A Systematic Evaluation of the Firing Temperatures of Archaeological Pottery from Catamarca, Argentina

Nora Inés Zagorodny, Cristina Volzone, Martín Morosi, and Bárbara Balesta

Introduction

Pottery firing systems in archaeological are usually addressed through different perspectives which include: macroscopic analysis (García Rosselló and Calvo Frías 2013; Feely 2010), ethnoarchaeology (García Rosselló 2009 and 2013; González Urquijo *et al.*, 2001; Vazquez Varela 2003), archaeometry (Zuluaga *et al.*, 2012), and experimental studies (Palamarczuk 2004).

Rye points out that: "The main aim of firing is to subject vessels to sufficient heat for a long enough time to ensure complete destruction of the clay-mineral crystals. The minimum temperature varies between about 500° and 700°C, depending on the type of clay. When heated above these temperatures, clays take on the characteristic properties of pottery: hardness, porosity, and stability under a wide range of chemical and physical conditions. The principal variables controlled by the potter during firing are the rate of heating, the maximum temperature, and the atmosphere surrounding the objects." (Rye 1981, 25)

There are two principal methods of firing pottery: open pits and kilns (Kingery 1997; Rice 1987; Rye 1981). The sample analyzed in this paper belongs to a *tinaja* (*sensu* Balfet *et al.*, 1983) fired usingthe first of these methods. Currently evidence of the kiln structures has not been found at sites in the Hualfín Valley (Belén Department, Catamarca, Argentina).

Open firing – according to available bibliography - reaches lower maximum temperatures with a faster heating rate. Kiln structures maintain these maximum temperatures for longer periods and require less fuel. Authors like Gosselain (1992) and Livingstone-Smith (2001) maintain that cooking procedures cannot be elucidated from determination of cooking temperatures on a vessel.

According to Kingery (1997), open-firing systems and simple kilns reach similar temperatures; the difference between them resides in the length of the process and temperature uniformity. Kilns built with permanent walls retain heat more efficiently and can reach temperatures approximately 100°C above those of open pits.

Several analytical methods can be applied for estimating firing temperature of pottery, including the color obtained by a second firing of ceramics, porosity, Mössbauer spectrometry, differential thermal analysis, and X-ray diffraction (XRD) (Brown 1980; Capel *et al.*, 1979; Guezzi 2011; López Milla and Contrerasa 2008; Mackenzie and Caillere 1979; Maggetti 1982; Maggetti *et al.*, 2010; Mosquera Díaz *et al.*, 2001; Newman 1987; Ortega *et al.*, 2005; Peña-Loza 2011; Rasmussen *et al.*, 2012; Shimada *et al.*, 2003a; Shimada *et al.*, 2003b; Tite 1969; Wagner *et al.*, 2000; Wagner and Kyek 2004) among many others.

Pottery porosity can be measured through different techniques such as water absorption, nitrogen adsorption, mercury intrusion porosimetry (MIP). The water adsorption method determines pore volume but not pore size distribution as does MIP method. A further advantage of MIP with respect to water absorption is that when a sample contains clay minerals, mercury does not expand as might occur with water, which then provides erroneous results. On the other hand, nitrogen adsorption can be used, although one must take into account that it defines micro to mesopores but not large macropores. Choosing one of these techniques depends on research objectives such as the type of material under study and the range of pore sizes to be determined. The IUPAC (International Union of Pure Applied Chemistry) types pore sizes as micro, meso, and macro according to diameter size: < 20Å; between 20–500Å, and >500Å, respectively (IUPAC, 1985).

X-ray diffraction, mercury intrusion porosimetry, and fired color analyses were combined here to propose an approximate firing temperature for a sample vessel fragment from Catamarca.

A Characterization of Belén Culture

Hualfín Valley is located in the center of Catamarca Province (Argentina); it comprises an area crossed by Hualfín River and tributaries. This valley was populated by different indigenous groups at least during the last two thousand years up to the arrival of Spanish conquest in the 16th century.

Belén culture refers to the prehistoric inhabitants of Hualfín Valley in the Argentinean Northwest. This culture has been dated to the Regional Developments Period which took place between AD 1000 and 1480 (González and Cowgill 1975).

