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Preliminary results of a multi-analytical study of ceramic technology in one of the earliest agropastoral villages of Northwestern Argentina (La Ciénega valley, ca. 200 BC- AD 900)

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ABSTRACT

The adoption of productive economies and settled life around the world constituted a pivotal moment in human history, one that reconfigured people's relationships with the environment, non-humans, and material culture, including the invention of ceramic technology. This paper presents a multi-analytical study of ceramic technology among early agricultural societies of Northwestern Argentina (ca. 200 BC-AD 900) obtained through a suite of chemical, physical, and mineralogical techniques. The results from pXRF, XRD, XRF, and SEM-EDS compositional analysis indicate homogeneity in the raw materials used. The results allowed us to observe textural and intended use performance differences among ware groups that expanded our knowledge about ceramic technology in early agricultural societies. We argue that textural and performance differences reflect distinct technologies choices used by ancient potters to enhance pots' performance in everyday food activities. We propose that a combined multiscalar consideration from the naked-eye examination of sherds to the elemental characterization of ceramics allows moving beyond isolated considerations of chemical composition or mineralogical characteristics to a single, integrated view of crafting as fulfilling important roles in everyday household tasks.

1. Introduction

The adoption of food production and settled life generated profound and novel transformations in people's everyday lives, human-environment relationships, and material practices, including the invention of new technologies. Particularly, the development of ceramic technology produced substantial improvements in terms of food preparation techniques, storage capacities, and transport, but also provided a new medium for the transmission and display of social identities, beliefs, and traditions (Potter, 2006). Thus, archaeological research on ceramic technology within farming groups holds a great potential to explore past social, economic, and cultural dynamics during a liminal moment of our history as species: the consolidation of early agricultural life (Bandy and Fox, 2010; Capriles, 2014; Leoni and Acuto, 2008; Hastorf, 2008; Roddick and Hastorf, 2010).

In Northwestern Argentina, the first agropastoral societies started to settle down and use ceramic containers around 200 BC (Formative Period) (González and Regueiro, 1960; Berberian and Nielsen, 1988; Olivera, 2001; Cremonte, 2003; Oliszewski, 2011; Salazar et al., 2021; Scattolin et al., 2009). Pottery studies are a well-established field within early agricultural societies' research in Argentinian Archaeology, perhaps because ceramic technology has been considered a relevant aspect in the overall transition to farming (Tarragó, 1999; Albeck, 2001; Olivera, 2001). These studies have traditionally focused on the stylistic and typological classification of Formative pottery (González, 1955; Berberián and Arguello de Dosh, 1988), although since the eighties, petrography has been a staple technique applied alone or in combination with traditional stylistic and functional analysis (Krapovickas, 1975; Tarragó 1999; Cremonte, 1996; Lazzari et al., 2017; Pereyra Domingorena, 2015). Other archaeometric techniques (i.e. XRD, XRF, SEM-

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EDS) have been scarcely implemented to date (Cremonte, 2003; Giusta, 2017; Lazzari et al., 2017; Franco, 2020; De Feo and Pereyra Domingorena, 2021, among others). Notably, few studies in the study region have integrated different methodologies into a single multi-analytical study for the elemental and mineralogical characterization of archaeological ceramics recovered from specific functional contexts.

This paper presents the analysis of technology and use practices of archaeological ceramics recovered from a food preparation space in one of the earliest agropastoral villages of Northwestern Argentina (NWA) obtained through a suite of chemical, physical, and mineralogical techniques. The results from pXRF, XRD, XRF, and SEM-EDS compositional analysis indicate homogeneity in the raw materials used. Petrographic analysis allowed to observe textural and intended use performance differences among differente wares, expanding the knowledge on early agricultural societies ceramic technology. We discuss that textural and performance differences within the ceramic assemblage reflect distinct technological choices used by ancient potters to enhance pots's performance in everyday food-related activities, with small variation in raw material sources. A multiscalar analysis of archaeological ceramics (from the naked-eye examination to elemental characterization) allowed us moving beyond isolated considerations of chemical and mineralogical composition to a single, integrated view of crafting as the result of technological choices made to enhance the functional performance of ceramic crockery.

1.1. Social aspects of pottery crafting

Research related to ceramic manufacture and functionality encompasses understanding how pottery was made, its performance characteristics, the manufacturing techniques, technological changes in time and space (if observable), and the possible edible resources processed in the containers (Buxeda I Garrigós and Madrid i Fernandez, 2016). Material science applied to ceramics holds great relevance to approaching these issues, yet its potential needs to be enhanced by a broader consideration of the sociocultural processes embedded in potting practices (Schiffer et al., 1994; Sillar and Tite, 2000; Tite, 2008), both in the research-design, the formulation of research questions, and the theoretical tool-kit.

During pottery crafting, potters make different 'choices' in raw materials, tools, and techniques from an universe of possibilities to create the pot (Sillar and Tite, 2000: 4). These technological choices enable researchers to analyze technology as socially and culturally embedded, and moreover woven in daily life practices (Echenique et al., 2021; Duistermaat, 2016; Roux, 2016). Thus, assisted by different sets of analytical techniques, researchers may reconstruct technological choices that offer insights into people's past lives (Echenique et al., 2021; Sillar and Tite, 2000). These choices reflect not only the ecological or economic environment of the potters, but also group' identities (Roux, 2016), local cultural perceptions about appropriate ways of 'crafting' (Sillar and Tite, 2000; Roddick and Hastorf, 2010), and space-landscape understandings (Michelaki et al., 2014; Ingold, 2001). In this paper, we investigate the technological choices embedded in the

