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Maize consumption in pre-Hispanic south-central Andes: chemical and microscopic evidence from organic residues in archaeological pottery from western Tinogasta (Catamarca, Argentina)

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ABSTRACT

Pre-Hispanic Andean societies depended economically on the cultivation of maize (*Zea mays*), the main staple food crop in the region after its introduction from highland Mexico. Here we report new data from residue analysis of potsherds recovered in archaeological sites in western Tinogasta, Catamarca province, Argentina, ca. 3rd to 16th centuries AD. Molecular and isotopic ($\delta^{13}C$ values) compositions of fatty acids and microscopically identified maize starch granules from organic residues absorbed in archaeological potsherds were compared with Andean ingredients and food residues obtained from experimental replica pots, where traditional recipes were cooked. Complex mixtures of lipids and starch remains observed in archaeological cooking pots indicated combinations of Andean ingredients such as llama, beans, *algarroba*, and maize, and suggest continuity in the domestic foodways through time. The distribution and $\delta^{13}C$ values of lipids preserved in vessels used for alcoholic beverage preparation, storage and transport in Inka sites suggested the possible consumption of two drinks with distinct patterns: traditional Andean maize beer (*chicha*) and a local fermented drink made from *algarroba* flour (*aloja*). This is potential evidence for consumption practices in festive contexts sponsored by the Inka state.

1. Introduction

1.1. Maize consumption in the Andes

Maize (*Zea mays*) has been a staple food for American societies since pre-Hispanic times (Johannessen and Hastorf, 1994; Staller et al., 2006); its domestication originated in highland Mexico ca. 8700 cal. BP and later spread to North and South America (Piperno et al., 2009). The earliest evidences of maize cultivation and consumption in South America are from the Pacific coastal regions of Peru and Ecuador ca. 3000 BC (Haas et al., 2013; Zarrillo et al., 2008). The expansion towards the south-central Andes occurred later (ca. 2000 BC), and new maize landraces characteristic to each region appeared (Oliszewski and Olivera, 2009). Archaeological evidences of maize based on microscopic remains of starch granules and phytoliths of *Z. mays* in the Southern Argentinean *Puna* (Catamarca province) date from ca. 2000 BC (Rodríguez and Aschero, 2007). Maize cobs and kernels found in several sites from 500 BC and 1st century AD in NW Argentina record the transition of hunter–gatherers to horticulture (Babot, 2005). Later periods showed an increasing dependence on maize as a staple food, as the population developed into complex organizations

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between the 1st and 14th centuries AD (Hastorf, 1990). During the Inka expansion between the 14th and 16th centuries AD the south-central Andes became the key region for state administered production and consumption of maize. Maize was consumed both in domestic contexts where food was shared by family members and was the setting for most of the basic socialization (Appadurai, 1981; Gumerman, 1997), and in centrally organized contexts where staple food was monopolized by the Inka administration. Consumption in the form of *chicha* beer was central for the symbolic domination of local societies (Logan et al., 2012), and has been associated to Inka festivities and rituals where the exhibition of maize and maize-based products was essential to the maintaining of power (Dietler, 2006; Goldstein, 2003; Hastorf and Johannessen, 1993; Moore, 1989).

In western Tinogasta (Catamarca province, Argentina) the earliest direct evidences of maize are archaeobotanic remains of cobs and kernels from Palo Blanco NH1 (AD 208–529) and Punta Colorada (AD 661–1020) (Fig. 1, sites 5 and 8). Later evidence comes from Batungasta (AD 1445–1558) and Lorohuasi (ca. AD 1550) (Fig. 1, sites 2 and 11). Three groups of local maize varieties were identified by morphological markers and ancient DNA: Andean complex, South American popcorns, and new races derived from varieties introduced from other regions after the Spanish conquest (Lia et al., 2007). Also, remains of *Z. mays* were found in funerary contexts at Las Champas (ca. AD 1400) and Bebé de la Troya (ca. AD 1330–1428) (Ratto et al., 2014). The importance of local cultivation was inferred from the agricultural installations at different altitude levels in the Fiambalá valley, such as those found for example at El Puesto 1 (Figs. 1 and 2E) (Orgaz and Ratto, 2013). These agricultural installations were used for food production by the agro-pastoralist societies during the first millennium AD, the Inka period ca. 14th to 16th centuries AD, and some are still in use today (Fig. 2F). During the 14th to 16th centuries AD, agricultural installations were reused and in some cases expanded in a context of interaction between local sociopolitical entities and foreign populations that were moved and established in the area by the Inka state (Orgaz and Ratto, 2013; Ratto and Boxaidós, 2012). Preliminary isotopic studies on bone collagen of bioarchaeological remains of individuals from the Fiambalá valley suggest differences in diet through time (Aranda et al., 2011). One case of a lactating individual from the first millenium 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. These results are preliminary, as more studies on the bone mineral fraction are currently in process. In summary, most of the archaeological evidence indicates an important reliance on maize in the Tinogastan diet, but the way in which this staple food was prepared and consumed in the study area is still largely unknown.

1.2. Detecting maize in food organic residues in archaeological ceramics

Organic residues are absorbed in ceramic vessel matrixes during food preparation or storage, thus allowing an exceptional preservation of organic compounds such as lipids and starch (Copley et al., 2005; Eerkens, 2005). Maize and maize-based food residues can be

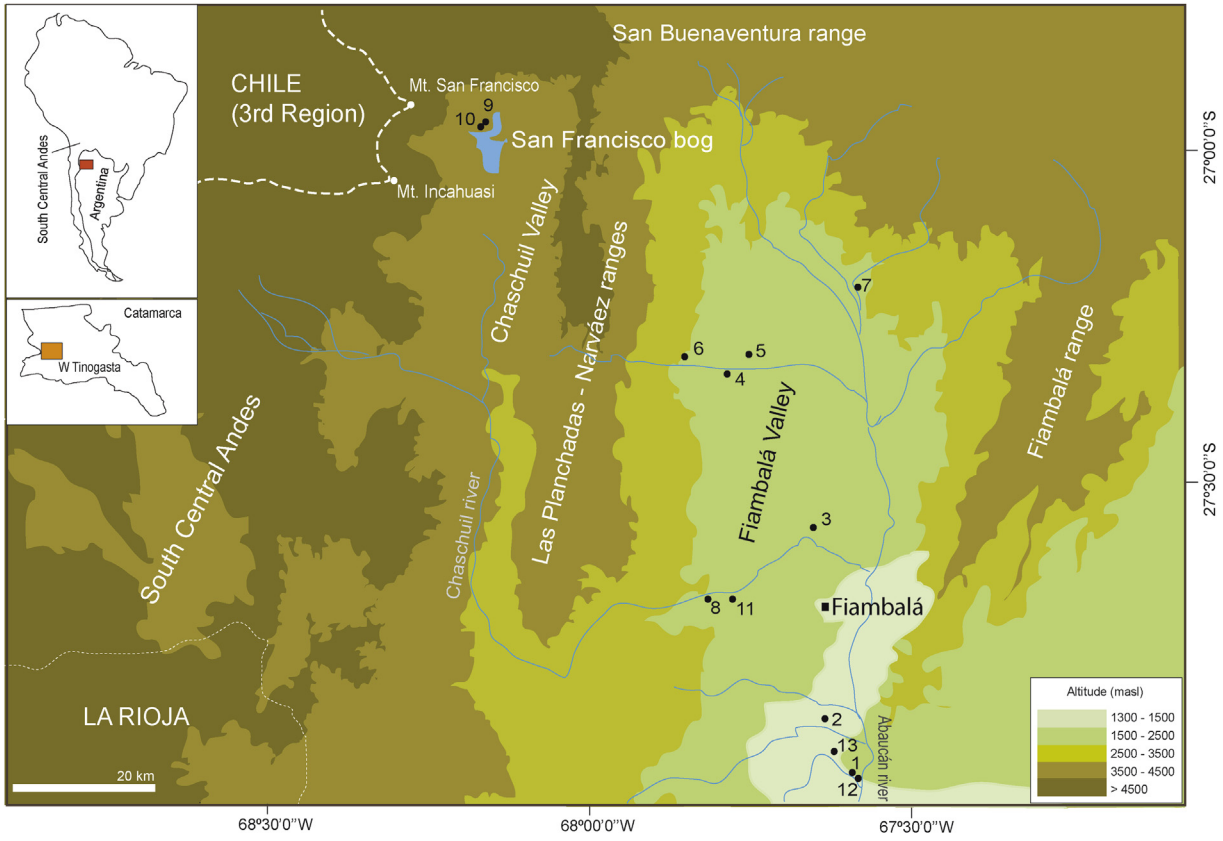

Fig. 1. Map of West Tinogasta Region, located in Catamarca Province, Argentina showing the location of the archaeological and experimental sites: 1, La Troya LT-V50; 2, Batungasta; 3, Mishma 7; 4, Quintar 1; 5, Palo Blanco NH1, NH3, NH4 and NH6; 6, Ojo del Agua; 7, Cardoso; 8, Punta Colorada; 9, San Francisco; 10, El Zorro; 11, Lorohuasi; 12, Experimental site; 13, El Puesto 1.

Fig. 2. Archaeological landscapes of West Tinogasta: A, view of San Francisco site in the Chaschuil region; B, detail of archaeological mud construction in Palo Blanco NH3 site in the Fiambalá region; C, detail of an archaeological floor at Palo Blanco NH6 site in the Fiambalá region; D, Replica cooking pots in experimental site in the Fiambalá region; E, Archaeological cultivation field at El Puesto 1 in the area of LTV-50 in the Fiambalá region; F, Maize cultivation in reclaimed archaeological terraces in Las Termas located outside the modern town of Fiambalá.

detected in archaeological ceramics by a combination of microscopic analysis of preserved starch granules, and molecular and stable carbon isotope analysis of preserved lipids. Starch grains can be recovered from the inner walls of cooking potsherds (Crowther, 2005; Saul et al., 2012). Maize starch remains have been microscopically identified under reflected normal and polarized light in different archaeological samples, including stone artifacts, sediments, and coprolites from cultural layers in Central and South America (Babot and Apella, 2003; Pearsall et al., 2004; Perry, 2004). Maize starch grains have also been recovered from cooking residues adhered to potsherds from North and South American archaeological sites (Boyd et al., 2008; Zarrillo et al., 2008).

Chemical characterization of lipids can be achieved by gas chromatography coupled to mass-spectrometry (Malainey et al., 1999; Reber and Evershed, 2004a). The fatty acids (FAs) profile and the relatively high stable carbon isotope ratio ($^{13}C/^{12}C$ expressed in $\delta^{13}C$ as ‰ vs. Vienna Pee Dee Belemnite limestone standard; VPDB) characteristic of C_4 plants can provide supporting evidence for the presence of residual maize oil in archaeological artifacts (Reber and Evershed, 2004b; Reber et al., 2004; Seinfeld et al., 2009). C_3 and C_4 plants have different photosynthetic pathways leading to distinct carbon isotope fractionations (Deines, 1980; O'Leary, 1993). C_3 plants use the Calvin–Benson cycle for CO_2 fixation and include most South American fruits, vegetables and cool season grasses, with $\delta^{13}C$ values clustering around −27‰ (Marshall et al., 2007). 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 $\delta^{13}C$ values cluster around −14‰ (Marshall et al., 2007). Although C_4 plants in NW Argentina also include pastures such as *Muhlenbergia fastigiata*, *M. atacamensis*, *Sporobolus rigens*, *Pennisetum chilense*, and *Eragrostris nigricans* which may have been used for herding, *Z. mays* was the main staple food consumed by humans derived from a C_4 plant that can be distinguished from other foodstuffs by its $^{13}C/^{12}C$ ratio (Reber and Evershed, 2004b; Tykot et al., 2009).

2. The western Tinogasta study area

Western Tinogasta is part of the south-central Andes located in the southwestern tip of Catamarca province in NW Argentina (Fig. 1). It is comprised by the Fiambalá region and the Chaschuil region, separated by Las Planchadas and Narváez ranges, and has diverse and contrasting eco-zones, including the mesothermal valley (1400–2400 m above sea level – masl), the piedmont (2400–3500 masl), the transitional *puna* (3500–4000 masl), and the Andes range (4000–6700 masl). Western Tinogasta is limited by the southern *puna* highlands in the north, the Abaucán river in the south, the Fiambalá mountain ranges in the east, Chile's III

Region in the northwest, and La Rioja province, Argentina, in the southwest.

The cultural landscape of pre-Hispanic western Tinogasta was characterized by discontinuous settlements of human populations due to environmental processes that made the area uninhabitable during certain periods (Ratto et al., 2013). Throughout the first millennium AD, western Tinogasta was populated by societies that developed herding and agricultural economies, while still maintaining hunting and gathering practices. Sparsely distributed villages were built with mud *tapia* (Fig. 2B) or stone *pirca* (Bonomo et al., 2010; Ratto et al., 2012). They were located both in the Fiambalá and Chaschuil regions, in different altitudinal levels and eco-zones, taking advantage of different local resources (Ratto, 2013).

Recent research shows that between the 10th and 13th centuries AD a synergy of environmental processes made lower eco-zones uninhabitable and 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 valley bottoms of the Fiambalá region were probably repopulated during the first centuries of second millennium AD when conditions slowly improved around the 13th century AD (Ratto, 2013). Then, when environmental conditions were optimal, the Inka expanded into the Fiambalá and Chaschuil regions ca. 14th to 16th centuries AD. The Inka domination process promoted the mobilization of people from other areas with new cultural characteristics as part of the territorial domination strategy (Ratto and Boxaidós, 2012). Towards the 17th century AD, the Spanish colonial installation created new politically unstable conditions and caused community relocations as the area underwent a new depopulation process (Ratto and Boxaidós, 2012).

In this study we selected samples from archaeological sites settled during the first or the second millennia AD, distributed along the Fiambalá and Chaschuil regions (Table 1; Fig. 2). In the following paragraphs we briefly introduce the sites from which samples were selected.

The sites dating to the first millennium AD (Table 1) include: (i) La Troya LTV-50 situated at 1380 masl and dated in AD 641–784; (ii) Palo Blanco archaeological village (Fig. 2B–C) composed of various habitation units located at 1900 masl, dated in AD 208–529 (Palo Blanco NH1), AD 427–599 (Palo Blanco NH4), AD 458–639 (Palo Blanco NH3), and AD 693–985 (Palo Blanco NH6); (iii) Punta Colorada placed at 2290 masl and settled during AD 661–1020; (iv) Cardoso set at 1970 masl and with architectural pattern and ceramic technology indicating it was settled between the last centuries of the first millennium AD and the first centuries of the second millennium AD (radiocarbon dates in process); (v) Ojo del Agua situated at 2400 masl and dated in AD 994–1047; and (vi) El Zorro located at 4050 masl and settled from AD 322–1019. All dates are calibrated from radiocarbon dates with 1 sigma standard deviation, and were taken from Ratto (2013).

