

PRELIMINARY STUDY OF CHEMICAL COMPOSITIONAL DATA FROM AMAZON CERAMICS

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ABSTRACT

Eighty seven ceramic samples from Açutuba, Lago Grande and Osvaldo archaeological sites located in the confluence of the rivers Negro and Solimões were submitted to chemical analysis using instrumental neutron activation analysis to determine As, Ba, Ce, Co, Cr, Cs, Eu, Fe, Hf, K, La, Lu, Rb, Na, Nd, Sb, Sc, Sm, Ta, Tb, Th, Yb, Zn, and U. The database were studied using the Mahalanobis distance, and discriminant analysis. The results showed that the ceramics of each site differ from each other in chemical composition and that they form three different groups. Chemical classification of the ceramics suggests that vessels were made locally, as only ceramics from the same area show homogeneity of data.

1. INTRODUCTION

Ceramic fragments are of the main category of artifacts used by archaeologists. The study of archaeological ceramics provides useful evidence for different aspects of ancient group lives [1,2]. However, the archaeologist work is limited to some extent to answering questions such as the contents of ancient vessels and the source of the raw materials, as well as aspects of trade and transport. This work calls for a multidisciplinary approach and, especially, for the help of chemical and petrographic analysis [1-6]. The chemical or mineralogical 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 sites [7,8].

In this work the concentrations of As, Ba, Ce, Co, Cr, Cs, Eu, Fe, Hf, K, La, Lu, Rb, Na, Nd, Sb, Sc, Sm, Ta, Tb, Th, Yb, Zn, and U in eighty seven ceramics fragments from three archaeological sites (Açutuba: 29, Lago Grande: 46 and Osvaldo: 12) located in the confluence of the rivers Negro and Solimões, Iranduba district, Manaus, were analyzed by means of instrumental neutron activation analysis, INAA. Neutron activation analysis is an important quantitative analytical technique with application in a broad range of discipline due

to its sensitivity, accuracy, precision, and versatility. The technique is a suitable method for analyzing many different types of samples. Archaeologists, in particular, have made extensive use of neutron activation analysis for the purpose of characterizing archaeological materials and determining their provenance [9].

Ceramics are of great importance, as they have turned out to be the most informative source concerning questions of cultural evolution in the Amazon. Through the different styles of vessels, a chronological sequence can be established covering a time span. Changes occurred in the economic way of life, as they did in the orientation of contact within the region. The issue of distribution and exchange of ancient ceramics from this place in Brazil arise questions concerning the origin of the manufactured vessels such as if were they made locally and others. The present study aims to contribute, by means of the chemical elements to provide data to answer these questions. Actually, this study offers the first archaeometric data for these sites.

2. EXPERIMENTAL

2.1. Sample Preparation and Standard

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 sherd as possible without drilling through the walls [8,10]. Eighty seven ceramics samples were analyzed. Finally they were dried in an oven at 105°C for 24 h and stored in desiccator.

The reference material Coal Fly Ash (NIST-SRM-1633b) was used as standard, and IAEA-Soil 7 Trace Elements in Soil was used to check samples in all analysis. These materials were dried in an oven at 105°C for 2 h and stored in desiccator until weighing [8,10].

2.2. Description of the Method

Generally speaking, NAA method is based on the properties of nuclei, in which a neutron incident interacts with a nucleus of the target element. During the sample bombardment with neutrons a small fraction of the nucleus from each one of the sample constituent 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 for each element. The measure of these gamma rays permits the qualitative and quantitative determination of the sample elements.

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

Two measurement series were carried out using Ge (hyperpure) detector, model GX 2020 from Canberra, resolution of 1.9 keV at the 1332.49 keV gamma peak of ^{60}Co , with S-100

MCA of Canberra with 8192 channels. As, K, La, Lu, Na, Nd, Sm, U, and Yb were measured after 7 days cooling time and Ce, Co, Cr, Cs, Eu, Fe, Hf, Rb, Sb, 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.