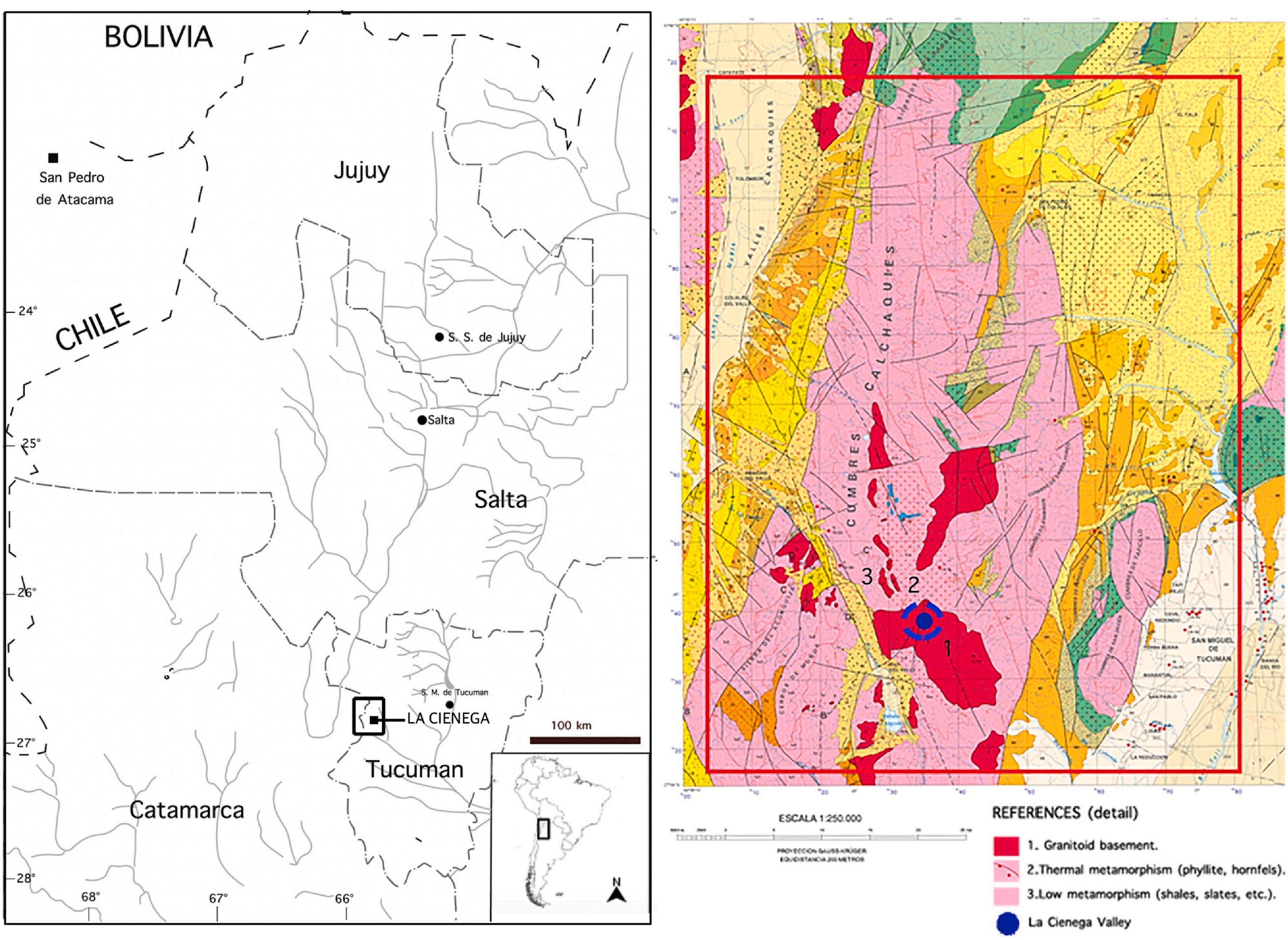

Fig. 1. A) regional map depicting the localization of la ciénega valley and nearby archaeological areas recalled in this article, b) geological map of the cumbres calchaquíes (detail, elaborated in base of the instituto geografico nacional, argentina).

manufacture of ceramic wares used in a specific setting, associated with food-related activities, to understand how technology was an important aspect of early villagers's daily life practices.

1.2. Case study: La Ciénega valley during the Formative period

La Ciénega is an intermontane ravine emplaced in NWA (Fig. 1). It is placed in late Paleozoic formations with granitoids rocks, mainly granites, granodiorites, and tonalites (Cremonte, 1996; López et al., 2019). In some sectors of the valley (e.g., Rio Las Piedras), the outcrops are exposed as large, rounded blocks of granitoids, granites with gneiss xenoliths, feldspathic quartz pegmatites, and schists (Cremonte, 1996; Sampietro Vattuone et al., 2009; Sampietro-Vattuone and Peña-Monné, 2019). These geological resources were used as raw materials for building houses and grinding stones by prehispanic and modern populations (Franco Salvi, 2020; Cremonte, 1996). Additionally, in the riverbanks, there are several clay sources of different colours and plasticity properties, whereas, in the floodplains, there are micaceous sands of fine to very coarse granulometry (Fig. 1b and Fig. 2) (Cremonte, 1996).

La Ciénega was first mentioned by A. Quiroga (1899) at the end of the nineteenth century, but the area was not systematically studied until the eighties of the 20th century, when Dr. B. Cremonte conducted her doctoral research focused on pottery crafting practices in the southern Calchaquí valleys (La Ciénega and Anfama valleys) during the first millennium AD (Cremonte, 1996,2003). Research resumed recently by the Equipo de Arqueología del Sur de las Cumbres Calchaquíes and the Tafí valley indigenous community with the goal of casting light in the long-term historical trajectories and everyday practices of agropastoral groups settled in the region (Salazar et al., 2021).

Prehispanic occupations in the valley sum up 150 household compounds, clustered in three sites scattered in the landscape and closely linked to agropastoral instalations (farmyars, crop fields, clearing fields, and grinding sectors) (Franco Salvi, 2020). Most of them were built and used during the Formative Period (*circa* 200 BC–AD 900) (Franco Salvi, 2020; Cremonte, 2003,1996).

Household compounds are composed of circular or semicircular structures built of local schists, without mortar joints attached to a central semi-public patio (Cremonte, 1996). The central room was often used for quotidian domestic tasks and is the only area connected to the outside and to other rooms (Salazar and Kuijt, 2016; Franco Salvi, 2019). The rest of the enclosures were designated to specific house chores: storage, food processing and consumption, tool making, and resting (Salazar, 2010; Molar, 2015; Franco Salvi et al., 2016). Associated with the residential units and farmyards, there are several grinding spaces located in open-air outcrops, made also with granitic raw materials (Franco Salvi, 2020).

In order to cast light on the nature of human occupations and socio-economic formations in La Ciénega, we conducted the first extended excavation of a complete residential unit in the area, in the *Lomita del Medio-U18* site (Franco Salvi, 2020). The household complex is composed of five rooms: R90 (resting or storage), R91 (multi-purpose), R93 (storage room), R94 (kitchen), and R89 (patio) (Franco Salvi, 2020). Three radiocarbon dates from the R93 and R94 indicate that the house was in use during the first half of the CE: 1486 BP ± 20 (D-AMS 041079), Cal. AD 551–640; 1543 ± 23 BP (D- AMS 041078, cal. 435—592 CE and 1700 ± 26 BP (D-AMS 044974) cal. AD 256–416 (Franco Salvi and Justiniano, 2020). Here we communicate the results of the ceramic analysis from one of these functionally-defined rooms, R94.

2. Materials and methods

In this paper, we consider samples from the R94 room in the *Lomita del Medio* residential unit. This room is the only one with a hearth in the entire household compound and it was interpreted as a processing, cooking, fermenting, and small-scale storage setting (Franco Salvi, 2020), likely a kitchen. The ceramic assemblage from the occupational floor was grouped into pottery types adopted from previous studies in the region considering temper type, texture, colour, surface decoration, and shape (Cremonte, 2003; Cremonte and Bugliani, 2009). Three typological groups were identified: coarse red, coarse grey, and fine grey (Fig. 3).