The sites from the second millennium AD (Table 1) include: (i) Quintar-1 (1750 masl) dated in AD 1123–1273, a period of environmental instability when most of the other sites in the mesothermal valley were abandoned; (ii) Mishma 7 (1760 masl) dated in AD 1405–1573 with local and Inka components; (iii) Batungasta (1480 masl) dated in AD 1445–1600, the central Inka administrative site of the region; and (iv) San Francisco (4000 masl) dated in 1400–1500 AD (Fig. 2A), a peregrination place used for feasting and religious activities previous to the ascension to Incahuasi Mt., where an Inka sacrificial site was found (Orgaz et al., 2007; Ratto et al., 2012).

In this paper we aimed to explore the potential changes and continuities in the maize preparation and consumption practices before and after the Inka state installation in order to provide insight into the foodways in western Tinogasta. We believe that domestic preparation of maize may have been part of the closely-knit social fabric of the agro-pastoral societies of the first millennium AD, and therefore domestic practices may have not changed substantially after the Inka occupation of the area during the second millennium. On the other hand, state sponsored public rituals and festivities may have suffered changes by power disputes when the Inka administration took over the area during the fourth and fifth centuries of the second millenium. These issues inspired us to carry out an exploratory study aimed to assess the potential occurrence of maize and maize-based food remains within organic residues preserved in the ceramic matrixes of archaeological pottery from western Tinogasta by molecular, carbon isotope and microscopic studies.

3. Materials and methods

3.1. Samples

Organic residues were obtained from twenty-two potsherd samples (n = 22) (Table 1). Samples were taken from lip, neck, body

Table 1
Archaeological sites and description of potsherds from West Tinogasta selected for residue analysis.

Sample	Sample code	Archaeological site	Region	Period	Vessel section	Functional category
A-01	LTV50-178-09	La Troya LT-V50	Fiambalá	First millenium AD	Lip	Cooking pot
A-02	LTV50-1344-04A	La Troya LT-V50	Fiambalá	First millenium AD	Neck/lip	Cooking pot
A-03	LT-BATH35	La Troya LT-V50	Fiambalá	First millenium AD	Lip-body	Cooking pot
A-04	VBAT-032	Batungasta	Fiambalá	Second millenium AD	Body	Cooking pot
A-05	VBAT-020-05	Batungasta	Fiambalá	Second millenium AD	Body	Cooking pot
A-06	VBAT-035-02	Batungasta	Fiambalá	Second millenium AD	Body/base	Cooking pot
A-07	M7-H1	Mishma 7	Fiambalá	Second millenium AD	Base	Cooking pot
A-08	M7-HA	Mishma 7	Fiambalá	Second millenium AD	Neck-body	Beverage-holding vessel (*Belén*)
A-09	Q1-E2-4849-Olla1	Quintar 1	Fiambalá	Second millenium AD	Body	Cooking pot
A-10	PB-NH1-05	Palo Blanco NH1	Fiambalá	First millenium AD	Base	Cooking pot
A-11	PB-NH3-228-21	Palo Blanco NH3	Fiambalá	First millenium AD	Body	Cooking pot
A-12	PB-NH6-11-30-2	Palo Blanco NH6	Fiambalá	First millenium AD	Body	Cooking pot
A-13	PB-NH4-14	Palo Blanco NH4	Fiambalá	First millenium AD	Base	Cooking pot
A-14	OA1-49-02	Ojo del Agua 1	Fiambalá	First millenium AD	Body	Cooking pot
A-15	Car-ES-647-01	Cardoso	Fiambalá	First millenium AD	Neck	Cooking pot
A-16	PC-2b-B	Punta Colorada	Fiambalá	First millenium AD	Body-base	Cooking pot
A-17	SF-R15-17	San Francisco	Chaschuil	Second millenium AD	Body	Beverage-holding vessel (*aribaloid*)
A-18	SF-R12-3	San Francisco	Chaschuil	Second millenium AD	Body	Beverage-holding vessel (*aribalo*)
A-19	EZ-067-04	El Zorro	Chaschuil	First millenium AD	Body	Cooking pot
A-20	VBAT-004-02	Batungasta	Chaschuil	Second millenium AD	Body	Cooking pot
A-21	SF-R12-51-ex1	San Francisco	Chaschuil	Second millenium AD	Body	Beverage-holding vessel (*aribalo*)
A-22	SF-R12-ex19	San Francisco	Chaschuil	Second millenium AD	Body	Beverage-holding vessel (*aribaloid*)

or base sections of the vessel silhouette according to availability for sampling and presence of macroscopic signs of absorbed residues, such as stains or oiliness. Previously, we tested lip, body and base sections of our three replica pots (R-11, R-12, R.-13, Table 2) and confirmed that although lipid concentration varied, FAs profiles were identical in all vessel sections. Therefore, we could safely compare archaeological absorbed residues from different vessel sections. In the field, potsherds were recovered using dustless gloves and immediately stored in aluminum foil paper to avoid contamination. In the lab, potsherds were carefully handled with dustless gloves and clean instruments. Potsherds had very little or no sediments attached to them given the extreme dryness of the sediment matrixes in the field. When present, it was carefully removed with clean brushes. All potsherds remained unwashed. Organic residues were absorbed into the matrixes of all samples, and no adhered crusts were visible. Samples of ca. 4×4 cm^2 were separated for residue analysis. Subsequent treatment is described in Section 3.2.

The potsherd samples were selected as representative of the vessel morphologies characteristic for specific functions. Following Skibo (1992) the vessel function was defined by technological and morphological characteristics, while vessel use by alteration evidences and presence of organic residues. The first functional category was "cooking pots" (Fig. 3, A-05, A-07 and A-11), characterized by a typical morphology showing a flat or round base, globular wide body, and slightly more restricted neck (Feely, 2013; Orgaz et al., 2007). The inner surfaces were untreated before firing, some were regular and smooth while others were irregular (Feely, 2010). The texture of the ceramic matrixes, in terms of particle size and porosity, ranged from medium to coarse (Feely, 2010). In addition, use-alteration macroscopic features indicating their use as cooking pots were visible, such as soot in the outer surfaces, and/or dark and oily stains in the inner surfaces. The second functional category was "beverage-holding vessels", including morphologies of *aribalos*, *aribaloids* and *Belén* vessels. *Aribalos* and *aribaloids* (Fig. 3, A-21) were characterized by their conic or semi-conic bases and constricted bottle-like necks (Orgaz et al., 2007) which indicate their function as liquid carriers. The inner surfaces were regular and smooth and the outer surfaces were treated with slip, polished and painted (Feely, 2010). The porosity of the ceramic matrixes in aribalos and aribaloids was medium to fine. Various authors have stated that *aribalos* and *aribaloids* were designed to store, transport and pour alcoholic beverages for Inka ritual festivities (Bray, 2004, 2003; Giovannetti et al., 2013; Jennings and Bowser, 2009; Moore, 2013). In western Tinogasta, *aribalos* had classic Inka shapes with provincial decorative style, while *aribaloids* had a slightly modified shape and a provincial Inka decorative style (Calderari and Williams, 1991). *Belén* vessels are characterized by typical morphologies with conic bases, ovoid bodies, and wide necks, as well as distinctive *Belén* style decoration (Orgaz et al., 2007). The only studied *Belén* vessel sample (Fig. 3, A-08) represents a local style coexisting during the Inka occupation (Calderari and Williams, 1991). The inner surface was irregularly smoothed while the outer surface was also smoothed, bathed in slip and painted, and the porosity of the ceramic matrix was medium. An ongoing debate exists on *Belén* vessel function, as it has been associated with beverage production, funerary purposes, grain storage, and cooking (Amuedo, 2012; Marchegiani et al., 2009). Therefore, the Belén vessel sample studied in this paper was cautiously and temporarily assigned to the "beverage-holding" category as we believe in this case residue analysis could provide some information on its possible use as a beverage production vessel.

Lipid concentrations (μg/g) were tested against vessel antiquity, function, vessel section, ceramic matrix texture and surface treatment. Non-parametric Mann Whitney and Kruskal–Wallis tests showed that age, function, vessel section, matrix texture, and surface treatment had no statistically significant ($P < 0.05$) effect on the concentration of absorbed and preserved lipids (see Appendix A, Supplementary data).

Reference samples including eleven ingredients from traditional Andean cookery ($n = 11$) and potsherds from three replica cooking pots ($n = 3$) were also studied (Table 2). Four maize landraces native from NW Argentina were chosen as C_4 plant references (*pisingallo*, *chullpi*, *dentado blanco*, and *capia*) and their starch granules were used as references for microscopic analysis. *Algarroba* pods and *alubia* beans from Catamarca province, NW Argentina, were selected as C_3 plant references and their starch granules were used as references for microscopic analysis. Bovine fat from the central Pampas and NW Argentina were selected as references for animals fed on a diet composed of C_3 pastures and some maize supplement. Samples of fat and jerky from llamas fed exclusively on C_3 pastures were obtained from the *puna* area of Jujuy province, NW Argentina. Three replica ceramic pots were used for cooking experiments conducted with traditional corn-based Andean recipes (Table 3, Fig. 2 D, Fig. 3 R-13, R-12). Replica pots were crafted by an artisan ceramist using traditional firing techniques. Shape and volumetric characteristics of the pots, proportions of raw materials, porosity of the matrix and inner surfaces were reproduced from archaeological cooking pottery recovered at the study area (Feely, 2013). In replica pot A *mazamorra* (a sweet porridge generally consumed for breakfast) was made with dry kernels of sweet maize (*Chullpi* corn) boiled in water. In replica Pot B *locro* (a thick stew consumed for lunch) was made with dry kernels of starchy maize (*Dentado Blanco* corn), dry beans, and fresh

Table 2
Reference samples selected for lipid analysis.

Sample	Material	Description	Locality	Altitude (masl)
R-01	Kernels	Pisingallo (*Zea mays* L., ARZM09063)	Tilcara, Jujuy	2460
R-02	Kernels	Chullpi (*Zea mays* L., ARZM09421)	Humahuaca, Jujuy	2500
R-03	Kernels	Dentado blanco (*Zea mays* L., ARZM12001)	Tiraxi, Jujuy	1750
R-04	Kernels	Capia blanco (*Zea mays* L., ARZM09370)	Maimará, Jujuy	2400
R-05	Pods	Algarrobo (*Prosopis alba* Girseb.)	El Alto, Catamarca	940
R-06	Seeds	Alubia bean (*Phaseolus vulgaris* L.)	Fiambalá Catamarca	1500
R-07	Fresh fat	Bovine fat I (*Bos taurus* L.)	Cruz Alta, Tucumán	1300
R-08	Fresh fat	Bovine fat II (*Bos taurus* L.)	San Antonio de Areco, Buenos Aires	100
R-09	Fresh fat	Llama fat (*Lama glama* L.)	La Candelaria, Jujuy	3950
R-10	Jerkey fat	Llama fat (*Lama glama* L.)	La Candelaria, Jujuy	3950
R-11	Potsherd	Replica pot A (*Mazamorra*)	Experimental site, Catamarca	1350
R-12	Potsherd	Replica pot B (*Locro*)	Experimental site, Catamarca	1350
R-13	Potsherd	Replica pot C (*Pochoclo*)	Experimental site, Catamarca	1350
R-14	Kernels	Pisingallo from Punta Colorada (*Zea mays* L.)	Punta Colorada site, Catamarca	2290

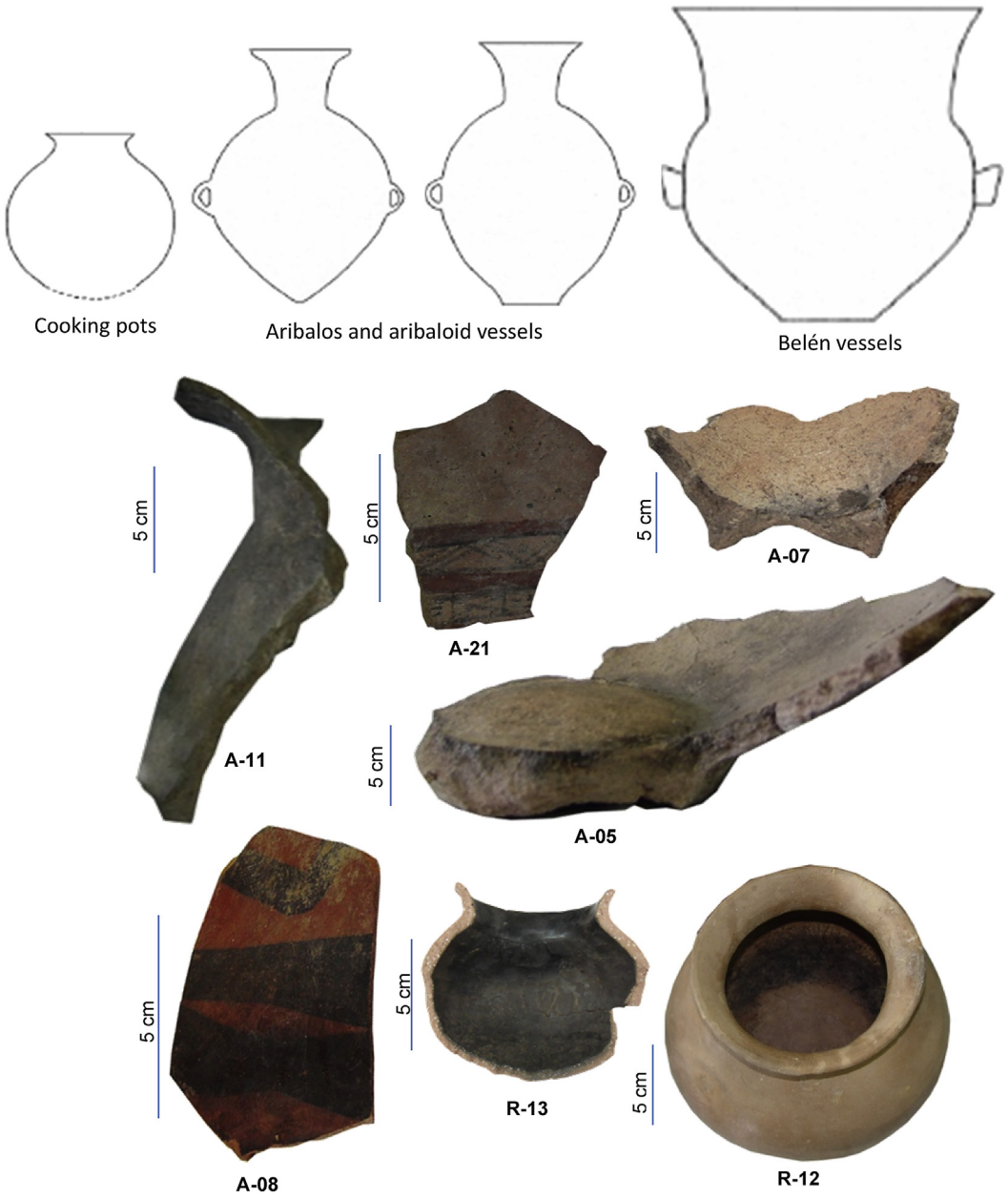

Fig. 3. Archaeological and replica potsherds from West Tinogasta. Photos taken before lipid and starch extraction at the Department of Organic Chemistry, University of Buenos Aires. Clockwise from top left: Silhouettes of typical cooking pots, *aribalos*, *aribaloids* and *Belén* vessels (from Orgaz et al., 2007); A-11, cooking pot from Palo Blanco NH3; A-21, *aribalo* vessel from San Francisco; A-07, cooking pot from Mishma 7; A-05, cooking pot from Batungasta; R-12, replica pot B; R-13, replica pot C; A-08, *Belén* vessel from Mishma 7.

beef fat. In replica Pot C *pochoclo* (popcorn) was made with kernels of another native race of maize (*Pisingallo*) from NW Argentina and a little fresh beef fat. The Maize Germplasm Bank of the Argentine National Institute for Agronomical Technology (INTA-Pergamino) provided the native landraces used in the recipes. Dry beans and beef fat were obtained from local farmers in the market of the modern town of Fiambalá, Catamarca, NW Argentina. In pre-Hispanic times, fat from llama, guanaco or vicuña would have probably been used but these products were not available at the time of the experimental cooking. The replica pots were broken and sherds were selected for microscopic starch analysis to obtain references of cooking alterations in starch granules (Crowther, 2012; Haslam, 2004; Raviele, 2011). Lipid analysis of the replica potsherds was performed to obtain references for the absorbed residues from cooked traditional Andean foods.

