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Preliminary molecular evidence of feasting in the Inca site of Fuerte Quemado-Intihuatana, Catamarca, Argentina

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ABSTRACT

Feasting was an important aspect of the domination strategy designed by the Inca Empire in the provinces. Hospitality banquets were the setting for negotiations between Cuzco and the annexed populations. Consumption of food and drink played a fundamental role in these feasts. In this paper we present the first study of organic residues recovered from ceramic vessels from the archaeological site of Fuerte Quemado-Intihuatana (Catamarca, Argentina), an important settlement of the *Collasuyu* province. Earlier functional studies proposed that these vessels were used to store and serve food and drink in commensal contexts. Results from this preliminary molecular study support this hypothesis because all the containers yielded organic residues. Chemical and isotopic studies suggest that food and different kinds of beers were held in these containers during festive events.

1. Introduction

Feasting was an important part of the Andean pre-Hispanic worldview and played a fundamental role in social cohesion, both in domestic and communal spaces. Festive events were total social facts that knit the fabric of economic, politic, and symbolic consumption practices (Dietler, 2006; Mintz and Du Bois, 2002). In pre-State decentralized Andean societies, food and drink for festive events were produced at a domestic or communal scale, and consumption practices were rooted in symmetric commensality and reciprocity (Logan et al., 2012). However, during the Inca expansion festive events were hosted by the State and consumption practices shifted towards asymmetrical commensalism (Bray et al., 2009; Dillehay, 2012; Moore, 2013). Production became specialized and organized by a central power, distribution was monopolized, and consumption took place in contexts of social segregation which crystallized hierarchies and unequal power relations (Bray, 2003; Hastorf, 1990).

Northwest Argentina was part of the *Collasuyu* southern Inca province during the 15th and 16th centuries AD, and festivities involving food and drink were often sponsored by the central State (Giovannetti et al., 2013; Leibowicz, 2013; Williams et al., 2005). Etnohistorical accounts suggest that the Inca drink of preference was *chicha* made form maize (*Zea mays*), although other fermented beverages were produced and consumed (Cobo, 1964). These beers were made from local resources such as mesquite or algarroba (*Prosopis*), mistol (*Ziziphus mistol*), chañar (*Geoffroea decorticans*), aguaribay or molle (*Schinus*), quinoa (*Chenopodium quinoa*), amaranth (*Amaranthus*), and peanut (*Arachis hypogaea*) (Biwer and VanDerwarker, 2015; Goldstein et al., 2009; Laffey, 2015). The availability of cultivated or gathered plants may have determined which raw material was used to produce drinks in each region. Also, the native fermentation recipes could have coexisted with the specialized production practices introduced by the Inca, such as the production of maize *chicha* at a large scale. As a consequence, one of the State's strategies was the uprooting and resettling of *mitimae* populations assigned to intensive agricultural production (Williams, 2000). The State offered sustenance-intoxication in the form of food and *chicha* beer in ritual contexts in order to mobilize workforce, to settle agreements with local authorities, and to destroy or re-signify local worship to the ancestors and other-than-human entities (Bray, 2012; Malpass and Alconini, 2010; Nielsen, 2010; Orgaz and Ratto,

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2015; Shimada, 2015; Sternfeld, 2007). Also, a syncretism between the local festivities and the new commensal practices introduced by the Incas has been proposed (Orgaz, 2012). Some local festivities with pre-Hispanic roots such as the *Chiqui*- where large amounts of *aloja* made from mesquite were consumed- have even survived up to the Colonial and Republican eras (Carrizo, 1942; Gentile, 2001; Karlovich, 2005).

The change in the scale of food and drink consumption during feasts implied a more complex organization of labor in order to carry out each of the many steps involved in raw material procurement and production. In the case of beers it included selection of seeds/pods/fruits, grinding, kneading, boiling, brewing, fermenting, decanting, straining, separating, storing, transporting, and serving (Cremonte et al., 2009; Hayashida, 2008; Parker and McCool, 2015). Simultaneously, large quantities of food were prepared for the banquet, including different kinds of roasts and stews (Hastorf, 2003).

The complex chain of food and drink production also implied the development of a specific ceramic assemblage for each step of the elaboration, storage, transport and service of foods and drinks. The Inca ceramic "culinary equipment" was designed not only to efficiently carry out its functional purpose, but also in some cases to be publicly exhibited during the libations and feasts. This was particularly true with the morphological types that were meant to be seen, such as the *aríbalos* and aribaloids, which boasted intricate decorations (Bray, 2003). Some local fineware such as *Santa María* vessels and *pucos* (bowls) could have also been used to serve food and drinks (Greco et al., 2012; Lantos et al., 2015; Orgaz, 2012). Other containers which were used for the first stages of production and decantation had no decoration. Because of their function, these vessels often had signs of soot and/or heavy stirring (Cremonte et al., 2009). Some of these pots may also have been multifunctional, and could have been used to both prepare stews and fermented drinks.

1.1. The archaeological site of Fuerte Quemado-Intihuatana in the Yocavil valley

The Yocavil valley is part of the Calchaquí valley system that is defined by the Sierra del Cajón mountainous chain to the West and the Calchaquí and Aconquija ranges to the East. The Santa María river runs along the valley North to South, and on each margin there are numerous alluvial cones from tributary streams that run into the main drainage system (Ruiz Huidobro, 1972). The valley is known for its numerous archaeological sites with prominent monumental constructions. During the 11th to 15th centuries, a complex social and political system developed in this region, which materialized in many large and highly populated settlements, increasingly complex organization of labor, specialized artisanship, and intricate funerary traditions (Tarragó and González, 2004; Tarragó et al., 1999). This was the social setting when the Inca Empire arrived at the end of the 15th century AD to the Yocavil valley. The domination strategies from Cuzco defined a new cultural pattern and a different spatial distribution of imperial assets in the annexed territories. This suggests a significant variability in the strategies and negotiations that took place between local and imperial societies, resulting in specific archaeological records in each of the sites located along the valley (González and Tarragó, 2005; Orgaz, 2014; Reynoso, 2003).

Fuerte Quemado-Intihuatana was a densely populated settlement located in the northern section of the Yocavil valley, Catamarca province, at an altitude of 1900 m.a.s.l. (Fig. 1). It was one of the largest administrative Inca sites in NW Argentina, and was declared Provincial Historical Site in 2006. In this place local and Inca cultures were negotiated and re-signified. It is. The site is defined by a building conglomerate that extends in a West-East direction from the summit of a rocky outcrop that is part of the Cajón chain to the slope and alluvial plain of the Simonita and Santa María rivers, covering a total area of three squared kilometers (Fig. 2A).