Hualfín and Abaucán Valleys constituted the core of Belén populations; moreover, Belén sites have been identified in Antofagasta de la Sierra - in the Catamarca Province Puna - and in association with Santa María populations in the Yocavil Valley. Scattered findings from this group were also registered in La Rioja Province, southern Salta Province, and in the Tafí Valley in the province of Tucumán.

Improvement of agricultural technology - through irrigation systems and the intensive use of natural resources – has been documented for this period. The main agricultural products were maize, squash, and beans. Belén populations practiced shepherding - which comprised camelid breeding, specifically of llamas - and hunting, mainly of wild camelids as guanaco and vicuña, and birds and rodents. This improvement in technological capacities and subsistence implied considerable demographic increases and concentration.

Belén groups received their name from vessels found at most sites called "*tinajas Belén*" (Figure 1). These vessels are tripartite shaped and painted Black on Red, reaching volumes of up to 20 liters. They show naturalistic - mainly snakes- and geometric decoration. Small hemisperical bowls, called *pucos*, are also found at these sites. They are smaller, decorated with the same colors, and with geometric and naturalistsic motifs – these latter are principally representations of armadillos. Some *tinajas* were used as domestic containers; others were deposited as offerings in tombs. Tombs were placed under big rocks or built of stone walls – these are known as *cistas*; infants were also buried in urns.

González and Cowgill (1975) proposed three different phases of Belén chronology. These phases were established on architectural and pattern characteristics:

Belén I (AD 1100-1300) was defined at the site Corral de Ramas, and was characterized by these authors by the presence of communal pit-houses made of mud brick walls.

Belén II (AD 1300-1480) was characterized at Cerrito Colorado (in the locality of La Ciénaga de Arriba). This phase was defined by defensive sites built with scattered stone-walled rooms.

Belén III (AD 1480-1535) represents a period of Inka influence on Belén culture. Characteristic populations during this phase were big villages with agglutinated rooms.

Based on site characteristics and radiocarbon dating, Belén phases have recently been questioned. New investigations (Balesta *et al.* 2011) have challenged this cultural evolutionary outline. At present, sites are thought as having different characteristics - and probably distinct hierarchies - based on their functionalities.

These phases were marked by political fragmentation into small regional polities; evidence of violence and conflict has been identified at several sites. Some were located in high places surrounded by walls and are called *pucaras* locally.

Aside from defensive sites located on the top of hills, new research (Wynveldt *et al.*, 2013) has identified other placement modalities such as: hilltop and ridgetop sites, sites on plateaus and mountain slopes, foothill sites, and sites on river terraces.

Belén Pottery

Studies of Belén pottery have addressed provenience analysis, production, composition, microstructural studies, morphometrics, decoration, use, and function (Balesta *et al.*, 2013; Basile 2009; Iucci 2009, 2010, 2013; Iucci *et al.* 2010; Manase and Páez 2006; Puente and Quiroga 2007; Volzone and Zagorodny 2014; Wynveldt 2007a, 2007b; 2008; Zagorodny *et al.*, 2010a, 2010b, 2013). Firing technology remains to be explored.

Belén pottery presents compact pastes, through firing, and black paint on red slips. There are three main morphologies: jars (*tinajas* or *urnas*), bowls (*pucos*), and pots (*ollas*; Figure 1).

Snakes constitute the most recurring motif. Ambrosetti (1896) pointed out that in the 20th century local inhabitants still believed that these reptiles were associated with lightning and rain; they were also considered as guardians of burials. These beliefs are consistent with the bonds, described in Calchaquí mythology, between snakes and lightning, and help to explain how and why they were depicted on urns.

Porosimetry studies on Belén *tinajas* were carried out using mercury intrusion and results show that porosity indexes oscillate around 40%. Pore size

