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A preliminary assessment of the provenance of ancient pottery through instrumental neutron activation analysis at the Monte Castelo site, Rondônia, Brazil

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Abstract
This research aims to contribute to the discussion of ceramic objects found at the Monte Castelo shellmound, an archaeological site located at south-west Amazonia, Brazil. This paper provides the results of a preliminary characterization of eighty-four pottery samples using instrumental neutron activation analysis (INAA) and dating by thermoluminescence (TL). The results showed the existence of three different chemical groups whose dates range from 3000 to 1500 BP. confirming changes in clay sources during that period. These results agree with previous studies about the technological changes and continuities in the Bacabal phase, one of the oldest ceramic cultures of South America.

Keywords Archaeometry · Pottery · Instrumental neutron activation analysis · Amazonia · Provenance analysis

Introduction

Pottery fragments are the most common type of artifact found in Amazonian archaeological sites due to their high resistance to soil conditions. Characteristics such as decoration, color, form and function usually are assumed to be chronological and cultural indicators and the analysis of these attributes can provide a myriad of information about the past [1]. Furthermore, the mineral and chemical composition of ceramics can be useful for better understanding the people that produced them [2]. These studies are based on the assumption that the ceramics of the same production series are chemically and mineralogically similar within a particular timeframe of the site or area of manufacture [3].

There are several analytical techniques that can be employed to determinate the chemical composition of archaeological ceramics. However, INAA employing γ-ray spectrometry is often more suitable than other techniques because the problems related to the dissolution of the sample are avoided. This aspect is very important when archaeological and cultural objects are analyzed. In addition, it is also possible to determine several elements simultaneously with high sensitivity, precision and accuracy [4–7]

This paper aims to study ceramic provenance in southwest Amazonia by means of chemical characterization of pottery sherds from the Monte Castelo shellmound. In order to establish the chronology of the samples, the thermoluminescence technique, TL, was used. This study provides new insights on the development of ceramic production in the Amazonia region through compositional analyses of the Bacabal ceramics.

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Archaeological framework

For many years the Amazon basin was considered to have been a demographic void [8–16]. Over the last few decades, however, archaeological research in the region has shown a very different picture. Archaeological remains found in different areas indicate a deep history of human occupation, proving that Amazonia was densely and diversely occupied for 1000 of years, and that the arrival of Europeans is directly linked to the demise of many native people [17].

In the Amazon, the oldest traces of pottery production are nearly always associated with shellmounds. Located in the alluvial plain of the Branco River, about 20 km from its confluence with the Guaporé River, the Monte Castelo shellmound is one of the oldest contexts of pottery production in the Amazon [10]. The first archaeological studies were made there by Miller in the 1980s, who found large quantities of ceramic fragments, lithic artifacts, plain remains, and abundant faunal remains, including snails of the genus *Pamacea* that constitute much of the matrix of the shellmounds [8]. In 2011, a research team from the Museum of Archaeology and Ethnology of the University of São Paulo, Brazil, resumed research on the site [9] and, besides ceramic materials [10, 12], in the last years new information is being published on microbotanic [13], macrobotanic [14] and zooarchaeological [15] remains. The dates obtained by ^{14}C allow the reconstruction of a long term sequence of cultural development since ca. 6000 years before present, BP.

The pottery classified as belonging to the Bacabal phase was produced with a paste tempered with *cauxí* (freshwater sponge) spicules, mixed with fine sands, and in some cases ground shell. Pots were manufactured by coiling with the addition of modelled apliqués [11], and high temperatures were achieved at firing [9]. Surfaces were smoothed, polished, and treated with red slip. The pot shapes are simple, with plane to concave bases and rims with simple contours. Mouth diameter and the thickness of the walls indicate that the pots had a wide range of dimensions, from small serving vessels to large cooking pots. Applied wings are commonly found, from the simplest varieties to the most stylized, including zoomorphic and anthropomorphic pieces.

Excision and incision were the most recurring types of plastic decoration. The most frequent type features motifs formed by zigzagging excisions, creating triangular areas filled by thin parallel incisions. This same motif is also found in decorations which are based on incised lines, in which the created areas are filled with dotted lines. Large, parallel and straight excisions abound, as do different types of incision and dotted lines, brushed decoration, and basketry impression.

