

Chemical characterization of Marajoara pottery by INAA

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Abstract

On the Amazon River there lies the world largest fluvial island known as Marajó where the descendents known as Marajora people lived. Many of these people were skilled artisans who have passed their art from father to son, generation after generation for centuries. The ceramic art is one of the most highly developed in the hemisphere, it is even extravagant and there is no doubt that the society maintained a relatively high level of complexity during near 1000 years in an environment that today supports only a sparse population dedicated mainly to cattle raising. This beautiful pottery is found in museums and private collections in Europe and North America as well as in Brazil.

The purpose of this work was to find a fingerprinting of the Marajoara pottery using instrumental neutron activation analysis, INAA, to do a characterization of the ceramic. To accomplish this task, 57 original ceramic fragments of Marajoara pottery were submitted to chemical analysis to determine Co, Cr, Cs, Eu, Fe, Hf, K, La, Lu, Na, Sc, Ta, Tb Th, Yb, Zn and U with the aim to find pottery chemical characteristics which have a solid database of trace elements to contribute to identifying fraud and illegal commerce of this pottery. First the data set was transformed into \log_{10} to compensate for the large difference of magnitude between major and trace elements. After that, the data were analyzed using the Mahalanobis distance using the lambda Wilks as critical value to identify the outliers. The similarities among the samples were studied by means of discriminant analysis. A stopping rule for the identification of redundant variables and the selection of variable subsets, preserving multivariate data structure for forward stepwise discriminant analysis is presented.

Introduction

The last decade has seen a surge in ceramic studies worldwide, covering such important topics as ceramic production,^{1,2} technological change,^{3,4} ceramic use and distribution⁵ and social boundaries.⁶ Recent studies have also examined the widespread association of potters with castes or castelike groups, ethnolinguistic units, or particular religious backgrounds.⁶ Several studies have discussed the relationship between daily life and vessel size.⁷

The application of chemical analytical techniques to archaeological questions has a long history that extends back to the late 1700s.⁸ Many of the earlier investigations dealt with the compositional characterization of objects to elucidate aspects of their properties, such as color, texture, plasticity, shrinkage and strength. The physical properties of fired ceramic material, most broadly conceived, are those that provide evidence of manufacture and those that characterize the appearance or functional capabilities of the finished piece. Mineralogical properties or chemical composition constitute a more homogeneous set of variables than do physical properties.

The chemical properties of ceramic materials typically are discussed in terms of major, minor and trace elements. The chemical analysis is based on the assumption that ceramics of the same production place are chemically and mineralogically similar while being distinguishable from ceramics of other production places.⁹ The concentration levels of a number of major elements are usually similar for different samples of sand or clay. For this reason it is necessary to consider the chemical composition and concentration levels of trace elements in the materials from which the pottery was manufactured.¹⁰ Several analytical techniques can be used to determine trace elements, however, INAA employing γ -ray spectrometry seems to be the most suitable analytical technique because in its purely instrumental form (INAA) is conceptually a simple analytical method, involving only two, completely separate processes, excitation and measurement. The excitation of the sample by exposure to neutrons as well as the subsequent measurement of the induced activity by a suitable detector are processes based on well-known physical laws, and the content of an element in the sample may be directly compared with an accurately known quantity of the same element. On the other hand, the method determines several elements simultaneously with high sensitivity, accuracy and precision.^{11,12}

In this work Co, Cr, Cs, Eu, Fe, Hf, K, La, Lu, Na, Sc, Ta, Tb Th, Yb, Zn, and U in 57 Marajoara pottery were analyzed using INAA. The elemental concentration data were studied by means of Mahalanobis distance to determine the outliers, and by forward stepwise discriminant analysis to establish the elemental fingerprinting.

Marajoara pottery

The name Majoara comes from Marajó Island on the Amazon river delta area in Brazil. The Island has about 50,000 km². In the 1950's, Meggers and Evans^{13,14} were the first to carry out a scientific study of Marajoara society. They were impressed by the highly elaborated ceramics and the monumentality of the mounds. They suggested that the Marajoara people had migrated from the Andes because they did not believe that social complexity could

emerge in a tropical forest environment. However, today several studies have shown that the Marajoara culture originated locally, emerging from a process of cultural change that occurred within communities that inhabited the area 3,500 years ago. The discrepancy between the archaeologists is due to the low agricultural potential. A detailed assessment of agricultural potential by the American State Organization recognized four categories of soil based on drained, effective depth, texture, fertility and extent of erosion: I Good; II Average, III restricted and IV unsuitable for cultivation. None of the Marajó soils was identified as I and II. Eighty-eight percent, including the region occupied by the Marajoara, was classified as Type IV, signifying that only low to very low returns can be obtained even during the first year.¹⁵

