

Archaeometric Studies of Ceramics from the São Paulo II Archaeological site

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Abstract

This paper aims at studying the elementary chemical composition of 70 ceramic fragments from the São Paulo II archaeological site. The site is located along the Solimões river, next to Coari city in the Brazilian Amazon. The characterization was performed using instrumental neutron activation analysis, INAA, in which were determined the concentration of Ce, Co, Cr, Cs, Eu, Fe, Hf, K, La, Lu, Na, Nd, Sb, Sm, Rb, Sc, Ta, Tb, Th, U, Yb and Zn in the samples. The Mahalanobis distance was used to study the presence of outliers. Three outliers were found using this procedure. The concentration data was studied next using multivariate statistical methods, such as cluster analysis, principal component analysis and discriminant analysis. The results showed three different chemical groups of samples regarding the similarity/dissimilarity between the samples. Ceramics from each group have been selected and dated using thermoluminescence, TL. The ages of the samples varied between 895 ± 92 and 1142 ± 100 A.D. The firing temperature of the ceramics was determined by electron paramagnetic resonance, EPR.

Introduction

The artifacts found in archaeological sites are related to ancient people who lived there long ago. The more abundant and important artifacts found in many areas worldwide are ceramics which combine, for the most part, durability with ubiquity. Several analytical methods including the nuclear and non-nuclear analytical methods are used to study of the characterization and the manufacture of artifacts [Munita et al., 2013]. Considering nuclear analytical methods, INAA has several advantages over other chemical analysis methods used in ceramic studies. The main advantages are ease of sample preparation, high precision and accuracy for bulk sample analysis, relative immunity to matrix effects, and the capacity to process large numbers of samples. Other strengths of INAA are its superior inter-laboratory comparability and its ability to validate new analytical methods and procedures [Glascok et al., 2007].

Chemical and physical analyses, can sometimes help to deduced a technical manufacturing process and, the provenance of the raw material can shed a light on the source, the trade routes and interactions between groups. The ancient techniques used may help in the discussion of the cultural level of a community. Highly developed techniques are most often found in structured social systems that are characterized by technical progress.

With this in mind, in this paper were analyzed 70 ceramics fragments from the São Paulo II archaeological site by INAA. The dataset was studied using multivariate statistical analysis, like cluster analysis, principal component analysis and discriminant analysis. Some samples were dated using the thermoluminescence method, TL, and the firing temperature was determined by electron paramagnetic resonance, EPR.

The archaeological area

The archaeological site of São Paulo II is located on the left bank of the Solimões river in the mid and lower part of the river in the municipality of Coari in Amazonas state, Brazil, covering an area of 5 ha.

The climate in the region is classified as equatorial hot and humid. The rainy season extends from December to May and the period of drought occurs from July to November.

The people that lived on the banks and affluents of the river occupied a vast territory, waged war and trade among themselves and had different languages [Tamanaha, 2012].

The Amazon has pre-pottery evidence dating back from 7510 b.C. to 2550 b.C [Costa, 2009]. Following this period, four stages of ceramist occupation associated to the Polychrome Tradition emerged [Schaan, 2001; Roosevelt, 1991; Meggers and Evans, 1957]: a) the Açutuba phase, around 450 B.C. and 360 b.C., b) Manacapuru phase, associated to the Incised Rim tradition, dating back to 425 b.C. and 650 b.C., c) Paredão phase, associated to the construction of manmade protection structures dating from 750 b.C. to 1250 b.C., d) The Guarita phase, which is the most recent and superficial occupation showing a rupture in the method of occupation and in the pottery production technique from around 800 b.C. and 1500 b.C. [Neves, 2008; Lima et al., 2006, Nimuendajú, 1981].

The polychrome Tradition is scattered in the Amazon and is associated to the people who speak a variation of Tupi. There are many hypotheses regarding the mobility and expansion of these people based on ethnographic, linguistic and archaeological data (Meggers and Evans, 1957, 1968, Meggers, 1977; Miller 1987].