The site's architectural remains were first described in the late 19th and early 20th centuries (Bruch, 1911; Lafone Quevedo, 1904). After a long hiatus, the investigations at Fuerte Quemado were resumed in the late 1970s and 1980s (Kriscautzky, 1999). In 2006 the locality was declared Provincial Historical Site by the government of Catamarca, Argentina. The extensive research at the site established that Fuerte Quemado-Intihuatana had a complex history of interaction between the local political entities and the Inca Empire. These interactions included both domestic activities related to the social reproduction of the inhabitants, as well as activities carried out in ceremonial contexts related to the Inca domination strategy (Kriscautzky, 1999; Orgaz and Kriscautzky, 2012; Orgaz, 2014, 2012).

Two types of occupation were established by cultural indicators such as architectural and ceramic styles: Local pre-Inca and Inca (Kriscautzky, 1999). Sectors I, II, III, V, and VI were built during the pre-Inca Late Intermediate period (11th to 15th centuries AD) by local societies and their occupation continued into the Inca period (15th and 16th centuries AD). These sectors are dispersed along the slopes and hillsides of the valley. Sector IV is located in the central urban area and sector VII is placed in the summit of the granitic outcrop. These last two sectors were built by the Inca when they expanded into the Yocavil valley.

In this paper we focus on samples from the ceramic assemblages recovered by Dr. Néstor Kriscautzky during the 1970s and 1980s excavations of two architectural features: Enclosure R-51 and Enclosure C-43 (Fig. 2B, C and D). Previous studies of the architecture, ceramic assemblage, botanic remains, and recovery of foreign objects, showed that these enclosures were specifically used for feasting (Orgaz, 2012, 2014).

Enclosure R-51 is located in the Sector V. It a spacious elliptic construction made from well finished stone walls, and it has no direct access in or out (Fig. 2B and C). A ceramic MNV (minimum number of vessels) of 31 was calculated in this assemblage, including fineware (three *aríbalos*, five aribaloids, eleven *pucos*, three *Santa María* vessels) and coarseware (two pedestal pots, two cone based globular pots, five globular pots) (Orgaz, 2014). The assemblage pointed towards storage and consumption of food and drinks. The small amount of cooking pots coupled with the existence of only one small hearth and no other cooking implements, suggested that food was not prepared in this enclosure. The food consumption practices carried out in Enclosure R-51 indicated a high level of social hierarchy and segregation, due to the low accessibility of this closed private space, the high amount of fineware (71%) *versus* coarseware (29%), the locally manufactured pottery that imitated Inca styles (aribaloids and pedestal pots), and the provincial style Inca pottery (*aríbalos*). It was proposed that this space was used for private commensal practices within a local elite residence (Orgaz, 2014).

Enclosure C-43 is located in sector IV (Fig. 2D). It is an open rectangular space built with stone and mortar with one wide opening on the Eastern side. In this space a ceramic MNV of 19 was calculated, including fineware (one *aríbalo*, five aribaloids, 12 *pucos*) and coarseware (one pedestal pot) (Orgaz, 2012). This assemblage was exclusively used for serving and consuming food and drink. No cooking pots or large storage vessels were found, and only a small hearth was detected, indicating that this space was not used to prepare food (Table 1). It was proposed that this building was dedicated to public commensal practices where food and beverages were shared (Orgaz, 2012).

Functional studies of the vessels found at R-51 and C-43 provided insight into the size, surface treatment, decoration, and use-alteration marks of the assemblage (Orgaz, 2014, 2012). Initial results indicated that:

a) *Aríbalos* and aribaloids may have been employed to store liquids due to their conic or semi-conic bases, constricted bottle-like necks, smoothed inner surfaces and slipped, polished, and painted outer surfaces (Fig. 3A). Various authors, based on ethno-historical records, have stated that *aríbalos* and aribaloids were designed and

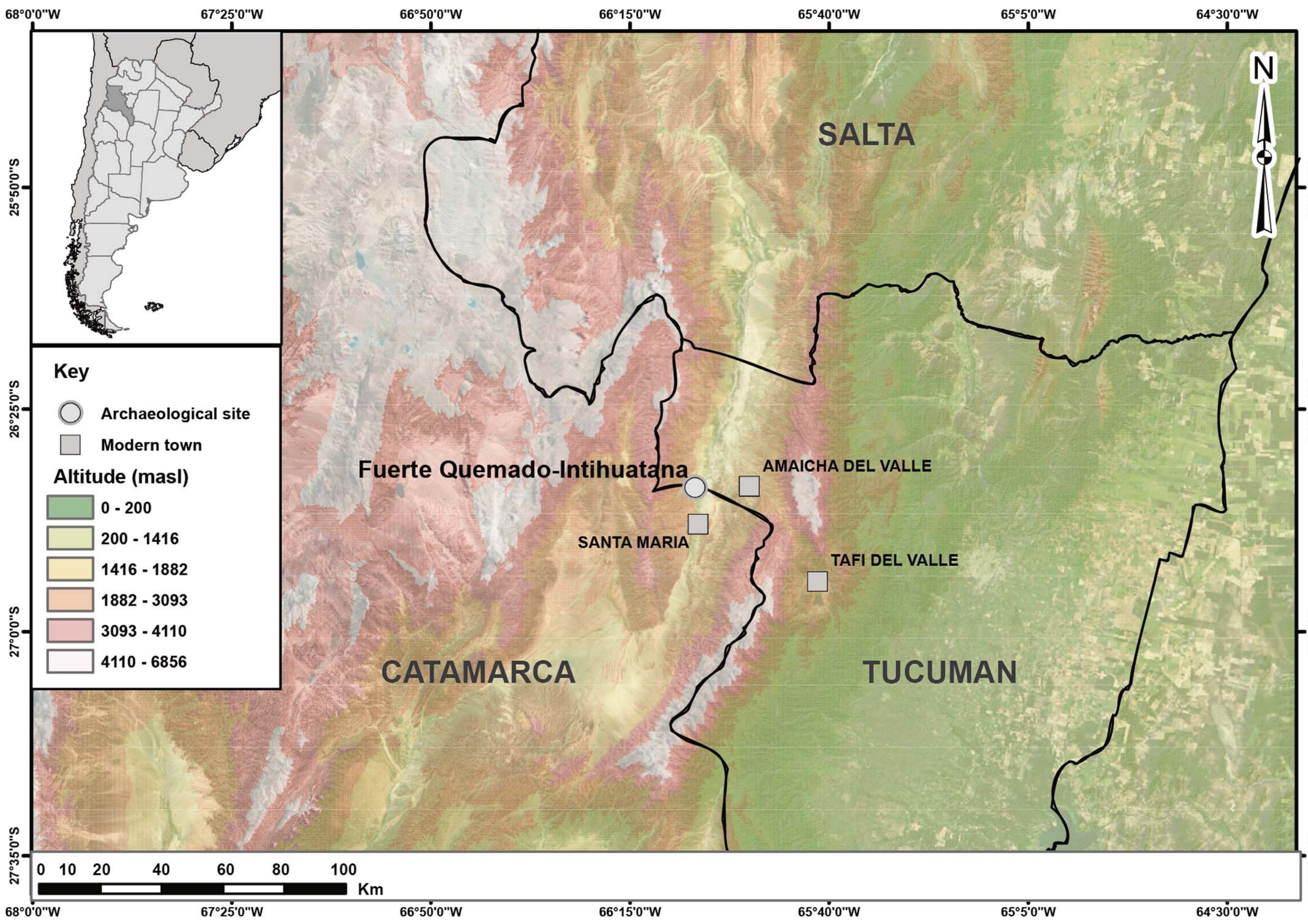

Fig. 1. Geographical location of the site Fuerte Quemado-Intihuatana.

