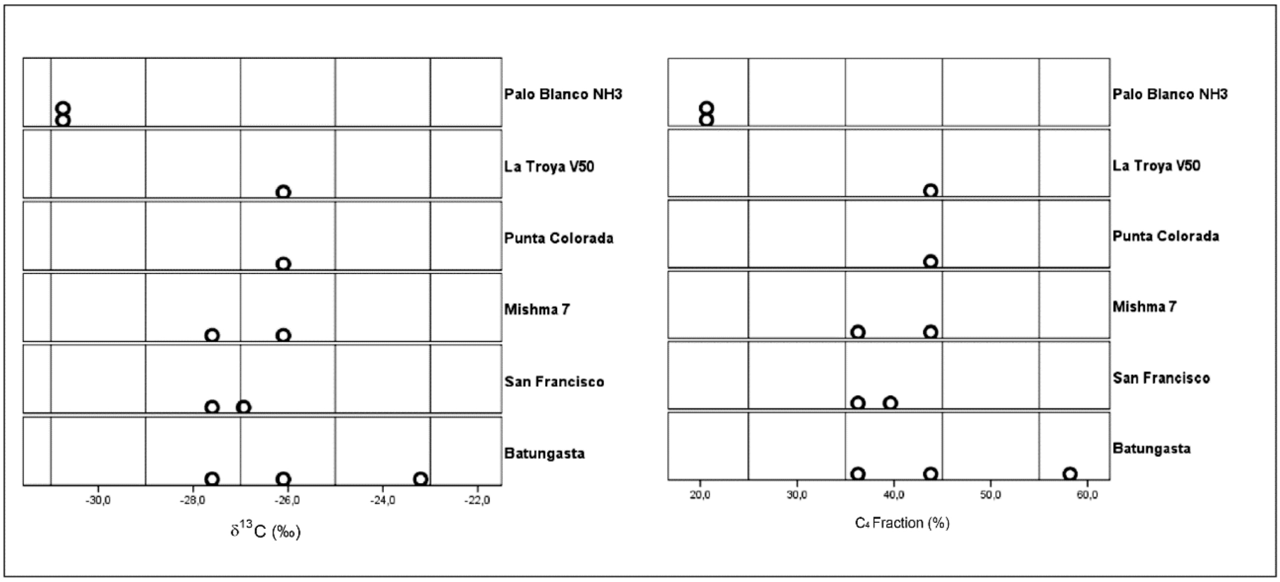

Figure 20. Distributions of $\delta^{13}C$ values and C_4 fraction of bulk lipid extracts from archaeological ceramic samples. The results are presented in relation to site chronology.

calibrated reference standards covering the entire ^{13}C range of the samples were also measured. Final results were expressed as $\delta^{13}C$, defined as:

$$\delta^{13}C = [(^{13}C/^{12}C)_{sample} / (^{13}C/^{12}C)_{V\text{-}PDB} - 1] \times 1000$$

where $\delta^{13}C$ is the isotopic deviation in ‰ and V-PDB is the international standard, (Coplen *et al.*, 2006; Gonfiantini 1978). The standard uncertainty is ±0.2‰.

The C_4 fraction in each sample was estimated with the following equation by Morton and Schwartz (2004):

$$P_{C4} = [(\delta_{sample} - \delta_{C3\ ref}) / (\delta_{C4\ ref} - \delta_{C3\ ref})] \times 100$$

where P_{C4} is the C_4 fraction in the sample, δsample is the $\delta^{13}C$ value of the sample, $\delta C_{3\ ref}$ is the most depleted modern reference value for C_3 plants and $\delta_{C4\ ref}$ is the most enriched reference value for C_4 plants (Seinfeld *et al.*, 2009). Given that the modern references and archaeological samples were all lipid extracts and therefore fractionation was equivalent in all cases, we considered the error reported by Hart *et al.* (2009) to be minimal. We also considered that modern samples are depleted on average -1.5‰ compared to archaeological samples from the pre-industrial period (Sonnerup *et al.*, 1999).