Since the first occurrence of Bacabal pottery in the shellmound, around 4300 BP, until the latest–dated to ca. 700 BP—the morphology of the vessels and the iconography depicted seems to be stable, while the kind of temper and the quantity added changes. The older strata present ground shells as temper, which disappear in more recent strata. One can also observe the increasing importance of sponge spicule temper from the middle period of the Bacabal phase onwards [11].

Experimental

INAA methodology

INAA is a nondestructive method that does not require complicated handling and where problems related to sample loss and contamination are avoided. During the procedure, a standard containing a known quantity of elements of interest is irradiated together with an unknown sample. The principle of the method are given elsewhere [3–7].

A total of 84 ceramic samples were analyzed. The external surface was removed with a fine bristle brush. After that, holes were made in the samples with a tungsten carbide rotary file attached to a variable speed drill. Around 500 mg of powdered sample were obtained from three to eight holes on the side surface of the ceramic fragment, preventing the drill from crossing over the walls [7].

The powder was dried for 24 h in an oven at 104 °C and stored in desiccators [7]. Approximately 100 mg of each sample were weighed in polyethylene bags and sealed with a sealing iron. Groups of up to seven ceramic powdered samples and two reference materials were wrapped in aluminum foil. The Standard Reference Material—NIST—SRM 1633b Coal Fly Ash was used as standard and the IAEA Reference Material, IAEA-Soil 7 Trace Elements in Soil, was used for analytical quality control [7].

The samples and the standards were irradiated in the swimming pool research reactor IEA-R1 of the Nuclear and Energy Research Institute, IPEN-CNEN/SP, at a thermal neutron flux of about 1×10^{12} cm^{-2} s^{-1} for 8 h. The measurements were carried out using a HPGe detector, model GX 2519 from Canberra, with a resolution of 1.90 keV at the 1332 keV gamma peak of ^{60}Co. The spectra were collected by a Canberra S-100 MCA with 8192 channels on an automated sample changer. The gamma-ray spectra analysis and the concentrations were carried out using the Genie-2000 NAA Processing Procedure from Canberra. Two measurement series were carried out. The elements Na, K, La, Sm, Yb, Lu and U were measured after 7 days of decay. The elements Sc, Cr, Fe, Co, Zn, Rb, Cs, Ce, Eu, Hf and Th were measured after 25–30 days of decay [18]

TL dating method

Dating by the TL (thermoluminiscence) technique uses the naturally occurring decay of U, Th and K in soil where at ceramic is buried to determine its age. The TL intensity of natural samples is proportional to the absorbed dose up to dating. In the case of ceramics, the ionic crystals are the quartz grains contained in the clay from which the ceramics are made [13]. The zeroing of the archaeological clock is defined by the moment the clay mould is heated to high temperature to produce ceramics [19–21]. The accumulated dose (D_{ac}) is determined from the ratio TL_{ac}/TL_0 where TL_{ac} is the TL value obtained from a given mass of quartz grains and TL_0 is the TL emitted by annealed quartz grains of the same mass. Finally, annual dose rate (D_{an}) is calculated from measurements of the radioactive elements within the material and its surroundings where the pottery was collected and from the radiation dose rate from cosmic rays [21]. The age of the pottery can be calculated by the D_{ac} divided by the D_{an}.

To obtain the pure quartz grains from each sample to determine the D_{ac}, approximately 1 mm of surface layer was removed by sawing with a diamond grinding wheel. After that, the material was carefully crushed in an agate mortar, dried and sieved. The powder with diameter between 0.080 and 0.0180 mm was subjected to chemical treatments with H_2O_2 30%, HF 20% and HCl 20% solutions to corrode a thin layer of the surface of quartz grains in order to clear the surface and to eliminate the possible presence of α-particle effects, carbonates and organic matter and separate the quartz as best as possible [18].

The quartz grains were used for TL measurements using the multiple aliquot regeneration procedure, MAR, assuming that the sensitivity to laboratory radiation was the same as it had been for radiation during burial [19–21].

The regenerative dose samples were then warmed from room temperature to a final temperature of 500 °C with a heating rate of 5 °C/s remaining at 200 °C for 10 s to erase the TL signal from the peaks of low temperatures that are very unstable [19–21]. The TL measurements were performed using a Risø TL/OSL Luminescence Reader Model TL/OSL DA-20 equipped with a $^{90}Sr/^{90}Y$ β radioactive source with a dose rate of 0.081 Gy/s.