The entire ceramic assemblage separed by typological groups was classified into family of fragments (FF) (Orton and Hughes, 2013), with a total of 110 groupings. From this classification, we selected a sample of 38 fragments for preliminary screening analysis by pXRF: coarse red wares (27 samples), fine grey wares (9 samples), and coarse grey (2 samples).

As presented in the following section, compositional homogeneity in the sample conduced us to select a sub-sample of 14 fragments to conduct further technological, mineralogical, and molecular studies as a part of a multi-analytical approach. Because of the sample set's compositional homogeneity observed by pXRF and time and money constrains for more detailed and expensive techniques, we considered that a smaller sample would allow us a deeper understanding of technological choices at several scales of analysis (from the elemental to the

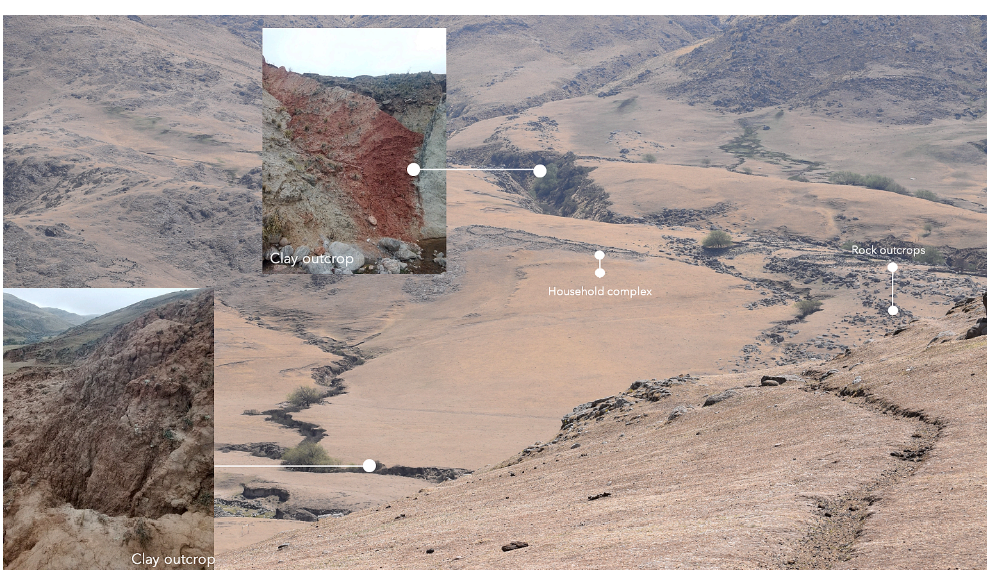

Fig. 2. La Ciénega valley lateral view with details of raw materials source location and residential units.

Fig. 3. Representative pieces of the typological groups analyzed here (images courtesy of J. Salazar and E. Berberián).

mineralogical). In selecting the subsample, we considered samples from each typological group depending on their frequency in the overall ceramic assemblage (for example, coarse red wares are present in higher quanties than coarse grey wares and therefore, we selected more specimens from the former group than from the coarse grey one).

2.1. Sample preparation

Ceramic sherds were initially sampled using a chasing tool and a hammer in order to separate a small fragment for thin-sectioning without contaminating the whole sample with a saw. Afterwards, ceramics were cleaned mechanically using a Dremel 3000 multi-tool with a diamond wheel point of 4.4 mm to remove soil residues and to remove any possible contamination due to handling and/or storage that might influence the chemical analyses. After cleaning, ceramics were ground with a mortar and pestle made of agate, which was cleaned with $CHCl_3$/MeOH (2:1) solution and milli-Q water between samples. The samples were ground until obtaining a fine powder of approximately less than 10 µm. Then, they were sub-sampled from approximately 2 g for chemical–mineralogical characterization. Sample preparation and experimental conditions for the different methods followed established procotols used in the Hercules Laboratory (Évora, Portugal) in similar research.

2.2. Optical microscopy and SEM-EDS

Sherds were brushed to remove any post-depositional sediment and then, cut perpendicular using a saw (Discoplan TS, Struers) achieving a minimum dimension of 3 cm. Then, samples were treated with epoxy resin (Epoxy Fix 25:3 epoxy: hardener ratio, Struers A/S). Samples were subjected to 24 h hardening time for consolidation in the resin. Next, samples were ground with abrasive papers of different roughness until 5 µm (Silicon Carbide Paper, SiC, FEPA P#, Struers). After, the grounded samples were glued to a glass slide (70x50 mm) using Araldite (1:1) and left on a hotplate at 100 °C for 2 h under pressure. Samples dried for 24 h and then, cut off the glass and re-sectioned with the saw (1–2 mm of sampled were left bonded to the glass).

After, samples were ground to 50–100 µm by hand with a sand sheet of P1000 first, and then P800 (Struers). Then, thin sections were polished to 30 µm thickness with a carborundum grit and water (Quinn, 2013). Because the samples were going to be re-used for VP-SEM-EDS analysis as well, there were not coated.

2.3. XRD

For XRD analysis, cleaned and powdered samples were put in glass disks.

2.4. XRF

The powdered samples used for XRD analysis were re-used for XRF. Therefore, powdered sample (1,2 g) was mixed with lithium iodide (12 g). The mixture was fused at 1065 °C and cast into a glass disk by using a Claisse Fluxer® LeNeoTM fusion instrument.

2.5. Experimental conditions

2.5.1. Optical microscopy

Mineral composition and particle size distribution of the thin-sections was performed using a Leica DM2500P polarized microscope. The methodology followed for mineral characterization contemplated the (1) crystal identification and (2) description of shape, size and approximate frequency (Echenique et al., 2018). Inclusions shape (roundness and sphericity) and matrix-temper ratio (approximate temper concentration) was estimated using printed comparative charts (Orton and Hughes, 2013). Similarly, size was recorded as approximate measures of large grain (>0.3 mm); medium (0.1 to 0.2 mm); or small (50 μm) (Echenique et al., 2018). Clay color was also recorded. Approximate amounts of each mineral, rock fragments, and pores were registered as: very abundant (70–100 %), frequent (40–70 %), common (40–10 %), rare (<10 %), and present. Images were captured with a Leica MC 170HD digital camera attached to the microscope.

2.5.2. SEM-EDS

Thin-sections were employed for micro-analysis with a variable pressure electronic microscope coupled with an energy dispersive detector (VP-SEM-EDS). The instrument used was a VP-SEM-EDS HITACHI S-3700 N, operating with an accelerating voltage of 20 kV and chamber pressure of 40 Pa. SEM images were captured in backscattering (BSE) mode in two ranges of magnifications (x100 and x300). The detector used for the chemical analysis was a Bruker XFlash 5010 Silicon Drift Detector (SDD) with a resolution of 126 eV at Mn Kα. Compositional distribution maps and point microanalysis was obtained by EDS elemental data and processed with Espirit 1.9 software.