2.3. Statistical Handling of the Data

The volume of compositional data generated by INAA studies of archaeological materials is substantial. As a result, multivariate statistical methods are required to quantify the similarities and differences between specimens and groups of specimens. In this work the chemical data from the samples were treated statistically by means of Mahalanobis distance and discriminant analysis. The Mahalanobis distance is defined as the squared Euclidean distance between the specimen and group centroid, divided by the group variance in the direction of the specimen. The Mahalanobis distance statistic incorporates information about the correlations between pairs of elements as derived by the off-diagonal terms of a variance-covariance matrix, which simple Euclidean distance does not. Thus, it permits calculation of the probability that a particular specimen belongs to a group based not only on its proximity to the group centroid but also on the rate at which the density of data points decrease away from the centroid in that direction. Discriminant analysis is a procedure to study differences between two or more groups of objects with respect to several variables. The compositional data are grouped by petrofacies. Individual samples are the objects and the elements are the variables. Heidke and Miksa[11] summarize the mathematical assumptions which underline discriminant analysis. They are: a) two or more groups; b) at least two cases per group; c) any number of discriminating variables, providing that it is less than the total number of cases minus two; d) discriminating variables are measured at the interval level; e) no discriminating variable may be a linear combination of other discriminating variables; f) the covariance matrices for each group must be (approximately) equal; and g) each group has been drawn from a population with a multivariate normal distribution on the discriminating variables.

Discriminant analysis provides the means to evaluate the degree of interpetrofacies compositional variability in a data set. In addition, functions derived from discriminant analysis can be used to predict the petrofacies membership of the ceramics. This use of discriminant analysis provides a rigorous statistic test to evaluate a ceramic assignment to a given petrofacies [11]. Finally, discriminant analysis provides a consistent, objective perspective on provenance; that perspective helps ceramicists arrive at a consensus regarding a ceramic composition and provenance.

3. RESULTS AND DISCUSSION

The precision of the analytical technique in archaeometric studies using trace elements is the basic premises in this kind of study. If an element used is not measured with good precision it can obscure real differences in concentration and, the discriminant effect of other well-measured elements tends to be reduced. In this work all the elements with relative standard deviation less than 10% were considered [7,12]. Then, the elements Na, Lu, U, Yb, La, Th, Cr, Cs, Sc, Fe, Eu, Ce and Hf were used in subsequent analysis. Table 1 shows the database of the three sites. The first step in the analysis was to transform the concentration data into base-10 logarithm values to compensate the large difference of magnitude between major and trace element [13]. After that, the data were analysed using the Mahalanobis distance (D)

Table 1. Concentration data from Açutuba, Lago Grande and Osvaldo archaeological sites, in µg/g, unless otherwise indicated.