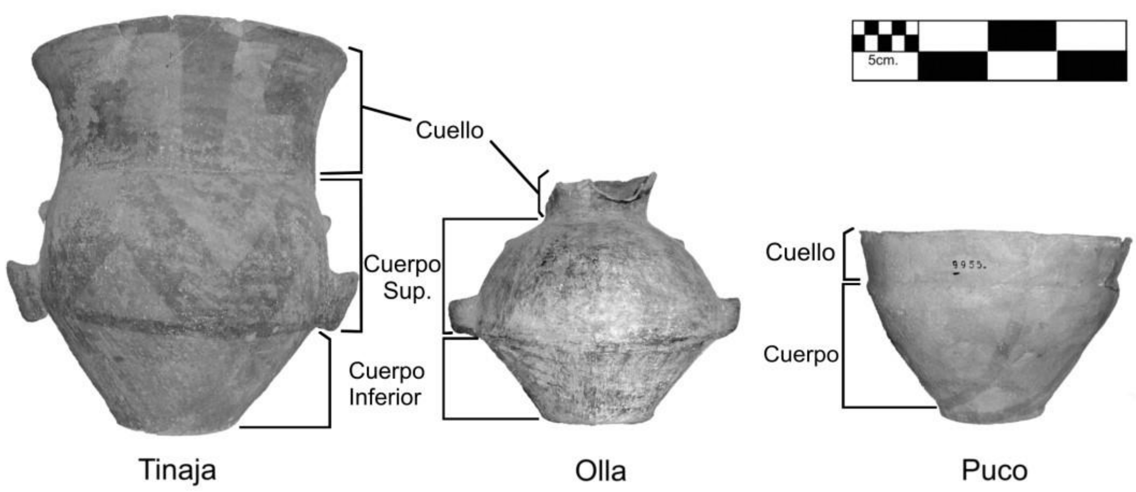

Figure 1. Morphologic categories in Belén pottery (Wynveldt, 2007a).

can be classified as macropore (IUPAC 1985; Zagorodny *et al.* 2010). This characteristic and the thinness of their walls confirms *tinajas* are light vessels and suitable to store water in domestic contexts.

Tinajas found in domestic areas and those deposited in funerary contexts show striking differences regarding their sizes. Morphometric analyses carried out on domestic *tinajas* confirm they are bigger and their profiles are more continuous in comparison with funerary specimens.

Ethnoarchaelogical observation was carried in La Ciénaga in the Hualfín Valley during some working days with Mrs. Antonia Sarapura, a local potter who manufactures archaeological replicas. Mrs. Sarapura collects clay in the riverbank near her house. After modeling and drying the vessels she digs a vertical hole in a low gully in order to protect the fire from the wind; next she puts the vessels alternating with dung and places a layer of dung on top (Figure 2). She then starts a fire and leaves the pots until the fire extinguishers - usually an entire night.

When the pots are cold she clears away the ashes and collects the objects. It is interesting to note that the pit does not leave traces, except for small effemeral short-lived ash accumulations. These kinds of activities in a dynamic environment such as the Hualfín Valley means that is very unlikely we will recover archaeological evidence of ceramic firing.

Figure 2. Antonia Sarapura placing ceramic pieces in a pit for firing, La Ciénaga de Abajo, Belén, Catamarca. Photo taken by our group in 2012.

Materials and Methods

The ancient pottery fragment was collected from archeological site of Cerro Colorado de la Ciénaga de Abajo located in Hualfin Valley, assigned to the Belén Culture during Regional Developments and Inka Periods or Late Period (AD 1000-1480) (Balesta and Zagorodny 2010; Sempé 1999). The site corresponds to a 150m fortified village with more than 100 rooms in different sectors, a large number of funerary structures, and walls arranged at different elevations (Wynveldt and López Mateo 2010).

The sample was recovered from a stone-lined tomb – called a *cista* by locals - which had been previously looted. Only the remains of broken

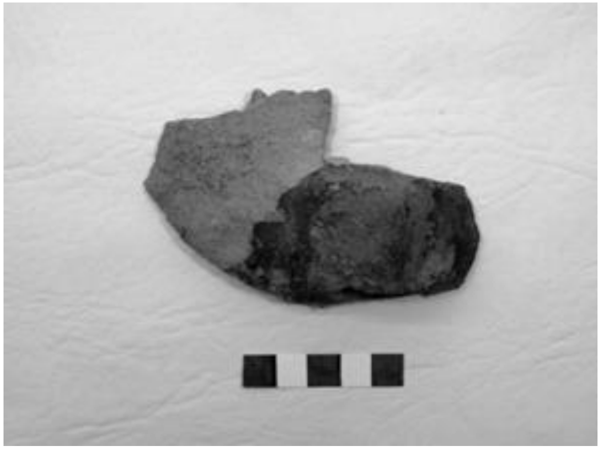

Figure 3. **A**nalyzed sample. Body vessel fragment which retains the handle.

pottery were found inside along with a few human bones, testimony of what had once been a burial.