The annual dose value was estimated from uranium, thorium and potassium concentrations obtained by INAA [3–7].

Results and discussion

The assumption in ceramic provenance analysis using chemical elements is that clay sources may be differentiated if the analytical technique used has a good precision and accuracy. If one element is not measured with good precision and accuracy, it can interfere in the real differences in concentration of the discriminating effect of other well-measured elements. The differences in the concentrations can be used to form ceramic compositional groups, because vessels manufactured from a given clay source will be more similar that those manufactured from a different source [12]

In this work, the precision of the analytical method was studied using the reference material IAEA-Soil 7, for which 14 independent determinations were made. Table 1 shows the elements, the mean, standard deviation (SD), the relative standard deviation, RSD, and the confidence interval. The results found were compared with the certified values.

Table 1 Results for IAEA Soil 7 determinations in mg/kg, unless otherwise indicated, n = 14

Element	Certified value	Mean ± SD	RSD (%)	Confidence interval
Na (g/kg)	2.40	2.35 ± 0.08	3.54	(2.30–2.50)
K (g/kg)	12.10	12.39 ± 0.70	5.62	(11.30–12.70)
La	(28)	30.81 ± 0.88	2.87	(27.00–29.00)
Sm	(5.1)	5.59 ± 0.27	4.77	(4.80–5.50)
Yb	(2.4)	2.29 ± 0.29	12.55	(1.90–2.60)
Lu	0.3	0.36 ± 0.04	10.47	(0.10–0.40)
U	(2.6)	2.45 ± 0.64	26.32	(2.20–3.30)
Sc	(8.3)	8.71 ± 0.19	2.14	(6.90–9.00)
Cr	(60)	68.74 ± 4.14	6.02	(49.00–74.00)
Fe (g/kg)	(25.7)	26.08 ± 0.72	2.75	(25.20–26.30)
Co	(8.9)	9.11 ± 0.40	4.37	(8.40–10.10)
Zn	(104)	93.03 ± 13.29	14.29	(101.00–113.00)
Rb	(51)	60.50 ± 13.72	22.69	(47.00–56.00)
Cs	(5.4)	4.97 ± 0.21	4.19	(4.90–6.40)
Ce	(61)	60.19 ± 0.99	1.64	(50.00–63.00)
Eu	(1)	1.02 ± 0.10	9.94	(0.90–1.30)
Hf	(5.1)	5.06 ± 0.60	11.90	(4.80–5.50)
Th	(8.2)	8.19 ± 0.51	6.24	(6.50–8.70)

In the table, the RSD values are in agreement with the values recommended. However, in this paper were used elements with RSD less than 10% for the interpretation of the results. This precision is considered appropriate by several authors for the choice of the chemical elements for studies of archeological objects using multivariate statistical methods [22]. Although Co had RSD 4.37, it was eliminated from the data set because its concentration can be affected by tungsten carbides files during sample preparation [23].

Results indicate that U precision was affected by the experimental conditions adopted and would need to be determined using epithermal NAA, ENAA. The measurement of zinc via ^{65}Zn using the peak of 1115.55 keV, $T_{1/2} = 245$ days is hardly possible in geological materials using INAA due to the strong influence of ^{152}Eu with a gamma peak in 1112.0 keV. Furthermore the Yb determination via ^{175}Yb using the peak of 396.32 keV and $T_{1/2} = 101$ h was affected by the energy of 398.2 keV from ^{233}Pa. Lastly the interference of ^{235}U fission in the determination of La and Ce was negligible because the U concentration did not exceed 5 ppm and the rare earth elements were not extremely low [24]

Consequently, the elements Na, K, La, Sm, Sc, Cr, Fe, Cs, Ce, Eu and Th were used in subsequent data analyses.