Table 3
Experimental cooking of Andean recipe formulations in replica pots.

	Recipe	Ingredients	Cooking time
Replica Pot A	*Mazamorra* (porridge)	Chullpi kernels (175 g); distilled water (1 L)	4 h
Replica Pot B	*Locro* (stew)	Dentado blanco kernels (425 g); alubia beans (250 g); bovine fat (25 g); distilled water (1 L)	4 h
Replica Pot C	*Pochoclo* (popcorn)	Pisingallo kernels (320 g), bovine fat (10 g)	15 min

3.2. Lipid extraction

Lipid extraction was carried out on all archaeological and reference samples (Tables 1 and 2). Powder of the dry samples (kernels, beans, pods) was obtained by grinding with a coffee mill.

The fresh animal fats were frozen and ground using a porcelain mortar and pestle. Replica potsherds and archaeological potsherds were first rinsed on both surfaces with dichloromethane. They were then broken into small fragments with a hammer and then ground in a porcelain mortar and pestle. Lipids were extracted with dichloromethane:methanol (1:1, vol/vol). All solvents were of chromatographic quality (Merck, Buenos Aires, Argentina and Merck Darmstadt, Germany) and pre-distilled before use. Each sample was placed in an ultrasound bath for 15 min and filtered; a few drops of distilled water were added, the organic phase was separated after centrifugation for 3 min (twice), evaporated at room temperature in the dark, weighed and then transferred with *n*-hexane to a 2 mL glass vial and stored at −20 °C. An aliquot of the extract was saponified with 1 ml of 1N potassium hydroxide in an ethanolic aqueous solution (9:1, vol/vol), at 60 °C for 2 h. After cooling at room temperature, the neutral fraction was extracted with 1.5 ml *n*-hexane and the aqueous fraction acidified with 1N HCl solution and extracted with 1.5 ml *n*-hexane. The organic phase was evaporated at room temperature in the dark and 0.5 ml of 1M boron trifluoride in methanol was added and heated at 60 °C for 8 min. The fatty acid methyl esters (FAME) were extracted with 2 × 0.75 ml of *n*-hexane and stored in 2 mL glass vials at −18 °C for gas chromatographic analysis. Trimethylsylil derivatives (TMS) of the neutral fraction were prepared by addition of 20 μl 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 as the archaeological samples.

3.3. Chemical characterization of acid and neutral lipids

Chemical characterization of FAMEs was performed with an Agilent (Palo Alto, USA) gas chromatograph 6890 coupled to an Agilent 5973 quadrupole mass selective detector (GC–MS). The system was equipped with an Agilent free FAs phase (HP-FFAP) fused silica capillary column (l50 m length, 0.20 mm i.d.) coated with nitroterephthalic acid modified polyethylene glycol stationary phase (film thickness 0.33 mm). He was used as carrier gas (1 ml/min flow rate) and manual injection was splitless at a temperature of 200 °C. After an initial period of 2 min at 100 °C, the column was heated to 240 °C at 5 °C/min followed by an isothermal period of 30 min. The MS was operated in the electron impact mode at 70 eV, source temperature of 250 °C, emission current of 1 mA and multiple-ion detection with a mass range from 50 to 600 amu. Compound identifications were carried out by comparing GC retention times of FAME standards ($C_{14:0}$, $C_{16:0}$, $C_{17:0}$, $C_{18:0}$, $C_{18:1}$, $C_{18:2}$, Sigma, St. Louis, MO, USA) and mass spectrometric fragmentation patterns. The abundances of FAME in the organic residues relative to total FAME were calculated from total ion chromatogram peak areas.

Chemical characterization of the TMS derivatives of the neutral lipids was performed by gas chromatography–mass spectrometry on a Shimadzu GCMS-QP5050A (Shimadzu Corporation, Kyoto, Japan). The system was equipped with an Agilent Ultra 2 capillary column (50 m length, 0.20 mm i.d.) coated with (5% phenyl)-methylpolysiloxane (film thickness 0.11 mm). He was used as carrier gas (0.9 ml/min flow rate) and manual injection was splitless at a temperature of 250 °C. After an initial temperature of 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.

3.4. Compound specific C isotope analysis of fatty acids (CSIA)

Compound specific stable carbon isotope analyses ($\delta^{13}C$ values) of the fatty acids (FAs) were performed on an Agilent 6890 GC coupled to a Thermo Fischer Delta V isotope ratio mass spectrometer by a combustion (C) interface III (GC-C-IRMS) under a constant He flow of 1.2 ml/min. The gas chromatograph was operated with an HP-FFAP column and same temperature program as used for GC–MS analyses. The combustion interface consisted of two ceramic furnaces, an oxidation reactor with CuO/NiO/Pt wires at 940 °C and a reduction reactor with Cu wires at 600 °C. Water was removed from the effluent gas by passing a Nafion tube (Perma Pure, Toms River, NJ, USA) with an annular back-flow of Helium. The background subtraction and calculations of $\delta^{13}C$ values were performed using ISODAT 2.5 software (Thermo Fischer Scientific, Bremen, Germany). The repeatability and intermediate precision of the GC-C-IRMS procedure, and the performance of the GC and combustion interface were evaluated every five analyses by co-injection of isotope standards (eicosanoic acid methyl ester, $\delta^{13}C_{20:0} = -24.3 \pm 0.2$‰ and naphtalene $\delta^{13}C_{20:0} = -26.1 \pm 0.3$‰) (Spangenberg et al., 2006). At least three replicate analyses of the reference and archaeological samples were carried out so averages and standard deviations could be calculated for each result. The accuracy of the GC-C-IRMS analyses was checked every ten analyses. The mean overall analytical error for the main FAMEs was 0.8‰ and ranged between 0.1 and 2.7‰. The stable carbon isotopic data are expressed in delta (δ) notation as the per mil (‰) deviations of the isotope ratio of the sample relative to that of a standard ($\delta^{13}C = [(R_{sample} - R_{standard})/R_{standard}] \times 1000$), where $R = {}^{13}C/{}^{12}C$. The stable isotope composition of carbon ($\delta^{13}C$ values) is reported relative to the Vienna Pee Dee Belemnite limestone (VPDB) standard. The isotopic shift due to the carbon introduced by methylation was corrected using a mass balance equation (Spangenberg et al., 1998). Post-industrial reference samples were corrected for anthropogenic CO_2 emissions by adding +1.6‰ to the isotopic values obtained in the GC-C-IRMS analysis (Spangenberg et al., 2006).

3.5. Statistical analyses

Principal component analysis (PCA), Hierarchical cluster analysis (HCA), and Discriminant analysis (DA) were performed by statistical analysis software SPSS 18.0 (IBM) and PAST (Hammer et al., 2001) to gain insight into the nature of the multivariate dataset obtained from lipid analyses of the archaeological samples.

3.6. Starch separation and microscopic analysis

Starch granules were extracted in a controlled clean hermetic environment to avoid contamination. Dustless gloves and sterile materials were used for each sample and the workspace was cleaned carefully between each sample extraction. An area of 4 × 4 cm of the inner surface of each archaeological sample was gently scraped with a scalpel, and the detached residue was sieved with an N°35 US mesh (0.5 mm opening) and the finer fraction was recovered. The same procedure was used for the replica potsherd samples on a different day and after the archaeological samples preparation to avoid cross-contamination. A tip of each sample was mounted on a glass slide with a solution of distilled water and glycerol (50%). Microscopic study of starch grains was carried out on a Lancet XSZ-148 microscope at 40× alternating normal and polarized light. The light intensity was fixed at seven and the diaphragm opening was set at five. Samples were analyzed by sweeping through the complete slide. Micrographic images were obtained with a Samsung HZ10W camera with 10.2 megapixel resolution, which was equipped with an adaptor to the eyepiece

lens. The digital processing of images was done with open-source software Image-J 1.44 (National Institute of Health, 2013). We recorded a number of variables such as granule morphology, size, *hillum* shape and position, extinction cross morphology under polarized light, striations, fissures, and growth rings. Identification was done at a species level whenever possible by comparison with modern reference starch samples of maize, bean and *algarroba* and published descriptions and photos of starch grains from other American native edible plants (Babot et al., 2007; Cortella and Pochettino, 1995; Giovannetti, 2013; Giovannetti et al., 2012, 2008; Holst et al., 2007; Lantos et al., 2014; Mickleburgh and Pagán-Jiménez, 2012; Piperno, 2009).

4. Results

4.1. Fatty acid and neutral lipids composition

Relatively high amounts of lipids were recovered from the replica cooking potsherds (4200 μg/g mean value) and substantially less in the archaeological Tinogasta potsherds, with a mean value of 200 μg/g for all vessel categories.

The FAs relative abundances, palmitic to stearic acid ratios (P/S = $C_{16:0}/C_{18:0}$), and lipid concentrations of the reference and archaeological samples are detailed in Table 4. The gas chromatograms showed a series of methyl esters of carboxylic acids in the C_{12}–C_{18} range (Figs. 4–5). The main saturated fatty acids were myristic ($C_{14:0}$), pentadecanoic ($C_{15:0}$), palmitic ($C_{16:0}$), margaric ($C_{17:0}$), and stearic ($C_{18:0}$) acids, maximizing at C_{16} and C_{18}. Small to trace amounts of lauric ($C_{12:0}$) acid occurred in most of the archaeological samples. The most abundant unsaturated fatty acids were palmitoleic ($C_{16:1}$), oleic ($C_{18:1}$), linoleic ($C_{18:2}$), and linolenic ($C_{18:3}$) acids. The FAs distributions of the lipids extracted from modern maize landraces (R-01, R-02, R-03, and R-04) were similar to previously published data (Spangenberg and Ogrinc, 2001; Woodbury et al., 1995). The P/S ratios for modern maize samples ranged from 6.9 to 12.4. The FAs profile for archaeological pisingallo maize (R-14) was typical for degraded vegetable oil, with a loss of unsaturated fatty acids and relative increase of palmitic and stearic acid abundances, which is reflected in a P/S ratio of 5.1. The

Table 4
Fatty acid relative abundances (%), palmitic acid to stearic acid ratios (P/S ratios), and lipid concentrations (μg/g) from reference samples and potsherds recovered at West Tinogasta.

Sample	Description	12:0	14:0	15:0	16:0	16:1	17:0	18:0	18:1	18:2	18:3	Other	P/S ratio	Lipid conc. (μg/g)
Reference samples														
R-01	Pisingallo				25.4			2.1	32.4	40.0			12.1	34600
R-02	Chullpi				20.2			2.9	32.0	43.6	1.3		6.9	54700
R-03	D. Blanco				28.9			2.3	32.1	36.7			12.4	44800
R-04	Capia				28.5			2.4	30.5	37.1	<1.0	<1.0 (6c-18:1)	12.0	41800
R-05	Algarrobo				43.2			22.3	23.4	11.2			1.9	9800
R-06	Alubia				19.5			2.9	7.2	31.2	37.0	2.2 (9t-18:1)	6.7	15700
R-07	Bovine I		8.0	<1.0	41.8	6.8	<1.0	10.5	29.9	3.0			4.0	986000
R-08	Bovine II		5.2	<1.0	33.1	6.5	<1.0	12.5	41.3	1.5			2.6	792800
R-09	Llama I	<1.0	7.9	1.62	32.3	5.0	1.2	16.4	30.0	1.1	<1.0	1.3 (14:0 12-methyl), 1.0 (16:0 14-methyl), 1.1 (9t,12t-18:2)	2.0	171400
R-10	Llama II	<1.0	7.8	1.95	31.8	6.1	1.0	14.5	29.3	3.4	<1.0	1.0 (16:0 14-methyl)	2.2	176100
R-11	Pot A				17.1			3.0	29.8	48.0	1.4	<1.0 (9t-18:1)	5.6	200
R-12	Pot B		12.4	1.78	47.6	1.8	1.8	19.7	13.5	1.3			2.4	8800
R-13	Pot C		8.0	1.24	37.9	2.1	1.7	21.0	24.7	<1.0		2.6 (9t-18:1)	1.8	3700
R-14	Pisingallo				60.6			12.0	24.3	3.2			5.1	3600
Archaeological samples														
A-01	La Troya LTV50; cooking pot	3.1	13.3	6.06	34.9	17.7	1.9	14.0	9.5				2.6	110
A-02	La Troya LTV50; cooking pot	2.7	14.0	5.63	49.9		2.0	25.8					1.9	50
A-03	La Troya LTV50; cooking pot	3.4	21.1	4.08	32.8	10.6	1.6	16.3	10.3				2.0	270
A-04	Batungasta; cooking pot	<1.0	5.9	3.43	44.0		2.8	41.4	1.9				1.1	430
A-05	Batungasta; cooking pot	2.9	13.3	3.60	39.8	6.0	1.3	28.0	5.1				1.4	80
A-06	Batungasta; cooking pot		4.9	3.46	56.7		2.6	30.9	1.5				1.8	340
A-07	Mishma 7; cooking pot		2.7		41.9		2.8	41.1	6.6	4.9			1.0	160
A-08	Mishma 7; beverage-holding vessel	5.3	16.8	7.34	44.0	9.5		11.0	6.0				4.0	190
A-09	Quintar 1; cooking pot		4.8	4.03	54.0		2.5	34.6					1.6	180
A-10	Palo Blanco NH1; cooking pot	8.4	23.6	7.29	37.7	4.2		7.9	3.1	1.8		2.9 (13:0)	4.8	190
A-11	Palo Blanco NH3; cooking pot	3.8	12.0	5.12	37.8	12.9	1.6	14.2	9.6	3.0			2.7	600
A-12	Palo Blanco NH6; cooking pot	3.6	7.5	2.46	31.3	1.5	1.4	17.0	29.4	2.4		3.6 (11c-16:1)	1.9	70
A-13	Palo Blanco NH4; cooking pot		9.7	3.72	51.7		2.0	32.8					1.6	90
A-14	Ojo del Agua; cooking pot	2.2	9.5	2.76	36.2	4.7	1.4	23.1	9.0	6.4		4.9 (11c-16:1)	1.6	330
A-15	Cardoso; cooking pot	3.2	13.5	6.95	45.9	10.1		7.9	7.1	1.6		3.8 (14:0 12-methyl)	5.8	200
A-16	Punta Colorada; cooking pot	4.0	14.1	5.86	43.9	8.3	1.8	13.4	7.5	1.3			3.3	130
A-17	San Francisco; beverage-holding vessel	2.1	12.7	6.47	46.7	7.0	2.2	14.0	6.7			2.1 (14:0 12-methyl)	3.3	200
A-18	San Francisco; beverage-holding vessel	4.2	14.8	6.08	39.0	12.4		10.5	10.0			3.0 (14:0 12-methyl)	3.7	170
A-19	El Zorro; cooking pot	<1.0	5.6	1.69	30.5	4.8	1.6	13.6	34.7	3.0		<1.0 (14:0 12-methyl), <1.0 (16:014-methyl), 2.0 (11c-16:1), <1.0 (11c-18:1)	2.2	140
A-20	Batungasta; cooking pot	3.8	10.9	2.94	34.6	6.0	1.1	11.2	27.3	1.4		<1.0 (14:0 9-methyl)	3.1	190
A-21	San Francisco; beverage-holding vessel	1.5	7.4	3.28	26.5	1.3	1.0	30.2	35.3			<1.0 (16:0 14-methyl), 1.7 (14:0 9-methyl), 8.3 (11c-16:1), 1.2 (11c-18:1)	0.9	430
A-22	San Francisco; beverage-holding vessel	4.8	9.8	3.87	29.7	2.4	1.0	9.0	21.2	2.3		5.1 (C10:0), 1.7 (14:0 9-methyl), 9.1 (11c-16:1)	3.3	120