2.5.3. XRD

Powdered samples placed in glass disks were analyzed in a Bruker D8 Discover X-Ray Diffractometer with Cu Kα source at 40 kV and 40 mA. Diffractograms were collected at a 2θ angular range of 3° – 75° with 0.05° step size and 1 s measuring time. The identification of minerals was conducted on the DIFFRAC.SUITE EVA software using the International Center for Diffraction Data Powder Diffraction File (ICDD PDF-2). The Reference Intensity Ratio (RIR) method (corundum as standard reference) was used for the semi-quantitative determination of mineral abundance (final values presented as a percentage relative to the presumed 100 % matrix of cyrstalline minerals). §.

2.5.4. pXRF

A portable X-Ray Fluorescence spectrometer (Bruker TRACER III-SD) equipped with a Rhodium anode X-ray tube was used for major elemental compositional characterization. Samples were analyzed in triplicates. The chemical data concerning the elements Al, Ca, Cr, Cu, Fe, K, Mn, Ni, Pd, Rb, Rh, S, Si, Sr, Ti, Y and Zr were collected by a silicon drift detector (SDD). All analyses were conducted at 40 kV and 35μA, in vacuum mode, without using a filter in the X-ray path and a 120 s live-time count. S1 pXRF software was used to record the data, and the ARTAX software to evaluate the spectra. Normalization of the chemical data was performed, converting count rates by dividing the Rh Ka peak values.

2.6. XRF

Compositional data was analyzed with a Energy Dispersive X-Ray Spectrometer (EDS-XRF, S2 Puma, Bruker). Quantification was obtained using 25 reference materials. Data was processed with Spectra Elements 2.0 (Bruker).

3. Results

3.1. Macroscopic characterization

Naked eye observation and under a stereomicroscope (20x to 40x) allowed us to get further information about firing conditions and textural attributes. The majority of the coarse red and fine grey wares were fired in oxidizing conditions, achieving earthy terracotta tones. In some cases, sherds in these groups exhibit greyish or darker colours on the surface, probably as a result of a change in the firing atmosphere during firing or because of an incomplete or poorly achieved reducing firing process. Opposite to it, coarse grey ceramics exhibited a fully achieved reducing firing, leading to grey color ceramics.

Drawings of each reconstructed pot were done in the software Adobe Illustrator and the 3D reconstructions and volume estimations in AutoCAD. We estimated each vessel's size and volume based on the projected diameter of the rim and with reference to excavated whole or reconstructable vessels from collections. Reconstruction of shapes allowed us to identify wares used for cooking and service activities, following Balfet and collaborators's (1992) classification (Table 1).

Morphologically, ceramics from coarse red are associated with big cooking and storage pots, with an inferred mouth larger than 20 cm. Estimated volume ranges from 4 to 15 L. In some cases, evidence of fire exposure, like sooted stains, can be observed in the external walls of the potsherds. Opposite to it, samples from fine grey are constituted by service wares such as small bowls (with a mouth larger than 20 cm) and jars, slightly polished on the surface. These small vessels might have had a capacity that varies between 1.5 and 15.0 l. These ceramics do not seem to have any evidence of fire exposure, which could strengthen the interpretation of service wares. Scratching marks were identified on the bottom of the bowls. Ceramics from coarse red and fine grey do not have any surface treatment or slip, and they are mainly utilitarian undecorated pots. Coarse grey, instead, included mostly jars decorated with rolling pins with carved dots or other motifs.

3.2. Initial screening by pXRF

The pXRF was used for an initial analytical assessment of the sample set and to optimize the sample selection for more sensitive and accurate elemental and mineralogical analysis (Hein et al., 2021) considering time and money constrains. Samples displayed high iron and moderate titanium, potassium and calcium content. They also exhibited low rubidium, strontium, zirconium, manganese and chlorine content. Coarse red, fine red, and fine grey groups did not exhibit pronounced differences in their mean concentrations and they clustered in a discrete group. A slight difference was observed in coarse grey samples, which exhibited higher concentrations of iron and low titanium and chlorine compared to the rest of the groups. Biplots plots are provided in Fig. 4.

The homogeneity of results observed by pXRF allowed us to select a sub-sample of 14 specimens from the coarse red (n = 7), fine grey (n = 5), and coarse gray (n = 2) groups. Selection criteria considering the representativity of each typological ground depending on their overall frequency in the ceramic assemblage. Then, ED-XRF and SEM-EDS analyses were performed to test the information collected by pXRF.

3.3. Petrographic analysis and grouping

Optical petrography was conducted on the samples in order to define textural attributes, such as temper size, distribution, and matrix-temper ratio, that could indicate different technological styles and choices in pottery production and or ceramic variability within the assemblage that may not be observed or individualized by chemical methods (XRF, XRD, SEM-EDS).

Samples were clustered in petrographic groups taking into consideration qualitative and semi-quantitative variables of textural analysis and mineralogical identification: 1) type and origin of non-plastic

Table 1
Classification of shapes following Balfet et al. (1992).

Typological group	Open		Closed		Restricted jar	Cooking pot		Non identificated	Total
	Bowl		Bowl						
	Small (≥20 cm)	Big (≤20 cm)	Small	Big		Open	Restricted/with neck		
Coarse red	5	7	3	4	9	16	16	20	80
Fine grey	12		4	1	4		2	1	24
Coarse grey					3			2	5
Other	1								1
TOTAL	18	7	7	5	16	16	18	23	110