The fragment corresponds to body fragment which retains the handle; its decoration was painted in Black on Red and came from a vessel fired in an open, oxidating environment (Figure 3). Its morphology corresponds to the jar type (Balfet *et al.* 1992).

The sample fragment was divided into segments. The resulting pieces were thermally treated to different temperatures ranging from 550°C to 1000°C. Each segment was examined were evaluated for color changes through direct visual comparison with the Munsell soil color chart (1954). Subsequently, segments were analyzed using X-ray diffraction and mercury intrusion porosimetry (Jozefaciuk *et al.* 2009; Morariu *et al.* 1977; Sanders 1973; Volzone and Hipedinger 1997; Volzone 2010; Volzone and Zagorodny 2014; Wolf 2002). The obtained results demonstrate changes in physical properties such as color and texture (pore size), and compositional properties (phase changes). Refiring permitted us to propose an approximate temperature for the original firing event.

Fragment segments were also submmitted to thin-section petrographic analyses. Petrographic results were published elsewhere (Zagorodny *et al.* 2010a; Zagorodny *et al.* 2010b) but in general terms, they indicate that plutonic rocks are dominant (70-76%), followed by volcanic inclusions (11-26%), while metamorphic inclusions are only present at low percentages (15%) or are absent, and vitroclastic inclusions range between 8 and the 27.5% (Figure 4).

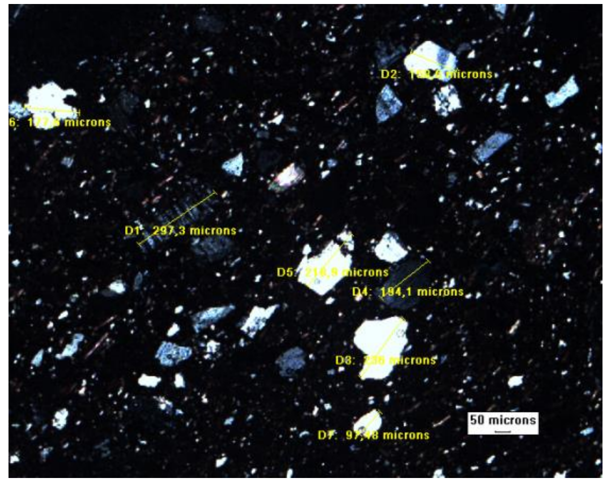

Figure 4. Photomicrograph of Tinaja Belén fragment (LAC 7.2). Cerro Colorado de La Ciénaga de Abajo, Belén, Catamarca.

In order to evaluate the original firing temperature of the ceramic fragment CCA2, segments measuring 0.5 x 0.5 x 1.0cm were refired to different temperatures - 550, 600, 700, 750, 800, 900, 1000 and 1100°C - for one hour in an oven with silicon carbide heating elements. The segments were labelled: CCA2-550, CCA2-600, CCA2-700, CCA2-750, CCA2-800, CCA2-900, CCA2-1000, and CCA2-1100 respectively.

Crystalline phases were identified by XRD using a Philips 3020 goniometer (Ni filtered, Cu Kα radiation). Step scan data were collected from 3-55° (2θ).

Mercury intrusion tests were performed by using a 2000 Carlo Erba Porosimeter with pressures ranging from 1 to 2000kg/cm2. Intrusion volumes were measured stepwise, increasing pressures and allowing equilibration at each pressure step.