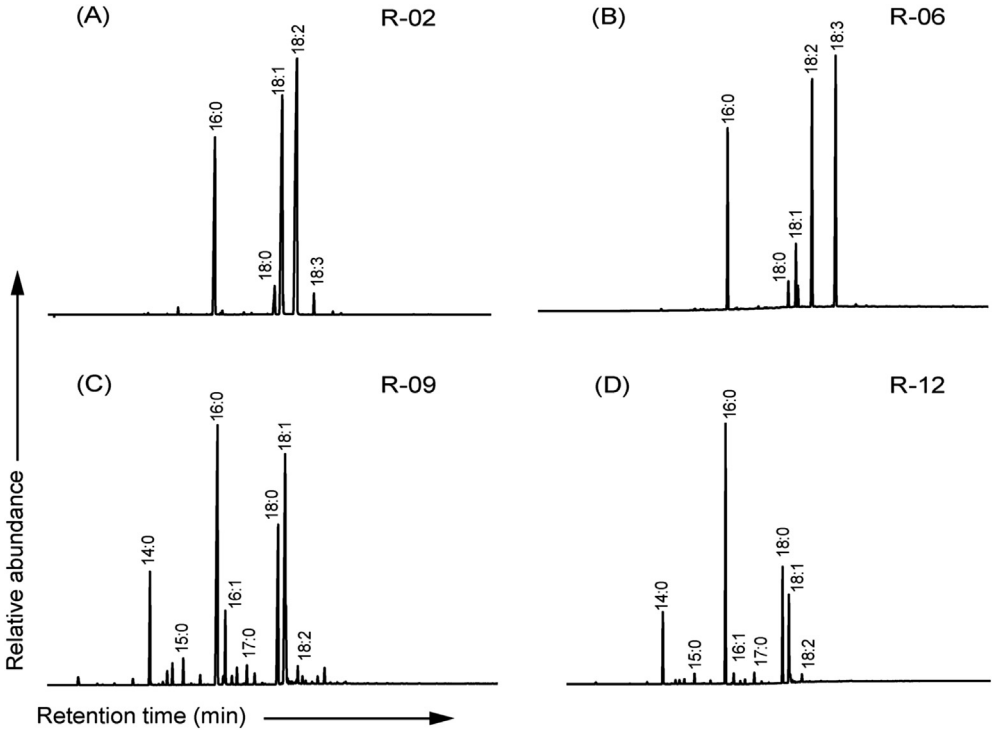

Fig. 4. Total ion chromatograms of fatty acid methyl esters extracted from reference samples: A, R-02 chullpi maize; B, R-06 alubia beans; C, R-09 llama fat; D, R-12 test pot B. The fatty acids are myristic (14:0), pentadecanoic (15:0), palmitic (16:0), palmitoleic (16:1), margaric (17:0), stearic (18:0), oleic (18:1), linoleic (18:2), and linolenic (18:3) acids.

algarroba (R-05) contained $C_{16:0}$, $C_{18:0}$ and $C_{18:1}$, followed by smaller amounts of $C_{18:2}$ (P/S ratio of 1.9), this FAs profile was similar to published data (Lamarque et al., 1994; Mazzuca and Balzaretti, 2003). The sample of native bean (R-06) showed abundant polyunsaturated fatty acids, especially $C_{18:2}$ and $C_{18:3}$, followed by $C_{16:0}$, $C_{18:1}$ and $C_{18:0}$ in lower abundance, and P/S = 6.7, also similar to published FAs profiles (Grela and Günter, 1995). Cow fat samples (R-07, R-08) showed high abundance of $C_{16:0}$ and $C_{18:1}$, presence of

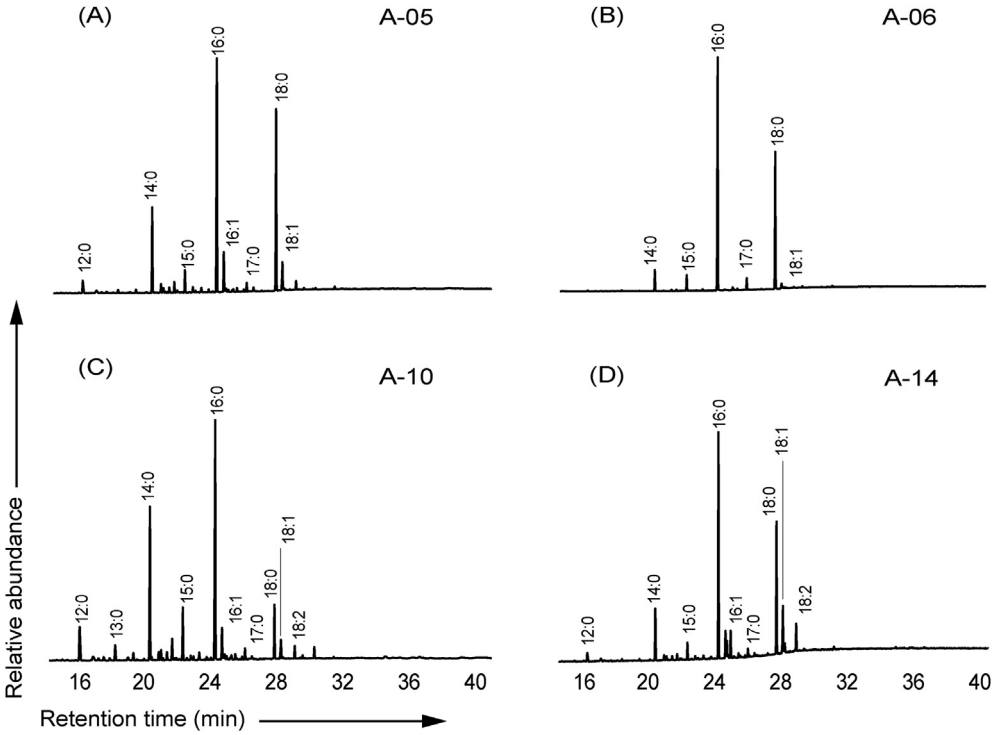

Fig. 5. Total ion chromatograms of fatty acid methyl esters extracted from archaeological samples: A and B, A-05 and A-06 cooking pots from Batungasta; C, A-10 cooking pot from Palo Blanco NH1; D, A-14 cooking pot from Ojo del Agua. The fatty acids are lauric (12:0), tridecanoic (13:0), myristic (14:0), pentadecanoic (15:0), palmitic (16:0), palmitoleic (16:1), margaric (17:0), stearic (18:0), oleic (18:1), and linoleic (18:2) acids.

odd-numbered FAs and a low P/S ratio ranging from 2.6 to 4.0, typical of animal fats (Evershed et al., 2002). Llama fat samples (R-09, R-10) had typical animal fat profiles with high $C_{16:0}$ and $C_{18:1}$ acids abundance and a low P/S ratio ranging from 2.0 to 2.2, and FAs typical of ruminant and pseudo-ruminant animals (e.g., American camelids) (Coates and Ayerza, 2004; Maier et al., 2007; Vázquez et al., 2008) such as lauric ($C_{12:0}$), pentadecanoic and margaric acids, and trace amounts of ramified *anteiso* methyl-tetradecanoic and methyl-hexadecanoic acids. Myristic and palmitoleic acids were present in all animal fats −both cow and llama-while absent in all vegetable oil samples. The FA distribution for replica pot A (R-11) showed a vegetable oil profile with little signs of degradation and a P/S ratio of 5.6 (Fig. 6, Table 4). Replica pots B (R-12) and C (R-13) showed FAs profiles of mixtures of vegetable and animal ingredients and few signs of degradation, with P/S ratios of 2.4 to 1.8, respectively. In these last two cases, the mixtures were dominated by the animal fat component with respect to the vegetable oil component. This indicated that lipid concentrations (μg lipid extracted in 1 g of whole raw ingredient) in the vegetable ingredients used in both recipes were much lower (15,700 μg/g to 54,700 μg/g) than those in the animal fat ingredient (793,000 μg/g to 986,000 μg/g), and explains why the P/S ratio in pots B and C is similar to cow fat rather than to maize or beans. This supports previous findings by Reber et al. (2004), known as "masking effect", where vegetable ingredients can be underestimated due to their lower lipid concentrations and higher abundance in more readily degradable unsaturated FAs. This is important for the interpretation of FAs profiles in archaeological samples.

The fatty acid distributions for the archaeological samples were typical of degraded fats and oils, and had a mean P/S ratio of 2.5 ± 1.3 (Fig. 7, Table 4). $C_{16:1}$ and $C_{18:1}$ acids were also found in most of the archaeological samples (A-01 to A-22). Small amounts of $C_{18:2}$ were only detectable in a few samples. This is to be expected since the polyunsaturated FAs would have decomposed under the oxidative conditions during vessel use and subsequent burial at the archaeological sites. Some archaeological samples had trace amounts of ramified methyl-tetradecanoic and methyl-hexadecanoic acids equivalent to those found in the llama reference samples (R-09 and R-10). The FAs profiles of archaeological samples from sites dated to the first and second millennia AD were mostly dominated by an animal fat signature comparable to the llama reference samples (R-09 and R-10).

GC−MS analysis of the trimethylsilylated neutral lipids of archaeological samples A-01, A-03, A-04, A-06, A-11, A-14, and A-18 showed the presence of 1-tetracosanol (24-OH) in all the samples, together with 1-pentacosanol (25-OH) (A-01, A-03, A-04), 1-hexacosanol (26-OH) (A-01, A-03, A-04, A-06, A-11), 1-octacosanol (28-OH) (A-01, A-03, A-04, A-06, A-14), and

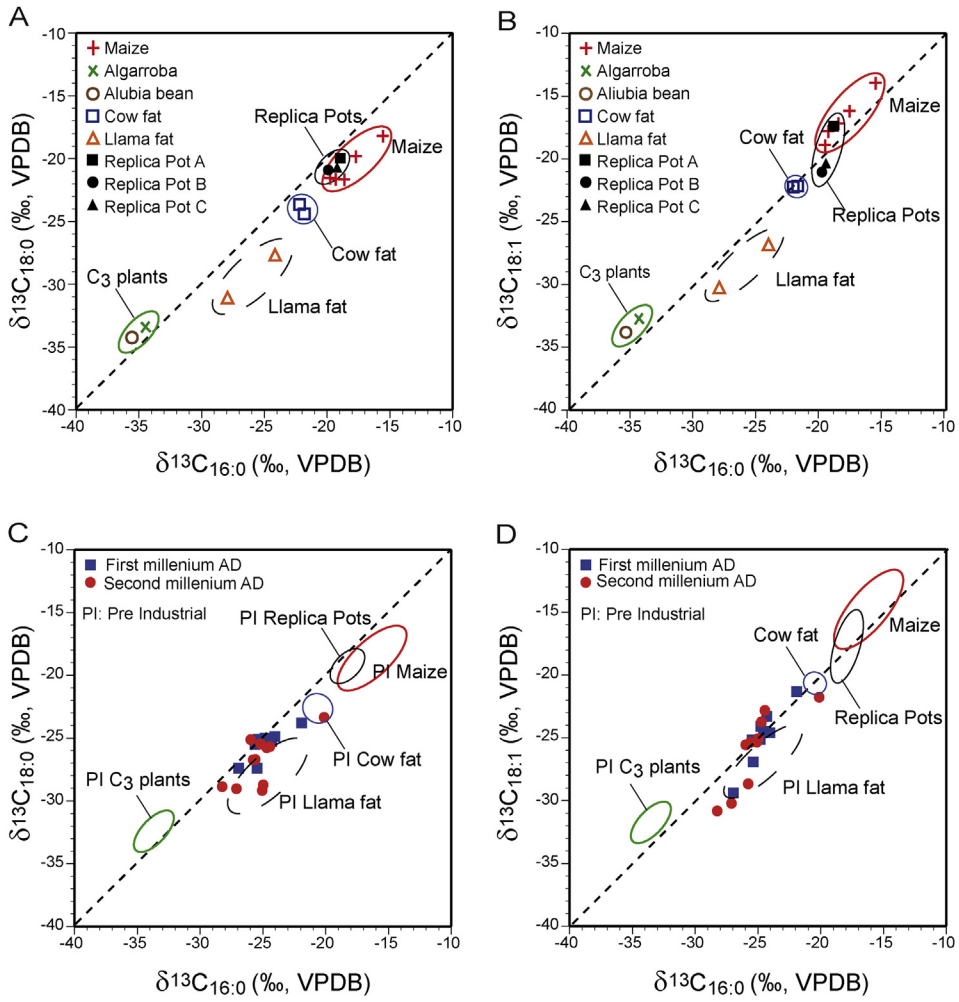

Fig. 6. Carbon isotope composition of: A, stearic acid ($\delta^{13}C_{18:0}$) versus palmitic acid ($\delta^{13}C_{16:0}$) of reference samples; B, stearic acid ($\delta^{13}C_{18:0}$) versus oleic acid ($\delta^{13}C_{18:1}$) of reference samples; C, stearic acid ($\delta^{13}C^{18:0}$) versus palmitic acid ($\delta^{13}C_{16:0}$) of reference samples; D, stearic acid ($\delta^{13}C_{18:0}$) versus oleic ($\delta^{13}C_{18:1}$) of reference samples. The fields for present-day reference samples were corrected to pre-industrial values for comparison with archaeological samples.