Differences in topography, soil, and vegetation between the southwestern and the northeastern of the Island are due to both tectonic movements related to the opening of the Atlantic passage to the Amazon during the Cretaceous period and the process of sedimentation during the Quaternary. While the southwestern portion received high amounts of sediments from the Amazon River during the Pleistocene, the northeastern part, a shoreline of emergence developed in Holocene deposits with a much lower rate of sedimentation.¹⁶

On Marajó Island, the dominance of certain plant species such as palms, wild cotton, and several fruits, including a wild variety of pineapple, may indicate that prehistoric populations cultivated these plants for their consumption.¹⁷ Studies of bone and dental analysis indicated staple consumption of plant protein, studies showed dental wear consistent with consumption of gritty food, which had caused heavy dental abrasion.¹⁸

Radiocarbon dates place the period of major growth and expansion of Marajoara culture between the 5th and 14th centuries. The Marajoara style seems to be related to different regions within the Marajoara domain, as well as to different chronological periods. The archaeologists noticed that the urns were buried together with other ceramic objects, such as stools, figurines, tangas (pubic covers), plates, vases, miniatures and eventually some lithic objects such as stone axes and a variety of ornaments. Its ceramics belong to the Polychrome Tradition, characterized by highly complex ceremonial wares in form and decoration. Decorative technique involve slip, painting, incision, excision and scraping. The ceramics are tempered with ground potsherds (grog) and two plain types also define two different types of paste that can be found in all decorative types. Some pottery tempered with crushed ashes of a tree bark known as caraipé (*Licania scabra*) are also found. The use of caraipé as temper material was introduced in the Island A.D. 500. This material was used in the Amazon Basin towards the end of the first millenium and it is associated with Polychrome Tradition.¹⁹

Experimental

Sample preparation and standard

All 57 ceramic samples were analyzed via INAA. The ceramic powder samples were obtained by cleaning the outer surface and drilling to a depth of 2-3 mm using a tungsten carbide rotary file attached to the end of a flexible shaft, variable speed drill. Depending on the thickness, 3 or 5 holes were drilled as deep into the core of the pottery as possible without drilling through the walls.¹¹ Finally, these materials were dried in an oven 105°C for 24h and stored in desiccator.

Constituent Elements in Coal Fly Ash (NIST-SRM-1633b) was used as standard and IAEA-Soil-7, Trace Elements in Soil, was used as check samples in all analysis. These materials were dried in an oven at 105°C for 24 h and stored in desiccator until weighing.²⁰

Description of the method

Generally speaking, NAA method is based on the properties of nuclei, in which a neutron incident interacts with the target element nucleus. During the neutron bombardment of the sample a small fraction of the nucleus from each one of the sample constituting elements will be transformed into unstable radioactive isotopes that decay with a half-life characteristic. During the decay these isotopes emit gamma rays with energies that are characteristic of each element. The measure of these gamma rays permits the qualitative and quantitative determination of the sample elements.

About 100 mg of ceramic samples, NIST-SRM-1633b and IAEA-Soil-7 were weighed in polyethylene bags and wrapped in aluminum foil. Groups of 8 samples and one of each reference material were packed in aluminum foil and irradiated in the research reactor swimming pool, IEA-R1m, from the IPEN-CNEN/SP at a thermal neutron flux of about $5 \times 10^{12} \text{ n}\cdot\text{cm}^{-2}\cdot\text{s}^{-1}$ for 8h.

Two measurement series were carried out using Ge (hyperpure) detector, model GX 2020 from Canberra, resolution of 1.90 keV at the 1332.49 keV gamma peak of ⁶⁰Co, with S-100 MCA of Canberra with 8192 channels. K, La, Lu, Na, U, and Yb were measured after 7 days cooling time and Co, Cr, Cs, Eu, Fe, Hf, Sc, Ta, Tb, Th, and Zn after 25-30 days. Gamma ray spectra analysis and the concentrations were carried out using the Genie-2000 Neutron Activation Analysis Processing Procedure from Canberra.