The São Paulo II site is unicomponential with pottery from the Guarita phase that shows rupture in the method of occupation and in the ceramic production techniques. The terrain is relatively flat formed by anthropogenic organic black earth [Kern and Kämpf, 1989], classified in soil assessments as Latosol, Argisol and Espodosol, with anthropogenic horizon A [EMBRAPA, 1999]. The soil has high pH, calcium, magnesium, zinc, manganese and phosphorous levels [Falcão et al., 2001] as compared with adjacent non-anthropogenic soils that do not have anthropic horizon A [Cunha et al. 2007].

The black earth is the result of intense human occupation and is construed as a chronological, cultural and social marker. In addition, to being an indicator of the increase of demographic density, and the establishment of sedentary occupations. The study of the black earth is essential in understanding how agricultural appeared and was established in the region.

Experimental

Sample preparation and description of the method

NAA is a method whereby samples are exposed to neutrons from a nuclear source, and a fraction of the nuclei from each element within the sample is transformed into unstable isotopes that decay with characteristic half-lives. The radioisotopes emit gamma rays characteristic of each isotope. Those gamma ray energies are measured by a high-purity germanium (HPGe) gamma ray spectrometer, their signals are amplified and the spectrum is measured.

The ceramic powder samples were obtained by cleaning the outer surface and drilling into it, using a tungsten carbide rotary file attached to the end of a variable speed drill with a flexible shaft. Five holes were drilled as deep into the core of the ceramic material as possible without drilling through the walls. Then, the materials were dried in an oven at 105°C for 24 h [Munita et al., 2013]. Seventy ceramic samples were analyzed.

Constituent Elements in Coal Fly Ash (NIST-SRM-1633b) were used as standards. IAEA-Soil-7, Trace Elements in Soil, was used to check samples in every analysis. These materials were also dried in an oven at 105°C for 2 h [Santos et al. 2009].

About 100 mg of samples, NIST-SRM-1633b and IAEA Soil-7 were irradiated in the research reactor pool, IEA-R1, from the IPEN-CNEN/SP, at a thermal neutron flux of about $5 \times 10^{12} \text{ cm}^{-2} \text{ s}^{-1}$ for 8 h.

Two measurement series were carried out using a Ge (hyperpure) detector, model GX 1925 from Canberra with a resolution of 1.90 keV at the gamma peak of ^{60}Co 1332.49 keV and S-100 MCA with 8192 channels. La, Na, Sm, and U were measured after 7 days cooling time and Ce, Cr, Eu, Fe, Hf, Nd, Sc, and Th, after 25-30 days. The gamma ray spectra analysis and the concentrations were carried out using the Genie-2000 Neutron Activation Analysis Processing Procedure from Canberra [Santos et al., 2009].

TL dating

The analytical procedure for TL dating has been described elsewhere [Aitken, 1985], however, it will be described briefly here. The ceramics can be considered as a number of crystalline inclusions, mainly quartz and feldspar. The environmental ionizing radiation excites the electrons of the atoms, which form part of the crystal lattice (e.g. of the quartz), and these excited electrons are ejected from the valence band and release energy (normally as photons) to recover their initial energy state (returning to the valence band). Since the crystal lattices are not perfect, mainly due to impurities in their composition (e.g. substitutions of Si atoms for Al, Fe, etc. in quartz), many of these electrons are trapped in stable potential holes called “electron traps” between the valence and the conduction bands. So, in an environment in which a mineral is exposed to constant ionizing radiation (e.g., inside a sediment), an accumulation of charge is produced, growing in a constant way over time. This charge accumulation is proportional to the energy absorbed by the mineral, and therefore, the energy dose received over the period time. The heat-stimulation results in the excitation of some of the trapped electrons, which recombines and return to the valence band, releasing the energy excess as light. This light emission is known as luminescence. This behavior of the electrons in crystals enables us to know the time elapsed since the moment the archaeological materials, in this case ceramics, were heated for the last time, i.e., fired during their preparation process. Therefore, after mineral is exposed to heat, the accumulated energy is released and the mineral is discharged, i.e., the “luminescence clock” is set to zero. After firing, the pottery starts receiving radiation. Once the ceramic fragment is not used any more, it is buried in sediment or deposited somewhere.

Therefore, the age calculation in luminescence requires estimating two factors: the equivalent dose (D_e) which is the absorbed dose, generally expressed in Gy (1Gy = 1J) and measured in a Luminescence reader and the annual dose (D_{an}), which is the rate of the received dose of ionizing radiation, expressed in mGy/year or Gy/ka. The ratio between both doses, D_e / D_{an} , provides the age.