Table 2 Range, mean and standard deviation for ceramic samples from Monte Castelo archaeological site in mg/kg, unless otherwise indicated

Element	Range	Mean ± SD[a]
Na, mg/g	0.31–1.45	0.71 ± 0.28
K, mg/g	5.11–21–07	10.89 ± 2.83
La	14.75–57.91	34.05 ± 9.26
Sm	2.72–25.25	6.74 ± 3.50
Sc	6.36–18.67	12.16 ± 2.54
Cr	30.44–76.73	51.87 ± 11.21
Fe, mg/g	9.47–30.07	16.60 ± 4.92
Cs	2.16–12.44	5.17 ± 2.03
Ce	30.39–117.36	63.62 ± 19.93
Eu	0.391–2.04	1.06 ± 0.38
Th	5.70–16.74	10.37 ± 2.43

[a]Mean and standard deviation of 84 individual samples

Table 2 shows the range, the mean and the standard deviation for elemental analyses of the 84 ceramic samples investigated in the present study. None of the elements considered contained missing values.

Previous authors [24, 25] have differed over whether mass fraction data for geological materials are distributed normally or lognormally. Ahrens [26] noted that if the dispersion in the data is small compared to the mean mass fraction, the lognormal distribution approximates a normal distribution. In this work the data set was rescaled to base10 logarithms because the assumption of lognormality is also convenient, the log transformations compensating for the large differences in magnitude between the high-concentration elements, such as Na, K and Fe, and the trace elements. Thus, log transformation creates a convenient quasi-standardization or rescaling of the original data that facilitates the application of various multivariate methods [27, 28]. Details on the calculations and reasoning behind these transforms can be found elsewhere [5, 27, 29].

After logarithmic transformation the data set was submitted to outlier tests. Many multivariate methods are sensitive to outliers and it is convenient to identify and remove them before analysis. The quadratic Mahalanobis distance matrix between each sample coordinates vector and the mean vector was calculated using the Statistic software. The values were compared with the lambda Wilk's critical value for the identification of multivariate outliers [28]. Only two samples were considered outliers and were removed of the data set.

A preliminary classification of the data set was made using hierarchical cluster analysis, HCA, to outline possible clusters or groups within the elemental data. This was performed with the program R using as similarity measure the squared mean Euclidean distance among the mass fraction of 11 elements and the linking criterion used was the Ward´s method. The grouping that resulted from chemical analysis of the sample is showed in the dendrogram in Fig. 1.

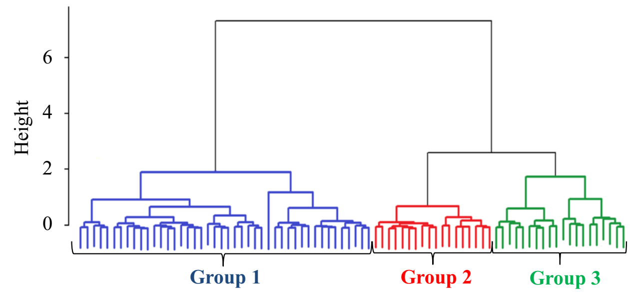

Fig. 1 Dendrogram of 84 samples from Monte Castelo archaeological site using Ward's method and squared mean Euclidean distances

The result of the cluster analysis outlined the separation of the samples into three very well-defined groups, henceforth referred to as Groups 1, 2 and 3, containing 44, 18 and 20 samples, respectively.

After identifying the chemical groups by HCA, a principal components analysis technique was used to confirm the samples in the data set that form each group [30]. Figure 2 shows PC1 vs PC2 and PC3. The three PCs explained 72.54% of the total variance.

Figure 2 shows that the samples cluster together in three groups in agreement with cluster analysis and with technological and stylistic characteristics.

Finally, a discriminant analysis was carried out in order to verify and confirm the data structure. The basis for all multivariate analyses is that all the elements included are independent variables [27]. This is not necessary true, but it can be tested using the pooled within-groups correlation matrix provided by discriminant analysis [27]. Figure 3 shows discriminant function 1 vs discriminant function 2.