used by the Inca to store, transport and pour alcoholic beverages during festivities (Bray, 2003; Moore, 2013). *Aríbalos* have classic Inca shapes with provincial decorative style, while aribaloids have a slightly modified shape and a provincial Inca decorative style (Calderari and Williams, 1991). We found no evidence of soot that could indicate these vessels were placed on hearths to prepare stews.

b) *Santa María* vessels were possibly used for storage, given their morphological features such as narrow bases, ovoid bodies, and wide necks, as well as distinctive slip and decorations. The large capacity and thick walls were linked to long term storage, and the everted thick lips were thought to be designed to cover the mouth with textiles or animal hide. The absence of soot and use-alteration marks -such as internal attrition- indicates that these vessels were not used to cook. (Fig. 3B). This local style coexisted with Inca styles during the State occupation (Calderari and Williams, 1991). An ongoing debate exists on the function of *Santa María* vessels, as they had been originally described as urns for funerary purposes, given their association to human remains (Marchegiani et al., 2009). However, several recent discoveries of *Santa María* vessels with visible use-alteration marks in domestic contexts point towards a wider range of functions, such as storage, processing or cooking (Piñeiro, 1996; Amuedo, 2012; Greco et al., 2012).

c) Globular and pedestal pots were potentially used to prepare or reheat foods, based on the soot marks on the external surfaces and the absence of decoration. In contrast, cone based globular pots were possibly used to store fermented beverages, given their large storage capacity, small mouth diameter and large maximum diameter that is optimum to prevent liquid spilling, conic base designed for the decantation of the fermentation residue, surface alteration due to burial of the conic base to stabilize the vessel for long periods of time, and absence of soot or decoration (Menacho, 2007; Valdez, 2002) (Fig. 3C).

d) *Pucos* were proposed to serve as bowls to consume foods or liquids, given their open shape, decoration and size (Orgaz, 2014) (Fig. 3D).

The preliminary functional characteristics of the vessels are hypothetical and require additional research into the organic residues preserved within the ceramic matrixes in order to provide further knowledge on their use, such as the foods and drinks prepared, stored, and served in enclosures used for feasting at Fuerte Quemado-Intihuatana. We selected ceramic samples that were good candidates for organic residue analysis. We chose to study samples which had: (a) absence of glue and/or marker labels; (b) well preserved surfaces and matrixes. Only seven fragments met these criteria. None of them had any adhered crusts, so residues were assumed to be absorbed in the matrix. Six sherds were from Enclosure R-51: one *aríbalo*, one aribaloid, one *Santa María* vessel, one cone based pot, and two *pucos*. One sherd from an aribaloid was from Enclosure C-43 (Table 2). Unfortunately, no sediment samples were available from the 1970s and 1980s excavations. Although the small sample size and lack of control sediments were not the ideal situation for organic residue analyses, these were the only samples available for analysis. Both enclosures R-51 and C-43 were completely excavated and there is no possibility to re-excavate in order to obtain new samples under modern conditions of retrieval and manipulation. Nevertheless, the seven samples that did qualify for organic residue analysis provided important information that would have been otherwise left unexplored.

In addition to the archaeological samples, we selected plant and

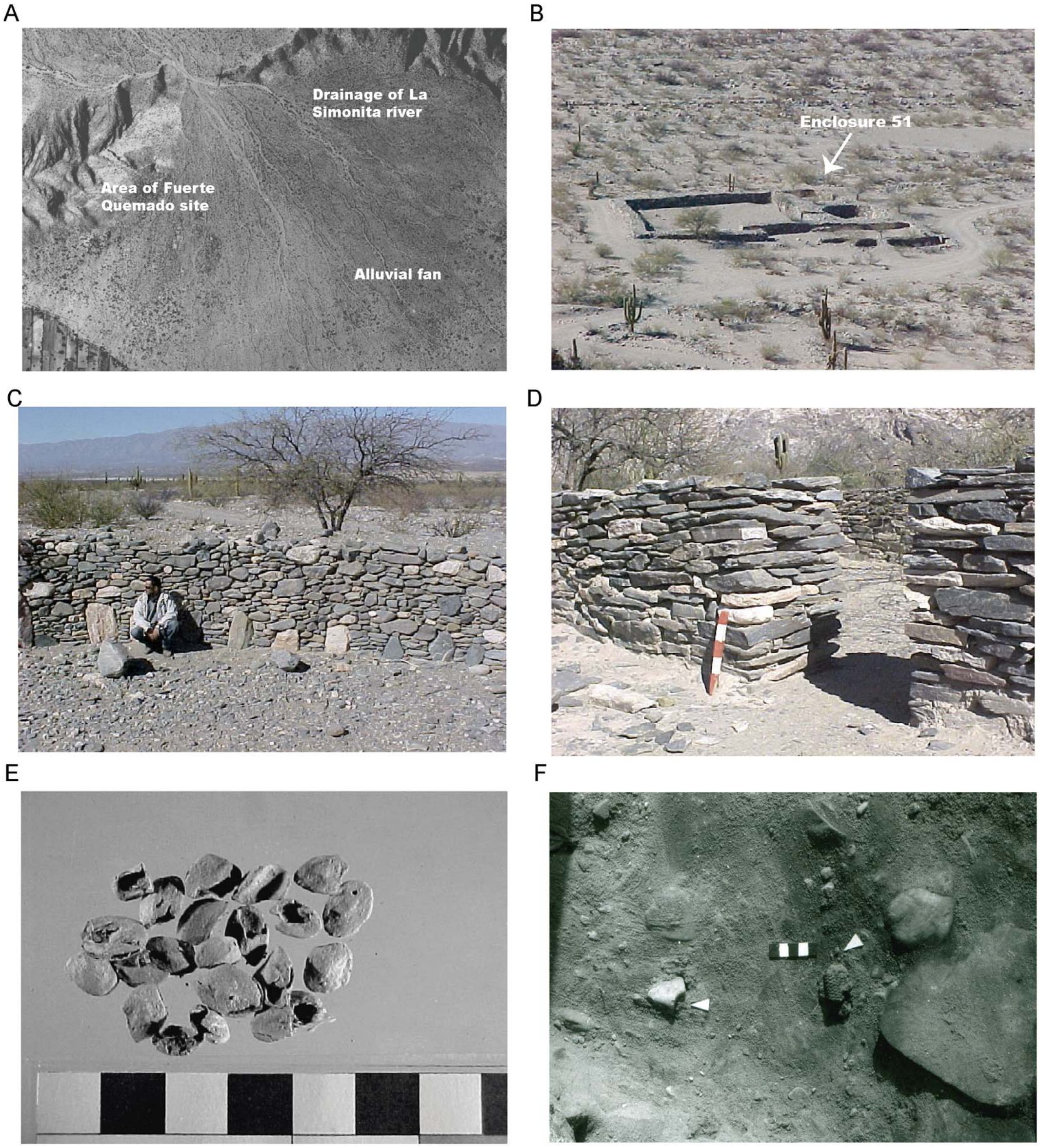

Fig. 2. Images from Fuerte Quemado-Intihuatana: A) Satellite image of site location and surrounding landscape; B) General view of Sector V and Enclosure 51; C) View of the entrance to Enclosure 51; D) View of Enclosure 43 in Sector IV; E) Mesquite botanical remains recovered from Sector IV; F) Maize carbonized botanical remains recovered from Sector IV.