Results and discussion

One of the basic premises for the use of a chemical composition analysis in ceramic samples is that the samples can be differentiated if the analytical technique has a good

sensitivity, precision and accuracy for the analyte in interest. If an element is not measured with good precision it may obscure real differences in concentration and the discriminating effect of other well-measured elements tends to be reduced. These small differences can be used to form groups of similar composition because manufactured artifacts with a determined raw material source will be more chemically similar than other ones, which were manufactured with different raw material.²⁰

NAA is a highly sensitive technique and it can determine the elements which are found in very low concentrations, such as ppm (parts per million) or ppb (parts per billion). This characteristic has contributed for NAA being recognized as the preferred technique by various researchers who analyzed samples for trace levels. Due to the fact that NAA is a highly sensitive technique, it is possible to quantify the small differences that may occur inside a geologic formation. Then the determination of the concentration of various elements for the trace level, which differs in their chemical properties, will tend to produce a highly specific “fingerprinter” for a source of raw material.

Another parameter related to the analytic properties of methods, is accuracy and precision. Accuracy is related to the real concentration of the sample element, a value that does not depend on the method used. The accuracy of the analytical methods is determined by means of the reference materials, where the concentration was determined by various analytical methods. NAA has great accuracy for various elements.^{21,22}

The determination of the analytic precision is of great importance and must be quantified. Precision is related to the capacity that the method has to reproduce the same result. The precision limitations can result from the sample inadequate preparation, either due to contamination with the same element is being determined or due to inhomogeneities problems.

The determination of all parameters in the analyses must be measured and quantified to make corrections before applying the method in real samples because these small variations may affect the power capacity to distinguish between the sample groups.

In this work, to evaluate the analytical process and to establish the chemical elements which can be used in the data interpretation, the elemental concentrations for the reference material IAEA-Soil-7 were statistically compared with the data found in our laboratory. The analytes with relative standard deviation, RSD, less than 10% were used.²³ All possible interference potentially occurring in gamma-ray spectrometry were considered and checked. A possible source of error in the determination of REE elements is the presence of significant amounts of fissile nuclides (²³⁵U and ²³³U). As it is well known, REE nuclides, especially among the light REE elements (La to Sm), are produced both by activation and by fission. In

the present work no interferences were expected in the samples because of the very low uranium fission in the determination of La and Ce. This interference is negligible when the uranium concentration does not exceed 5 ppm.¹¹ Although Co had RSD less than 10%, it was not included in the data set because the concentration can be affected by tungsten carbides files.²⁴ The determination of Zn is not reliable as a consequence of a strong γ -ray interference by ⁴⁶Sc and ¹⁸²Ta. Using this criteria, the elements Na, K, Lu, U, Yb, La, Th, Cr, Cs, Sc, Fe, Eu, Hf, and Tb were used in the subsequent data analysis.

Table 1 shows the concentration results for the ceramic samples manufactured by Marajoara population. None of the elements considered presented missing values. In all, 57 samples were analyzed.

Initially, the results were transformed to \log_{10} to compensate for the large differences of magnitudes between the measured elements for the trace level and for the larger ones. The \log_{10} transformation of data before a multivariate statistical method is common. One reason for this is a belief that within the manufacturing raw material, elements have a natural log-normal distribution and that normality of the data is desirable. Another reason is that a logarithmic transformation tends to stabilize the variance of the variables and would thus give them approximately equal weight in an unstandardized multivariate statistical analysis. All individual determinations in each data set were tested for discordant results. The Mahalanobis distance, D_i , is suggested by many authors as a method for detecting outliers in multivariate data.²⁵ For each of the n observations (samples) in p variable data set, a distance value D_i was calculated. Let \bar{x} be the sample mean vector and let S be the sample covariance matrix,

$$S = \sum_{i=1}^n (x_i - \bar{x})(x_i - \bar{x})^T / (n-1) \quad \text{and} \quad D_i = \sqrt{\{(x_i - \bar{x})^T S^{-1} (x_i - \bar{x})\}}$$

for $i = 1, \dots, n$, where $(x_i - \bar{x})$ is the difference vector between the measured values in one group and the mean values of the other group. Wilks²⁶ suggested the use of

$$p(n-1)^2 F_{p, n-p-1} / n(n-p-1 + p F_{p, n-p-1})$$

to calculate the critical values for D_i when searching for a single outlier. Wilks used the so called scatter ratios to search for outliers in multivariate normal data. To search for a single outlier, the author calculates a scatter ratio R_i ,

$$R_i = |A_i| / |A| \quad \text{where} \quad A = \sum_{i=1}^n (x_i - \bar{x})(x_i - \bar{x})^T, \quad \text{and} \quad |A| = \text{determinant}(A), \quad A_i \text{ is calculated as for}$$

A with observation i eliminated from the sample. The most outlying observation is the one with the smallest scatter ratio R_i , where $R_1 = \min\{R_i\}$, i.e. the observation whose removal leads to the greatest reduction in $|A|$. This procedure at confidence level of 95% was applied at data set. As it can be seen in Table 1 the D values were calculated for all samples as shown in the Table. When the D calculated in the sample was higher than D critical value the sample was eliminated and calculated a new D , i.e., in Table 1 the samples eliminated were 8, 17, and 43. The procedure ended when the D critical was higher than D calculated. Then three samples were outliers and eliminated.