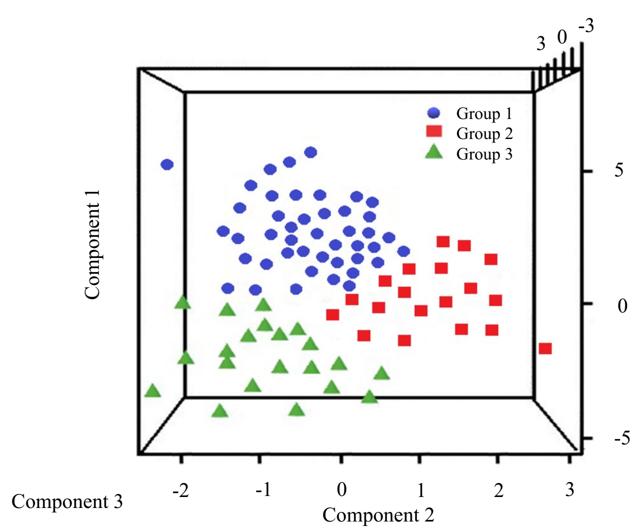

Fig. 2 3D principal component analysis

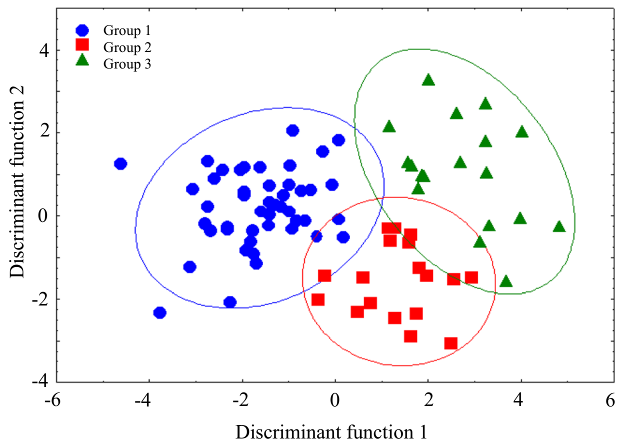

Fig. 3 Discriminat function 1 vs. discriminant function 2

As can be seen, each cluster is formed by a chemically relatively homogeneous showing a degree of chemical similarity among the samples.

These results clearly show that compositionally the pottery samples are discriminated by at least three kinds of clay, that may be identified in the area. Previous work had demonstrated some possible local clay sources for Monte Castelo ceramics and parameters for the Bacabal clay were mapped [9].

The chemical groups are in agreement with the stylistic diversity of whole vessels found at the site that were studied by means of techno typological method. The compositional data were able to reinforce the group divisions made stylistically and suggests the likelihood of at least three clay sources of the pottery from Monte Castelo. To see if the samples are from the same period, two samples of each group were submitted to TL analysis for dating.

Following its stratigraphic provenance and from the stylistic point of view, the diversity within the three compositional ceramic groups suggest that the samples would be from different time periods. In this case, the compositional data would serve to reinforce the group divisions based on style and chronology indicating different periods.

Table 3 shows the results for the accumulated dose and annual dose, both in μGy, and the age of the samples in years.

As can be seen in Table 3, the ages of the samples vary according to the groups. The samples of the first group are the oldest and their ages vary from 2408 to 3220 BP.

For samples of group 3, the ages range from 1446 to 1523 BP However, the ceramics from group 2, range in age from 1859 to 1947 BP. The age of the ceramics of the three groups are covered by previous archaeological chronologies that place the Bacabal phase from ca. 4300 BP extending to 700 BP [8, 9].

Table 3 Accumulated dose, D_{ac}, annual dose rate D_{an} and age obtained

Group	Sample	D_{an} (μGy)	D_{ac} (μGy)	Age (years BP)
1	1	1767 ± 122	5.69 ± 0.15	3220 ± 238
	2	2197 ± 168	5.29 ± 0.06	2408 ± 186
2	3	2106 ± 159	4.10 ± 0.10	1947 ± 154
	4	2862 ± 190	5.32 ± 0.20	1859 ± 142
3	5	3153 ± 135	4.56 ± 0.096	1446 ± 69
	6	2804 ± 156	4.27 ± 0.073	1523 ± 89

Conclusions

The compositional variation exposed by the chemical analysis of ceramics from the Monte Castelo archaeological site suggested the likelihood of not one but rather of three Bacabal periods of ceramic production, which is in agreement with technological studies about the site. Stylistically, the three compositional ceramic groups show diversity and suggest that the samples would be from different time periods [9, 11]. The compositional data were able to reinforce the group divisions made stylistically and indicate three production periods of the Bacabal pottery of the site.

This research provides a solid framework that can be employed in the future to carry out a more detailed and extensive study of the Monte Castelo ceramics. Further archaeometric analyses based on this high-resolution compositional technique (INAA) can produce data that, together with other proxies from the Amazonian archaeological record, will help to clarify the geography of cultural the networks that existed in the deep indigenous history of South America.

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