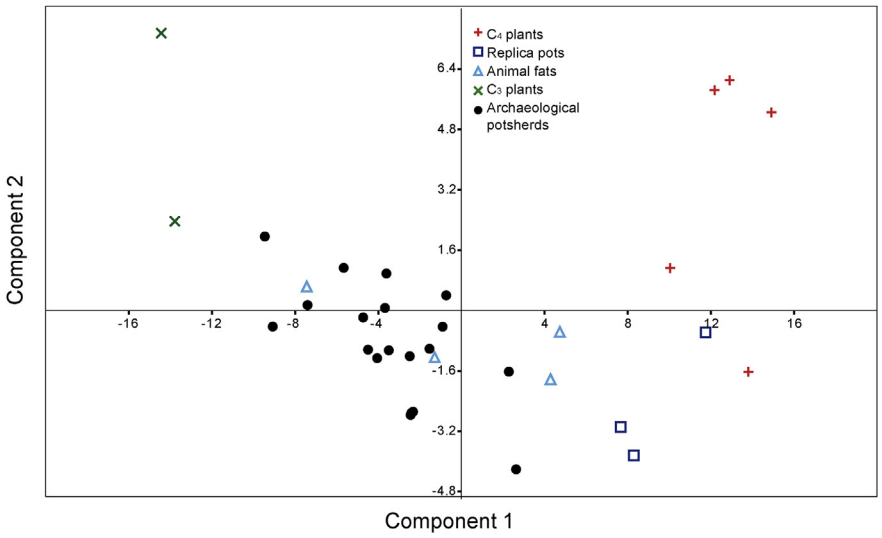

Fig. 7. Principal component analysis of main fatty acid isotopic values and palmitic/stearic ratio. N = 34. Variable 1 ($\delta^{13}C_{16:0}$); variable 2 ($\delta^{13}C_{18:0}$); variable 3 ($\delta^{13}C_{18:1}$); variable 4 (P/S ratio). Explained variance: PC1 (86.7%); PC2 (10.83%). Loadings: variable 1 (PC1: 0.55, PC2: −0.22); variable 2 (PC1: 0.49, PC2: −0.17); variable 3 (PC1: 0.63, PC2: −0.05); variable 4 $C_{16:0}/C_{18:0}$ (PC1: 0.25, PC2: 0.96).

triacontanol (30-OH) (A-04, A-06, A-14). These long-chain alcohols are characteristic of plant waxes (Kolattukudy, 1970). Neither the archaeological samples nor the maize reference sample (R-03, *dentado blanco* maize) contained *n*-dotriacontanol (32-OH), a long-chain alcohol that was proposed as a biomarker of North American maize (Reber and Evershed, 2004b; Reber et al., 2004). Samples A-01 and A-03 showed the presence of cholesterol, a lipid biomarker for meat, together with minor cholesterol degradation products. Sitosterol, a lipid biomarker for plants, was identified in sample A-11 as well as in maize and bean reference samples, which also contained campesterol and stigmasterol. The identification of cholesterol together with long-chain alcohols 24-OH, 25-OH, 26-OH, and 28-OH in archaeological samples A-01 and A-03 points to a mixture of vegetable and animal ingredients in these cooking pots.

4.2. Stable carbon isotope composition of individual fatty acids

Results of CSIA of the main FAs from lipid extracts of reference and archaeological samples are presented in Table 5. The $\delta^{13}C$ values of the main FAs for maize (R-01, R-02, R-03, R-04, and R-14) were within the published range (Spangenberg and Ogrinc, 2001; Woodbury et al., 1995). Mean values for $C_{16:0}$, $C_{18:0}$, $C_{18:1}$, and $C_{18:2}$ were −16.9 ± 1.0‰, −19.2 ± 1.0‰, −15.5 ± 1.3‰ and −16.5 ± 1.2‰, respectively. The C_4 leguminous plant samples (bean R-05 and *algarroba* R-06) had mean $\delta^{13}C$ values for $C_{16:0}$, $C_{18:0}$, $C_{18:1}$, and $C_{18:2}$ between −33.4 and −31.6‰. Bovine fat samples (R-07 and R-08) had values between −22.4 and −20.4‰ for $C_{16:0}$, $C_{18:0}$, $C_{18:1}$ and −29.1‰ for $C_{18:2}$. The fat samples of llamas (R-09 and R-10) fed exclusively on C_3 grasses had mean $\delta^{13}C$ values between −27.5 and −24.5‰ for $C_{16:0}$, $C_{18:0}$, $C_{18:1}$, and similarly to bovine fat had ^{13}C-depleted $C_{18:2}$ acids (−29.9‰). The lower mean values for $C_{18:2}$ compared to the other main adipose FAs may be explained by a contribution of endogenous synthesized ^{13}C-depleted *cis*-9,*trans*-11$C_{18:2}$ acid (Richter et al., 2012a, 2012b).

Replica pot A (R-11) had $\delta^{13}C$ values ranging from −19.0‰ to −17.4‰. In replica pots B and C (R-12, R-13), the $\delta^{13}C$ value of the FAs represented mixtures of animal and vegetable fats. They had similar $\delta^{13}C$ values for $C_{16:0}$, $C_{18:0}$, $C_{18:1}$, varying between −21.0‰ and −19.1‰. In replica pot B, the relatively lower $\delta^{13}C_{18:2}$ value (−24.6 ± 1.7) is explained by the mixture of the recipe components: maize (−16.5 ± 1.2‰), bean (−34.8 ± 0.6‰) and bovine fat (−29.1 ± 4.5‰). In replica pot C, the $\delta^{13}C_{18:2}$ value (−17.7 ± 0.4‰) is dominated by maize.

The $\delta^{13}C$ values of the main FAs of the lipids extracted from the archaeological samples (A-01 to A-22) were −25.6 ± 1.6‰ ($C_{14:0}$), −25.1 ± 1.7‰ ($C_{15:0}$), −25.0 ± 1.7‰($C_{16:0}$), −25.3 ± 1.9‰ ($C_{16:1}$), −26.3 ± 1.9‰ ($C_{17:0}$), −26.1 ± 1.7‰ ($C_{18:0}$), −25.5 ± 2.7‰ ($C_{18:1}$), and −31.7 ± 6.3‰ ($C_{18:2}$). The mean $\delta^{13}C$ values of each sample ranged from −28.8 to −21.5‰, indicating different biological sources of the organic residues.

The isotopic compositions of the main FAs in archaeological samples were compared with those from reference samples (Evershed, 2008; Spangenberg et al., 2006). We observed complex mixtures of different biological origins in the western Tinogasta archaeological samples. Biplots of the reference samples $\delta^{13}C_{16:0}$ values against $\delta^{13}C_{18:0}$ and $\delta^{13}C_{18:1}$ (Fig. 6 A and B) showed a clear separation between vegetable oils from C_3 and C_4 plants. Animal fats were positioned in between C_3 and C_4 plants. Replica pot A was positioned in both biplots near maize reference samples. Replica pots B and C were positioned close to maize reference samples in biplot A, and in an intermediate position between maize and cow fat in biplot B. Biplots C and D of archaeological samples (Fig. 6C and D) showed that most isotopic values fell within the range of pre-industrial llama fat reference values. Only a few samples with higher values were positioned closer to the pre-industrial values for cow fat (A-09, A-14, A-18 and A-19). These higher values could indicate mixtures with a greater proportion of C_4 plant oils and a lower proportion of C_3 plant oils and animal fats.

4.3. Statistical analyses

Principal component analysis (PCA) was carried out on 33 samples (14 reference samples corrected to preindustrial values and 19 archaeological samples). We selected four variables for multivariate analysis: the main fatty acid $\delta^{13}C$ values ($\delta^{13}C_{16:0}$, $\delta^{13}C_{18:0}$, and $\delta^{13}C_{18:1}$) and palmitic acid to stearic acid (P/S) ratios. The first two principal components (PC) explain 97.5% of the total variance; PC1 explains 86.7% and PC2 10.8%. For PC1, the greatest loading was observed for $\delta^{13}C_{18:1}$ (0.63), followed by $\delta^{13}C_{16:0}$ (0.55), $\delta^{13}C_{18:0}$ (0.49), and P/S ratio (0.25). For PC2, the greatest loading was observed for P/S ratio (0.96), followed by $\delta^{13}C_{16:0}$ (−0.22),

Table 5
$\delta^{13}C$ values (‰ *vs.* VPDB) of the main fatty acids in lipids from reference samples and archaeological potsherds recovered at West Tinogasta.

Sample	Description	$\delta^{13}C_{14:0}$	$\delta^{13}C_{16:0}$	$\delta^{13}C_{18:0}$	$\delta^{13}C_{18:1}$	$\delta^{13}C_{18:2}$
Reference samples						
R-01	Pisingallo		−17.7 ± 0.3	−19.7 ± 0.4	−16.3 ± 0.4	−16.6 ± 0.4
R-02	Chullpi		−19.7 ± 0.4	−21.4 ± 0.5	−18.9 ± 0.3	−18.6 ± 0.1
R-03	D. Blanco		−18.6 ± 0.2	−21.6 ± 0.5	−17.2 ± 0.1	−17.7 ± 0.1
R-04	Capia		−19.2 ± 0.5	−21.5 ± 1.5	−17.8 ± 0.8	−17.6 ± 0.4
R-05	Algarroba		−34.5 ± 0.4	−33.4 ± 0.3	−32.5 ± 1.2	−34.5 ± 1.7
R-06	Alubia		−35.5 ± 0.2	−34.3 ± 0.3	−33.8 ± 0.8	−34.8 ± 0.6
R-07	Bovine I	−23.5 ± 0.9	−22.2 ± 0.1	−23.6 ± 0.1	−22.3 ± 0.1	−33.8 ± 0.8
R-08	Bovine II	−23.4 ± 0.5	−21.8 ± 0.3	−24.4 ± 0.4	−22.2 ± 0.1	−27.5 ± 1.4
R-09	Llama I	−24.3 ± 0.3	−24.2 ± 0.1	−27.2 ± 0.3	−26.5 ± 0.2	−28.4 ± 0.1
R-10	Llama II	−27.9 ± 0.9	−28.0 ± 0.4	−31.0 ± 0.3	−30.1 ± 0.7	−34.7 ± 3.0
R-11	Pot A		−19.0 ± 0.2	−19.0 ± 1.0	−17.4 ± 0.7	−18.4 ± 0.6
R-12	Pot B	−19.6 ± 0.2	−20.0 ± 0.3	−20.9 ± 0.1	−21.0 ± 0.7	−24.6 ± 1.7
R-13	Pot C	−19.1 ± 0.1	−19.7 ± 0.1	−20.6 ± 0.2	−20.3 ± 0.8	−17.7 ± 0.4
R-14	Pisingallo		−15.6 ± 0.1	−18.3 ± 0.1	−13.9 ± 0.4	−19.7 ± 2.1
Archaeological samples						
A-01	La Troya LTV50; cooking pot	−25.2 ± 0.3	−24.3 ± 0.4	−25.3 ± 0.3	−23.3 ± 0.4	
A-02	La Troya LTV50; cooking pot	−25.7 ± 0.2	−24.6 ± 0.4	−25.0 ± 0.1		
A-03	La Troya LTV50; cooking pot	−27.6 ± 0.7	−24.0 ± 0.4	−24.8 ± 0.3	−24.6 ± 0.4	
A-04	Batungasta; cooking pot	−25.9 ± 0.2	−25.0 ± 0.2	−28.7 ± 0.2	−25.1 ± 1.3	
A-05	Batungasta; cooking pot	−27.4 ± 0.9	−26.0 ± 0.4	−25.1 ± 0.7	−25.6 ± 1.5	
A-06	Batungasta; cooking pot	−26.0 ± 0.2	−25.1 ± 0.3	−29.2 ± 0.1	−25.3 ± 0.3	
A-07	Mishma 7; cooking pot	−26.5 ± 1.3	−25.6 ± 0.6	−26.7 ± 0.6	−25.4 ± 1.4	
A-08	Mishma 7; beverage-holding vessel (*Belén*)	−24.9 ± 0.1	−24.7 ± 0.3	−25.8 ± 0.3	−23.8 ± 0.3	
A-09	Quintar 1; cooking pot	−23.0 ± 1.2	−24.8 ± 2.4	−25.6 ± 2.7		
A-10	Palo Blanco NH1; cooking pot	−25.3 ± 1.1	−24.7 ± 1.6	−25.5 ± 1.3	−24.1 ± 1.1	−34.6 ± 1.2
A-11	Palo Blanco NH3; cooking pot	−26.1 ± 0.3	−25.4 ± 0.1	−25.1 ± 0.1	−27.0 ± 0.1	−36.5 ± 2.6
A-12	Palo Blanco NH6; cooking pot	−25.4 ± 1.1	−26.9 ± 0.9	−27.4 ± 0.9	−29.4 ± 1.0	
A-13	Palo Blanco NH4; cooking pot	−26.3 ± 0.3	−25.6 ± 0.2	−25.5 ± 0.1		
A-14	Ojo del Agua; cooking pot	−24.0 ± 1.0	−24.8 ± 0.9	−25.0 ± 0.8	−25.1 ± 0.4	
A-15	Cardoso; cooking pot	−26.7 ± 0.6	−24.5 ± 0.2	−25.3 ± 0.1	−24.5 ± 0.7	−24.5 ± 0.1
A-16	Punta Colorada; cooking pot	−27.7 ± 0.1	−25.5 ± 0.3	−27.4 ± 0.2	−25.2 ± 0.3	−31.9 ± 2.9
A-17	San Francisco; beverage-holding vessel (*aribaloid*)	−25.5 ± 1.5	−24.5 ± 1.3	−25.7 ± 1.5	−22.8 ± 1.6	
A-18	San Francisco; beverage-holding vessel (*aribalo*)	−21.7 ± 1.2	−20.2 ± 0.4	−23.4 ± 2.0	−21.8 ± 3.5	
A-19	El Zorro; cooking pot	−23.5 ± 0.5	−21.9 ± 0.4	−23.8 ± 0.4	−21.3 ± 0.1	−23.6 ± 0.8
A-20	Batungasta; cooking pot	−28.8 ± 0.4	−28.3 ± 0.1	−28.9 ± 0.3	−30.8 ± 0.1	−31.1 ± 1.6
A-21	San Francisco; beverage-holding vessel (*aribalo*)	−25.5 ± 0.6	−27.1 ± 0.8	−29.0 ± 0.5	−30.2 ± 0.3	
A-22	San Francisco; beverage-holding vessel (*aribaloid*)	−25.0 ± 1.3	−25.8 ± 1.5	−26.8 ± 1.7	−28.7 ± 1.7	−28.3 ± 2.9

$\delta^{13}C_{18:0}$ (−0.17), and $\delta^{13}C_{18:1}$ (−0.05). PC1 represents mainly the FAs $\delta^{13}C$ values, while for PC2 the P/S ratio was the most significant. We observed superposition of most archaeological samples with pre-industrial reference values for llama and cow fats (Fig. 8).

