

Study of the firing temperature of Marajoara archaeological ceramics by electron paramagnetic resonance associated to INAA data

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ABSTRACT

The Marajo Island is the largest island of the Marajo 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 1350 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 São Paulo and samples collected in the Marajo 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 no statistical differences in the firing temperature between the samples from Archaeology and Ethnology Museum and the samples collected in the Marajo Island.

Keywords: Marajoara ceramic, Marajo Island, INAA, EPR, multivariate statistical analysis.

1. INTRODUCTION

The Marajó archipelago is located in the Pará State and east part of Amazon. With 1200 islands registered, the largest of them is Marajó island which have 50.000 km², being also the largest fluvial island in the world [1, 2].

Archaeological research in Marajo begun in the decade of 1940, from the works of Meggers and Evans [3]. Six consecutive archaeological phases were established as a proposed hypothesis to the origin of the Marajoara society. The first phase, known as phase *Ananatuba*, inhabited the island between 1500 and 1000 BC. The second, *Mangueiras*, held between 1100 and 900 BC. Although there are no reliable data for the third phase, *Acauã*, it is believed that occupied the island around 1000 BC. The fourth phase, *Formiga*, inhabited the island between 0 and 800 BC. The Phase *Marajoara* occupied the island from 400 to 1350 BC and the sixth and last phase, *Aruã*, succeeded Marajoara phase [2].

Archaeological records show that on the island of Marajo and prior to the Marajoara phase, there were small towns, which lived from 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 a few decorative details. After 400 AD, there were societies organized on the island. 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, 4].

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 artefacts. 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 [1, 3].

Marajoara people also built on Marajo a lot of hills, called stiff. Over these elevations homes and cemeteries were built and a large quantity and variety of ceramics were found. Marajoara phase reached its apogee in his archaeological artifacts, which show exuberance and variety in decoration. The large amount and high quality of these materials had been characterized Marajoara society as complex, given its specialization and highly preserved stratigraphy [5].

Therefore, systematic research to obtain information about the distribution, activities and interactions of the Marajoara culture populations, as well as to determine the nature and numbers of different occupations types is relevant to better understand this society [5]. 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. Importantly, such information can help archaeologists to better understand the lifestyle of complex forms of development of ancient societies, serving as support for their works [6].

The research area that studies archaeological artifacts by means of physical and chemical techniques is named Archaeometry [7]. In an empirical form, the archaeometry studies had been begun in 1795, when Martin Klaproth determined the chemical composition of ancient objects, such as glasses and coins [8]. In the same way, by means of analytical techniques, Humphry Davy, Karl Gobel, Michael Faraday, Friedrich Kekulé, J. Berzelius, Louis Pasteur, among others, over the time also provided significant contributions to the History and Archaeology [9].

In the XIX century, sporadically, approaches physics and chemistry begun to be applied to the analysis of archaeological ceramics, due to its extreme durability [1]. 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, making also a form of investigation of the originality of artistic works [10].

In this perspective, the achievements in the field of the Archaeometry in the XVIII and XIX centuries allowed to create a close relationship between the humanities and exact sciences, intensifying in the last 50 years [6, 11]. The chemical composition, mineralogy and age are the main parameters investigated in Archaeometry [6, 12]. However, little focus has been given to the technology employed in the ceramic's production, as firing temperature, hardness, density, etc 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, the chemical elementary concentrations of ceramics were determined and submitted to multivariate statistical analysis. The goal was to determine differences in its chemical composition, once this fact indicates different sources of raw material used in the manufacture of artefacts. From the different groups formed, samples belonging to the groups were chosen to determine their firing temperatures by Electron Paramagnetic Resonance (EPR).

2. EXPERIMENTAL

2.1 Neutron Activation Analysis

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 [13]. 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.

2.2 Electron Paramagnetic Resonance

In archaeometric studies, Electron Paramagnetic Resonance (EPR) can be employed for dating, as well as, determining firing temperature of ceramics, among other applications [14, 15]. To determine firing temperature the EPR spectrum of a ceramic it is necessary analysis for different temperatures looking for changes that can be correlated to the temperature. 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 equation 1 (resonance condition).