The application of statistic methods in experimental results aims to classify and to order the objects related to each other in function, exclusively from its chemical composition. Various multivariable statistical techniques have been developed. However, discriminant analysis maximizes the difference between two or more groups and it is based on the fact that principal variance-covariance matrix is an accurate representation of the total variance and covariance.

With the purpose of studying the similarities and the dissimilarities between the ceramic, the results were submitted for discriminant analysis. The intention was to group similar samples according to their characteristics (variables). The purpose, therefore, is to consider several simultaneously related variables, all of them having equal importance in the beginning of the analysis. Another consideration is that the data must follow a normal distribution. Generally speaking, if each studied variable follows a normal distribution, the resulting distribution of the group of all the variables will also be normal.

Figure 1 shows the discriminant function 2 versus discriminant function 1 for all the studied ceramic samples. The results showed that the separation is 100%. In other words, there is a clear difference in the chemical composition between the ceramics.

In general when characterizing samples by means of chemical elements for the first time, such as ceramic samples, the analyst measures a large number of variables, many of which may not be very informative. In fact, some may even be unrelated to the issue at hand, and blur the picture instead of making it clearer. In subsequent studies the analyst may wish to measure fewer variables for several reasons such as to save time, in cases where measurement time is important or in order to reduce cost or effort; etc. Therefore, hopefully those variables which are most relevant will be determined without losing essential information and the less productive information will be removed.

A procedure for the identification of redundant variables for the Marajoara ceramic is presented here as well as the selection of variable subsets preserving multivariate data

structure. Forward stepwise discriminant analysis, was used by selecting those variables which are in some sense adequate for characterization purposes.

Assuming that p variables have been measured on each of n samples, and that the essential dimensionality of the data to be used in any comparison, is k , then the procedure is based on the fundamental partition equation:^{27,28}

$$T = B + W$$

where T is the matrix of the total variation in the data set, consisting of B , variation between the groups, and W , the variation within the groups. A formal discussion of these matrix components is given by Cooley and Lohnes.³⁰ By using these relationships one can use minimum trace- W , determinant- W , or maximization of trace $W^{-1} B$.³⁰ In this paper the minimum trace- W was used to determine which variable is important by comparison with the critical value at 95% confidence level. A criterion for assessing a particular variable x_{p+1} increases the separation provided by the variables x_1, \dots, x_p which is obtained by means of an analysis of covariance, treating x_{p+1} as the response, and x_1, \dots, x_p as covariants. Then x_{p+1} provides significant additional information at level α if the partial F statistic for the value of the $(p+1)$ th variables is given by³¹

$$F = \frac{n - k - p}{k - 1} \left(\frac{A_p}{A_{p+1}} - 1 \right)$$

where A_p is the value of Wilk's Lambda for MANOVA based on the first p variables³¹ with F distribution with $(k - 1)$ and $(n - k - p)$ degrees of freedom if the $(p + 1)$ th variable does not bring about a significant improvement in discrimination among the groups.

In others words, for each variable, the F statistic is computed. The variable corresponding to the lower of these statistics is first selected. Variables are then added one at a time based on an examination of partial F statistic. The procedure terminates when none of the selected variables can be excluded, and no further variables can be included.

The procedure was applied on the data set (Table 1). The partial F statistic involved in the data base for forward selection procedure is shown in Table 2. The variable with the small partial F statistic was selected. The smallest value in the first column was 1.1×10^{-11} corresponds to the variable Eu (for $Yb = 6.9 \times 10^{-10}$, $La = 2.9 \times 10^{-7}$, $Lu = 7.9 \times 10^{-6}$). For the sake of discussion, this is compared with the critical value at 95% of confidence level (0.3761). Hence Eu can be selected. The smallest partial F statistic in the second column is 0.0001 and it corresponds to the variable Tb. The comparison of this with the critical value at 95% of confidence level (0.3761) leads to the selection of Tb. The procedure terminates at step 8 when the smallest partial F statistic is higher than the critical value at 95% of confidence level (0.3761). Thus the variables selected are Lu, U, Yb, Cs, Fe, Eu, and Tb.