Hierarchical cluster analysis (HCA; Ward's method) grouped samples in four clusters at an Euclidean distance of 19 (Fig. 9). Cluster 1 grouped three modern maize landraces: *pisingallo*, *dentado blanco*, and *capia blanco*. Cluster 2 grouped samples of modern *chullpi*, archaeological *pisingallo*, replica potsherds, cow fat, and two archaeological samples (A-18 and A-19). Cluster 3 included modern llama fat and archaeological samples A-01, A-03, A-04, A-05, A-06, A-07, A-08, A-10, A -11, A-14, A-15, A-16, and A-17. Cluster 4 grouped *algarroba*, bean and llama jerkey with archaeological samples A-12, A-20, A-21, and A-22. Cooking pots were mostly included in Cluster 3 (A-15, A-10, A-01,A-03, A-14, A-05, A-07, A-11, A-16, A-04, and A-06), followed by Cluster 4 B2 (A-12 and A-20) and finally Cluster 2 (A19). Vessels used to produce, store and/or transport beverages were classified into Cluster 4 (A-08, A-21 and A-22), Cluster 2 (A-18), and Cluster 3 (A-17).

In order to test if the grouping of samples achieved by PCA and HCA was significant, we carried out a discriminant analysis (DA). The first three components resulting from PCA were used for such purpose. The samples were hypothetically assigned to the four groups that resulted from HCA. Three discriminant functions and four predictive groups were generated. The first two functions are plotted in Fig. 9. Overall, 97% of the original groups were correctly classified. Predictive groups 1, 2 and 4 had 100% correct classification, so their membership was identical to that in clusters 1, 2 and 4, respectively. Predictive group 3 had 92.9% correct classification, while 7.1% of the cases were classified into predictive group 4. Canonic correlations and Wilk's Lambda values were satisfactory (value in caption of Fig. 9), indicating that the predictive model created by DA is reliable. The result of this analysis proves that the four HCA groups are significant and relevant for this study.

4.4. Microscopic identification and characterization of starch grains

Starch grains were recovered in five of the 22 archaeological potsherds and three replica potsherds. Maize starch granules were found in three replica potsherd samples R-11, R-12 and R-13 (Fig. 10 A, B, E, F), and additionally bean starch granules were found in R-12 (Fig. 10 M, N). Some starch granules in R-13 showed signs of heat alteration due to the gelatinization of the amylose and amylopectin layers, and could be observed microscopically by the loss of the extinction cross under polarized light (Fig. 10) (Raviele, 2011). Maize starch granules were also found in samples A-05, A-06, A-13, and A-14 (Fig. 10C, D, G, H, I, J, K, L). They were identified by the polyhedral or semi-polyhedral morphologies, maximum diameters ranging from 10 to 20 μm, pointed *hillum* in a central position, central fissures, straight angled polarized cross, and polarized light intensity of 4–6 in the 1–10 scale (Piperno and Holst, 1998). Sample A-06 contained a bell-shaped granule typical of maize (Fig. 10K, L) (Holst et al., 2007). Sample A-06 also contained one starch granule of *Phaseolus vulgaris* L. (bean) which was identified by its kidney-shaped morphology, a 33 μm maximum diameter, a polarized light intensity of 8, an eccentric pointed *hillum*, and the straight

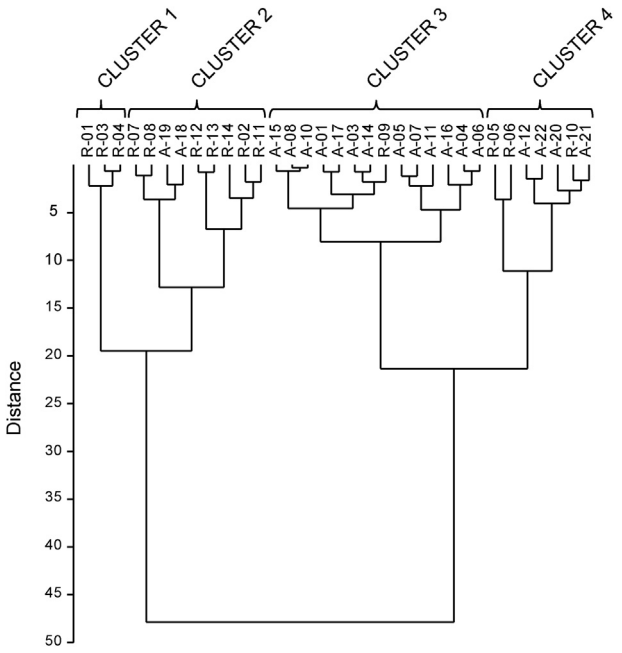

Fig. 8. Hierarchical cluster analysis (Ward's method) of main fatty acid isotopic values and palmitic/stearic ratio. Cluster dissimilarity was set at a Euclidian distance of 19. N = 33. Variable 1 ($\delta^{13}C_{16:0}$); variable 2 ($\delta^{13}C_{18:0}$); variable 3 ($\delta^{13}C_{18:1}$); variable 4 ($C_{16:0}/C_{18:0}$). Correlation coefficient: 0.7104. Cluster 1: samples R-01, R-03, R-04. Cluster 2: samples R-07, R-08, A-19, A-18, R-12, R-13, R-14, R-02, R-11. Cluster 3: samples A-15, A-08, A-10, A-01, A-17, A-03, A-14, R-09, A-05, A-07, A-11, A-16, A-04, A-06. Cluster 4: samples R-05, R-06, A-12, A-22, A-20, R-10, A-21.

angled polarized cross (Fig. 10 O, P) (Babot et al., 2007; Piperno and Holst, 1998). A granule of *Prosopis* L. genera (*algarroba*) was also observed in sample A-06, which was identified by its irregular faceted morphology, a maximum diameter of 31 μm, an eccentric *hillum*, a polarized light intensity of 4, and the thin broken arms of the polarization cross which thickened towards the ending (Fig. 10S, T) (Acosta et al., 2013; Giovannetti et al., 2008; Lema et al., 2012). The occurrence of starch granules from maize, bean, and *algarroba* in sample A-06 provided evidence for a mixture of vegetable ingredients. This could result from food recipes including the three ingredients, or from multiple uses of the vessel. The starch granules recovered in sample A-11 could not be identified at a species or genera level because they did not present any characteristic features and showed signs of degradation such as a partial loss of the extinction cross under polarized light.

5. Discussion

Starch and lipid organic residues from archaeological potsherd samples dating from the first millennium AD (A-01, A-02, A-03, A-10, A-11, A-12, A-13, A-14, A-15, A-16, A-19) indicated that the most common cooking practice was stewing a mixture of ingredients in the same pot. These mixtures could possibly be the result of a combination of ingredients being stewed together, or of multiple uses of the vessels for cooking separately each of the different ingredients. The organic mixtures included different proportions of South American camelid fat (such as llama, guanaco or vicuña), C_3 plants such as bean or *algarroba* and C_4 plants such as maize. The latter ingredient was detected by the presence of maize starch granules rather than by distinct FAs profiles and $\delta^{13}C$ values. This suggests that (i) the relative proportions of maize in the stews were not dominant, or (ii) that the contribution of maize lipids was masked by other ingredients, such as animal fat. Similar patterns were observed in two of our experimental replica pots (B and C), where the characteristic signature of maize in complex mixtures was observed mostly by relative high abundance of $C_{18:2}$ with high $\delta^{13}C$ values, which can be rarely found in archaeological samples due to early diagenetic degradation. It is worth to point out that $C_{18:2}$ was found in small amounts in 8 of the 22 samples (A-10, A11, A-12, A-15, A-16, A-19, A-20; A-21), indicating that they were exceptionally well preserved. In these cases, only A-15 and A-19 had less negative $\delta^{13}C$ values that could indicate maize oil origin. The other samples had $\delta^{13}C$ typical for C_3 plants. Maize starch granules were found in samples A-13 from Palo Blanco NH4 (AD 427–599) and A-14 from Ojo del Agua (AD 994–1047) located at

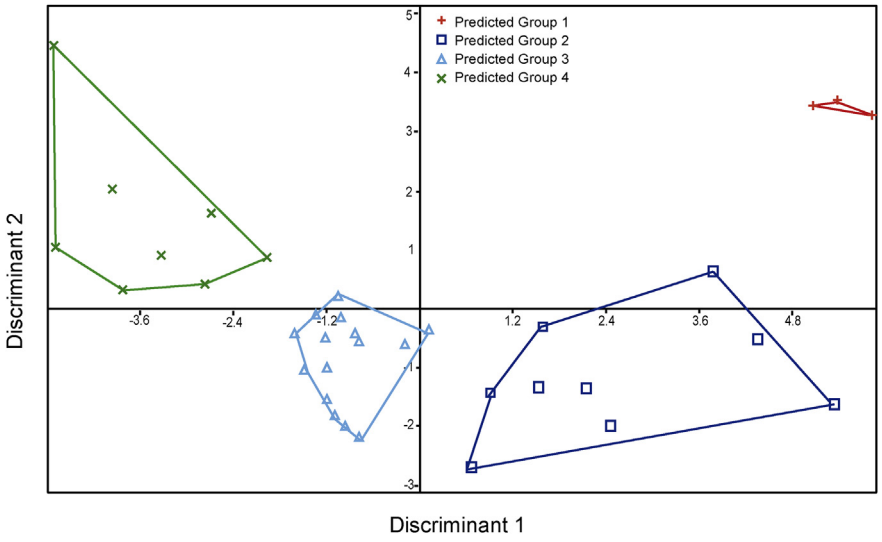

Fig. 9. Discriminant Analysis carried out using the first three components resulting from Principal Component Analysis. Samples were hypothetically assigned to the groups resulting from Hierarchical Cluster Analysis (4 clusters). N = 33. Correction was carried out by Mahalanobis distance. Graph shows the two first discriminant functions and four predictive groups. Predictive group 1: 100% correct classification of members of Cluster 1. Predictive group 2: 100% correct classification of members of Cluster 2. Predictive group 3: 92.9% correct classification of members of Cluster 3 and 7.1% classified in Predictive Group 4. Predictive group 4: 100% correct classification of members of Cluster 4. Overall, 97% of the original groups were correctly classified. Canonic correlation: 0.948 (Discriminant Function 1), 0.843 (Discriminant Function 2), 0.340 (Discriminant Function 3). Wilk's Lambda: 0.026 (Function 1–3); 0.256 (Funtion 2–3); 0.885 (Function 3).

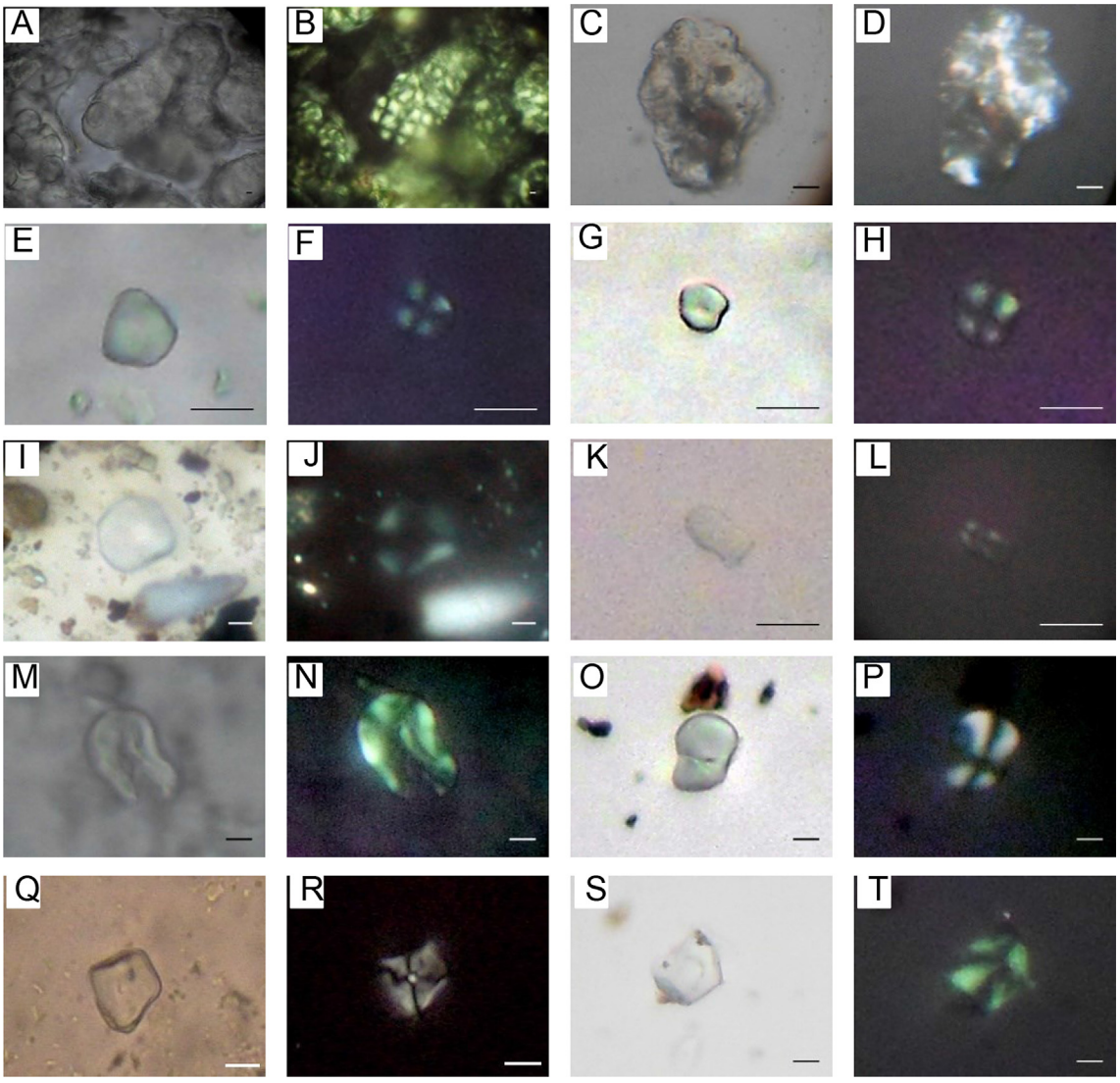

Fig. 10. Starch grains from archaeological and replica pots: A and B, modern maize starch granule aggregates from R-11 under normal and polarized light; C and D, archaeological maize starch granule aggregates from A-06 under normal and polarized light; E and F, modern maize polyhedral starch granule from R-11 under normal and polarized light; G and H, archaeological maize polyhedral starch granule from A-06 under normal and polarized light; I and J archaeological maize starch granule from A-14 under normal and polarized light; K and L, archaeological maize bell-shaped granule from A-06 under normal and polarized light; M and N, modern bean starch from R-11 under normal and polarized light; O and P, archaeological bean starch granule from A-06 under normal and polarized light; Q and R, modern *algarroba* starch granule under normal and polarized light; S and T, archaeological *algarroba* starch granule from A-06 under normal and polarized light. Bars represent 10 μm.