Site	Na(%)	Lu	U	Yb	La	Th	Cr	Cs	Sc	Fe(%)	Eu	Ce	Hf
Açutuba	0,03	0,37	2,02	2,30	26,30	11,92	54,12	4,92	10,84	3,78	0,54	51,71	15,76
Açutuba	0,19	0,37	3,52	2,76	36,69	14,24	62,95	4,83	15,94	4,02	1,23	75,66	6,94
Açutuba	0,04	0,24	2,07	1,69	22,96	8,08	45,06	7,79	10,88	2,14	0,67	44,22	3,53
Açutuba	0,30	0,44	4,29	2,81	37,18	13,19	59,36	4,67	14,73	3,31	1,18	72,81	8,21
Açutuba	0,22	0,44	4,14	3,15	40,27	14,44	65,07	9,46	15,81	4,00	1,36	72,96	8,77
Açutuba	0,35	0,35	3,89	2,55	31,58	13,88	62,23	2,86	14,36	5,03	0,95	60,03	7,23
Açutuba	0,18	0,33	3,27	2,34	22,81	14,82	62,57	7,74	13,65	3,33	0,74	46,23	7,63
Açutuba	0,15	0,33	3,34	2,53	34,66	15,30	64,68	10,13	15,03	3,35	0,95	62,91	7,37
Açutuba	0,05	0,34	3,04	2,48	36,21	14,26	57,61	7,85	13,96	3,07	1,02	74,96	8,85
Açutuba	0,03	0,18	2,26	1,33	10,40	7,93	46,00	7,59	9,55	2,16	0,34	19,69	3,69
Açutuba	0,09	0,34	3,26	2,57	22,99	10,51	60,52	11,76	13,77	2,86	0,70	44,78	4,65
Açutuba	0,05	0,30	3,10	2,30	33,30	13,00	65,40	9,20	13,30	2,50	0,80	72,10	8,00
Açutuba	0,07	0,30	3,70	2,40	28,70	15,00	53,00	14,20	12,70	2,30	0,80	63,70	6,00
Açutuba	0,20	0,50	2,70	2,70	36,50	13,50	60,80	5,70	14,70	3,00	1,03	70,80	6,60
Açutuba	0,20	0,60	3,60	3,20	40,00	15,00	64,70	8,90	15,70	4,00	1,40	78,00	8,30
Açutuba	0,20	0,50	3,50	3,00	41,00	14,80	64,60	8,40	15,40	3,50	1,20	79,00	8,20
Açutuba	0,20	0,60	5,30	2,90	40,20	14,30	66,70	10,10	15,50	3,50	1,30	78,00	6,90
Açutuba	0,20	0,50	4,20	3,50	44,10	14,30	66,00	11,20	16,20	3,50	1,40	80,00	7,30
Açutuba	0,04	0,40	3,60	2,00	27,40	13,10	53,00	6,80	12,80	2,60	0,50	48,00	7,20
Açutuba	0,03	0,30	1,90	1,50	21,00	8,00	38,70	6,20	8,60	1,60	0,40	47,60	4,30
Açutuba	0,01	0,50	4,10	3,00	23,00	14,40	65,50	1,10	11,30	3,90	0,60	48,00	14,20
Açutuba	0,10	0,30	2,90	1,80	27,20	10,90	48,40	8,90	11,60	3,00	0,70	54,20	7,50
Açutuba	0,05	0,30	3,70	1,80	27,40	13,80	67,00	7,90	16,00	3,80	0,60	49,20	6,20
Açutuba	2,00	0,40	3,50	2,80	35,30	12,40	56,00	10,60	14,40	3,00	1,20	84,20	7,00
Açutuba	0,20	0,40	4,40	3,10	43,90	15,30	59,10	12,00	16,20	3,30	1,40	105,00	9,10
Açutuba	0,07	0,40	3,40	2,10	35,50	13,10	63,40	7,10	14,40	3,10	0,80	65,70	6,70
Açutuba	0,20	0,50	4,20	3,70	67,50	14,70	63,40	6,20	15,00	3,70	1,90	136,60	8,50
Açutuba	0,10	0,40	3,20	2,70	38,00	12,30	50,40	5,20	11,10	2,80	0,90	74,00	12,00
Açutuba	0,20	0,40	4,00	3,00	39,50	14,30	60,80	10,70	15,10	3,50	1,30	77,00	6,90
L. Grande	0,04	0,30	3,50	2,40	38,10	11,80	57,00	11,30	13,00	2,50	0,98	70,30	4,70
L. Grande	0,20	0,40	4,10	2,20	40,00	13,70	63,00	13,00	15,50	4,10	1,20	77,00	6,50
L. Grande	0,15	0,40	3,40	2,70	37,60	12,10	54,80	12,10	14,00	3,50	1,30	69,90	3,40
L. Grande	0,06	0,50	3,90	2,80	41,30	14,20	68,10	12,60	15,00	3,20	1,00	77,30	8,90
L. