J Radioanal Nucl Chem (2015) 306:721–727
DOI 10.1007/s10967-015-4183-5

Archaeometric studies of ceramics from the São Paulo II archaeological site

Rogério Baria[1] · Nilo F. Cano[2] · Betzabel N. Silva-Carrera[3] · Shigueo Watanabe[3] · Eduardo G. Neves[4] · Sonia H. Tatumi[2] · Casimiro S. Munita[1]

Received: 11 February 2015 / Published online: 17 May 2015

Abstract This paper aims at studying the elementary chemical composition of 70 ceramic fragments from the São Paulo II archaeological site by instrumental neutron activation analysis. 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. The firing temperature of the ceramics was determined by electron paramagnetic resonance.

Keywords Archaeological ceramics · São Paulo II archaeological site · INAA · TL · EPR

✉ Nilo F. Cano
nilocano@if.usp.br; nilo.cano@unifesp.br

Rogério Baria
rogeriounesp@uol.com.br

Betzabel N. Silva-Carrera
nsilvacarrera@gmail.com

Shigueo Watanabe
watanabe@if.usp.br

Eduardo G. Neves
edgves@usp.br

Sonia H. Tatumi
sonia.tatumi@unifesp.br

Casimiro S. Munita
camunita@yahoo.com

[1] Instituto de Pesquisas Energéticas e Nucleares, IPEN-CNEN/SP, São Paulo, Brazil

[2] Departamento de Ciências do Mar, Universidade Federal de São Paulo, Santos, SP, Brazil

[3] Instituto de Física, Universidade de São Paulo, São Paulo, SP, Brazil

[4] Museu de Arqueologia e Etnologia, Universidade de São Paulo, São Paulo, SP, Brazil

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 the characterization and the manufacture of artifacts [1]. Considering nuclear analytical methods, instrumental neutron activation analysis (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 [2].

Chemical and physical analyses can sometimes help to deduce 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, the present paper analyzes 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 TL method and the firing temperature was determined by EPR.

The archaeological area

The archaeological site of São Paulo II is located in the mid and lower part of the Solimões River which flows 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 affluent of the river occupied a vast territory, waged war and trade among themselves and had different languages [3].

The Amazon has pre-pottery evidence dating back from 7510 B.C. to 2550 B.C [4]. Following this period, four stages of ceramist occupation associated to the Polychrome Tradition emerged [5–7]: (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. [8–10].

The polychrome Tradition is scattered in the Amazon and is associated with 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 [7, 11–13].

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 [14], classified in soil assessments as Latosoil, Argisoil and Espodosoil, with anthropogenic horizon A [15]. The soil has high pH, calcium, magnesium, zinc, manganese and phosphorous levels [16] as compared with adjacent non-anthropogenic soils that do not have anthropic horizon A [17].

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

Instrumental neutron activation analysis (INAA)

Instrumental neutron activation analysis (INAA) 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 and their signals are amplified and the spectrum is measured.

The powder samples were obtained by cleaning the outer surface of the ceramics 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 as possible into the core of the ceramic material without drilling through the walls. Then the materials were dried in an oven at 105 °C for 24 h [1]. 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 [18].

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 5×10^{12} $cm^{-2}s^{-1}$ 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. K, La, Lu, Na, Nd, Sb, Sm, U and Yb were measured after 7 days cooling time and Ce, Co, Cr, Cs, Eu, Fe, Hf, Rb, Sc, Ta, Tb, Th and Zn, 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 [18].

TL dating

TL is a widely used method for dating pottery [19–21]. Dating by TL technique is a particular application of dosimetry in which there is a source of constant irradiation (the natural radioactivity from radionuclides in the soil where the ceramics is buried). In the case of ceramics, the ionic crystals are the quartz grains contained in the clay from which the ceramics are made. The zeroing of the

archaeological clock is defined by the moment the clay mould is heated to high temperature to produce ceramics.

Therefore, the age calculation in luminescence requires estimating two factors: the equivalent dose (D_e) which is the absorbed dose, generally expressed in Gy 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. 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 [21].

In order to obtain pure quartz grains from each fragment to estimate D_e, about 1 mm of surface layer was first removed 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 ceramics powder with diameter between 0.080 and 0.0180 mm was subjected to chemical treatments with H_2O_2, HF and HCl to eliminate the possible presence of organic matter and some inorganic particles and separate the quartz as best as possible [22, 23]. The quartz grains obtained after chemical treatments were used for TL measurements, using the additive dose procedure assuming that the sensitivity to the laboratory radiation was the same as it has been for radiation during burial [24].