The D_{an} dose rate determinations were performed by analyzing the U, Th and K concentrations using INAA, on about 200 mg of crushed sample (100 mesh). With this data it is possible to calculate the beta and alpha doses in the samples and the internal gamma dose based on the conversion factors (Adamiec and Aitken, 1998)

In order to obtain pure quartz grains from each fragment to estimate D_e , was first removed 1 mm from the surface layer under red light conditions, by sawing carefully using a diamond grinding wheel and, soaking the blade in water to avoid overheating. The resulting material was carefully crushed in an agate mortar and then dried and sieved. The fraction of 0.080-0.0180 mm was used and was subjected to a “quartz inclusion” procedure that consists of treating the sample with HCl 20% solution to eliminate the carbonates and with H_2O_2 to eliminate the possible presence of organic matter. After these steps, the samples were treated with HF 20% solution in several steps to eliminate feldspars and leave the quartz, eliminating the external layer of quartz to clear the surface and to eliminate the α -particle effects [Li, 2001]. When the proportion of quartz is low and it is impossible to obtain pure quartz, an enriched-quartz polymineral fraction is obtained, which may be used for dating.

The TL signals were performed using an automated TL/OSL system, model 1100-series of Daybreak Nuclear Instruments Inc. TL measurements were taken with a heating rate of 10 °C/s and using two optical filters (Kopp and BG-39) under nitrogen atmosphere.

EPR

EPR spectroscopy can be used to find the firing temperature of ceramics [Bensimon, et al. 1998, Bensimon et al., 1999, Mangueira et al., 2011]. It is based on the absorption of microwave radiation by paramagnetic centers in the ionic crystal. The g-factor of some paramagnetic centers can vary with high annealing temperatures. Such is the case for the signal associated to Fe^{3+} , contained in the ceramics.

Several papers have already discussed chemical methods used to reduce the intensity of the EPR signal due to Fe^{3+} [Bensimon et al. 1998, Ferreti et al., 2002, Duttine et al., 2003]. This Fe^{3+} signal is usually large and hides the E' -center signal used in EPR dating. In this paper was used the method proposed by Watanabe et al. 2008. First was used an additional chemical treatment with 40% HNO_3 solution for 40 minutes, followed by washing with Milli-Q water, to remove Fe agglomerates by dilution.

For the EPR measurements was used a Bruker EMX EPR spectrometer operating at X-band frequency with 100 kHz modulation frequency. One hundred

milligrams of powdered sample were used for each measurement. Diphenil picryl hydrazyl (DPPH) was used for calibration of the g values of the defect centers.

Results and discussion

The analytical quality control of the analysis was tested using 18 independent determinations of the reference material IAEA Soil-7 with the purpose to study the precision of the INAA method. The clays in the different sampling loci of the region may not differ greatly in composition therefore, the method of analysis must be sensitive enough to cope with this problem. Then, the results from IAEA Soil-7 were compared to the certified values. The results showed that most elements had a precision of $\leq 10\%$. This precision is considered by several authors as appropriate for the choice of chemical elements for archaeometric studies using multivariate statistical methods [Bishop et al., 1990]. Since the Co determination showed a precision of less than 10% it was eliminated in the dataset due to contamination by tungsten carbide during the sample preparation [Munita et al., 2013].

The determination of Zn is not reliable as a consequence of a strong γ -ray interference by ^{46}Sc and ^{182}Ta . The interference by the ^{235}U fission in determining the La, Ce and Nd was negligible because the U concentration did not exceed 5 ppm and the rare earth elements were not extremely low [Glascok, 1992]. Nd, Rb and Sb showed a good precision; however, previous studies have shown that there are no reliable elements to include in the database due to the natural heterogeneity [Santos et al., 2009]. Therefore, the elements used in the subsequent studies were Na, La, Yb, Lu, Sc, Cr, Fe, Ce, Eu, Hf and Th.

None of these elements contained missing values. Range, mean and standard deviation are shown in Table 1.

The elemental concentration data was converted to base log 10 to normalize element contributions and to reduce the impact of the differences in the concentrations of some of the major elements.