To improve spectrometer sensitivity, the EPR spectrum usually is observed as the first derivative of the absorption spectrum as a function of the magnetic field, B . 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 [16].

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

where:

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

2.3 Sample preparation and analytical procedure for INAA and EPR

Initially, for INAA analysis, 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 [17].

The results showed that, apart from preventing contamination, it is not also necessary to destroy the fragment [18,19]. Then, with the objective of assuring the chemical homogeneity, about 5 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 [18].

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 $8.92 \times 10^{12} \text{ n.cm}^{-2}\text{s}^{-2}$. 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.

For EPR analysis, each sample was divided into 11 aliquots of 70mg 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, 450, 500, 550, 600, 650, 700, 750, 800 e 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 e 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 9,4GHz (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 4000 G was used in two minutes and the constant of time was 100ms. The spectra were recorded at room temperature.

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. P.P. Hilbert and Felisberto Camargo, in the 50's, on the archaeological sites *Furinho*, *Cuieiras*, *Pascoval dos Mello*, *Pacoval* and *Fazenda São Marcos*.

These pieces, originally, belonged to Paulista Museum and, in 1989 was incorporated in to the Marajoara ceramic MAE, the collection of which is part of the Amazon Collection [20]. The remaining 45 samples were

provided from Marajó Museum and 15 contemporary samples produced and marketed in the Marajo 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 e Zn were determined by NAA in 8 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 [21]. Co was eliminated due the contamination by means of tungsten carbide during the sample preparation [17].

The ^{65}Zn , with peak of energy 1115,55 keV and the ^{182}Ta with peak of energy 1121,30 keV also were eliminated, because they suffer interference in the gamma rays spectrum of the peak 1120,5 keV do ^{46}Sc , resulting in the reduction of its precision [22]. 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 \log_{10} 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 first is explained by the fact that a normal logarithmical distribution of the elements exists. The other is the difference magnitude between elements, which it 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,

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

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, 5 sample outliers were found and removed from the data set. Due to space limitations, this work shows only the results of elementary concentrations for 109 samples of ceramic fragments analyzed, as well as, for values of *Mahalanobis* distance and outlier's removal.

5.1 Cluster analysis

The cluster analysis is a multivariate statistical technique named of unsupervised [25]. 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 5 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 Marajo island belong to the group 3. *Figure 1* 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.

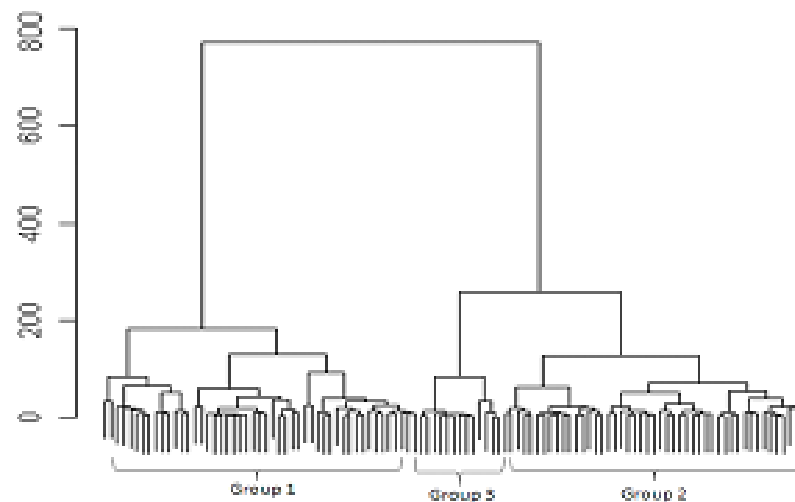


Figure 1. Dendrogram of the ceramics samples using square Euclidean distance and Ward's method

5.2 Principal component analysis

Principal component analysis (PCA) is a multivariate statistical technique. It is mean that there is knowledge with relation to the groups formed by samples. The principal objectives of the principal component analysis are the reduction of the dimensionality of the data, the achievement of interpretable combinations of the

variables [26]. Figure 2 shows that no occurred variation in relation to the number of groups formed by dendrogram. The group 3 it is a little bit distance of the other groups.