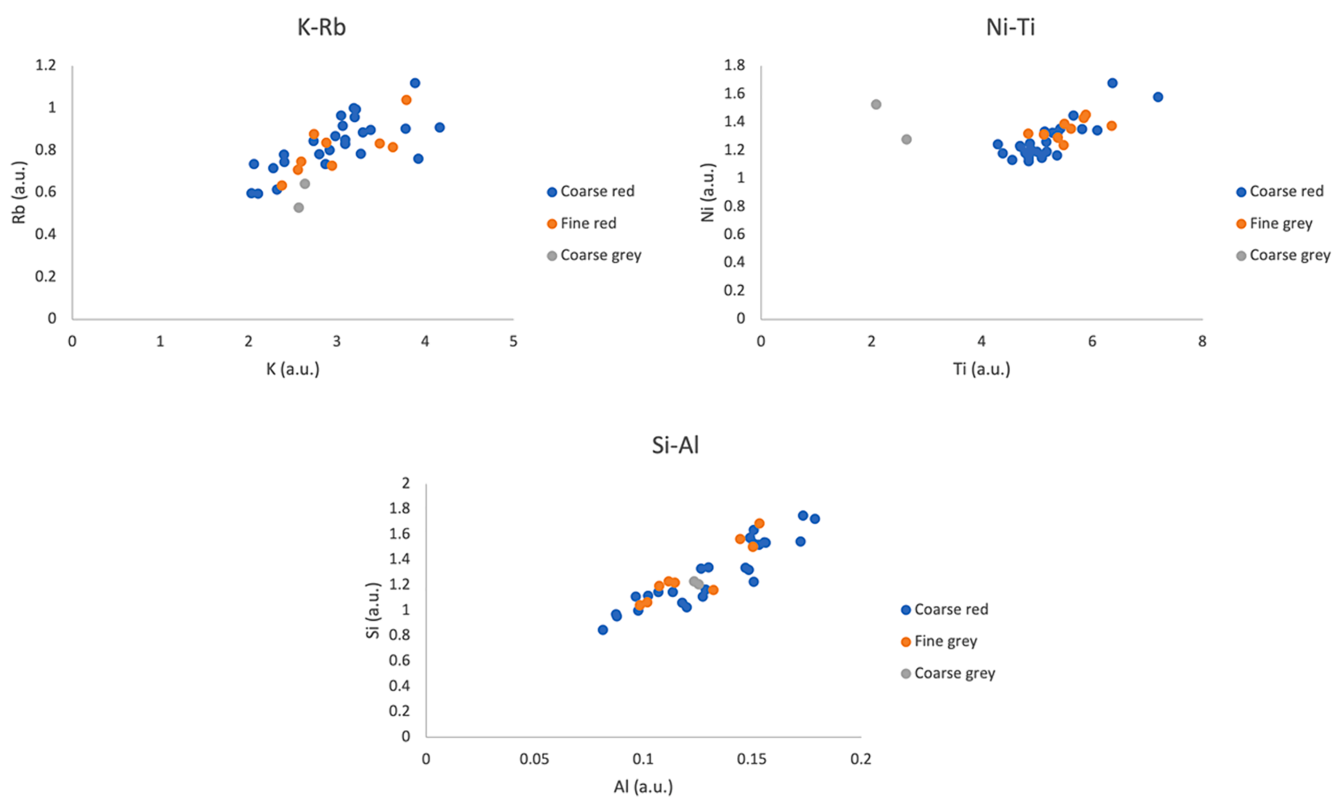

Fig. 4. Pxrf biplots. samples cluster in a discrete and homogenous group, except for coarse grey group samples that are slightly deployed in ti.

inclusions (mineral clasts and lithoclasts) 2) matrix-temper ratio and 3) temper sorting. The petrographic analysis of thin sections allowed us to recognize three main groups: group A (coarse granite), group B (fine), and group C (coarse metamorphic) (Fig. 7), which will be defined and characterized in the following. These groupings match the typological groups defined initially: group A correspond mainly to coarse red wares, group B to fine grey wares, and group C to coarse grey wares.

These groupings align well with petographic data obtained by Cremonte (1996) in ceramics from two residential units in La Ciénega (*El Pedregal* and *Rio Las Piedras*), including 15 samples subjected to optical petrography, NAA, SEM-EDS, and XRD. Cremonte's descriptions of her petrographic groups 1 (*castaño grueso*), 2 (*Anfama gris grueso*), 3 and 4 (*gris* and *rosado finos*) overlap with our classification (group A, C, and B respectively in our petrographic groups), and therefore, we consider these groupings as reflecting very similar technological choices for pottery making within La Ciénega Valley's households. Notably, Cremonte observed a very similar frequency in the typological groups, with a predominance of coarse red and fine wares over coarse grey wares (only 2 fragments of the 15 specimens were classified as coarse grey or *Anfama gris grueso*).

3.3.1. *Group A: Coarse granitic*

Group A is composed of 8 of the 14 samples under study. The temper-matrix ratio ranges between 30 % and 35 %, and temper sorting ranges from poorly to moderately when assessed visually. Inclusions have middle to high sphericity and are subangular to angular. Temper grains are mainly quartz and feldspars crystals. Smaller temper inclusions were identified as biotite, muscovite, plagioclases, K-feldspar, iron oxides, and opaque minerals. The presence of volcanic glass is minoritarian to absent, and in most cases, it was difficult to identify certainly. This group is characterized by the presence of rock inclusions, mainly granite and granitoid lithoclasts, with a scarce presence of metamorphic fragments.

Group A is characterized by heterogeneously thick fabrics with light to darker brownish colour and no surface treatments. Some samples (i.e., LDM_R94_m17) present non-desegregated sediments forming darker-earth spots. The maximum grain size is 0.4 mm in this group, although most inclusions range from 0.1 mm to 0.2 mm. Fig. 7a presents a microphotograph illustrating the textural attributes and temper inclusions of this group.

Two cases (LDM_R94_m15 and LDM_R94_m10, possible storage vessels) have a coarser texture with bigger size inclusions (more than 0.5 mm). A particular case is the sample LDM_R94_m10. It presents poor to very poorly tempered sorting with middle and big angular non-plastic inclusions (between 1 mm and 1.5 mm). The main inclusions are quartz, biotite, muscovite, plagioclases and K-feldspars. Iron, opaque minerals and apatite are present in minor amounts. The sample has a coarse heterogeneous texture with granitoid rocks and metamorphic lithoclasts (slates). The temper-matrix ratio is around 30 %, and it was a light brownish color.

3.3.2. Group B: Fine granitic

Group B is composed of 4 samples. It is characterized by well-sorted temper distribution, contrasting with group A's texture, even when they are likely from the same raw material (see group C). Additionally, samples of group B have abundant inclusions and a temper-matrix ratio between 40 % and 50 %, as can be seen in Fig. 7b. Samples in the group are characterized by homogeneously distributed thin fabrics with light greyish to dark brown colour, and generally, they correspond to reduced firing vessels.

The temper roundness ranges from subangular to angular inclusions, with middle to high sphericity. Inclusions in this group have a maximum grain size of 50 µm. Quartz and feldspars are the major mineral inclusions in the group, with minor quantities of biotite. The small size of the grains made it difficult to distinguish, more certainly, K-feldspar and plagioclases. Muscovite and iron oxides are absent. Also, there is a total absence of lithoclasts in the group.

3.3.3. Group C: Coarse metamorphic

Group C includes 2 samples. It is characterized by poorly sorted temper distributions and subangular to angular non-plastic inclusions. The maximum grain size of inclusions is 2.5 mm. The temper-matrix ratio is around 30 %. Temper grains and rock inclusions are metamorphic lithoclasts (slates and schists). Fig. 7c presents the microphotographs of sample LDM_R94_m13 where the textural features and main mineral constituents can be observed. Biotite and feldspar are present but in minor amounts. The group is characterized by very heterogeneously distributed thick fabrics with a dark greyish or brownish colour.

3.4. Mineralogical composition

Point analysis performed by SEM-EDS allowed mineralogical identification of temper grains, corresponding to quartz, feldspars, iron oxides, titanium oxides, and monazite. The XRD data suggested similar mineralogical composition across the samples (Fig. 5). The main mineral phases encountered by XRD are quartz (SiO_2), K-feldspars ($KAlSi_3O_8$) and plagioclases ($NaAlSi_3O_8$ - $CaAl_2Si_2O_8$), and amphiboles $(Na,K)(Ca,Na)_2(Mg, Fe, Al)_5(Al, Si)_8O_{22}(OH, F)_2$. Representative diffractograms of each group can be seen in Fig. 3. Semi-quantitative determination by reference intensity ratio (RIR) of the bulk composition of the samples also demonstrated small variation within the sample set and thereby, compositional groups were not defined. Quartz is encountered across all samples, between 30 and 50 %. In B cases, the feldspar values slightly increase when quartz diminishes and the other way around.

Temper inclusions were identified preliminarily by petrography and then confirmed by point microanalysis in SEM-EDS. Samples from A and B groups display K-Feldspars (microcline). Coarse red exhibits Na-plagioclases (albite, andesine) and Ca plagioclases (e.g. anorthite). Whereas Na-plagioclases in B are mainly oligoclase/andesine and albite. Coarse grey samples exhibit poorly defined feldspars grains, mainly albite, anorthite, and sanidine. Overall, the more abundant feldspar is oligoclase, anorthite, albite and sanidine. It was not possible to identify amphiboles by the point microanalysis.

3.5. Elemental characterization

The chemical data obtained by ED-XRF was used to establish the elemental composition of samples and to define elemental patterns (major and minor elemental compositions) regarding the typological groups across the assemblage and SEM-EDS was applied with the aim of illustrating the elemental distribution of samples. The samples display a homogeneous pattern of silica, aluminium, potassium, iron, calcium, sodium, and titanium across all samples in ED-XRF and SEM-EDS. The determination of minor elements in the samples by ED-XRF did not allow to establish compositional groups or sharp inter-group differences, maintaining the homogeneity observed in pXRF. Nevertheless, group B samples exhibited higher Sr and Zn values but lower Zr amounts (Fig. 6a).

Through ED-XRF, the major elemental composition expressed in oxides allowed us to observe the predominance of silicon and aluminium as the two main oxides (not presented in Fig. 6b). The silicon oxide is always predominant without any significant deviation. The silicon and the aluminium oxides are linked to the aluminosilicate fraction of the clay material. Calcium oxide maintains similarly low levels across all groups, with the exception of one group C sample that is slightly enriched (LDM_R94_m27) (Fig. 6b). Group C samples exhibited slightly higher values of phosphorus oxide, whereas the remaining groups' values range from 2,20 % to 3,70 %. Sodium oxide is present in small amounts ($<3\%$), and there does not exist any relation between typological, although group B values cluster tightly when compared with coarse groups (A and C).

4. Discussion

The petrographic analysis allowed us to differentiate three groups of ceramics based on mineralogical and textural differences between samples: group A (coarse granitic), group B (fine granitic), and group C (metamorphic). In most cases, these petrographic groups fit well with the typological groups previously established: coarse red (A, coarse granitic), fine grey (B, fine granitic), and coarse grey (C, metamorphic), with a few exceptions (fine grey wares classified as coarse red due to the size of temper inclusions in the petrographic analysis).

The predominance of granitic inclusions in groups A and B point to a local origin of these wares probably made with clays and sands obtained from the riverbank or clay deposits nearby, although they were differentially sorted and ground to achieve different textural attributes. Indeed, La Ciénega Valley is placed on a granitic-granitoid region with abundant raw material sources readily available for pottery production (Cremonte, 1996). Group C wares were made of metamorphic materials that are not found locally, and therefore they are interpreted as non-local materials, although their minoritary number within the assemblage seclude stronger conclusions.

The elemental and mineralogical analysis of samples by XRF, XRD, and SEM-EDS did not show appreciable compositional differences between groups, which suggest the homogenous use of the same raw materials within each typology. Furthermore, there is not an appreciable difference in the feldspars's composition between both groups identified by SEM-EDS, composed mainly by K-Feldspars and Na-rich feldspars such as oligoclase, andesine and albite, compatible with Cremonte's previous observations (Cremonte, 1996,2003). A slight divergence in this pattern is the presence of anorthoclase inclusions in group A samples, which are not present in other groups. Although we did not analyze clay and sand samples from La Ciénega in this study, Cremonte (1996, 2003) conducted XRD studies on reference materials locally sourced. These displayed similar mineralogy to archaeological ceramics: quartz, K-feldspar (orthoclase and microcline), oligoclase, biotite, muscovite, volcanic glass, hornblende, diopside, and granitic rocks (Cremonte, 2003,1996), all of which sustain our hypothesis of the local origin of La Ciénega's ceramics.

Indeed, it is important to recall that our conclusions align well with a previous study in other households complexes in La Ciénega Valley and adjacent Anfama and Tafí Valleys (Cremonte, 1996,2003). To consider samples from published data helps to elucidate the extent to which potting practices were developed within La Ciénega's village and between households. Considering Cremonte's previous results, we confirm a strong emphasis on the local production of utilitarian wares used for