Table 1
Ceramic assemblages from Enclosures R-51 and C-43 in Fuerte Quemado-Intihuatana.

Ceramic morphological type	Enclosure R-51	Enclosure C-43
Aríbalo	3	1
Aribaloid	5	5
Pedestal pot	2	1
Cone based globular pot	2	0
Globular pot	5	0
Puco	11	12
Santa María vessel	3	0
Total	31	19

animal reference samples following the findings in the archaeobotanical and zooarchaeological record of Fuerte Quemado-Intihuatana. These include: maize (dentado blanco; *Zea mays* var. *indentata* L.), mesquite (*Prosopis nigra* Griseb.), chañar (*Geoffroea decorticans* Gill. ex Hook. & Arn.; Burkart), and llama (*Llama glama* L.) (Table 2, Fig. 2E and F).

1.2. *Chemical analyses of culinary organic residues*

Organic residues resulting from the preparation, storage, transport and service of foods and beverages can be well preserved in the porous matrixes of ceramic containers (Copley et al., 2005). Absorbed lipid residues are complex mixtures which form during the container's multiple uses during its life history (Evershed, 2008; Skibo, 1992). Residues can be the unintentional result of culinary activities, or they can be the result of intentional coating of inner surfaces in order to impregnate the pores and avoid evapo-transpiration of liquids (Henrickson and McDonald, 1983; Otero, 2006; Schiffer, 1990; Skibo, 1992).

The characterization of lipid residues from foods and beverages has been successfully achieved by a combination of chemical and isotopic analyses, applying methods such as gas chromatography–mass spectrometry (GC–MS) and bulk or compound specific isotope ratio mass spectrometry (IRMS) (Colombini and Modugno, 2009; Evershed, 2008).

In this paper we studied the lipid residues recovered from ceramics from Fuerte Quemado-Intihuatana. Chemical characterization of fatty acids and neutral lipids was done by GC–MS. Isotopic analysis of bulk lipids (total lipid extracts) was done by elemental analysis-isotope ratio

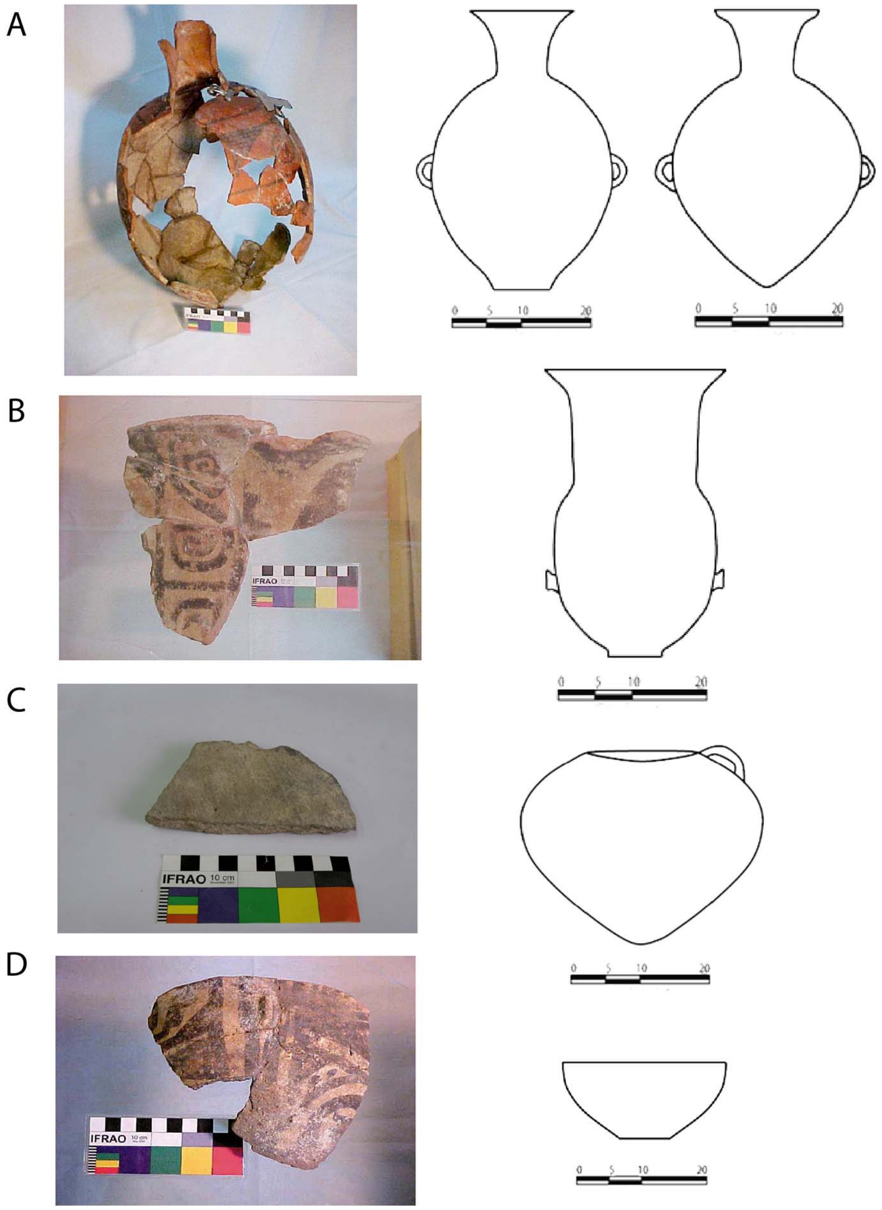

Fig. 3. Morphological types of vessels studied in this paper: A). *Santa María* vessel; B) *aríbalo* and aribaloid; C) puco; D) conic base pot.

mass spectrometry (EA-IRMS). Results from both methods were combined in order to identify the origins of lipids within complex mixtures.

2. Materials and methods

Lipid extraction was carried out on all archaeological and reference samples (Table 1). Powder of the dry samples (maize kernels, mesquite pods, chañar fruits) was obtained by grinding with a coffee mill. The fresh llama fat was frozen and ground using a porcelain mortar and pestle. Archaeological potsherds were first rinsed on both surfaces with chloroform:methanol (2:1; vol/vol), they were then broken into small fragments with a hammer and ground in a porcelain mortar and pestle.