To determine how well these subsets capture all the information of the Marajoara pottery, Figure 1 shows the plot for discriminant function 2 versus discriminant function 1 for all the variables (Na, K, Lu, U, Yb, La, Th, Cr, Cs, Sc, Fe, Eu, Hf, and Tb). Figure 2 shows the plot of discriminant function 2 versus discriminant function 1 using the variables selected (Lu, U, Yb, Cs, Fe, Eu, and Tb). The comparison of Figures 1 and 2 confirms that discriminant analysis based on seven variables produce similar results to a discriminant analysis using all variables.

Conclusion

The procedure presented in this paper provided a useful descriptive tool to do a chemical characterization of the Marajoara pottery. The results showed that the elemental fingerprinting of the Marajoara ceramic are Lu, U, Yb, Cs, Fe, Eu, and Tb . The paste composition of the pottery analyzed was diverse, reflecting both spatial and temporal differences in clay selection and in the kind and amount of temper used.

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Table 1- Results for Marajoara potteries in µg/g, unless otherwise indicated, and Mahalanobis distance

Sample	Na, %	K(%)	Lu	U	Yb	La	Th	Cr	Cs	Sc	Fe(%)	Eu	Hf	Tb	D_1	D_2
1	0,45	2,40	0,70	3,90	3,80	62,60	18,00	87,30	9,70	18,90	5,70	2,30	6,10	2,10	12,41	15,24
2	0,50	1,40	0,60	3,60	3,50	48,90	17,60	93,20	7,90	18,60	4,30	1,60	6,40	0,90	11,82	13,10
3	0,37	1,80	0,60	3,90	4,10	55,20	19,80	101,20	9,00	20,60	5,50	2,00	5,90	0,90	8,96	10,80
4	0,54	2,70	0,70	4,70	3,90	59,50	18,70	86,10	9,20	18,90	5,00	2,10	5,80	1,90	12,82	13,24
5	0,47	2,10	0,50	3,70	3,50	50,00	16,70	84,50	8,80	17,90	5,00	1,90	5,00	1,30	9,95	12,06
6	0,61	2,30	0,50	3,40	3,30	48,00	17,30	88,40	7,00	17,00	5,10	1,40	6,50	0,70	11,06	10,95
7	0,47	2,00	0,50	2,90	3,30	50,00	16,40	82,00	8,30	17,70	4,80	1,80	4,80	0,60	16,31	16,40
8	0,57	2,20	0,50	3,30	0,50	49,90	17,90	85,70	8,90	17,60	6,20	1,80	6,20	0,90	31,18	
9	0,39	1,60	0,50	4,50	3,40	47,50	17,30	85,30	7,80	18,30	5,00	1,80	5,60	1,10	12,47	12,33
10	0,55	1,90	0,60	4,40	3,90	56,90	20,50	95,40	10,60	20,10	6,00	1,90	6,90	1,10	8,37	8,49
11	0,54	4,20	0,60	3,90	4,00	57,70	19,30	91,40	9,60	20,40	4,90	1,80	6,90	1,30	16,62	16,65
12	0,66	3,20	0,60	3,70	3,90	59,00	19,40	96,40	9,50	19,80	4,90	1,90	7,00	1,10	6,26	6,01
13	0,39	2,21	0,70	4,90	5,00	64,50	19,20	93,10	7,50	18,30	5,50	2,30	9,80	1,50	10,37	14,85
14	0,39	2,21	0,60	4,50	3,30	55,90	20,00	91,30	8,80	19,80	5,40	1,70	5,80	1,20	10,15	11,59
15	0,45	2,21	0,60	4,10	4,20	69,60	21,80	107,10	9,00	21,60	5,70	2,40	6,70	1,50	10,51	16,83
16	0,36	3,20	0,70	4,50	4,00	57,70	19,60	93,10	8,10	19,60	5,30	1,80	6,60	1,20	9,15	11,23
17	0,50	2,21	0,50	3,20	3,30	47,60	17,90	83,10	91,40	18,40	3,90	1,60	6,40	0,50	49,96	
18	0,50	1,60	0,60	4,40	4,50	61,10	17,10	90,40	8,20	17,10	5,10	2,00	8,70	1,50	10,56	10,05
19	0,40	1,50	0,70	3,80	5,00	66,20	18,00	89,10	7,20	17,10	5,10	2,40	8,70	1,60	17,85	18,21
20	0,50	2,30	0,60	4,70	3,60	61,90	19,60	93,90	10,00	19,20	5,50	1,90	6,70	1,10	4,53	6,94
21	0,40	2,30	0,60	4,90	4,10	63,30	19,30	98,10	11,10	20,30	5,40	1,90	6,90	1,10	5,56	6,09
22	0,40	2,10	0,60	4,30	4,00	65,50	19,40	103,80	9,70	19,90	5,60	2,20	7,40	1,10	6,85	9,03
