

Study of the Firing Temperature of Marajoara Archaeological Ceramics by EPR Associated to INAA Data

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Abstract: The Marajó Island is the largest island of the Marajó archipelago, located in the north of Brazil. In this region, at the end of 19th century, it was identified an archaeological society named Marajora, which occupied the island from 400 to 1,350 AD. It was characterized by a ceramic style recognized by its beauty and exuberance. Until this moment, the physical and chemical analysis studies in this ceramics are scarce and this kind of studies may help archaeological studies performed in the island. With this purpose, this work presents a preliminary study of the firing temperature in the ceramics. For that, the elementary concentrations of 109 samples from Museu de Archaeology and Ethnology Museum of the University of São Paulo and samples collected in the Marajó Island were studied. The samples were analyzed using instrumental neutron activation analysis in which the elemental concentration of Ce, Co, Cr, Cs, Eu, Fe, Hf, K, La, Lu, Na, Nd, Rb, Sc, Sm, Ta, Tb, Th, U, Yb and Zn were determined. The interpretation of the results was made using multivariate statistical approaches and showed the existence of three chemical groups. Some samples of each cluster were chosen for the determination of their firing temperatures by electron paramagnetic resonance, in order to verify if different firing temperatures was associated with different elemental groups of ceramic samples. The results showed that there are no statistical differences in the firing temperature between the samples from Archaeology and Ethnology Museum and the contemporary samples of the Marajó Island.

Key words: Marajó island, archaeological ceramics, INAA, EPR, multivariate statistical analysis.

1. Introduction

The Marajó Archipelago is located in Pará State, Brazil, and occupies the eastern part of the Brazilian Amazônia. It is limited northwest by the Amazon river—the largest river in the world by waterflow, south by Pará River, southeast by Tocantins River and northeast by the Atlantic Ocean (Fig. 1). The archipelago is formed by several beaches of great

beauty, and is also known for having the largest buffalo herd in Brazil. The biggest island in the region is Marajó with an area of 50.000 km², which makes it the largest fluvial island in the world. In Fig. 1, it is possible to observe the Belém city, which is the capital of the Pará State.

Archaeological researches in Marajó begun in 1940, from the works of Betty Meggers and Clifford Evans [1]. Six consecutive archaeological phases were established to the origin of the Marajoara society, 2004. The archaeological phases Ananatuba,

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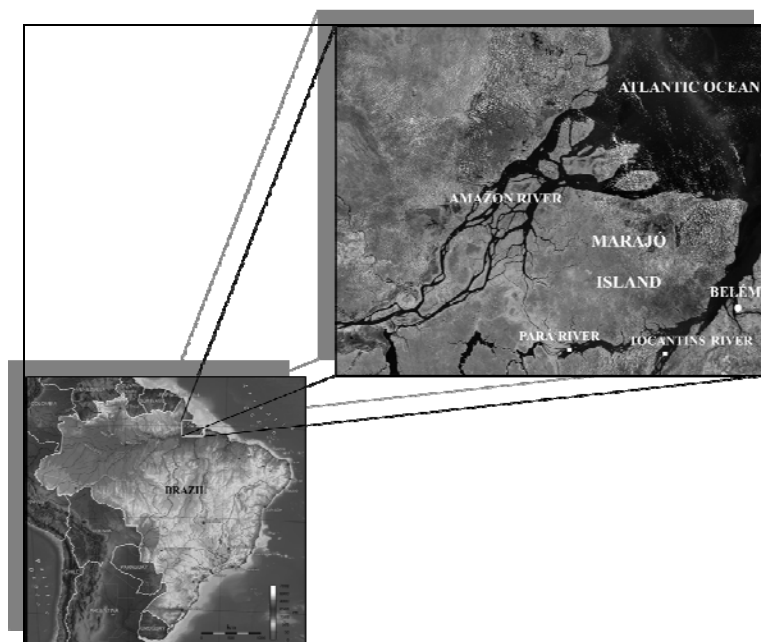


Fig. 1 Localization of the Marajó Island [2].

Mangueiras, Acauã, Formiga, Marajoara e Aruã occupied the island in a period between 1,500 BC and 1,500 AC, where the Marajoara phase was the most expressive relative to the manufacture of a large amount, as well as quality, variety and exuberance of its archaeological artifacts.

Records show that on the island of Marajó and prior to the Marajoara phase, there were small towns, which lived on fishing, hunting and cultivation of manioc. The pottery produced at this time is found often in the form of heavy vessels of thick walls, employed for domestic uses and few decorative details. After 400 BC, there were societies organized on the island [1]. Because of having to adapt to the devastating effects of climate, the Marajoara peoples built barrage damming rivers, creating ingenious hydraulic systems and causing great changes in the landscape [3].

The strong rains, as well as floods during throughout half of the year, followed by severe drought, led the native peoples to adapt to this situation as well as to express their lifestyle in the patterns of design of the ceramic artifacts. It is common to find in the ceramics painting expressions

of the aquatic animals, as well as those relating to the mythology Marajoara such as crabs, lizards, scorpions, turtles and snakes. In ceramics, there is also emphasis on abstract representations and geometric designs of animals and humans [4].

Marajoara people also built a lot of hills on Marajó called tesos, which are huge buildings constituted by several interspersed floors. During a period of 1000 years, tesos were considered homes, as well as political and ceremonial centers of those people. These characteristics are revealed in the quality of the ceramics impregnated of creativity, details in both decoration and form, density and how they were found on the tesos. That area provides a vast field for research. From the archaeological point of view, there are several studies published in this aspect. However, the scarcity of information about the physical and chemical analysis of Marajoara pottery still dominates.

The research area that studies archaeological artifacts by means of physical and chemical techniques is named archaeometry [5]. In an empirical form, the archaeometry studies had begun in 1795, when Martin Klaproth determined the chemical

composition of ancient objects, such as glasses and coins [6].

In the XIX century, sporadically, approaches physics and chemistry begun to be applied to the analysis of archaeological ceramics, due to its extreme durability [7]. The analysis of ceramic fragments collected was, initially, directed to provenance studies, by means of the determination of the chemical composition. But, the discovery of the X-ray in 1895, by Wilhem Konrad Roentgen, enabled the radiographs of paintings, also making a form of investigation of the originality of artistic works [8].

In this perspective, the achievements in the field of the Archaeometry in the XVIII and XIX centuries allowed to create close relationship between the humanities and exact sciences, intensifying in the last 50 years [9, 10]. The chemical composition, mineralogy and age are the main parameters investigated in archaeometry [9, 11]. However, little focus has been given to the technology employed in the ceramic's production, as firing temperature, hardness, density, as well as with relation to conditions in which it was made.

In this study, 109 Marajoara ceramic samples fragments were analyzed. With the application of INAA (instrumental neutron activation analysis), the chemical elementary concentrations of ceramics were determined and submitted to multivariate statistical analysis. From the different groups of ceramics formed, samples belonging to the groups were chosen to determine their firing temperatures by EPR (electron paramagnetic resonance). The characterization of archaeological and contemporary ceramics in the present work has the objective of applying techniques to determine some parameters to observe possible differences between those ceramics, contributing in their identification. Moreover, the information obtained in this work may also help the museums to analyze received pieces with doubtful provenance, where the physical and chemical analyzes can be a support to typological analysis (such as color,

design standard, tempers, artifact form) when the latter is not enough.

2. Material and Methods

2.1 Sample Preparation and Analytical Procedure for INAA

Initially, for determination by means of INAA, the ceramic fragments were washed with water using a brush of fine bristles. Then, the external surface of the ceramics was cleaned with tungsten carbide drill bits, with the preventing any contamination in the analysis, since the fragment collected was dirty. However, tungsten carbide cleaning can causes alteration in the elementary concentrations of Co and Ta and this possibility must be considered. In the literature there are works reporting the relation of the contamination effect and the homogeneity of the samples to be analyzed [12].

The results showed that, apart from preventing contamination, it is not also necessary to destroy the fragment [13, 14]. Then, with the objective of assuring the chemical homogeneity, about five holes in different internal parts of each of the fragments were made. A mass of 500 mg was obtained in the form of powder from each fragment and this amount of sample was dried in 104 °C for 24 hours [13].

For the total powder mass of 500 mg, 120 mg was separated for INAA analysis. This material was weighed in polyethylene packaging and sealed with iron, with the same amount of Standard Reference Material NIST-SRM 1633b, used as standard. These packaging were mingled with sheets of aluminum paper. After this procedure, the samples and standard were irradiated for 1 h at the IEA-R1 reactor of IPEN-CNEN/SP, under a thermal neutrons flux of $8.92 \times 10^{12} \text{ n}\cdot\text{cm}^{-2}\cdot\text{s}^{-2}$. Two countings were carried out: K, La, Lu, Na, Nd, Sb, Sm, U and Yb were determined after seven days of decay. After 25-30 days of decay, Ce, Co, Cr, Cs, Eu, Fe, Hf, Rb, Sc, Ta, Tb, Th and Zn were determined.