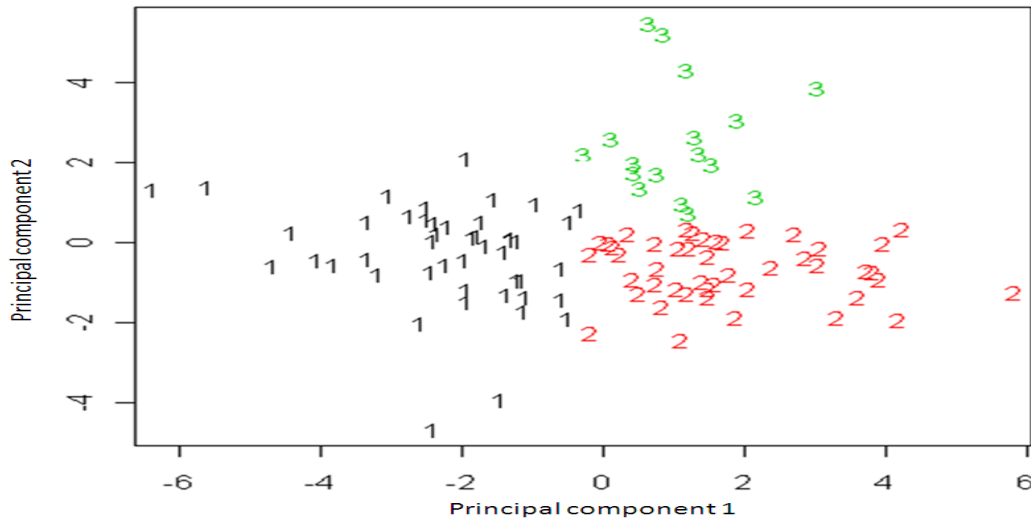


Figure 2. Principal component analysis 1 versus principal component 2 of the ceramic samples

After the separation of the samples in groups, from of its chemical compositions, 9 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, 5 samples were chosen in the group 1, 1 sample of the group 2 and 3 samples of the group 3.

Figure 3, a Boxplot [27] shows that the samples of the group 1 present firing temperatures that oscillate between 500 e 600°C. In the group 2, the unique sample analyzed present firing temperature about 650°C. And in the group 3, the samples present firing temperatures that vary between 510 e 650°C. Preliminary, it is possible to observe that the firing temperature of the group 1 is smaller than the firing temperature of the groups 2 e 3. In the future, the number of samples will be increased and nonparametric tests will be carried out for comparison of the medians among firing temperatures.

Figures 4 to 12, show all the plots with the obtained firing temperature determined for each ceramic sample analyzed. In these Figures, we see a plateau θ region, and a decreasing region. The firing temperature is considered as been the first point after the plateau θ region. This meaning that this in-laboratory annealing was higher than the previously firing temperature carried out by the ancient's cultures.

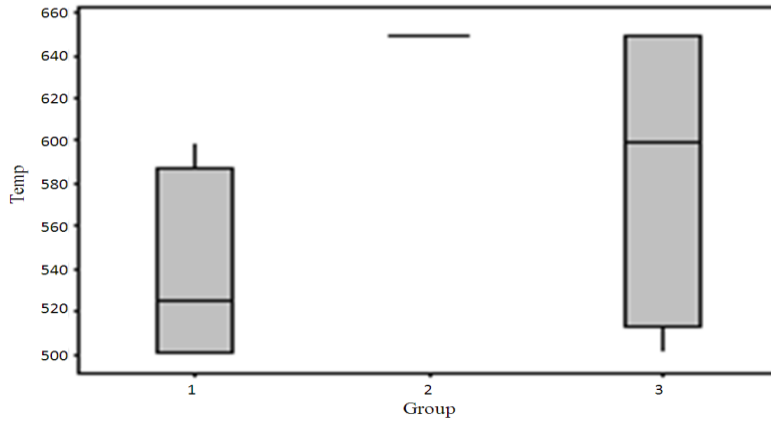


Figure 3. Boxplot for the firing temperatures of Marajoara ceramic separated by compositional groups.