1900 masl and 2400 masl, respectively. One cooking pot (A-19) from El Zorro (AD 322–1019) located at 4050 masl showed higher $\delta^{13}C$ values (−23.3 ± 1.4‰), suggesting a contribution of maize residues. Given that maize cannot be grown for food purposes at such altitude, it might have been transported by herders from the lower Fiambalá region for sustenance during flock tending in the San Francisco *vega* (or highland bog). Also in sample A-19, the $\delta^{13}C_{14:0}$ value (−23.5 ± 0.5‰) falls within the ranges of modern samples of cows raised on C_3 pastures and fattened on maize before slaughter. The high isotopic value for this fatty acid of animal origin can be explained by the ancient herding practice of feeding llama herds with maize stubble during certain annual fallow periods (Dantas et al., 2014) or the use of other C_4 pastures to feed the animals.

In samples from the second millenium AD we observed continuity in the domestic cooking practices from the previous period. Cooking pots from Mishma 7 and Batungasta showed mixtures of llama, C_3 plants such as bean or *algarroba*, and C_4 plants such as maize, cooked either simultaneously or sequentially. On the other hand, samples from San Francisco and Mishma 7 that were associated with Inka rituals and festivities showed some changes in the way maize was prepared and consumed. Beverage-holding vessels used to store, transport and serve alcoholic beverages in Inka rituals had different chemical and isotopic profiles. While A-18 had $\delta^{13}C$ value comparable to C_4 plants, A-21 and A-22 had $\delta^{13}C$ values comparable with C_3 plants, and A-08 and A-17 had intermediate isotopic composition, suggesting a mixture of C_3 and C_4 plants (Table 5). The relatively higher $\delta^{13}C$ values in sample A-18 could indicate preparation of maize beer *chicha*, while the relatively low $\delta^{13}C$ values in samples A-21 and A-22 could indicate the preparation of another typical local fermented drink called *aloja* produced from the flour of *algarroba* pods. The mixed isotopic values in A-17 could indicate possible alternation between *chicha* and *aloja* storage and/or transport. The presence of some FAs of animal origin (myristic acid and ramified fatty acids) in samples A, 17, A-18, A-21 and A-22 could indicate the use of South American camelid fat to

make the inner walls of the containers impermeable and avoid beverage loss by diffusion through the ceramic matrix (Henrickson and McDonald, 1983; Schiffer, 1990; Skibo, 1992; Volzone and Zagorodny, 2014). The constricted necks, large volumes and absence of soot in these containers do not support their use for cooking stews.

The variations in the types of alcoholic beverages produced are potential evidence of the survival of certain local practices as a resistance to those imposed by the Inka administration. The production and consumption of traditional Andean *chicha* beer and local Catamarcan *aloja* drink in the same ritual scenarios are indication of some permeation of the Inka practices by local cultural traditions (Orgaz, 2012). This is supported by the ethno-historical accounts of consumption of *aloja* during the early colonial period as part of local rituals carried out in defiance of the Spanish rule, which considered these practices as witchcraft and persecuted them (Arana, 1999; Carrizo, 1942; Faberman, 2005; Quiroga et al., 1999). Moreover, mixtures of imperial *chicha* and local *aloja* were identified in *Belén* vessels and *aribaloids*, which are both beverage-holding vessels that mix local and Inka design and technological elements.

6. Conclusion

The three archaeometric techniques (FAs distributions, compound specific C isotope analysis, and microscopy of starch grains) used to study the organic residues preserved in ceramic potsherds from western Tinogasta, Catamarca, Argentina, allowed us to identify lipid and starch granule origins. Domestic foodways throughout the 3rd to 16th centuries AD were based on mixing a variety of ingredients into the stew pots, and cooking practices changed little through time. On the other hand, ritual feasting practices were part of the public life and therefore more subjected to the political transformations during the Inka domination period and later Hispanic conquest. Nevertheless, our results suggest that the survival of local traditional beverage production and consumption practices may have been a form of resistance to the Inka administration. In sum, residue analysis opened a window into the strong sociological and political significance of domestic and public foodways of pre-Hispanic agro-pastoral societies in the south-central Andes.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.jas.2014.12.022.

References

Acosta, I., Leiva, G., Malec, L., 2013. Estudio de herramientas líticas del humedal del Paraná inferior. Análisis de los gránulos de almidón. Cuad. Inst. Nac. Antropol. Pensam. Latinoam. Ser. Espec. 1, 174–184.

Amuedo, C., 2012. Las conexiones entre las prácticas mortuorias de infantes y los alimentos. La materialidad y los significados generados en el movimiento cotidiano. In: Babot, M.P., Marschoff, M., Pazzarelli, F. (Eds.), Las Manos En La Masa. Arqueologías, Antropologías E Historias de La Alimentación En Sur-amárica. Corintios 13, Córdoba, pp. 667–692.

Appadurai, A., 1981. Gastro-politics in Hindu South Asia. Am. Ethnol. 8, 494–511. http://dx.doi.org/10.1525/ae.1981.8.3.02a00050.

Arana, M., 1999. El Tiempo de la Algarroba. In: Aschero, C.A., Korstanje, M.A., Vuoto, P. (Eds.), Los Tres Reinos: Prácticas de Recolección En El Cono Sur de América. Instituto de Arqueología y Museo, Universidad Nacional de Tucumán, Tucumán, pp. 197–203.

Aranda, C., Ratto, N., Luna, L., 2011. Dieta de individuos de contextos funerarios de valles mesotérmicos del oeste Catamarqueño (dpto. Andalgalá, Belén y Tinogasta, Catamarca, Argentina). In: Guitérrez, S., Hocquet, F.P., Rojas, M., Sepúlveda, M., Silva-Pinto, V. (Eds.), Libro de Resúmenes Del III Congreso Latinoamericano de Arqueometría. Presented at the III Congreso Latinoamericano de Arqueometría. Universidad de Tarapacá, Arica, pp. 110–111.

Babot, M.P., 2005. Plant resource processing by Argentinian Puna hunter-gatherers (ca. 7000-3200 B.P): microfossil record. Phytolith. Bull. Soc. Phyt. Res. 17, 9–10.

Babot, M.P., Apella, M.C., 2003. Maize and bone: residues of grinding in Northwestern Argentina. Archaeometry 45, 121–132. http://dx.doi.org/10.1111/1475-4754.00099.

Babot, M.P., Oliszewski, N., Grau, A., 2007. Análisis de caracteres macroscópicos y microscópicos de Phaseolus vulgaris (Fabaceae, Faboideae) silvestres y cultivados del noroeste argentino: una aplicación arqueobotánica. Darwiniana 45, 149–162. http://www.ojs.darwin.edu.ar/index.php/darwiniana/article/view/87.

Bonomo, N., Osella, A., Ratto, N., 2010. Detecting and mapping buried buildings with ground-penetrating radarat an ancient village in northwestern Argentina. J. Archaeol. Sci. 37, 3247–3255. http://dx.doi.org/10.1016/j.jas.2010.07.025.

Boyd, M., Varney, T., Surette, C., Surette, J., 2008. Reassessing the northern limit of maize consumption in North America: stable isotope, plant microfossil, and trace element content of carbonized food residue. J. Archaeol. Sci. 35, 2545–2556. http://dx.doi.org/10.1016/j.jas.2008.04.008.

Bray, T., 2003. Inka pottery as culinary equipment: food, feasting, and gender in imperial state design. Lat. Am. Antiq. 14, 3–28.

Bray, T., 2004. La alfarería imperial inka: una comparación entre la cerámica estatal del área de Cuzco y la cerámica de las provincias. Chungará 36, 365–374. http://www.jstor.org/stable/27802278.

Calderari, M., Williams, V., 1991. Re-evaluación de los estilos cerámicos del noroeste argentino. Comechingonia 9, 75–95.

Carrizo, J.A., 1942. Cancionero Popular de La Rioja. Baiocco y Cía., Buenos Aires.

Coates, W., Ayerza, R., 2004. Fatty acid composition of llama muscle and internal fat in two Argentinian herds. Small Rumin. Res. 52, 231–238. http://dx.doi.org/10.1016/j.smallrumres.2003.07.002.

Copley, M.S., Bland, H.A., Rose, P., Horton, M., Evershed, R.P., 2005. Gas chromatographic, mass spectrometric and stable carbon isotopic investigations of organic residues of plant oils and animal fats employed as illuminants in archaeological lamps from Egypt. Analyst 130, 860–871. http://dx.doi.org/10.1039/B500403A.

Cortella, A.R., Pochettino, M.L., 1995. Comparative morphology of starch of three Andean Tubers. Starch – Stärke 47, 455–461. http://dx.doi.org/10.1002/star.19950471202.

Crowther, A., 2005. Starch residues on undecorated Lapita pottery from Anir, New Ireland. Archaeol. Ocean. 40, 62–66. http://dx.doi.org/10.1002/j.1834-4453.2005.tb00586.x.

Crowther, A., 2012. The differential survival of native starch during cooking and implications for archaeological analyses: a review. Archaeol. Anthropol. Sci. 4, 221–235. http://dx.doi.org/10.1007/s12520-012-0097-0.

Dantas, M., Figueroa, G.G., Laguens, A., 2014. Llamas in the Cornfield: prehispanic Agro-Pastoral System in the Southern Andes. Int. J. Osteoarchaeol. 24, 149–165. http://dx.doi.org/10.1002/oa.2351.

Deines, P., 1980. The isotopic composition of reduced organic carbon. In: Fritz, P., Fontes, J.C. (Eds.), The Terrestrial Environment. Elsevier Scientific Publishing Company, New York, pp. 239–406.

Dietler, M., 2006. Alcohol: anthropological/archaeological perspectives. Annu. Rev. Anthropol. 35, 229–249. http://dx.doi.org/10.1146/annurev.anthro.35.081705.123120.

Eerkens, J.W., 2005. GC–MS analysis and fatty acid ratios of archaeological potsherds from the western great basin of North America. Archaeometry 47, 83–102. http://dx.doi.org/10.1111/j.1475-4754.2005.00189.x.

Evershed, R.P., 2008. Organic residue analysis in archaeology: the archaeological biomarker revolution. Archaeometry 50, 895–924. http://dx.doi.org/10.1111/j.1475-4754.2008.00446.x.

Evershed, R.P., Dudd, S.N., Copley, M.S., Berstan, R., Stott, A.W., Mottram, H., Buckley, S.A., Crossman, Z., 2002. Chemistry of archaeological animal fats. Acc. Chem. Res. 35, 660–668. http://dx.doi.org/10.1021/ar000200f.

Faberman, J., 2005. Las Salamancas de Lorenza. Magia, Hechicería y Curanderismo en el Tucumán Colonial. Siglo Veintiuno, Buenos Aires.

Feely, A., 2010. Tradición cerámica y límites sociales en el valle de Fiambalá (Dto. Tinogasta, Catamarca) (Tesis Doctoral inédita). Universidad de Buenos Aires, Buenos Aires.