2.2 Sample Preparation and Analytical Procedure for EPR

For EPR analysis, each sample was divided into 11 aliquots of 70 mg resulting in a total of 99 aliquots. The 11 aliquots of each sample were submitted to ten different annealing temperatures. The following temperatures were used: 400 °C, 450 °C, 500 °C, 550 °C, 600 °C, 650 °C, 700 °C, 750 °C, 800 °C and 850 °C, respectively. This interval was chosen assuming that the firing temperature was in this interval, once that the most of the archaeological ceramics has firing temperatures between 500 °C and 850 °C. A heating rate of 10 °C per minute was chosen.

After been kept in the stove by about 20 minutes and achieve the temperature previously determined, the samples were removed from the furnace and waited until reach room temperature. The samples analyses were carried out in a Varian EPR spectrometer operating in the frequency of 9.4 GHz (X-band) with modulation frequency of 100 kHz and amplitude 0.4 mT. The samples were placed in glass tubes with about 4.3 mm of diameter. For eliminate the possible interference of the glass tubes, the signal with empty tube was performed and then, subtracted from the signal obtained with the set sample plus tube. A field sweep of 4,000 G was used in two minutes and the constant of time was 100 ms. The spectra were recorded at room temperature.

2.3 Instrumental Neutron Activation Analysis

INAA is based on the measurement of the induced radioactivity in the sample elements, by means of the irradiation with neutrons. This phenomenon occurs due to nuclear reactions, when a neutron is captured by the target forming a compound nucleus in an excited state. This interaction results in the formation of a radioisotope [15, 16]. For this work, the particles of interest are the gamma ray, emitted according to the half-life of each radioisotope. Thus, being the energy of gamma rays and half-life characteristic parameters for each radioisotope, it is possible to obtain the

qualitative and quantitative determination of the elements present in the samples.

2.4 Electron Paramagnetic Resonance

In archaeometric studies, EPR can be employed for dating and determining firing temperature of ceramics, among other applications [17, 18]. EPR is spectroscopy technique able to measure the absorption of microwave radiation by paramagnetic species, such as Fe(III), Cu(II) and Mn(II). The EPR spectrometer is constituted mainly by source of microwaves and an electromagnet that can generate a magnetic field which vary linearly. The radiation absorption is observed when the intensity of the magnetic field satisfies the Eq. (1) (resonance condition).

$$h \times \nu = g \times \beta \times B_o \quad (1)$$

where,

h = Planck's constant; ν = frequency of the microwaves radiation (Hz); β = Bohr magneton; B_o = magnetic field.

To improve spectrometer sensitivity, the EPR spectrum is usually observed as the first derivative of the absorption spectrum as a function of the magnetic field. Knowing the resonance frequency and its respective magnetic field, the value of the g factor becomes determined. The value of g factor of paramagnetic centers is very sensitive its electronic neighborhood. In this manner, the method to determine the firing temperature of a sample is based on the fact that changes in the EPR spectrum will appear only when the sample is heated above the firing temperature. Therefore, the structure of the material will be affected and the EPR spectra resultant will show a variation in the g value [19].

3. Results and Discussion

A total of 109 ceramic samples were analyzed. 49 samples were provided from Museum of Archaeology and Ethnology of the University of São Paulo-MAE-USP. The majority of this material was collected by H. Schultz, et al. in the 50's, on the archaeological sites Furinho, Cuieiras, Pascoal dos

Mello, Pacoval and Fazenda São Marcos. These pieces belonged to Paulista Museum, and, in 1989 were incorporated into the Marajoara ceramic MAE, the collection of which is part of the Amazon Collection [7]. The remaining 45 samples were provided from Marajó Museum and 15 contemporary samples produced and marketed in the Marajó Island.

Initially, the concentrations of Ce, Co, Cr, Cs, Eu, Fe, Hf, K, La, Lu, Na, Nd, Rb, Sc, Sm, Ta, Tb, Th, U, Yb and Zn were determined by INAA in eight samples of the reference material IAEA-Soil 7 Trace elements in soil, with the aim of studying the precision and accuracy for each of the elements.