The TL experiments 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 4 °C/s and using two optical filters (Kopp and BG-39) under nitrogen atmosphere.

The peak at around 350 °C (actually it is a superposition of peaks at 325 and 375 °C) has been used since long by many authors for the same dating purpose. Hence, we did not carry out the plateau test.

Firing temperature

EPR spectroscopy can be used to find the firing temperature of ceramics [25]. It is based on the absorption of microwave radiation by paramagnetic centers in the pottery. 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. The value of g-factor of the Fe^{3+} center is very sensitive to its electronic environment. The method to determine the firing temperature of a sample using the Fe^{3+} center is based on the fact that the Fe^{3+} EPR spectrum will change when the sample is heated at high temperatures. At these temperatures the structure of the material will be affected and the resultant EPR spectra of Fe^{3+} ion will show a variation in the g-value [25–27].

EPR measurements were carried out with 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 of the region may not differ greatly in composition and therefore the method of analysis must be sensitive enough to cope with this problem. 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 [28]. 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 [1].

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 [29]. Nd, Rb and Sb showed a good precision;

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

[a] Mean and standard deviation of 70 individual samples

however, previous studies have shown that there are no reliable elements to include in the database due to the natural heterogeneity [18]. Therefore, the elements used in the subsequent studies were Na, La, Yb, Lu, Sc, Cr, Fe, Ce, Eu, Hf and Th.

As can be seen from the Table 1, none of these elements was missing from the elemental analyses assays of the 70 ceramic samples investigated in the present study.

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 [30, 31].

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.

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 [1, 32]:

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

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 [1], 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 [33]. 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

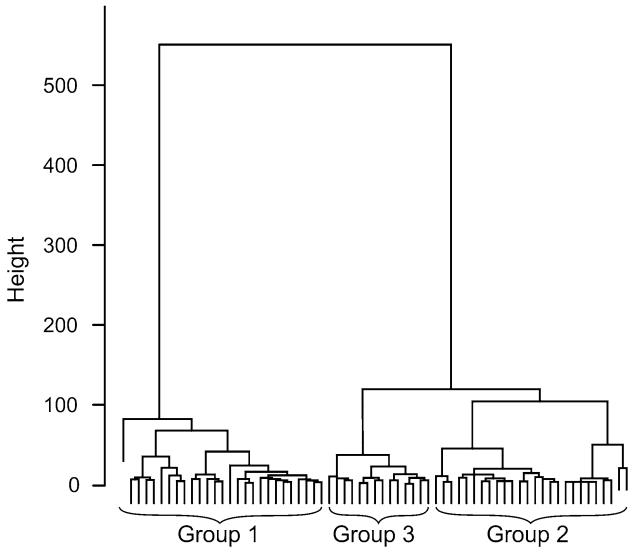

Fig. 1 Dendrogram of the ceramics samples using squared Euclidean distance and Wards method, $n = 67$

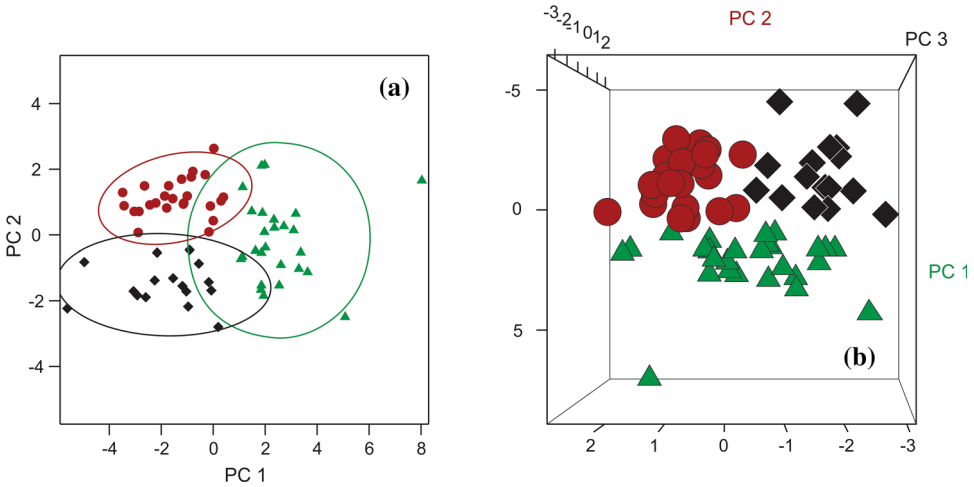

Fig. 2 **a** The plot of the two principal components and **b** 3D plot of the three principal components obtained by the PCA of the dataset of São Paulo II archaeological site. The *ellipses* represent a 95 % confidence region

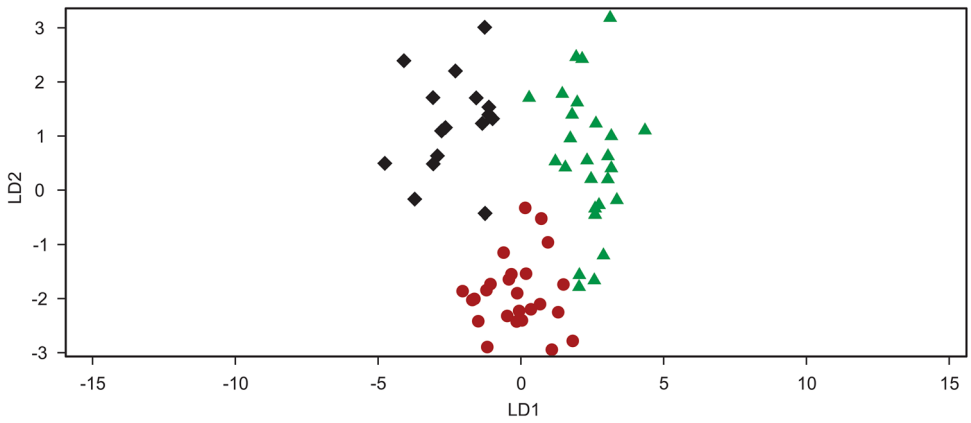

Fig. 3 Discriminant function 1 versus discriminant function 2

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 × 11 matrix, in which the columns represented the analyzed elements and the rows the samples using Ward́s method and square Euclidean distance. The Ward́s 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 Fig. 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 Fig. 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 considerer several simultaneously related variables, all of them having equal importance at the beginning of the analysis.

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

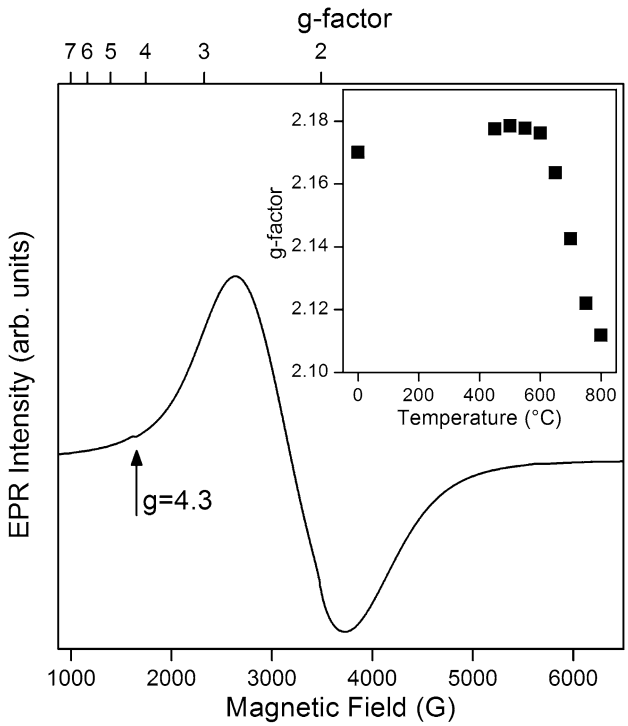

Fig. 4 EPR spectrum of the sample 1 without chemical treatment. The variation of g-factor of Fe^{3+} with heating temperature of the experimental sample is shown in *inset* of figure

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

Table 3 U, Th and K concentrations, annual dose, equivalent 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_e (Gy)	Age (years A.D.)
1	1	4.43 ± 0.28	8.11 ± 0.52	2.50 ± 1.31	3.35 ± 0.17	2.77 ± 0.46	1184 ± 142
2	2	2.98 ± 0.25	14.44 ± 0.93	1.49 ± 0.57	2.58 ± 0.12	2.61 ± 0.26	1000 ± 112
3	2	3.14 ± 0.25	11.67 ± 0.64	1.03 ± 0.19	2.06 ± 0.08	2.51 ± 0.19	793 ± 102
4	3	2.65 ± 0.48	9.97 ± 0.73	0.29 ± 0.04	1.29 ± 0.13	1.37 ± 0.13	947 ± 140