23	0,50	2,10	0,60	4,30	4,10	60,10	20,10	102,30	9,70	19,80	4,80	1,70	7,90	1,10	7,34	7,71
24	0,30	1,60	0,60	3,70	4,70	56,90	18,60	97,10	8,70	19,10	4,40	2,30	6,60	1,50	12,37	13,13
25	0,50	2,20	0,50	4,30	3,90	54,10	19,30	88,00	10,00	18,70	5,80	1,70	9,30	1,20	14,41	14,13
26	0,40	2,60	0,70	3,90	4,50	65,20	21,60	99,30	10,90	20,80	5,30	2,10	7,40	1,60	9,25	9,82
27	0,50	1,80	0,60	3,00	4,10	52,80	17,80	88,60	9,90	17,80	5,80	1,60	10,60	1,10	13,54	14,65
28	0,40	1,90	0,60	4,80	3,90	60,40	20,00	95,90	9,20	19,30	5,30	2,00	7,70	1,40	3,82	5,54
29	0,50	2,60	0,60	3,80	3,60	46,80	16,60	75,20	7,00	15,90	5,20	1,50	9,80	0,60	18,68	18,19
30	0,50	2,20	0,50	4,70	3,40	48,50	17,40	86,50	7,30	17,00	5,10	1,50	6,80	0,80	8,63	8,72
31	0,50	2,10	0,50	4,00	3,30	51,80	19,80	100,00	8,60	19,00	5,00	1,40	6,20	0,80	14,61	14,23
32	0,50	2,20	0,50	4,50	3,90	58,20	18,80	97,90	8,20	18,70	4,80	1,70	6,90	1,20	9,17	8,79
33	0,50	2,40	0,60	5,70	4,40	60,10	20,70	97,60	11,20	20,40	6,00	2,00	6,60	1,10	9,00	15,18
34	0,40	2,20	0,50	3,40	3,90	56,40	20,30	93,10	10,40	19,40	5,60	1,90	7,00	0,90	11,40	12,50
35	0,40	2,50	0,50	5,00	4,30	58,90	19,90	95,30	9,20	18,70	5,00	1,90	8,70	1,00	11,04	11,21
36	0,60	3,20	0,50	3,50	4,00	55,60	18,40	89,20	8,90	18,60	5,10	1,80	7,40	0,80	5,31	6,00
37	0,50	2,70	0,50	4,50	3,90	52,70	20,80	95,10	7,50	18,80	4,90	1,80	6,70	1,00	14,33	15,04
38	0,60	1,60	0,50	3,10	4,10	58,70	16,90	88,60	8,20	17,80	5,30	1,90	6,40	1,40	12,60	12,36
39	0,20	1,10	0,70	3,00	4,10	44,10	16,10	91,30	8,00	17,00	4,20	1,80	7,10	1,20	27,61	28,60
40	0,40	1,50	0,50	3,60	3,60	53,50	15,90	83,50	7,70	17,30	4,90	1,60	6,30	0,90	11,48	11,60
41	0,50	1,70	0,50	3,60	4,00	57,60	17,00	85,60	9,70	18,00	5,50	1,90	8,00	1,00	9,45	9,22
42	0,40	1,50	0,50	3,80	3,40	43,90	16,20	86,70	6,70	17,30	6,50	1,60	5,50	1,10	18,72	21,78
43	0,40	1,30	0,60	3,10	0,50	59,10	19,00	101,20	5,90	19,70	6,10	1,90	8,90	1,30	36,03	
44	0,60	1,80	0,60	3,80	4,20	59,80	18,90	97,30	10,90	19,80	6,10	1,90	6,40	1,20	6,78	8,90
45	1,20	1,80	0,50	4,20	3,30	53,40	18,20	101,30	10,30	19,60	4,70	1,70	6,70	0,90	9,15	14,36
46	0,40	1,90	0,60	5,00	3,70	60,80	18,20	101,20	11,30	20,50	5,10	2,00	7,00	0,60	21,86	22,13
47	0,50	1,60	0,50	4,30	3,80	51,60	17,80	96,90	9,10	18,30	5,80	1,70	7,80	0,90	7,83	7,37
48	0,50	2,40	0,50	3,00	3,20	49,90	16,20	72,50	7,60	16,60	5,10	1,70	6,90	0,90	15,28	16,84
49	0,50	3,10	0,60	3,00	4,00	51,90	17,50	88,60	9,20	17,80	6,00	1,60	10,30	0,80	10,55	10,16
50	0,40	2,50	0,60	3,60	4,30	62,40	19,10	103,20	9,70	21,00	6,40	1,80	6,80	1,00	11,88	17,92
51	0,30	2,00	0,60	4,60	4,00	61,00	18,20	90,60	10,40	18,30	5,90	2,00	7,30	0,90	7,54	9,59
52	0,50	2,50	0,60	3,50	4,10	52,50	18,00	91,10	8,90	18,00	7,00	1,80	9,40	1,10	10,90	10,95
53	0,60	2,30	0,60	3,30	3,20	52,40	18,80	93,40	8,20	18,00	6,30	1,80	8,00	0,90	8,00	19,28
54	0,50	2,90	0,60	3,60	3,40	50,70	18,90	93,90	10,20	18,60	6,40	1,60	8,10	0,50	11,58	12,52
55	0,40	3,10	0,60	3,50	3,90	58,40	19,80	99,60	11,60	20,60	6,10	1,90	8,20	1,10	8,15	8,59
56	0,70	2,50	0,60	3,20	3,70	57,20	18,80	91,10	10,50	19,20	5,60	1,70	7,40	0,40	17,81	17,53
57	0,60	2,90	0,60	3,20	4,20	55,60	17,60	88,90	11,40	18,00	5,60	1,90	8,70	1,20	8,19	14,35