The results showed that the concentration determined for most elements have a precision $\leq 10\%$. This precision is considered appropriate for the choice of chemical elements for studies of archaeological objects, using multivariate statistical analysis [20]. The element Co was eliminated due the contamination by means of tungsten carbide during the sample preparation [12].

The ^{65}Zn , with peak of energy 1,115.55 keV and the ^{182}Ta with peak of energy 1,121.30 KeV were also eliminated, because they suffer interference in the gamma rays spectrum of the peak 1,120.5 keV (^{46}Sc), resulting in the reduction of its precision [21]. Even though the elements Nd, Rb and Sm were also obtained with good precision, previous studies showed that they are not reliable elements to insert in the database, due to the great dispersion in the ceramics concentrations. Therefore, the used elements were Ce, Cr, Cs, Eu, Fe, Hf, K, La, Lu, Na, Sc, Tb, Th, U and Yb.

Following, the concentrations of the ceramics samples were transformed in a logarithmic scale by taken lg of concentration values. This transformation before applying multivariate statistical techniques is a usual procedure in archaeometric studies and there are two reasons for this: the one is explained by the fact that a normal logarithmical distribution of the elements exists [22]. The other is the difference magnitude between elements, which was found in

percentage and trace level. After this, the detection of the outliers was done by means of Mahalanobis distance, D_i^2 , a method used when numerous variables are determined [23]. Considering a database with n samples and p measured variables, the distance D_i^2 is calculated by:

$$D_i^2 = (X_i - \bar{X})^T S^{-1} (X_i - \bar{X}) \quad (2)$$

where,

$$S = \sum_{i=1}^n (X_i - \bar{X})(X_i - \bar{X})^T / n - 1 \quad (3)$$

and T is the transposed matrix, X_i is the observation of interest, \bar{X} is the vector of the average and S represents the covariance-matrix.

For each sample in the variables set, D_i^2 was calculated using the lambda Wilks criterion as critical value [24]. The samples with values overtaking the critical value were eliminated and the same procedure was repeated for the remaining samples. The procedure finished when the samples showed values of Mahalanobis distance lower than the critical value. By means of this procedure, five sample outliers were found and removed from the data set.

3.1 Cluster Analysis

The cluster analysis is a multivariate statistical technique named of unsupervised [25, 26]. This means that there is no knowledge with relation to the groups formed by the samples. The main objective is to form groups with similar samples more possible homogeneous internally and more heterogeneous among the different groups in accordance with their characteristics.

After the elimination of five outlier's samples, the 104 ceramic samples results were submitted to cluster analysis. 52 ceramic samples from MAE and 28 ceramic samples provided by Marajó Museum belong to the Group 1. Samples from MAE and from Marajó Museum belong to the Group 2, with 21 samples of each Museum. And, 15 contemporary samples of the Marajó island belong to the Group 3. Fig. 2, a dendrogram, shows that samples were separated in

three groups, indicating similarity in the chemical composition among the samples gathered in each group and dissimilarity among the groups.

3.2 Principal Component Analysis

The principal objectives of the principal component analysis are the reduction of the dimensionality of the data, and the achievement of interpretable combinations of the variables [27]. After the prior identification of the data structure by means cluster analysis, the data were studied by principal components. The reason for this is that cluster analysis does not consider the correlation structure among the variables, and it is just a tool for preliminary analysis of the correlated data. In this study, the principal component analysis corroborated the identification of the groups suggested by dendrogram in the cluster analysis. Fig. 3 shows the scores of the first principal component against the second one for all the ceramic samples analyzed. These components represent together most of the fraction of the system variance.

After the separation of the samples in groups, from its chemical compositions, nine ceramic samples belonging to the distinct groups were chosen for determination of its firing temperatures by EPR. The objective is analyzing possible differences of firing temperature in the samples that present differences with relation to its chemical composition. For this, five samples were chosen in the Group 1, 1 sample of the Group 2 and Group 3 samples of the Group 3.

Figs. 4-6, show all the plots with the obtained firing temperature determined for each ceramic sample analyzed.