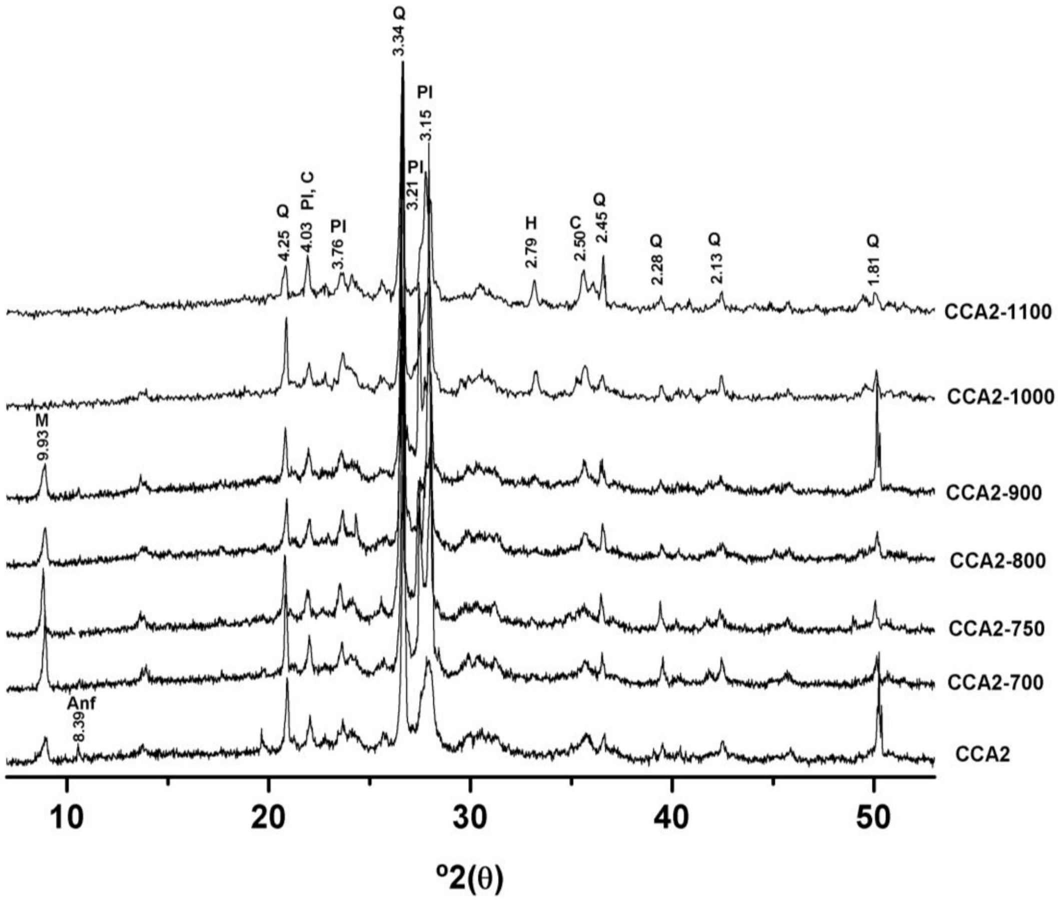

Figure 5. XRD Spectra. C = cristobalite, H = hematite, M = muscovite, Pl = plagioclase, Q = quartz.

Pores radii were measured within a range of 37 to 75,000Å according to definitions determined by the International Union of Pure and Applied Chemistry (IUPAC 1985). The volume of mercury V (mm3/g) intruded at a given pressure P (kg/cm2) provides a measure of pore volume. Intrusion pressure was translated to an equivalent pore radius (rp) in Å following the Washburn (1921) equation: $rp = -0.2\ \gamma\ \cos\alpha/P$; where γ is the mercury surface tension (485dynes cm-1) and α is the mercury/solid contact angle (taken as 141.3° for all studied minerals). In these conditions, the equipment determines pore radii from 37 to 75,000Å (0.0037-7.5µm) or pore diameter from 74 to 150,000Å (0.0074-15µm).

Results

Characterization by X-Ray Diffraction

Figure 5 provides XRD spectra of the sample fragment before and after refiring to 1100°C. Figure 6 lists the mineralogical phases and compositional changes to each segment after thermal treatments from 700°C to 1100°C. The crystalline phases of the unrefired CCA2 fragment - which had not been subjected to heating treatment - are quartz (SiO_2), plagioclase ($(Na,Ca)(Si,Al)_4O_8$), muscovite ($KAl_2(AlSi_3O)_{10}(OH)_2$), amphibole ($(NaCa_2(Mg,Fe,Al)_3Si,Al)_8O_{22}(OH)_2$) and cristobalite ($SiO_2$). The XRD of CCA2-550 and CCA2-600 were not included in Figure 5 because their spectra were similar to CCA2. The same occurred to all segments heated up to 700°C. In sum, the mineralogical composition of CCA2 did not change from 550 to 700°C.