$D_{critical}$

29,52 29,07

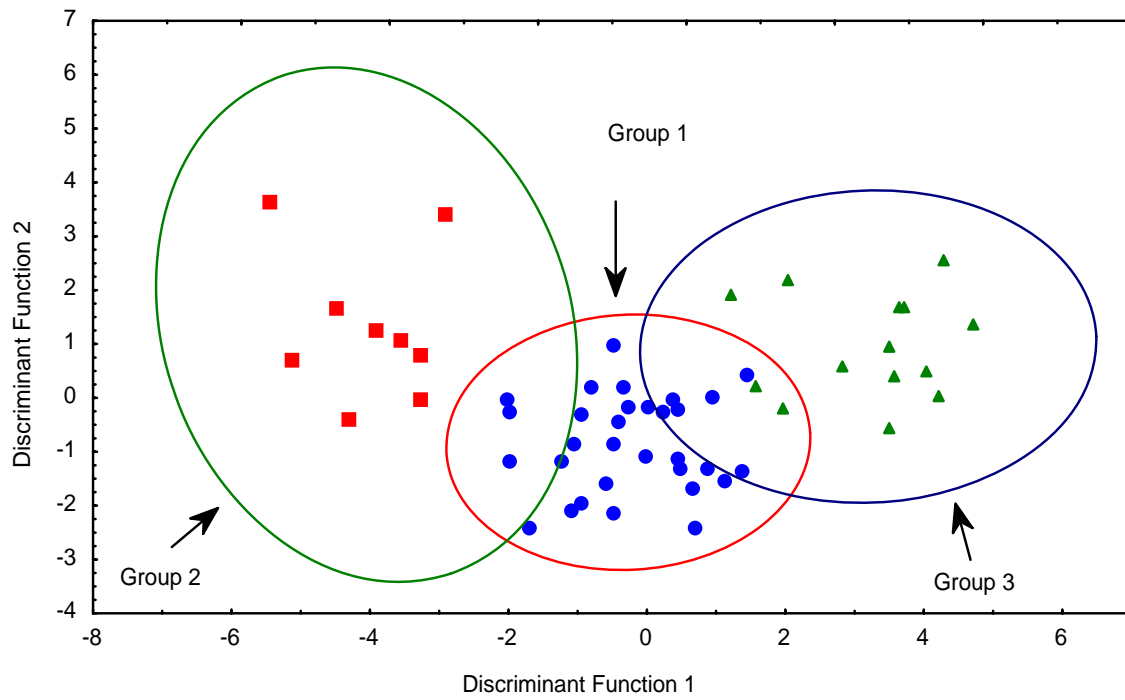


Figura 1. Discriminant functions for Marajoara samples using all the variables (Na, K, Lu, U, Yb, La, Th, Cr, Cs, Sc, Fe, Eu, Hf and Tb). Ellipses represents a confidence level of 95%.