Figs. 4-6 present the behavior of the g_{ex} factor in function of the firing temperature for nine ceramic samples analyzed by EPR. It was identified a sudden variation of the g_{ex} factor in the region between 500 °C and 650 °C. It indicates that the firing temperatures determined for nine ceramic samples analyzed are within that interval. The firing temperature did not present statistically significant differences for samples

of different groups. More number of samples will be analyzed to verify the possibility of differentiation between the archaeological and contemporary ceramics by means of their firing temperatures.

The firing temperatures determined may be relevant to study the ceramics manufacture technology. The Marajoaras might have utilized a stove to produce their artifacts, once under these conditions it is possible to keep a better control of the high temperatures. In the case of firing of pottery in outdoor bonfires, it is more difficult to control and achieve higher temperatures due to uncontrolled natural factors.

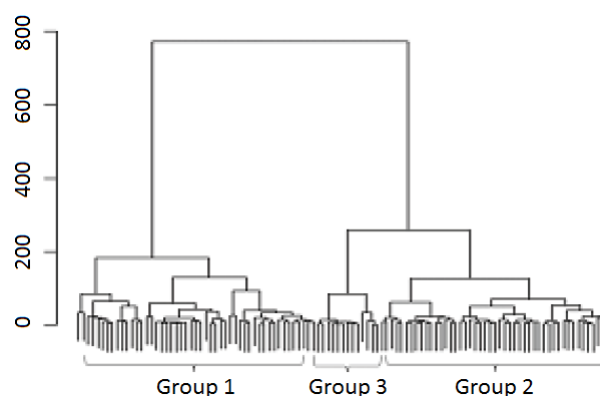


Fig. 2 Dendrogram of the ceramics samples using square Euclidean distance and Ward's method.

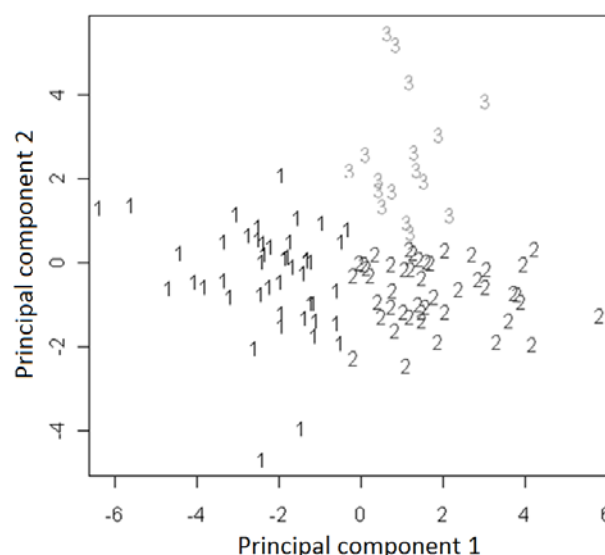


Fig. 3 Principal component analysis 1 versus principal component 2 of the ceramic samples.

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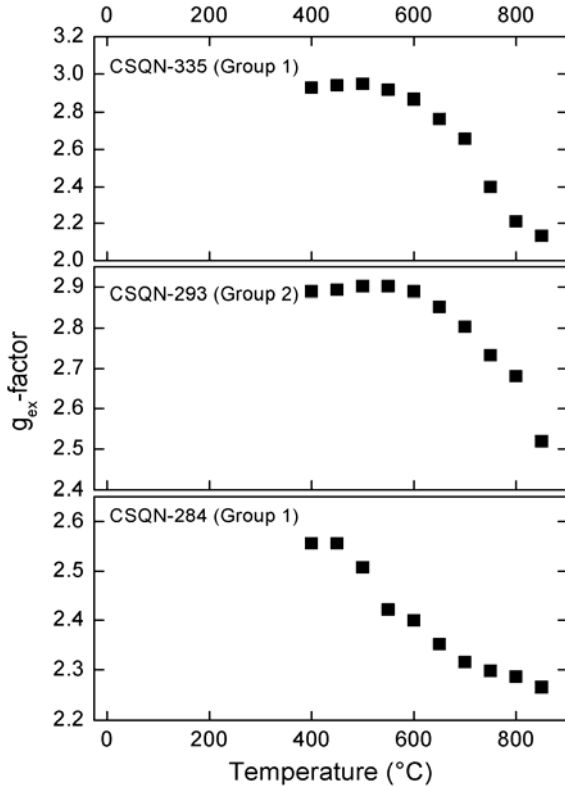


Fig. 4 g_{ex} factor in function of the temperature of reheating for the samples CSQN335, CSQN293 and CSQN284.