In turn, the dataset was submitted to outlying tests using the Mahalanobis distance. Outliers can have a considerable influence on multivariate statistical methods because they can disturb homogeneous groups.

Table 1. Range, mean and standard deviation for ceramic samples from São Paulo II archaeological site, in µg/g, unless otherwise indicated.

Element	Range	Mean ± SD ^a
Na, %	0.03 - 0.57	0.18 ± 0.14
La	17.88 – 73.64	38.89 ± 9.67
Yb	1.67 – 4.65	2.96 ± 0.53
Lu	0.28 – 0.78	0.48 ± 0.08
Sc	9.67 – 22.2	16.28 ± 2.46
Cr	47.33 – 104.69	70.89 ± 10.40
Fe, %	1.07 – 4.96	3.25 ± 0.92
Ce	35.59 – 153.24	75.59 ± 18.67
Eu	0.45 – 2.95	1.20 ± 0.37
Hf	3.25 – 10.91	6.18 ± 1.71
Th	7.58 – 17.94	13.99 ± 2.12

^aMean and standard deviation of 70 individual samples

The Mahalanobis distance is an important measure in statistics and has been suggested by many authors as the best method for detecting outliers in multivariate data. For each of the n samples and p variables, the *Mahalanobis* distance (D_i) was taken from the sample to the centroid, as calculated by the expression [Munita et al. 2013 20]

$$D_i = \sqrt{(x_i - \bar{x})' S^{-1} (x_i - \bar{x})} \quad (1)$$

where $'$ is the transpose matrix; $S = \sum_{i=1}^n (x_i - \bar{x})' (x_i - \bar{x})$ is the variance-covariance sampling matrix and, $(x_i - \bar{x})$ is the vector of difference between the concentrations measured in one group and the concentrations measured in the other group. Each one of these values is compared with the critical value, cv , which can be calculated using the lambda Wilks criteria [Munita et al., 2013], calculated as follows:

$$\frac{p(n-1)^2 F_{p, n-p-1; \alpha/n}}{n(n-p-1 + pF_{p, n-p-1, \alpha/n})} \quad (2)$$

where p is the number of variables; n is the number of samples and F , is the F test called “Fisher distribution” ($F = s_1^2/s_2^2$ where s_1^2 and s_2^2 are the sample variances), with p degrees of freedom at a significance level of α/n , $\alpha = 0.05$.

When the value found by expression (1) is larger than the critical value produced by expression (2), the sample is considered to be an outlier [Oliveira et al., 2010]. Thus, the *Mahalanobis* distance values for each sample were calculated and compared to the critical value. In accordance with the *Mahalanobis* distance rule, three ceramics sample outliers were found and removed from the data. Due to the small size of this compositional group, it is difficult to know whether it is a real group. We believe that the outlier samples could be related to contamination processes that may have occurred during burial.

The data from the remaining 67 ceramics samples were submitted to cluster analysis on a 67 x 11 matrix, in which the columns represented the analyzed elements and the rows the samples using Ward’s method and square Euclidean distance. The Ward’s method was employed because it tends to form groups with high internal homogeneity and takes account of the cluster structure. In all statistical studies the software used was R version 2.4.1, 2012.

In Figure 1, the dendrogram shows that the samples were classified into three primary groups, linked at different levels of similarity. With such a large number of samples, the identifiers normally displayed along the bottom of the diagram became unreadable. A clear distinction exists between the two branches of the dendrogram, A and B, which indicates a high level of dissimilarity.

As can be seen in Figure 1, the samples were separated into three groups that were very similar in chemical composition among the samples of each group. This fact allowed for three distinct sources of raw materials that were used in ceramics production at the São Paulo II site.

With the purpose of studying the similarities and dissimilarities between ceramics, the results were submitted to principal component analysis, PCA, and

discriminant analysis, DA. The intention was to group similar samples according to their characteristics (variables). The purpose, therefore, is to consider several simultaneously related variables, all of them having equal importance at the beginning of the analysis.