Table 2. Results for forward selection procedure

Variable	<i>p</i> -values for entry							
	Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7	Step 8
Na	0,0430	0,3588	0,4104	0,6453	0,8649	0,9934	0,9966	0,9993
K	0,2174	0,2281	0,2746	0,7169	0,6551	0,2583	0,3943	0,4352
Lu	0,0000	0,0148	0,0135	0,0033	0,0060			
U	0,0274	0,1483	0,1798	0,2092	0,1927	0,2268	0,2317	
Yb	0,0000	0,0009	0,0018	0,0024				
La	0,0000	0,0316	0,0283	0,5719	0,6914	0,6042	0,6353	0,8200
Th	0,0018	0,0269	0,0239	0,4949	0,5990	0,4411	0,4531	0,8616
Cr	0,0011	0,0136	0,0049	0,1361	0,2924	0,2960	0,3262	0,5296
Cs	0,0001	0,0002	0,0001					
Sc	0,0002	0,0021	0,0021	0,4093	0,3967	0,2655	0,2404	0,4682
Fe	0,1994	0,0551	0,0815	0,1167	0,0516	0,0330		
Eu	0,0000							
Hf	0,6331	0,2866	0,1042	0,1469	0,7749	0,9515	0,7871	0,8116
Tb	0,0000	0,0001						
Critical value	0,3761							

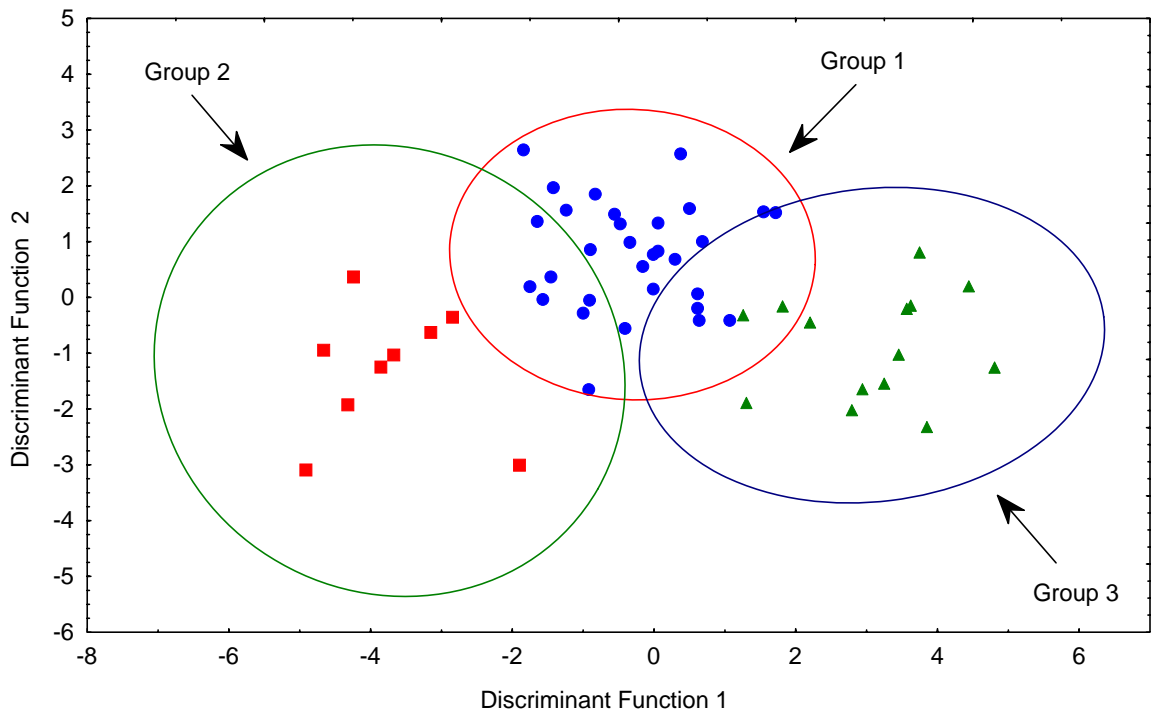


Figura 2. Discriminat functions for Marajoara samples using the selected variables (Eu, Tb, Cs, Yb, Lu, Fe and U). Ellipses represents a confidence level of 95%.