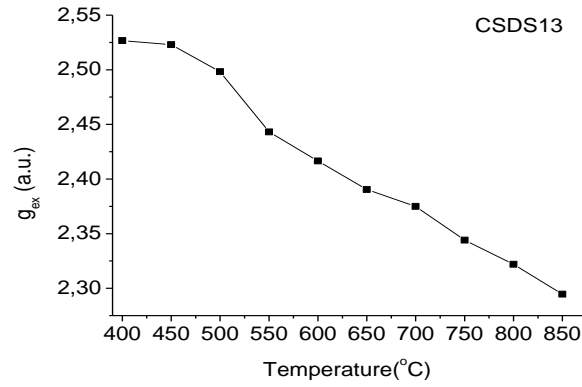


Figure 4: Group 1. Firing temperature: 500°C

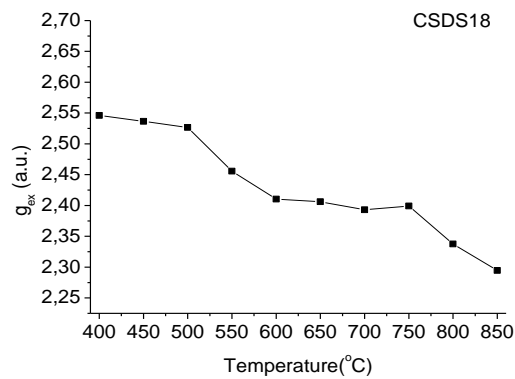


Figure 5. Group 1. Firing temperature: 550°C.

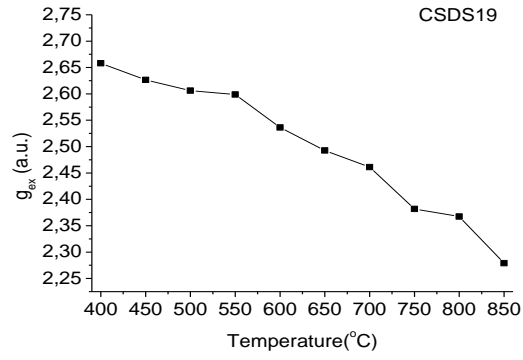


Figure 6. Group 1. Firing temperature: 600°C.

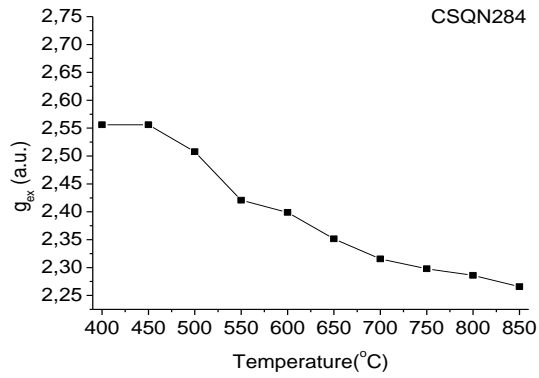


Figure 7. Group 1. Firing temperature: 500°C.