Table 2
Description of archaeological and reference samples in this study.

Sample	Code	Location	Context	Type of sample
A1	FQ-R51-SN	Fuerte Quemado-Intihuatana, Catamarca	Section V, Enclosure 51	Aribaloid
A2	FQ-C43-SN	Fuerte Quemado-Intihuatana, Catamarca	Section IV, Enclosure 43	Aribaloid
A3	FQ-R51-183	Fuerte Quemado-Intihuatana, Catamarca	Section V, Enclosure 51	*Aríbalo*
A4	FQ-R51-279	Fuerte Quemado-Intihuatana, Catamarca	Section V, Enclosure 51	*Santa María* vessel
A5	FQ-R51-268	Fuerte Quemado-Intihuatana, Catamarca	Section V, Enclosure 51	Cone based globular pot
A6	FQ-R51-353	Fuerte Quemado-Intihuatana, Catamarca	Section V, Enclosure 51	*Puco*
A7	FQ-R51-319	Fuerte Quemado-Intihuatana, Catamarca	Section V, Enclosure 51	*Puco*
R1	n/a	Tiraxi, Jujuy	n/a	Maize (Dentado Blanco; *Zea mays* var. *indentata L.*)
R2	n/a	El Alto, Catamarca	n/a	Mesquite (*Prosopis nigra* Griseb.)
R3	n/a	El Alto, Catamarca	n/a	Chañar (*Geoffroea decorticans* Gill. Ex Hook. & Arn.; Burkart)
R4	n/a	La Candelaria, Jujuy	n/a	llama (*Lama glama* L.)

Table 3

Bulk lipid isotopic values and fatty acid methyl ester (FAME) profiles of archaeological samples and modern references samples in this study. References: $\delta^{13}C$ bulk lipid values ($\delta^{13}C$), C_4 fraction ($\%C_4$), capric acid (C10:0), lauric acid (C12:0), miristic acid (C14:0), 12-methyl-tetradecanoic acid (12*Me*-C14:0) pentadecanoic acid (C15:0), 14-methy-pentadecanoic acid (14*Me*-15:0), palmitoleic acid (C16:1), palmitic acid (C16:0), 14-methyl-hexadecanoic acid (14*Me*-C16:0), margaric acid (C17:0), linolenic acid (C18:3), linoleic acid (C18:2), oleic acid (C18:1), stearic acid (C18:0), eicosanoic acid (C20:0), docosanoic acid (C22:0), tetracosanoic acid (C24:0), DA (dicarboxylic acid), *corrected values.

Sample	A1	A2	A3	A4	A5	A6	A7	R1	R2	R3	R4
Description	Aribaloid	Aribaloid	*Aríbalo*	*Santa María* vessel	Cone based globular pot	*Puco*	*Puco*	Maize	Mesquite	Chañar	llama
$\delta^{13}C$	− 27.1	− 27.1	− 30.2	− 28.2	− 25.2	− 28.0	− 30.2	− 17.0*	− 31.1*	− 27.8*	− 31.1*
$\%C_4$	28.4	28.4	6.4	20.6	41.8	22.0	6.4				
C10:0			1.5								
C12:0	2.1		2.1		0.2		2.6				0.4
C13:0	2.1		0.7				0.6				
C14:0	10.6	5.9	13.3	16.9	10.0	16.3	16.7				6.5
12Me-14:0	1.7	1.3	2.5				0.4				1.6
C15:0	5.7	2.1	4.9	1.4	2.9	4.0	5.1				1.4
14Me-15:0			0.6				0.7				
C16:1			3.6		2.0				3.3		4.8
C16:0	34.7	30.2	39.6	36.5	43.2	40.5	43.2	27.3	46.3	15.0	33.0
14Me-16:0	1.1		2.1								0.8
C17:0	2.2	1.9			1.6		1.3				1.0
C18:3								0.8	1.5	10.4	
C18:2								28.5	10.6	40.3	0.7
C18:1	3.0	4.2	7.7	19.5	12.5	19.6	5.3	38.1	30.7	31.3	33.0
C18:0	23.8	30.6	20.3	25.7	26.6	17.1	21.3	5.2	7.7	3.1	16.8
C20:0	0.8	0.7			0.5						
C22:0	0.8	0.6									
C24:0	0.7										
C16:0/C18:0	1.5	1.0	1.9	1.4	1.6	2.4	2.0	5.3	6.0	4.8	2.0
Hexanodioic acid	0.3	2.1					1.2				
Octanodioic acid	1.9	5.3									
Nonanodioic acid	5.5	11.3	1.2		0.5	2.5	1.6				
Decanodioic acid	1.2	2.1									
Undecanodioic acid	1.1	1.8									
Dodecanodioic acid	0.8										

Lipids were extracted with chloroform:methanol (2:1; vol/vol) (Folch et al., 1957). All solvents were of chromatographic quality and pre-distilled before use. Each sample was placed in an ultrasound bath for 15 min (twice) and filtered; a few drops of distilled water were added, the organic phase containing the total lipid extract (TLE) was separated after centrifugation for 3 min (twice), evaporated under a soft nitrogen stream, weighed and then transferred to a 2 mL glass vial and stored at − 18 °C. An aliquot of the TLE was saponified with 1 mL of 4% potassium hydroxide in an ethanolic aqueous solution (2:1, vol/vol), at 60 °C for 2 h (Colombini et al., 2003). After cooling at room temperature, the neutral fraction was extracted with 1.5 mL *n*-hexane and the aqueous fraction acidified with 2 N HCl solution to pH 3 and extracted with 1.5 mL diethyl ether. The ethereal phase containing the free fatty acids was evaporated under N_2 stream and 0.5 mL of 20% boron trifluoride in methanol was added and heated in a boiling water bath for 3 min. After cooling, 1.5 mL of chloroform and a drop of water was added, and the organic phase containing the fatty acid methyl esters (FAME) was recovered and stored in 2 mL glass vials at 4 °C for GC–MS analysis. Trimethylsilyl derivatives (TMS) of the neutral fraction were prepared by addition of 20 mL of *N,O*-bis (trimehtylsilyl) triflouroacetamide (BTSFA) with 1% trimethylchlorosilane (TMCS) (Supelco) and heating at 60 °C for 20 min. After cooling, the TMS derivatives were dried under a soft stream of nitrogen, *n*-hexane was added and the solution stored at 4 °C. Samples were analyzed within 24 h of derivatization. Procedure blanks for lipid extraction, saponification, methylation, and TMS derivatization were prepared and analyzed.