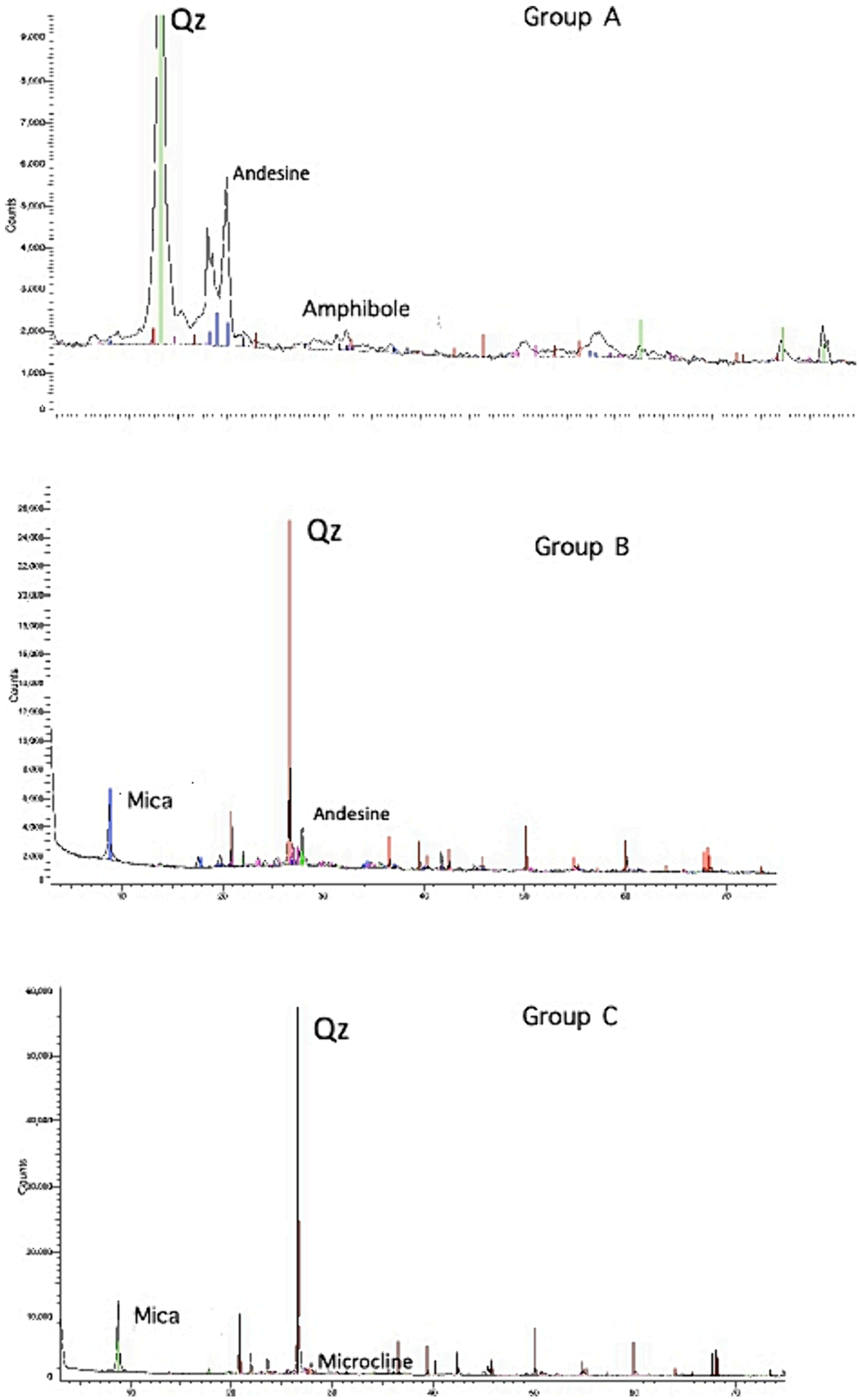

Fig. 5. XRD diffractograms from each petrographic group.

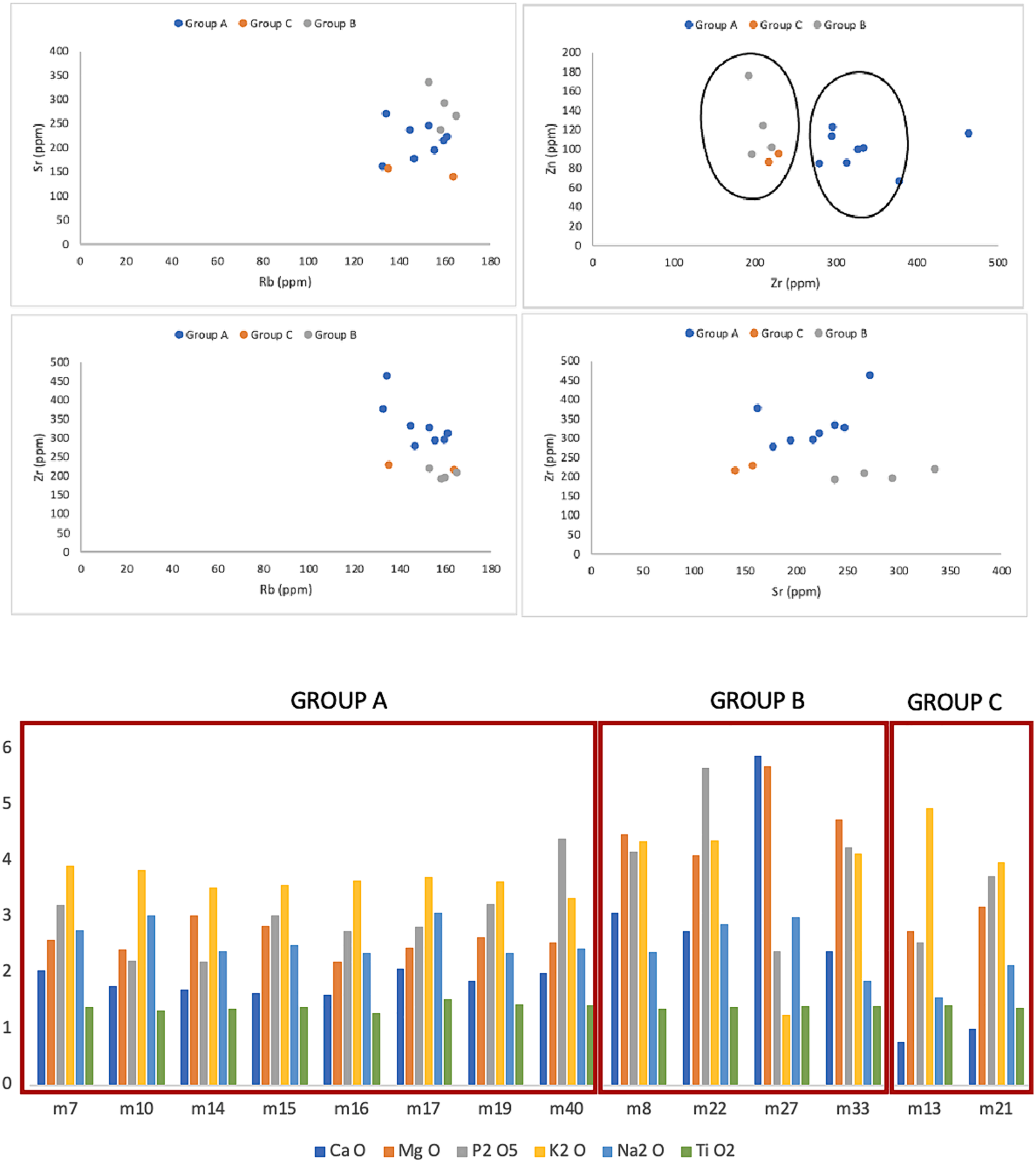

Fig. 6. ED-XRF data a) Trace elements biplots showing the lack of variation between groups. It can be observed, however that group C samples are enriched in Zr when compared with group A and B samples b) Oxide concentration (excluding those related to the Al-Si fraction of the clay material). It can be observed that group C specimens are slightly enriched in P oxides.

everyday cooking and serving activities (groups A and B). In Cremonte's classification, the coarse red and greyish group is formed by fragments with quartz, feldspars, biotite, muscovite, and granitic lithoclasts as the main mineral inclusions (Cremonte, 2003), a description that matches the results presented here. In some cases, the micas can be more exposed to the surface, giving it a shinier look, but this is not related to a higher amount of mineral temper but due to a better smoothing of the pottery wall (Cremonte, 2003). Opposite to it, the *gris fino alisado* (fine) group did not exhibit temper additions, and it is characterized by a thicker clayey matrix (Cremonte, 2003), corresponding to group B in our classification.