A slight increase in cristobalite and a reduction in amphibole were observed from heating to 750°C and 800°C. Compositional changes became more apparent at 900°C, where amphibole disappeared, hematite (Fe2O3) appeared, and plagioclase increased. At 1000 °C, muscovite began to disappear, and at 1100°C, a significant structural change was observed involving the reduction of quartz and increasing cristobalite. Consequently, the main changes were observed and 750°C and 1000°C. The changes observed by XRD suggest that the original jar was fired at around 700°C, no higher than 750°C.

Color Firing

The most important changes to segment colors were coincident with after each refiring, as shown in Figure 6. CCA2 and CCA2-700 had color 7.5YR, while segements refired from 750°C to 1000°C, presented color values of 5.5YR, and refiring a 1100°C caused a significant color change to 10R 3/4. The difference among pieces heated to between 750 and 1000°C are degrees of intensity in the Munsell color chart (6/6, 6/8, 5/8; 1954). Changes in color are related to the appearance or disappearance of certain compositional elements; these changes were observed at different phases as the product of increasing temperatures (e.g., the presence of iron; Mirti and Davit 2004).

Textural Characterization by Mercury Intrusion

Cumulative pore volumes as a function of pore radii are shown in Figure 7 which confirms the high contribution of a narrow range of pore sizes. This behavior has also been observed in other fine vessels (Zagorodny *et al.* 2010b). The curve of the CCA2 sample (corresponding to the segment without refiring) established a distribution similar to all other segments up to 1000°C. An important contribution (65%) to pore volume of pores with radii of well-dimensioned size (between 4000-5000Å) is clear. The total pore volume of all segments is found between 250 and 280 mm3/g.

An important change was observed with the 1100°C refiring event. The pore volume reduced almost by one-third, indicating an almost total collapse of pores in the sample. The key area of the cumulative curve was expanded in the bottom half of Figure 7 in order to evaluate changes to pore size in each pieces subjected to thermal treatment. This allowed us to pinpoint differences among the samples.

The original sample (CCA2) curve is coincident with that of the segment refired to 700°C (CCA2-700); both show an average pore size of 4300Å. A shifting to larger pore sizes of 4730, 4800, 4800,

<table>
<tr><th>Sample</th><th>Quartz
(75-2315)</th><th>Cristobalite
(75-0923)</th><th>Plagioclase
(41-1480)</th><th>Muscovite
(77-2255)</th><th>Amphybole
(71-1060)</th><th>Hematite
(72-0469)</th><th colspan="2">Munsell Color</th></tr>
<tr><td>CA2</td><td>xxx</td><td>X</td><td>xx</td><td>xx</td><td>xx</td><td>n.d.</td><td colspan="2" rowspan="2">7.5YR 7/4</td></tr>
<tr><td>CCA2-700</td><td>xxx</td><td>X</td><td>xx</td><td>xx</td><td>xx</td><td>n.d.</td></tr>
<tr><td>CCA2-750</td><td>xx</td><td>xx</td><td>xx</td><td>xx</td><td>n.d.</td><td>n.d.</td><td rowspan="4">5.5R</td><td>6/6</td></tr>
<tr><td>CCA2-800</td><td>xx</td><td>xx</td><td>xx</td><td>xx</td><td>n.d</td><td>n.d.</td><td>6/8</td></tr>
<tr><td>CCA2-900</td><td>xx</td><td>xx</td><td>xx</td><td>xx</td><td>n.d.</td><td>x</td><td rowspan="2">5/8</td></tr>
<tr><td>CCA2-1000</td><td>xx</td><td>xx</td><td>xx</td><td>n.d</td><td>n.d.</td><td>xx</td></tr>
<tr><td>CCA2-1100</td><td>x</td><td>xxx</td><td>xxx</td><td>n.d.</td><td>n.d.</td><td>xxx</td><td colspan="2">10YR 3/4</td></tr>
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Figure 6. Phases found by XRD analysis of sample CCA2 before and after thermal treatment. The bracketed number refers to reference spectra. X = scarce; xx = abundant; xxx = very abundant; n.d. = not detected.