Chemical characterization of FAME by GC–MS was performed with a Shimadzu GCMS –QP5050A (Kyoto, Japan). The system was equipped with a Zebron ZB5 capillary column (Phenomenex, 5% phenyl- 95% dimethylpolysiloxane, 30 m length, 0.25 mm i.d., 0.25 μm film thickness). Helium was used as carrier gas (0.9 mL/min continuous flow rate) and manual injection was in split mode at a temperature of 250 °C. After an initial temperature at 110 °C, the column was heated to 280 °C at 10 °C/min followed by an isothermal period of 45 min. The MS was operated in the electron impact mode at 70 eV, source temperature of 290 °C. Compound identifications were carried out by comparing retention times of FAME standards and mass spectrometric fragmentation patterns. The relative abundances of individual FAME to total FAME in lipid extracts were calculated from total ion chromatogram (TIC) peak areas.

Chemical characterization of TMS derivatives of neutral lipids was carried out in a Shimadzu GCMS – QP5050A (Kyoto, Japan). The system was equipped with an Ultra 2 capillary column (Agilent, 5% phenyl-methylpolysiloxane, 50 m length, 0.20 mm i.d., 0.11 μm film thickness). Helium was used as carrier gas at a continuous flow rate of 0.9 mL/min. The injection was manual and in split mode at a temperature of 250 °C. The initial temperature was 100 °C, the column was heated to 240 °C at 10 °C/min followed by an isothermal period of 25 min, and then heated to 280 °C at 4 °C/min, followed by and isothermal period of 30 min. The MS was operated in the electron impact mode at 70 eV with a source temperature of 290 °C. Compound identifications were carried out by comparing retention times of sterol standards and mass spectrometric fragmentation patterns.

For EA-IRMS anlayses, an aliquot of the TLE was weighed (*ca.* 150 μg) and transferred to tin capsules. Samples were combusted in a Carlo Erba elemental analyzer coupled to a Thermo Delta V Advantage isotope ratio mass spectrometer by means of a CONFLO IV interface, using helium as carrier gas. A pure CO_2 standard was measured before every analysis. Three calibrated reference standards that cover the complete ^{13}C range were also measured every few analyses. The internal error was calculated in ± 0.2‰. Isotopic values were expressed in delta notation (δ) as per mil (‰) and calculated as the isotopic deviation of the samples from the international standard “Viena Peedee belemnite” (V-PDB) (Coplen et al., 2006; Gonfiantini, 1978).

$$\delta^{13}C = [(R_{\mathrm{sample}} - R_{\mathrm{standard}})/R_{\mathrm{standard}}] \times 1000)$$

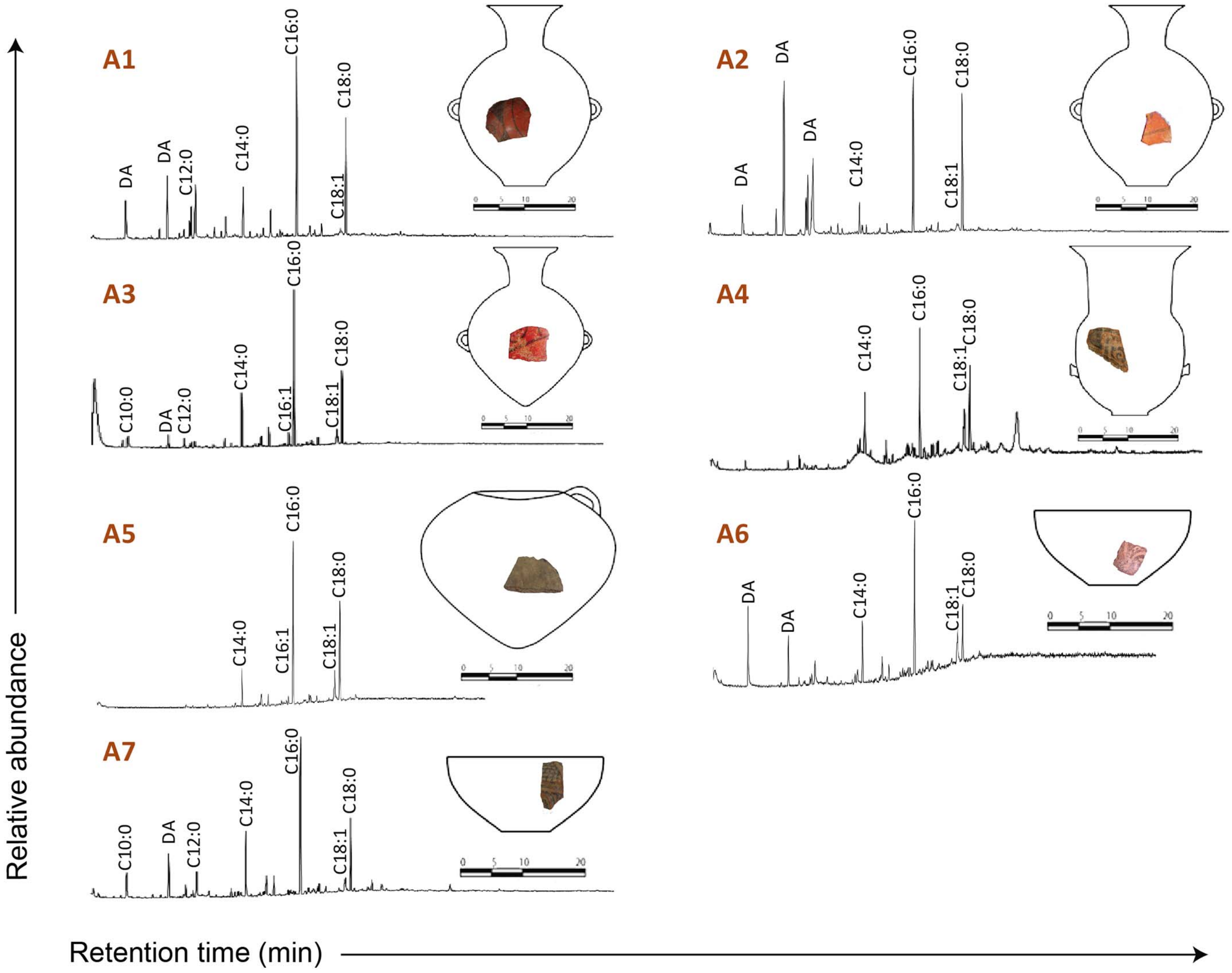

Fig. 4. Total ion current chromatographs of fatty acid methyl ester samples analyzed by GC–MS.

where $R = {}^{13}C / {}^{12}C$ and the standard is V-PDB.

The C_4 fraction of each sample was calculated using the following equation by Morton and Schwarcz (2004):

$$PC_4 = [(\delta_{sample} - \delta_{C3\ reference})/(\delta_{C4\ reference} - \delta_{C3\ reference})] \times 100$$

where PC_4 is the fraction of C_4 in the sample, $\delta_{samplea}$ is the $\delta\ {}^{13}C$ value of the archaeological sample, $\delta_{C3\ reference}$ is the lowest value obtained from C_3 plant reference samples, and $\delta_{C4\ reference}$ is the highest value obtained from C_4 plant reference samples. Given that modern and archaeological samples are all lipid extracts, we considered fractionation to be equivalent, thus the error reported by Hart et al. (2009) was dismissed. We also considered that modern samples are depleted in 1.6‰ in comparison to archaeological samples from the pre-Industrial era (Sonnerup et al., 1999). Hence, modern reference samples were corrected in this way for comparative purposes.