The technological differences in similar chemical and mineralogical composition (as observed by physico-chemical techniques such as pXRF, ED-XRF, XRD, and SEM-EDS) arise when considering paste preparation and intended function. In the analyzed assemblage, the main distinction between groups A and B lies in their textural characteristics. There is a

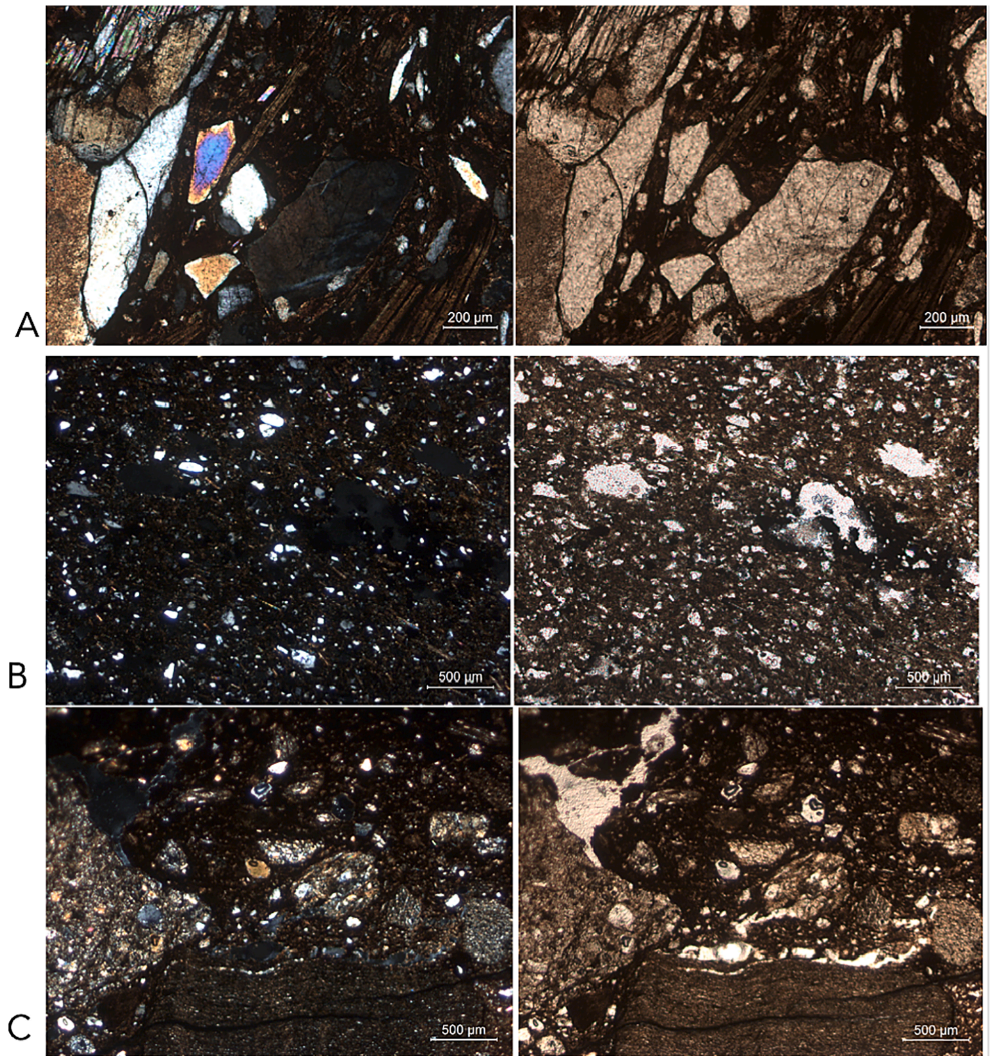

Fig. 7. Petrography microphotographies a) group A b) group B and c) group C.

notable difference in the matrix/temper ratio and inclusion sizes, although the chemical and mineralogical composition remains the same. We suggest that the correlation between typological-petrographic groups and intended function (inferred by morphological characteristics) indicates a relation between technological choices and practical reasons, as observed in other works (Sillar and Tite, 2000; Tite, 2001; Skibo, 2013). The evidence presented here suggest that technological choices observed in petrography is closely linked to the intented function of the pots.

Indeed, cooking vessels should be heat-resistant, thick and porous to achieve thermal shock resistance (Rice, 1987; Bowen and Harry, 2019; Eramo, 2020). The technological characteristics of group A vessels allow us to identify them as cooking pots used for food processing. These vessels have big dimensions and capacity, open-mouth and constricted neck shapes and smoothed walls. Similarly, group C ceramics are vessels with coarser pastes, nevertheless, it is hard to ascribe them to cooking or hot processing functions. They could have been used as containers for already prepared foods on special occasions, although their use as non-utilitarian vessels cannot be excluded. Group B samples correspond to service vessels. Morphologically, group B was classified as open bowls and constricted neck jars. Samples are thin-walled, tempered and with smoothed or slightly polished surfaces. The predominance of small bowls could indicate that they were implicated in small-scale everyday gathering activities of the household group.

The above indicate a strong correlation between pottery production and food processing activities conducted at a specific setting within the household, indicating how both dimensions, production and consumption, were intimately bounded (Mills 2016). The evidence analyzed might suggest that the intended uses and everyday activities in which pots were going to participate were determinant aspects of the technological choices made by potters, emphasizing suitability and performance in the selection of raw materials and pot making.

5. Conclusions

Chemical, mineralogical and petrographic analyses provide complementary information. By petrography, we were able to identify three groups, two associated with the local geology of La Ciénega (granitic inclusions) and the other one (metamorphic inclusions), most possibly a non-local production. Nonetheless, it was not possible to maintain this differentiation in further elemental and mineralogical analysis where data inhibited the grouping of samples. Petrography provided important information about possible provenance and related to technological and intended use features of pottery.

The homogeneity within each typological group in our sample-set and the published data suggest the presence of long-term ways of doing utilitarian ceramics for food processing and preparation. We finally suggest that the repetition of crafting practices through the use of the same recipes and the exploitation of the same raw material sources might be explained by potters' interest in enhancing the functional and performance characteristics of ceramic vessels used for food preparation and consumption. More research in the future expanding the dataset could provide important insights to understanding the role of long-standing crafting traditions in the conformation of early agricultural societies.

Authors' contributions.

A. Wrote the main manuscript text, conducted the experiments, did the data acquisition/interpretation, prepared figures and tables.

B. Conducted ED-XRF experiments and oversaw SED-EDS data acquisition.

C. Conducted archaeological fieldwork, collaborated in sample selection and macroscopic analyses.

D. Oversaw the experiments, reviewed data acquisition and interpretation.

E. Oversaw pXRF experiments.

All authors reviewed the manuscript.

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Availability of data and materials.

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CRediT authorship contribution statement

Agustina Vazquez Fiorani: Conceptualization, Data curation, Formal analysis, Funding acquisition, Investigation, Methodology, Resources, Software, Visualization, Writing – original draft, Writing – review & editing. **Anna Tsoupra:** Investigation, Methodology, Writing – review & editing. **Julian Salazar:** Funding acquisition, Investigation, Project administration, Resources, Supervision, Writing – review & editing. **José Mirão:** Supervision, Writing – review & editing. **Massimo Beltrame:** Supervision.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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