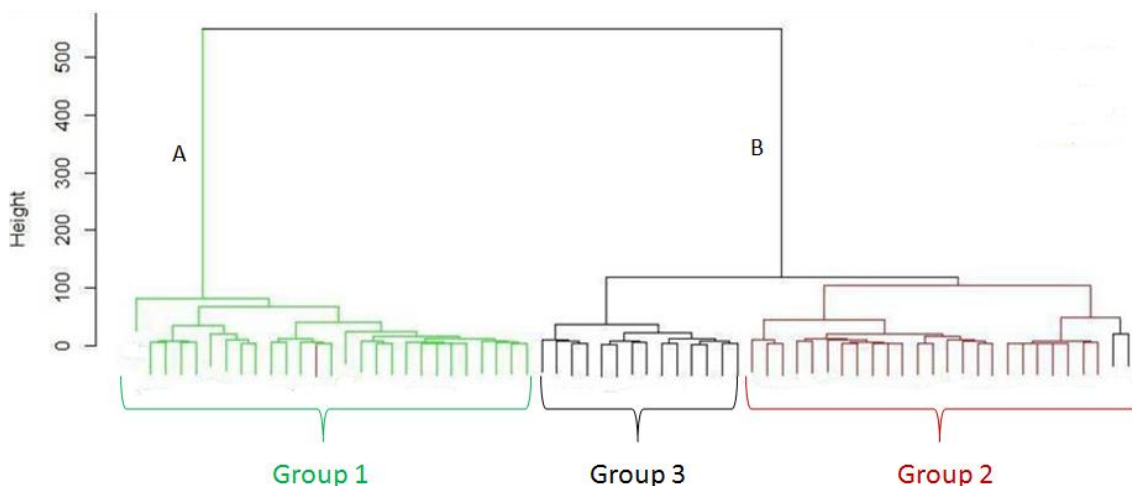


Figure 1. Dendrogram of the ceramics samples using squared Euclidean distance and Ward's method, $n = 67$.

The 3D plot of the first three principal components obtained by the PCA of the São Paulo II dataset is showed in Figure 2. The three PCA explained 83.5 % of the total variance. The fig. confirms the existence of three different chemical groups obtained by cluster analysis.

In order to confirm the latter assumption the data was submitted to discriminant analysis. The basis for all multivariate analyses is that all the elements included are independent variables. This is not necessarily true, but it can be tested using the pooled within-groups correlation matrix provided by discriminant analysis.

After identifying the cluster within samples, discriminant analysis was used to isolate the variables which can most effectively reveal the differences between cluster and establish a discriminant function for this purpose.

The plot obtained by discriminant function 1 vs. discriminant function 2 is shown in Figure 3.

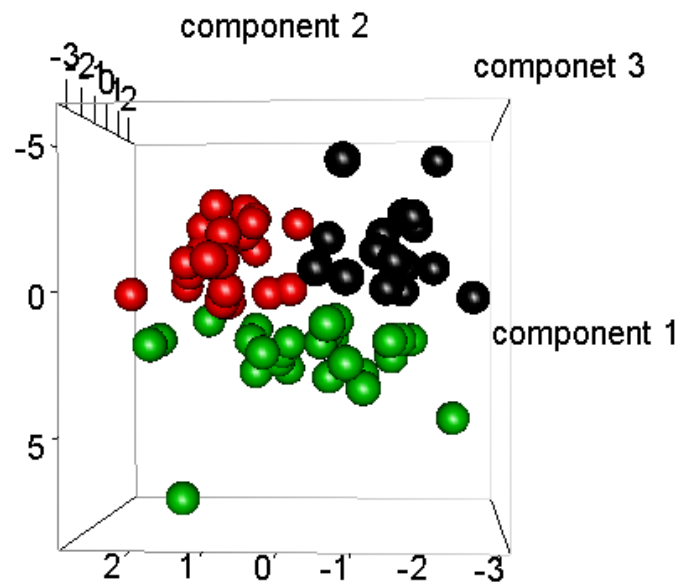


Figure 2. 3D plot of the three principal components.

Fig. 3 shows three groups of samples which indicates that the raw materials used in the manufacture of the analyzed ceramics are different.

In order to verify the firing temperature of the ceramics, five samples, two of group 1 and 2 and one of group , were studied by EPR. The results are shown in Table 2.