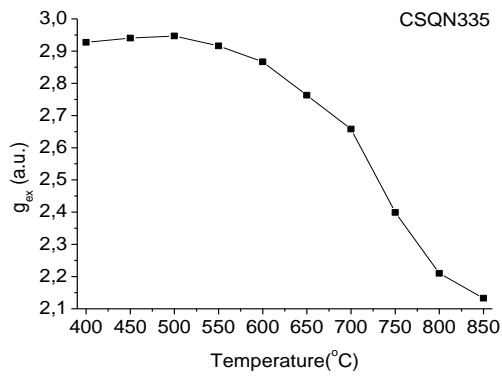


Figure 8. Group 1. Firing temperature: 500 < 650°C

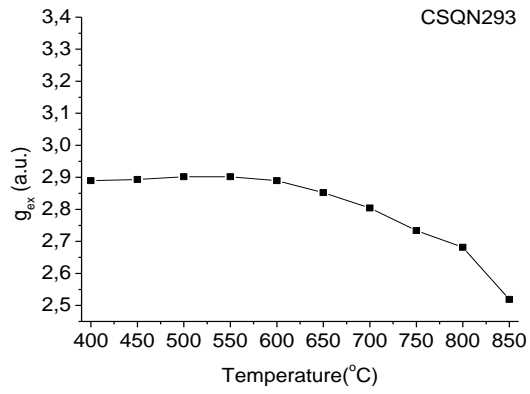


Figure 9. Group 2. Firing temperature: 650°C.

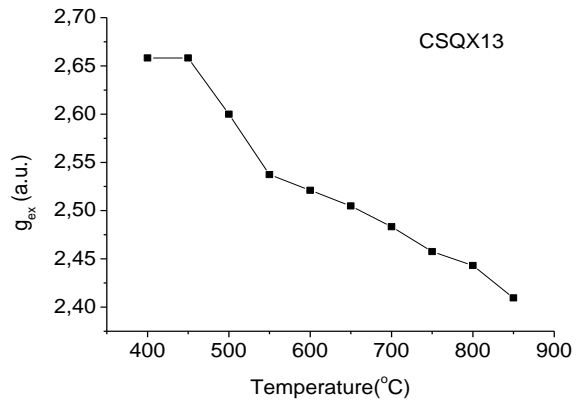


Figure 10. Group 3. Firing temperature: 500°C

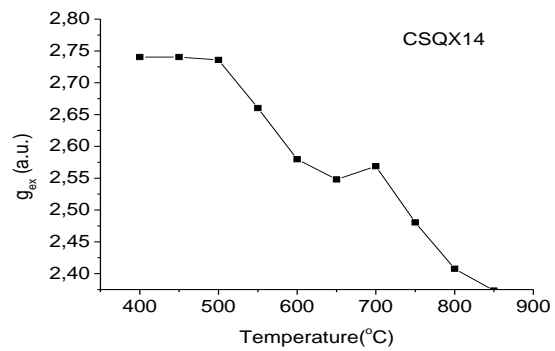


Figure 11. Group 3. Firing temperature: 550°C

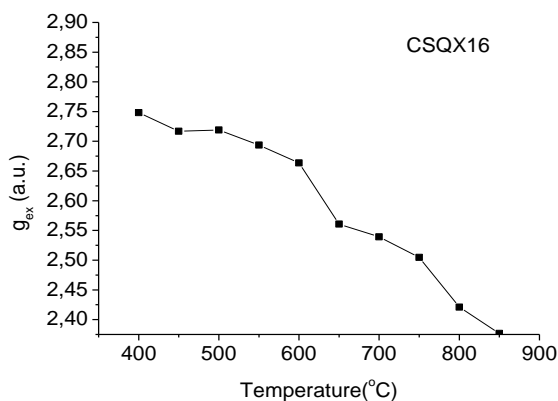


Figure 12. Group 3. Firing temperature: $500 < \text{temperature} < 600^{\circ}\text{C}$

4. CONCLUSIONS

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 e Yb were used.

The interpretation by means of multivariate techniques showed the existence of three distinct groups of Marajoara archaeological ceramics, indicating three distinct raw material sources used in the production of these artifacts. In two groups formed, there was mix of ceramics from MAE and the ceramics from Marajó Museum and a unique raw material source was used for production of the ceramics of each other of these groups. And, in the third group, there are only contemporary ceramics. From the chemical point of view, these ceramics are quite different of the ceramics belonging to the two other groups. It's necessary to associate this information with archaeological interpretations, for a more comprehensive understanding.

The knowledge of the ceramic firing temperature was not sufficient to differentiate the ceramics of MAE, Marajó Museum and the ceramics collected in the Marajó island, once that it presented similar temperatures. This could indicate that the firing temperature didn't change along the years. It's necessary 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 number of samples.

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