3. Results

Results from chemical analyses showed that all seven vessels (A1-A7) had organic residues resulting from contact with foods and/or drinks (Table 3 and Fig. 4).

The gas chromatograms from the archaeological vessels showed a series of methyl esters of carboxylic acids in the C_{10}–C_{24} range (Table 3). The most abundant saturated fatty acids (FA) were capric ($C_{10:0}$), lauric ($C_{12:0}$), myristic ($C_{14:0}$), palmitic ($C_{16:0}$), and stearic ($C_{18:0}$) acids, maximizing at C_{16} and C_{18}. The unsaturated fatty acids were palmitoleic ($C_{16:1}$) and oleic ($C_{18:1}$) acids. In some archaeological samples trace amounts of pentadecanoic ($C_{15:0}$), margaric ($C_{17:0}$), and branched *iso* and *anteiso* carboxilic acids (12-methyltetradecanoic acid, 14-methylpentadecanoic acid and 14-methylhexadecanoic acid) were found. The presence of odd chained FA, both linear and branched, in samples A1, A2, A3, and A7 suggests the presence of ruminant animal fat (Martínez Marín et al., 2010; Spangenberg et al., 2006). In this archaeological context, South American camelids are the most probable sources (Lantos et al., 2015; Maier et al., 2007; Miyano et al., 2017; Vázquez et al., 2008). Although bacterial contamination cannot be discarded as a possible source of branched fatty acids (Dudd et al., 1998), this is unlikely given that: (a) preliminary analysis by HPLC-ESI of samples from Fuerte Quemado showed that lipids are preserved predominantly as intact triacylglycerols, and some of these intact triacylglycerols contain odd chain fatty acids (Lantos et al., 2017); (b) other microbial biomarkers such as ergosterol are absent in all samples; (c) odd chained fatty acids, both linear and branched, were indentified in our llama reference sample. Small amounts of eicosanoic ($C_{20:0}$), docosanoic ($C_{22:0}$) and/or tetracosanoic ($C_{24:0}$) were found in three samples (A1, A2, A5). These long chain fatty acids could indicate the presence of plant lipids as well as lipids from fish or shellfish. Given that access to marine or fresh water resources is highly unlikely due to the location of the site and the absence of zooarchaeological remains of these resources (Kriscautzky, 1986), plants are the most probable origin of these fatty

Table 4
Neutral lipids (TMS derivatives) identified in archaeological and reference samples in this study.

Sample	Code	Neutral lipids
A1	FQ-R51-SN	Cholesterol, pentacosane, hexacosane, heptacosane
A2	FQ-C43-SN	Cholesterol
A3	FQ-R51-183	Cholesterol
A4	FQ-R51-279	Cholesterol
A5	FQ-R51-268	Cholesterol, cholesta-3,5-dien-7-one
A6	FQ-R51-353	Cholesterol
A7	FQ-R51-319	No signal
R1	Maize	Campesterol, dihidrocampesterol, stigmasterol, sitosterol, sitostanol or stigmastanol
R2	Mesquite	Campesterol, stigmasterol, sitosterol
R3	Chañar	Sitosterol stigmastanol
R4	Llama	Cholesterol

acids[1]. Dicarboxylic acids (hexanodioic, octanodioic, nonanodioic, decanodioic, undecanodioic, and dodecanodioic acids) were found in 6 of the 7 vessel samples. These dicarboxylic acids can be the oxidation products of longer mono and polyunsaturated FA, which are indicators of degraded vegetable oils, as well as the products of hydrolysis of cutin and suberin, which are components of plant cuticular waxes.

Neutral lipids (NL) were found in most archaeological vessels (6:7) (Table 4). Cholesterol and/or its degradation product (cholesta-3,5-dien-7-one) (Gómez et al., 2016) were detected. This information supports the presence of animal fats in the residues. Plant sterols were not found in the archaeological samples. This is not uncommon in archaeological samples given that the sterol concentration in plant food products is very low. No alkanols were found in any of the archaeological samples. Small amounts of alkanes (pentacosane, hexacosane, heptacosane) were found in one sample (A-1), but alkanes are also common in sediments, and can be present in archaeological samples as the result of contamination. Given that no sediment samples were available for analysis, we cannot use alkanes as reliable markers of plant lipids.

When comparing the FA profiles (Table 3) with NL profiles (Table 4) of archaeological and modern reference samples, we can suspect that archaeological lipids are degraded mixtures of oils and fats. Plant reference samples (R1 maize, R2 mesquite, and R3 chañar) have higher amounts of unsaturated FA such as linoleic ($C_{18:2}$) and linolenic ($C_{18:3}$) acids that naturally disappear in archaeological samples due to oxidation processes (Maier et al., 2005; Regert et al., 1998). Lipid profiles for maize, mesquite and chañar were similar to published data (Lamarque et al., 2000, 1994; Woodbury et al., 1995). The neutral lipids found in maize were campesterol, dihydrocampesterol, stigmasterol, sitosterol, and sitostanol or stigmastanol. In mesquite campesterol, stigmasterol, and sitosterol were detected. Neutral lipids from chañar were sitosterol and stigmastanol. Llama reference sample (R4) showed a typical animal fat profile comparable to reported data (Coates and Ayerza, 2004), and also had trace amounts of the branched *iso* an *anteiso* carboxylic acids combined with odd numbered fatty acids, identical to those found in the archaeological samples. Cholesterol was the only neutral lipid found in this sample.

The palmitic/stearic ratio ($C_{16:0}/C_{18:0}$) was calculated for all samples. Both $C_{16:0}$ and $C_{18:0}$ are abundant saturated fatty acids with similar oxidation rates and therefore constitute good indicators of animal or plant lipid origin in a sample (Colombini et al., 2005; Eerkens, 2005; Malainey et al., 1999). Although microbial breakdown of lipids can affect the palmitic/stearic ratio ($C_{16:0}/C_{18:0}$) causing ratios of degraded plant lipids to look similar to animal fat ratios, the palmitic/stearic ratio was still calculated as a proxy in our samples, given that the microbial contribution was low (Lantos et al., 2017). We observed variations of this ratio in the archaeological samples, suggesting that different substances or mixtures may have been contained in each vessel.

The $\delta^{13}C$ values of bulk lipid extracts and the C_4 fraction calculations were not homogenous and showed variations between archaeological samples (Table 3). The archaeological sample with the highest $\delta^{13}C$ value and C_4 fraction was A5 (− 25.2‰, 41,8%), followed by A1 and A2 (− 27.1‰, 28.4%), A6 (− 28.0‰, 22%), A4 (− 28.2‰, 20,6%), A3 and A7 (− 30.2‰, 6,4%). These values were compared with $\delta^{13}C$ values of bulk lipid extracts of reference samples. Maize is a C_4 plant and obtained a corrected value of − 17.0‰. Mesquite and chañar are C_3 plants and obtained corrected values of − 31.1‰ and − 26.2‰, respectively. The llama sample had a corrected value of − 31.1‰.