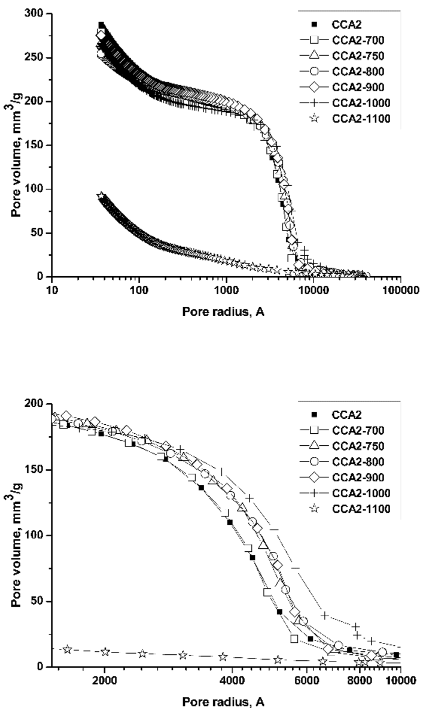

Figure 7. Cumulative pore volume vs pore radius. The top image captures the complete studied range. The bottom image is a detail of the top image, highlighting important changes.

and 5300Å was found after refiring to 750, 800, 900, and 1000°C respectively. Pore volume then collapsed when the sample was heated to 1100°C. This increase in pore size might be the result of a coalescence of smaller pores.

Discussion

X-ray diffraction and mercury porosimetry analyses indicate that the unrefired segment coincided with the segements treated to 700°C since their mineral phases and contributions to pore size are similar. This suggests that the vessel was originally fired to between 700°C and 750°C, since important changes were only observed at higher temperatures. The increase in average pore size after 750°C could be attributed to dehydroxylation of muscovite (Cole and Crook 2006). The textural collapse at 1100°C can be related to the total disappearance of muscovite, with concurrent increases in hematite and plagioclase phases, and the presence of vitreous phases, inferred by the increase in the baseline in the diffractogram corresponding to that temperature.

The thermal treatment generated a reduction in quartz through its transformation into cristobalite, while the plagioclase phase remained and muscovite disappeared. The amphibole also disappeared and the iron component generated a hematite phase. Some components of the initial phases of the fragment which were not transformed may have remained in amorphous phase as the increased background to the diffractogram suggests.

As we have stated previously, changes in color were the product of changes in phases due to application of thermal treatments. It could be established that in the original and treated segments (up to 700°C), the color remained 7.5YR according to the Munsell scale (1954); in segments refired to between 750°C and 1000°C, segments shifted intensities within 5.5YR. After heating to 1100°C, color changed to 10R.This increase in a reddish color can be attributed to the newly-formed hematite phase, as shown by the XRD analysis.

Conclusions

The original firing temperature of the selected sample was established through the application of several techniques during refiring events: X-ray diffraction, mercury intrusion, and color change measurement. The results indicate that firing temperature reached 700 to 750°C.

Constituent phases analyzed by XRD were: quartz, plagioclase, muscovite, amphibole, and trace cristobalite. Refining to 900°C caused the disappearance of amphiboles and initiated hematite. At 1100°C, quartz reduction coincided with an increase in cristobalite, the disappearance of muscovite, and an increase in hematite and plagioclase.

Changes in color ranged from 7.5YR to 10R on the Munsell cale as the segments were treated from 700 to 1100°C.

The original vessel showed a contribution, in pore volume, of 65% in sizes around 4000Å. The heat-treated at 1100ºC dramatically collapsed the pore volume.

The reported firing temperature (700°C and 750°C) allowed the production of light porous vessels in spite of their size. It should be remembered that these vessels were built to store any content, so diminishing their weight would require less physical effort to move them.

These conclusions are added to those already presented in previous published papers (Wynveldt *et al.* 2006; Zagorodny *et al.* 2010a) showing the results of compositional analysis of this type of ceramic paste; it was noted that most of the samples include volcanic glass, mainly as pumice fragments in some cases accompanied by shredded vitreous inclusions, which contribute their own porosity to lighten vessels.

This paper attempts to contribute an experimental approach to determine the firing temperature of a ceramic vessel. Knowing the cooking temperature of a vessel fragment does not imply that the entire vessel reached the same temperature, however, it does facilitate inferences about pottery manufacturing practices.

Acknowledgements

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