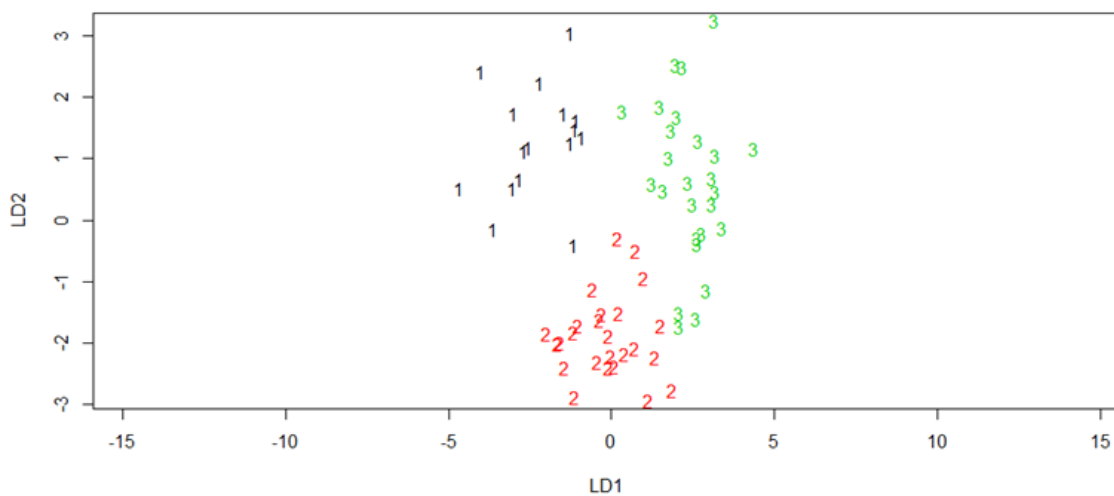


Figure 3. Discriminant function 1 vs. discriminant function 2.

The results show that there are not differences in the firing temperature in the ceramic samples from the São Paulo II archaeological site.

Table 2. Firing temperature of the ceramics samples from the São Paulo II archaeological site.

Sample	Group	Firing temperature, °C
1	1	600 ± 50
2	1	600 ± 50
3	2	650 ± 50
4	2	650 ± 50
5	3	600 ± 50

Following the study, four samples, one from group 1, two of group 2, and one of group 3 were submitted to thermoluminescence dating. Table 3 shows the U, Th and K concentration obtained by INAA, accumulated dose, annual dose and the year of the 4 samples of the three different chemical groups.

Table 3 shows that the average dates of the samples vary between 895 and 1142 a.C., a time interval of 247 years. The uncertainties in the ages vary between 10% and 9% for sample 3 and 1, respectively. The São Paulo II archaeological dates agree with the dates obtained by ^{14}C dating [Lima 2006, 2008].

Table 3. U, Th and K concentrations, annual dose, accumulated dose, and the age of the samples from the São Paulo II archaeological site.

Sample	Group	U, ppm	Th, ppm	K, %	D _{an} , mGy/y	D _{ac} , Gy	Age (years A.D.)
1	1	4.43±0.28	8.11±0.52	2.50±1.31	3.35±0.17	2.91±0.30	1142±100
2	2	2.98±0.25	14.44±0.93	1.49±0.57	2.58±0.12	2.54±0.16	1027±785
3	2	3.14±0.25	11.67±0.64	1.03±0.19	2.06±0.08	2.30±0.17	895±92
4	3	2.65±0.48	9.97±0.73	0.29±0.04	1.29±0.13	1.33±0.13	978±141

The TL dates in Table 3 suggest that the occupation of the site would extend until the 10th century. However, more samples should be analyzed to check the time span for the site, for more conclusive considerations.

Conclusion

The Guarita ceramic from the São Paulo II archaeological site analyzed by INAA and studied by cluster, principal components and discriminant analysis showed three distinct chemical groups. The differences among ceramics from the site studied can be understood in terms of cultural influences in the preparation of the ceramic paste, changes in land use and in organization of ceramic production or of the availability of raw materials. The firing temperature determined by TL of the samples of each group was 600 ± 50 °C and the dating studied by EPR varies between 895 to 1142 A.C. The results found in this study are consistent with others papers conducted in the Central Amazon.

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