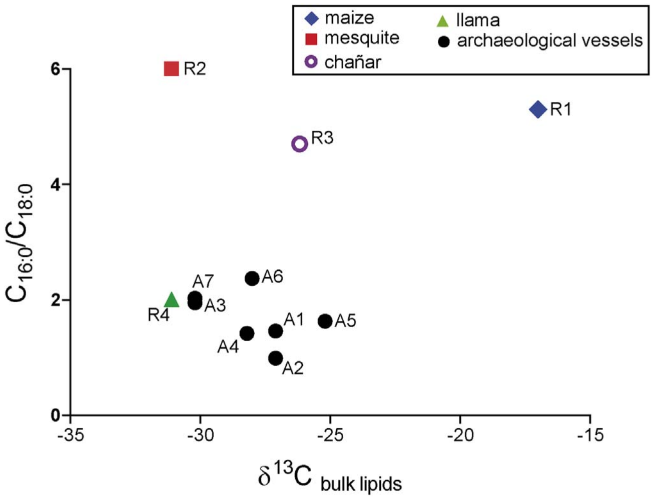

Fig. 5. Relation between the palmitic/stearic ratio ($C_{16:0}/C_{18:0}$) and the $\delta^{13}C$ value of lipids from archaeological samples and modern reference samples.

A biplot of bulk lipid $\delta^{13}C$ values and the palmitic/stearic ratio ($C_{16:0}/C_{18:0}$) is shown in Fig. 5. Most archaeological samples are plotted near to the llama reference sample. Palmitic/stearic ratios are very similar to llama values (ranging from 1.0 to 2.4 on the y-axis), although $\delta^{13}C$ values have more variation (ranging from − 30‰ to − 25‰ on the x-axis) indicating different mixtures of animal fats with C_3 and/or C_4 plant lipids.

4. Discussion

Results from chemical and isotopic analysis allowed further insight into the use of the vessels from feasting contexts in the enclosures R-51 and C-43 in Fuerte Quemado-Intihuatana.

Aribalos and aribaloids had a hypothetical function as containers to store and transport a variety of beverages. This study found that the lipid residues from this group of vessels (A1, A2, and A3) were complex mixtures. In all samples an important component of camelid fat was found, evidenced by the FA profiles, the odd and branched FA, the low palmitic/stearic ratios, and the presence of cholesterol. The data points towards the use of camelid fat to seal the inner surfaces. Also in some samples there is some evidence of plant oils, such as long chained saturated FA (C_{22}–C_{24}) and dicarboxylic acids which are oxidation products of unsaturated fatty acids of possible plant origin. The isotopic analysis indicated C_4 fractions ranging from 28.4% to 6.4%. The variation in isotopic values suggests that different kinds of beers could have been stored in the vessels, such as maize *chicha*, mesquite *aloja* or chañar beer. Higher C_4 fractions could indicate a preference for storage of *chicha* (samples A1 and A2), while lower C_4 fractions could indicate a preference for storage of *aloja* or chañar beer (sample A3). The use of these vessels to store different kinds of beers is novel, notwithstanding the small sample size studied here. Future analyses from similar Inca contexts in Norwestern Argentina could broaden our knowledge and challenge traditional views on exclusive *chicha* storage in *aríbalos* and aribaloids (Bray, 2003; D'Altroy and Hastorf, 1984; D'Altroy, 2001; Leibowicz, 2013; Rowe, 1944). The choice of endogenous beers as part of the feasting practices could indicate that local societies had an active participation in the production of highly valued drinks (Figueroa and Dantas, 2006; Greene, 2003; Martínez, 1998; Rosso, 2015; Saignes, 1993).

It was proposed that *Santa María* vessels were used for storage. The analysis of one *Santa María* vessel (A4) showed that the lipid residues were dominated by animal fats. This was based on the FA profile, the low palmitic/stearic ratio, and the presence of cholesterol. The isotopic analysis indicated an estimated C_4 fraction of 36.2%, possibly indicating some input of C_4. The type of food or drink that was stored in this vessel cannot be established. However results confirmed that it was used for a culinary purpose.

Cone based globular pots were thought to store fermented beverages. The residue analysis of one sample (A5) pointed towards a mixture of animal fats and vegetable oils. Animal fat was identified by the FA profiles, the low palmitic/stearic ratio, and the presence of cholesterol. Also there is some evidence of plant oil, such as the presence of eicosanoic acid ($C_{20:0}$). The high amount of oleic acid (C18:1) and azelaic (nonanodioic) acid which is an oxidation product of oleic acid could be both from plant or animal lipids. The isotopic analysis indicated an estimated C_4 fraction of 41.8%, suggesting an important input of C_4 plant oils. This supports the hypothesis that cone based globular vessels were used for the last stage of fermentation and decanting of maize beer (*chicha*). The data also points towards the use of animal fat to seal the inner surfaces before its use as a storage vessel.

Pucos were proposed as bowls for serving foods or drinks. The results from this study showed that both samples A6 and A7 had complex mixtures of plant and animal lipids. Animal lipids were identified by the FA profile, the palmitic/stearic ratio and the presence of cholesterol. In sample A7 camelid fat was identified by the odd and branched FA, although in sample A6 the source of animal fat could not be identified. Oxidation products of unsaturated fatty acids were found in both samples. The isotopic analysis indicated C_4 fractions ranging from 24.4% to 37.1%. This variation could indicate that different foods or drinks were served in each container. Further identification of the foodstuffs was not possible, but results confirmed that *pucos* were used to serve and consume food and/or drink.

5. Conclusion

In this paper, we studied organic residues from seven ceramic vessels from enclosures used for feasting at the archaeological site of Fuerte Quemado-Intihuatana (Catamarca, Argentina) during the Inca period. Chemical and isotopic analyses showed that all the containers were used to store and serve foods and different kinds of beers in these enclosures. The results obtained from this study are the first molecular evidence of feasting in Fuerte Quemado-Intihuatana. Although our results are preliminary, they contribute to the knowledge on the commensal practices during the Inca conquest.

6. Notes

1. The site is set in an Andean environment, where there is no access to marine products and there are no lakes in the area to exploit aquatic sources. Rivers are semi-permanent streams that dry up or filter down to underground water systems during the dry season and flood during the rainy season. There is little or no fish wildlife in these rivers. The zooarchaeological record is dominated by camelid remains, with small amounts of iguana and bird bones, and complete absence of fish bones (Kriscautzky, 1986). Only five *Spondylus* shells were recovered in Sector V, but they have no marks indicating they were processed to obtain the mollusk for food. The clean shells were likely transported from the Pacific coast as religious objects or jewels of high symbolic value (Murra, 1975). Orgaz (Orgaz, 2014) has suggested that these highly valued shells were gifts for the local authorities

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