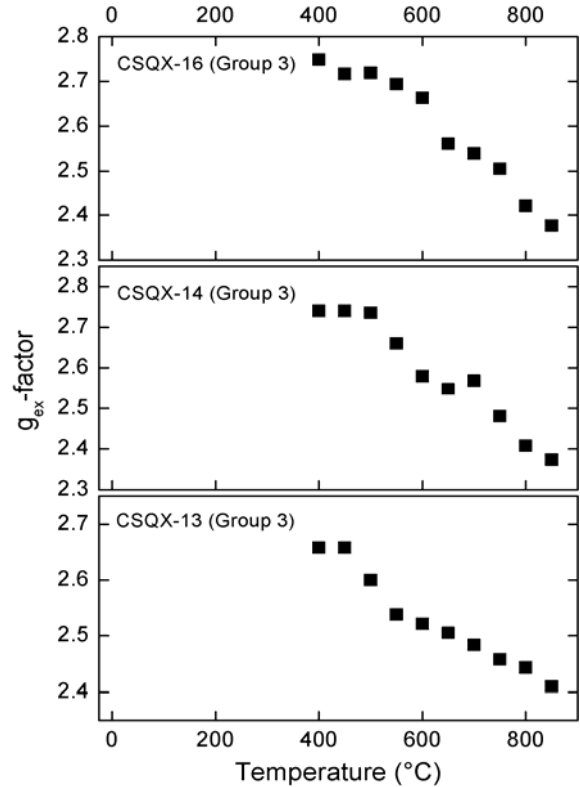


Fig. 6 g_{ex} factor in function of the temperature of reheating for the samples CSQX16, CSQX14 and CSQX13.

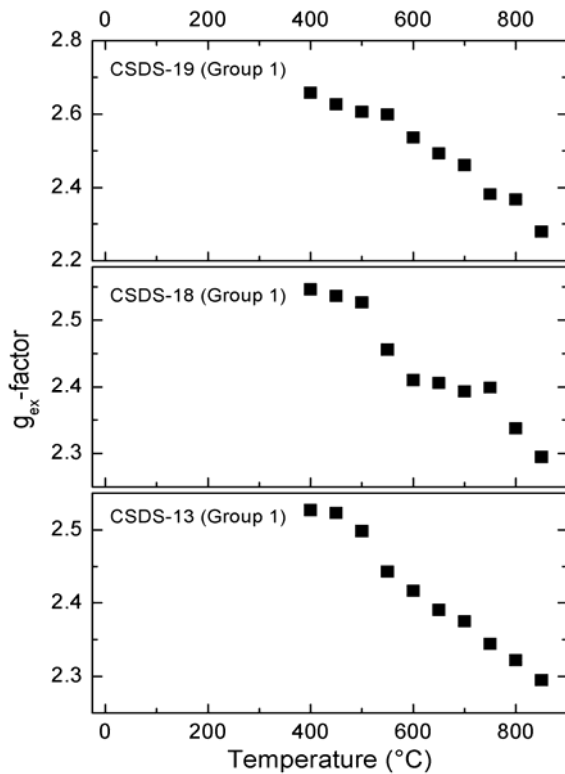


Fig. 5 g_{ex} factor in function of the temperature of reheating for the samples CSDS19, CSDS18 and CSDS13.

4. Conclusion

Neutron activation analysis was extremely important for the determination of several chemical elements, found in lower concentrations in the archaeological ceramics. The results obtained in this work allow concluding that the analytical method is appropriate for this study, showing good precision and accuracy. In 104 Marajoara archaeological ceramic samples, the elementary concentrations of Ce, Cr, Cs, Eu, Fe, Hf, K, La, Lu, Na, Sc, Tb, Th, U and Yb were used.

The interpretation by means of multivariate techniques showed three chemically distinct groups of Marajoara ceramics. In two groups formed, there was mix of ceramics from MAE and the ceramics from Marajó Museum. And, a third group formed only with contemporary ceramics.

The knowledge of the ceramic firing temperature was not sufficient to differentiate the ceramics of MAE, Marajó Museum and contemporary ceramics of

the Marajó island, once that it presented similar temperatures. This could indicate that the firing temperature did not change along the years. It is necessary to analyze these ceramics by other analytical techniques and carry out comparisons between the results. The next analysis includes X-ray diffraction for study mineralogical composition and also dating of the ceramics, using more numbers of samples.

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