Preliminary Results and Discussion

Results of EA-IRMS analysis and C4 fraction estimations are presented in Figure 19. As expected, reference lipid samples of C_4 modern maize landraces had the most enriched $\delta^{13}C$ values varying from -15.9‰ to -14.8‰. These values are depleted in relation to standards for whole kernels that range from -11‰ to -9‰ (Killian Galván *et al.*, 2014). On the other hand, reference lipid samples of C_3 plants were in the range -34.9‰ to -32‰ which also is more depleted than the whole edible parts of these same species. In an intermediate position were the $\delta^{13}C$ values for bovine fat. The $\delta^{13}C$ value of sample from a bovine fed mostly on C_3 pastures was -20.8‰, while that of the fat from a bovine fed mostly on C_4 pastures was slightly more enriched at -19.8‰. The $\delta^{13}C$ value for llama jerky was -28.6‰ which leads us to infer that it was fed on C_3 pastures and its diet was not complemented with corn products or C_4 pastures. It is worth mentioning that the isotopic signals diminish with higher trophic levels, so that less differentiated values are expected from herbivores than plants (Gannes *et al.*, 1997).

The $\delta^{13}C$ values and C_4 fractions of extracted lipid residues from archaeological samples showed variations. The results from Palo Blanco NH3 (AD 458-639) had values which fell in the range of C_3 food products. The other two samples from La Troya V50 (AD 641-719) and Punta Colorada (AD 661-1020) pointed towards a mixed preparation of C_3 and C_4 food products. The samples from Mishma 7 (AD 1414-1573), Batungasta (AD 1445-1558) and San Francisco (AD 1400-1500) also pointed towards a mixed consumption, except for one case from Batungasta which had more enriched levels pointing towards a greater C_4 use.

Statistical analyses were carried out to observe trends between $\delta^{13}C$ values, chronology, and vessel type. All numerical analyses are exploratory, given the limited sample size (N=11).

Statistically, no significant trend was observed between $\delta^{13}C$ values and site chronology, which was determined by Pearson's x^2 test (bilateral asymptotic significance: 0.279; obtained value: 6.294) using the SPSS 19 software (IBM, 2010). Nevertheless, the distinctly negative values of Palo Blanco NH3 contrast with one markedly enriched value from Batungasta, while the remaining samples are in an intermediate position (Figure 20). This information is insufficient to propose an increase of maize dependence through time, especially considering the restricted sample size, but it does pose the question of a greater access to maize in Batungasta compared to other locations. Also, the lower isotopic values in Palo Blanco could respond to a greater access to animal products rather than a maize-based diet complemented with some C_3 plants (e.g. beans, peppers, squash, quinoa, algarroba, etc.) and limited animal products as seen in most sites from the first millennium AD, in contrast to site from the Inka period (thirteenth to mid-fourteenth centuries AD).

In terms of morphological and functional properties of the samples, we observed no trends when comparing vessel morphological type and isotopic patterns.

Everyday cooking pots and ritual vessels such as aryballos, aryballoid, and Belén vessels did not separate into two distinct groups. This was contrary to our expectations, because we had predicted a higher C_4 signal in ritual vessels used for maize beer (*chicha*) production and consumption found in sites dedicated to ritual functions such as San Francisco (Orgaz *et al.*, 2007; Orgaz 2012). This could not be inferred from the results obtained in this study, possibly due to the small sample of ritual ceramic wares analysed. An alternative hypothesis that remains for future studies is the use of the ritual vessels for the production and consumption of maize chicha and algarrobo aloja alcoholic drink, resulting in mixtures of C_4 and C_3 signals which are coherent with the values obtained in samples of ritual vessels. Also, animal fat may have been added post-firing to the inner walls of vessels in order to make them impermeable. This would have contributed to the mixed isotopic signals observed in our aryballos, aryballoid, and Belén vessels samples.

Conclusion

In this paper we studied the use and consumption practices of C_3 and C_4 food products in bulk lipid extracts from ceramic samples in West Tinogasta region. This preliminary analysis showed a great variability of use and consumption practices which could be explained by the multiple recipes and functions a pot had during its use life resulting in organic residue ‘palimpsests’. No statistically significant correlation existed between site chronologies and isotopic signals, but a differential access to maize in the Batungasta Inka site was recognized. Another idea prompted from this study is that consumption and storage of both aloja and chicha alcoholic beverages occurred in the same festive wares, which could explain the mixed isotopic signals in this special kind of vessels. In sum, the present study demonstrated the usefulness of carbon stable isotope analysis on bulk lipid residues and triggered hypothesis for future studies on the foodways of pre-Hispanic West Tinogasta societies.

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