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 versus discriminant function 2 is shown in Fig. 3. This shows three groups of samples which indicate 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 3, were studied by EPR. The EPR spectrum of the raw sample 1 (ceramic powder) is shown in the Fig. 4. We observed a broad absorption around $g = 2$; this line is characteristic of Fe^{3+} ion in an octahedral site. Another EPR line of low intensity was detected in the region of $g = 4.3$, typical of Fe^{3+} in an orthorhombic site. The behavior of the g-factor as a function of the temperature of the pottery sample 1 is shown in the inset of Fig. 4. For all the pottery samples, g value changed above 500–600 °C, indicating that the firing temperature for production of ceramics reached 600–650 °C. The results of five samples are shown in Table 2. The results show that there are no differences in the firing temperature in the ceramic samples from the São Paulo II archaeological site.

Following this study, four samples, one from group 1, two of group 2 and one of group 3 were submitted to TL dating. The TL glow curves of the quartz grains extracted from the pottery samples through chemical treatments with H_2O_2, HF and HCl exhibit three TL peak at 120, 200 and 350 °C. The TL peak at 350 °C (superposition of the peaks at 325 and 375 °C) was used for determination of D_e by additive method. Table 3 shows the U, Th and K concentration obtained by INAA, D_e, D_{an} and the age of the four samples of the three different chemical groups. Table 3 shows that the average ages of the samples vary between 895 and 1142 A.D., 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 [9, 34].

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

Conclusions

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 EPR of the samples of each group was 600 ± 50 °C and the dating studied by TL varies between 895 and 1142 A.D. The results found in this study are consistent with others papers conducted in the Central Amazon.

Acknowledgments This work was supported by the grant from Comissão Nacional de Energia Nuclear—CNEN and CNPq.

References

1. Munita CS, Barroso LP, Oliveira PMS (2013) Variable selection study using Procrustes analysis. Open J Archaeom 1:31–35
2. Glascock MD, Speakman RJ, Neff H (2007) Archaeometry at the University of Missouri research reactor and the provenance of obsidian artefacts in North America. Archaeometry 49:343–357
3. Ocupação Polícroma no Baixo e Médio rio Solimões, Estado do Amazonas. http://www.teses.usp.br/teses/disponiveis/71/71131/tde-05062012-104830/pt-br.php. Accessed 10 October 2014
4. Arqueologia das campinaranas do baixo rio Negro: em busca dos pré-ceramistas nos areais da Amazônia Central. http://www.teses.usp.br/teses/disponiveis/71/71131/tde-29072009-145147/pt-br.php. Accessed 10 October 2014
5. Schaan D (2001) Into the labyrinths of Marajoara pottery: status and cultural identity in prehistoric Amazonia. In: McEwan C,

Barreto C, Neves EG (eds) Unknown Amazon: culture in nature in ancient Brazil. The British Museum, London
6. Roosevelt A (1991) Moundbuilders of the Amazon, geophysical archaeology of Marajó Island. Academic Press, San Diego
7. Evans C, Meggers BJ (1968) Archaeological investigations on the Rio Napo. Smithsonian Institution Press, Washington
8. Neves EG (2008) Digesta: escritos acerca do direito, do pensamento jurídico, da sua metodologia e outros. In: Silverman H, Isbell W (eds) Handbook of South American Archaeology. Springer, New York
9. Lima HP, Neves EG, Petersen JB (2006) A fase Açutuba: Um novo complexo cerâmico na Amazônia central. Arqueol Suramericana 2:26–52
10. Nimuendajú C (1981) Mapa Etnohistórico. Fundação Nacional Pró-Memória - IBGE, Rio de Janeiro
11. Meggers BJ, Evans C (1957) Archaeological investigations at the mouth of the Amazon. Smithsonian Institution Press, Washington
12. Meggers BJ (1977) Vegetational fluctuation and prehistoric cultural adaptation in Amazonia: some tentative correlations. World Archaeol 8:287–303
13. Miller ET (1987) In: Nuñez L, Meggers BJ (eds) Investigaciones paleoindias al sur de la linea ecuatorial. San Pedro de Atacama, Universidad del Norte, Chile
14. Kern DC, Kämpf N (1989) O efeito de antigos assentamentos indígenas na formação de solos com terra preta arqueológicas na região de Oriximiná - PA. Rev Bras Ciência Solo 13:219–225
15. Silva FC (2003) Manual de análises químicas de solos, plantas e fertilizantes. EMBRAPA, Brasilia
16. Falcão NPS, Carvalho EJM, Comerford N (2001) In: Congresso da Sociedade de Arqueologia Brasileira, XI. Grupo de trabalho: Terras Pretas Arqueológicas na Amazônia: Estado da Arte, Rio de Janeiro
17. Cunha TJF, Madari BE, Benites VM, Canellas LP, Novotny EH, Moutta RO, Trompowsky PM, Santos GA (2007) Fracionamento químico da matéria orgânica e características de ácidos húmicos de solos com horizonte a antrópico da amazônia (Terra Preta). Acta Amazonica 37:91–98
18. Santos JO, Munita CS, Toyota RG, Vergne C, Silva RS, Oliveira PMS (2009) The archaeometry study of the chemical and mineral composition of pottery from BraziÍs Northeast. J Radioanal Nucl Chem 281:189–192
19. Cano NF, Machado NG, Gennari RF, Rocca RR, Munita CS, Watanabe S (2012) TL dating of pottery fragments from four archaeological sites in Taquari Valley. Radiat Eff Defects Solids 167:947–953
20. Cano NF, Ribeiro RB, Munita CS, Watanabe S, Neves EG, Tamanaha EK (2014) Dating and determination of firing temperature of ancient potteries from São Paulo II archaeological site Brazil by TL and EPR techniques. J Cult Herit. doi:10.1016/j.culher.2014.05.010
21. Cano NF, Turbiani-Filho IT, Munita CS, Souza MC, Angulo R, Watanabe S (2013) TL dating of sediments from Ilha do Mel, Brazil. Quat Int 306:137–145
22. Zimmerman DW (1971) Thermoluminescent dating using fine grains from pottery. Archaeometry 13:29–52
23. Watanabe S, Farias TMB, Gennari RF, Ferraz GM, Kunzil R, Chubaci JFD (2008) Chemical process to separate iron oxides particles in pottery sample for EPR dating. Spectrochim Acta A 71:1261–1265
24. Wintle AG (1997) Luminescence dating: laboratory procedures and protocols. Radiat Meas 27:769–817
25. Bensimon Y, Deroide B, Clavel S, Zanchetta JV (1998) Electron spin resonance and dilatometric studies of ancient ceramics applied to the determination of firing temperature. Jpn J Appl Phys 37:4367–4372
26. Bensimon Y, Deroide B, Zanchetta JV (1999) Comparison between the electron paramagnetic resonance spectra obtained in X- and W-bands on a fired clay: a preliminary study. J Phys Chem Solids 60:813–818
27. Mangueira GM, Toledo R, Teixeira S, Franco RWA (2011) A study of the firing temperature of archeological pottery by X-ray diffraction and electron paramagnetic resonance. J Phys Chem Solids 72:90–96
28. Bishop RL, Canouts V, Crown PL, Atley SP (1990) Sensitivity, precision, and accuracy: their roles in ceramic compositional data bases. Am Antiq 55:537–546
29. Glascock MD (1992) Characterization of ceramics at MURR by NAA and multivariate statistics. Prehistory Press, New York
30. Glascock MD, Neff H (2003) Neutron activation analysis and provenance research in archaeology. Meas Sci Technol 14:1516–1526
31. Baxter MJ, Freestone C (2006) Log-ratio compositional data analysis in archaeometry. Archaeometry 48:511–531
32. Hazenfratz R, Mongelo G, Munita CS, Neves EG (2012) Comparison of INAA elemental composition data between Lago Grande and Osvaldo archaeological sites in the central Amazon: a first perspective. J Radioanal Nucl Chem 291:43–48
33. Oliveira PMS, Munita CS, Hazenfratz R (2010) Comparative study between three methods of outlying detection on experimental results. J Radioanal Nucl Chem 283:433–437
34. História das caretas: a tradição borda incisa na Amazônia Central. http://www.teses.usp.br/teses/disponiveis/71/71131/tde-22092008-125315/pt-br.php. Accessed 10 October 2014