Feely, A., 2013. Los modos de hacer las vasijas: elecciones técnicas y estilos tecnológicos del oeste tinogasteño (Catamarca). In: Ratto, N. (Ed.), Delineando Prácticas de La Gente Del Pasado: Los Procesos Sociohistóricos Del Oeste Catamarqueño. Sociedad Argentina de Antropología, Buenos Aires, pp. 69–133.
Giovannetti, M., Spina, J., Páez, M.C., Cochero, G., Rossi, A., Espósito, P., 2013. En busca de las festividades del Tawantinsuyu: Análisis de los tiestos de un sector de descarte de El Shincal de Quimivil. Intersecc. antropol. 14, 67–82. http://ref.scielo.org/kr9m37.
Giovannetti, M.A., 2013. Propuesta para la recolección de microvestigios arqueobotánicos en morteros fijos. Comechingonia 17, 189–199. http://ref.scielo.org/kv5ngz.
Giovannetti, M.A., Lantos, I., Defacio, R., Ratto, N., 2012. Construcción de un banco de almidones de variedades nativas de Zea mays L. del noroeste argentino: Propuesta metodológica y primeros resultados. In: Babot, M.P., Marschoff, M., Pazzarelli, F. (Eds.), Las Manos En La Masa: Arqueologías, Antropologías E Historias de La Alimentación En Suramárica. Corintios 13, Córdoba, pp. 361–385.
Giovannetti, M.A., Lema, V.S., Bartoli, C.G., Capparelli, A., 2008. Starch grain characterization of Prosopis chilensis (Mol.) Stuntz and P. flexuosa DC, and the analysis of their archaeological remains in Andean South America. J. Archaeol. Sci. 35, 2973–2985. http://dx.doi.org/10.1016/j.jas.2008.06.009.
Goldstein, D., 2003. From stew-eaters to maize-drinkers. The Chicha economy and the Tiwanaku expansion. In: Bray, T. (Ed.), The Archaeology and Politics of Foods and Feasting in Early States and Empires. Klewer Academic/Plenum Publishers, New York, pp. 143–172.
Grela, E.R., Günter, K.D., 1995. Fatty acid composition and tocopherol content of some legume seeds. Animal Feed Sci. Technol. 52, 325–331. http://dx.doi.org/10.1016/0377-8401(94)00733-P.
Gumerman, G., 1997. Food and complex societies. J. Archaeol. Method Theory 4, 105–139. http://dx.doi.org/10.1007/BF02428056.
Haas, J., Creamerb, W., Huamán Mesía, L., Goldstein, D., Reinhard, K., Vergel Rodríguez, C., 2013. Evidence for maize (Zea mays) in the Late Archaic (3000–1800 B.C.) in the Norte Chico region of Peru. Proc. Natl. Acad. Sci. 110, 4945–4949. http://dx.doi.org/10.1073/pnas.1219425110.
Hammer, O., Harper, D.A.T., Ryan, P.D., 2001. PAST: paleontological statistics software package for education and data analysis. Palaeontol. Electron. 4, 1–19. http://palaeoelectronica.org/2001_1/past/issue1_01.htm.
Haslam, M., 2004. The decomposition of starch grains in soils: implications for archaeological residue analyses. J. Archaeol. Sci. 31, 1715–1734. http://dx.doi.org/10.1016/j.jas.2004.05.006.
Hastorf, C.A., 1990. The effect of the Inka State on Sausa agricultural production and crop consumption. Am. Antiq. 55, 262–290. http://dx.doi.org/10.2307/281647.
Hastorf, C.A., Johannessen, S., 1993. Pre-Hispanic political change and the role of maize in the Central Andes of Peru. Am. Anthropol. 95, 115–138. http://dx.doi.org/10.1525/aa.1993.95.1.02a00060.
Henrickson, E.F., McDonald, M.M.A., 1983. Ceramic form and function: an ethnographic search and an archeological application. Am. Anthropol. 85, 630–643. http://dx.doi.org/10.1525/aa.1983.85.3.02a00070.
Holst, I., Moreno, J.E., Piperno, D.R., 2007. Identification of teosinte, maize, and Tripsacum in Mesoamerica by using pollen, starch grains, and phytoliths. Proc. Natl. Acad. Sci. 104, 17608–17613. http://dx.doi.org/10.1073/pnas.0708736104.
Jennings, J., Bowser, B.J., 2009. Drink, Power, and Society in the Andes. University Press of Florida Gainesville.
Johannessen, S., Hastorf, C.A. (Eds.), 1994. Corn and Culture in the Prehistoric New World. Westview Press, Boulder.
Kolattukudy, P., 1970. Plant waxes. Lipids 5, 259–275.
Lamarque, A.L., Maestri, D.M., Grosso, N.R., Zygadlo, J.A., Guzmán, C.A., 1994. Proximate composition and seed lipid components of some Prosopis (leguminosae) from argentina. J. Sci. Food Agric. 66, 323–326. http://dx.doi.org/10.1002/jsfa.2740660309.
Lantos, I., Giovannetti, M.A., Ratto, N., 2014. Alcances y limitaciones para la identificación arqueológica de gránulos de almidón de razas nativas de Zea mays (Poaceae) del noroeste argentino. Darwiniana nueva ser. 2, 75–95. http://dx.doi.org/10.14522/darwiniana.2014.21.551.
Lema, V.S., Della Negra, C., Bernal, V., 2012. Explotación de recursos vegetales silvestres y domesticados en Neuquén: implicancias del hallazgo de restos de maíz y algarrobo en artefactos de molienda del holoceno tardío. Magallania (Punta Arenas) 40, 229–247. http://dx.doi.org/10.4067/S0718-22442012000100013.
Lia, V.V., Confalonieri, V.A., Ratto, N., Cámara Hernández, J.A., Miante Alzogaray, M.A., Poggio, L., Browns, T.A., 2007. Microsatellite typing of ancient maize: insights into the history of agriculture in southern South America. Proc. R. Soc. Bot. 274, 545–554. http://dx.doi.org/10.1098/rspb.2006.3747.
Logan, A.L., Hastorf, C.A., Pearsall, D.M., 2012. "Let's drink together": early ceremonial use of maize in the Titicaca basin. Lat. Am. Antiq. 23, 235–258. http://dx.doi.org/10.7183/1045-6635.23.3.235.
Maier, M.S., Faria, D.L.A., Boschín, M.T., Parera, S.D., Bernal del Castillo, M.F., 2007. Combined use of vibrational spectroscopy and GC–MS methods in the characterization of archaeological pastes from Patagonia. Vib. Spectrosc. 44, 182–186. http://dx.doi.org/10.1016/j.vibspec.2006.09.003.
Malainey, M.E., Przybylski, R., Sherriff, B.L., 1999. Identifying the former contents of late precontact period pottery vessels from Western Canada using gas chromatography. J. Archaeol. Sci. 26, 425–438. http://dx.doi.org/10.1006/jasc.1998.0344.
Marchegiani, M., Palamarczuk, V., Reynoso, A., 2009. Las urnas negro sobre rojo tardías de Yocavil (Noroeste argentino): reflexiones en torno al estilo. Bol. Mus. Chil. Arte Precolomb. 14, 69–98. http://www.scielo.cl/pdf/bmchap/v14n1/art05.pdf.
Marshall, J.D., Brooks, J.R., Lajtha, K., 2007. Sources of variation in the stable isotopic composition of plants. In: Michener, R., Lajtha, K. (Eds.), Stable Isotopes in Ecology and Environmental Science. Blackwell, Malden, MA, pp. 22–61.
Mazzuca, M., Balzaretti, V.T., 2003. Fatty acids, sterols and other steroids from seeds of Patagonian Prosopis species. J. Sci. Food Agric. 83, 1072–1075. http://dx.doi.org/10.1002/jsfa.1510.
Mickleburgh, H.L., Pagán-Jiménez, J.R., 2012. New insights into the consumption of maize and other food plants in the pre-Columbian Caribbean from starch grains trapped in human dental calculus. J. Archaeol. Sci. 39, 2468–2478. http://dx.doi.org/10.1016/j.jas.2012.02.020.
Moore, J.D., 1989. Pre-hispanic beer in Coastal Peru: technology and social context of prehistoric production. Am. Anthropol. 91, 682–695. http://dx.doi.org/10.1525/aa.1989.91.3.02a00090.
Moore, J.D., 2013. Power and drink. Rev. Anthropol. 42, 3–14. http://dx.doi.org/10.1080/00938157.2013.761931.
National Institute of Health, 2013. ImageJ 1.47, Image Processing and Analysis in Java.
O'Leary, M.H., 1993. Biochemical basis of carbon isotope fractionation. In: Ehleringer, J.R., Hall, A.E., Farquhar, G.D. (Eds.), Stable Isotopes and Plan Carbon-water Relations. Academic Press, San Diego, pp. 19–28.
Oliszewski, N., Olivera, D.E., 2009. Variabilidad racial de macrorrestos arqueológicos de Zea mays (Poaceae) y sus relaciones con el proceso agropastoril en la Puna Meridional Argentina (Antofagasta de la Sierra, Catamarca). Darwiniana 47, 76–91. http://www.ojs.darwin.edu.ar/index.php/darwiniana/article/view/42.
Orgaz, M., 2012. Chicha y aloja. Inkas y autoridades locales en el sector meridional del valle de Yocavil - Catamarca - Argentina. Surandino Monográfico, Prohal Monográfico. segunda ser. 2, 1–38. http://www.filo.uba.ar/contenidos/investigacion/institutos/ravignani/prohal/surandino2/surandino2_articulos.html.
Orgaz, M., Feely, A., Ratto, N., 2007. La cerámica como expresión de los aspectos socio-políticos, económicos y rituales de la ocupación Inka en la puna de Chaschuil y el valle de Fiambalá (dpto. Tinogasta, Catamarca). In: Nielsen, A.E., Rivolta, M.C., Seldes, V., Vázquez, M., Mercolli, P. (Eds.), Procesos Sociales Prehispánicos En El Sur Andino. Editorial Brujas, Córdoba, pp. 239–250.
Orgaz, M., Ratto, N., 2013. Memoria y apropiación en paisajes agrícolas. In: Gordillo, I., Váquer, J.M. (Eds.), La Espacialidad En Arqueología: Enfoques, Métodos Y Aplicaciones. Abya-Yala, Quito, pp. 191–226.
Pearsall, D.M., Chandler-Ezell, K., Zeidler, J.A., 2004. Maize in ancient Ecuador: results of residue analysis of stone tools from the Real Alto site. J. Archaeol. Sci. 31, 423–442. http://dx.doi.org/10.1016/j.jas.2003.09.010.
Perry, L., 2004. Starch analyses reveal the relationship between tool type and function: an example from the Orinoco valley of Venezuela. J. Archaeol. Sci. 31, 1069–1081. http://dx.doi.org/10.1016/j.jas.2004.01.002.
Piperno, D.R., 2009. Identifying crop plants with phytoliths (and starch grains) in Central and South America: a review and an update of the evidence. Quat. Int. 193, 146–159. http://dx.doi.org/10.1016/j.quaint.2007.11.011.
Piperno, D.R., Holst, I., 1998. The presence of starch grains on prehistoric stone tools from the humid Neotropics: indications of early tuber use and agriculture in Panama. J. Archaeol. Sci. 25, 765–776. http://dx.doi.org/10.1006/jasc.1997.0258.
Piperno, D.R., Ranere, A.J., Holst, I., Iriarte, J., Dickau, R., 2009. Starch grain and phytolith evidence for early ninth millennium B.P. maize from the Central Balsas River Valley, Mexico. Proc. Natl. Acad. Sci. 106, 5019–5024. http://dx.doi.org/10.1073/pnas.0812525106.
Quiroga, L., Aschero, C.A., Korstanje, M.A., Vuoto, P., 1999. Los dueños de los montes, aguadas y algarrobales. Contradicciones y conflictos coloniales en torno a los recursos silvestres. Un planteo del problema. In: Los Tres Reinos: Prácticas de Recolección En El Cono Sur de América. Instituto de Arqueología y Museo, Universidad Nacional de Tucumán, Tucumán, pp. 217–226.
Ratto, N., 2013. A modo de introducción: la articulación de estudios arqueológicos, paleoambientales e históricos en el oeste tinogasteño (Catamarca). In: Ratto, N. (Ed.), Delineando Prácticas de La Gente Del Pasado: Los Procesos Socio-Históricos Del Oeste Tinogasteño. Publicaciones de la Sociedad Argentina de Antropología, Buenos Aires, pp. 17–45.
Ratto, N., Boxaidós, R., 2012. Arqueología y Etnohistoria. La construcción de un problema de investigación (Abaucán, Tinogasta, Catamarca). Rev. Mem. Am. 20, 187–220. http://ref.scielo.org/t47xsj.
Ratto, N., Carniglia, D., Coll, L., 2012. Ocupación del área de "Los Seismiles" desde tiempos Formativos a recientes (Dpto. Tinogasta, Catamarca): nuevas evidencias. Relac. la Soc. Argent. Antropol. 37, 207–216. http://www.saantropologia.com.ar/relacionescoleccion/37%201%20-%202012/10%20Ratto.pdf.
Ratto, N., Lema, V.S., López, M.L., 2014. Entierros y orfendas: prácticas mortuorias, agrícolas y culinarias en los siglos XIII y XIV en Tinogasta (Catamarca, Argentina). Darwiniana, nueva ser. 2, 125–143. http://dx.doi.org/10.14522/darwiniana.2014.21.zzz.
Ratto, N., Montero, C., Hongn, F., 2013. Environmental instability in western Tinogasta (Catamarca) during the Mid-Holocene and its relation to the regional cultural development. Quat. Int. 307, 58–65. http://dx.doi.org/10.1016/j.quaint.2012.09.014.
Raviele, M.E., 2011. Experimental assessment of maize phytolith and starch taphonomy in carbonized cooking residues. J. Archaeol. Sci. 38, 2708–2713. http://dx.doi.org/10.1016/j.jas.2011.06.008.
Reber, E.A., Dudd, S.N., van der Merwe, N.J., Evershed, R.P., 2004. Direct detection of maize processing in archaeological pottery through compound-specific stable

isotope analysis of n-dotriacontanol in absorbed organic residues. Antiquity 78, 682–691.
Reber, E.A., Evershed, R.P., 2004a. Identification of maize in absorbed organic residues: a cautionary tale. J. Archaeol. Sci. 31, 399–410. http://dx.doi.org/10.1016/j.jas.2003.09.008.
Reber, E.A., Evershed, R.P., 2004b. How did Mississippians prepare maize? The application of compound-apecific carbon isotope analysis to absorbed pottery residues from several Mississippi Valley sites. Archaeometry 46, 19–33. http://dx.doi.org/10.1111/j.1475-4754.2004.00141.x.
Richter, E.K., Spangenberg, J.E., Klevenhusen, F., Soliva, C.R., Kreuzer, M., Leiber, F., 2012a. Stable Carbon Isotope composition of c9,t11-conjugated linoleic acid in cow's milk as related to dietary fatty acids. Lipids 47, 161–169. http://dx.doi.org/10.1007/s11745-011-3599-0.
Richter, E.K., Spangenberg, J.E., Willems, H., Kreuzer, M., Leiber, F., 2012b. Stable carbon isotope composition of perirenal adipose tissue fatty acids from Engadine sheep grazing either mountain or lowland pasture. J. Anim. Sci. 90, 905–913. http://dx.doi.org/10.2527/jas.2011-4284.
Rodríguez, M.F., Aschero, C.A., 2007. Archaeological evidence of zea mays L. (Poaceae) in the Southern Argentinean Puna (Antofagasta de la Sierra, Catamarca). J. Ethnobiol. 27, 256–271.
Saul, H., Wilson, J., Heron, C.P., Glykou, A., Hartz, S., Craig, O.E., 2012. A systematic approach to the recovery and identification of starches from carbonised deposits on ceramic vessels. J. Archaeol. Sci. 39, 3483–3492. http://dx.doi.org/10.1016/j.jas.2012.05.033.
Schiffer, M.B., 1990. The influence of surface treatment on heating effectiveness of ceramic vessels. J. Archaeol. Sci. 17, 373–381. http://dx.doi.org/10.1016/0305-4403(90)90002-M.
Seinfeld, D., von Nagy, C., Pohl, M.D., 2009. Determining Olmec maize use through bulk stable carbon isotope analysis. J. Archaeol. Sci. 36, 2560–2565. http://dx.doi.org/10.1016/j.jas.2009.07.013.
Skibo, J.M., 1992. Pottery Function, a Use-alteration Perspective. Plenum Press, New York.
Spangenberg, J.E., Jacomet, S., Schibler, J., 2006. Chemical analyses of organic residues in archaeological pottery from Arbon Bleiche 3, Switzerland – evidence for dairying in the late Neolithic. J. Archaeol. Sci. 33, 1–13. http://dx.doi.org/10.1016/j.jas.2005.05.013.
Spangenberg, J.E., Macko, S.E., Hunziker, J., 1998. Characterization of olive oil by carbon isotope analysis of individual fatty acids: implications for authentication. J. Agric. Food Chem. 46, 4179–4184. http://dx.doi.org/10.1021/jf980183x.
Spangenberg, J.E., Ogrinc, N., 2001. Authentication of vegetable oils by bulk and molecular Carbon Isotope Analyses with emphasis on olive oil and pumpkin seed oil. J. Agric. Food Chem. 49, 1534–1540. http://dx.doi.org/10.1021/jf001291y.
Staller, J.E., Tykot, R., Benz, B., 2006. Histories of Maize: Multidisciplinary Approaches to the Prehistory, Linguistics, Biogeography, Domesticacion, and Evolution of Maize. Left Coast Press, Walnut Creek.
Tykot, R.H., Falabella, F., Planella, M.T., Aspillaga, E., Sanhueza, L., Becker, C., 2009. Stable isotopes and archaeology in central Chile: methodological insights and interpretative problems for dietary reconstruction. Int. J. Osteoarchaeol. 19, 156–170. http://dx.doi.org/10.1002/oa.1065.
Vázquez, C., Maier, M.S., Parera, S.D., Yacobaccio, H., Solá, P., 2008. Combining TXRF, FT-IR and GC–MS information for identification of inorganic and organic components in black pigments of rock art from Alero Hornillos 2 (Jujuy, Argentina). Anal. Bioanal. Chem. 391, 1381–1387. http://dx.doi.org/10.1007/s00216-008-2038-4.
Volzone, C., Zagorodny, N., 2014. Mercury intrusion porosimetry (MIP) study of archaeological pottery from Hualfin Valley, Catamarca, Argentina. Appl. Clay Sci. 91–92, 12–15. http://dx.doi.org/10.1016/j.clay.2014.02.002.
Woodbury, S.E., Evershed, R.P., Rossell, J., Griffith, R., Farnell, P., 1995. Detection of vegetable oil adulteration using gas chromatography combustion/isotope ratio mass spectrometry. Anal. Chem. 67, 2685–2690. http://dx.doi.org/10.1021/ac00111a029.
Zarrillo, S., Pearsall, D.M., Raymond, J.S., Tisdale, M.A., Quon, D.J., 2008. Directly dated starch residues document early formative maize (Zea mays L.) in tropical Ecuador. Proc. Natl. Acad. Sci. 105, 5006–5011. http://dx.doi.org/10.1073/pnas.0800894105.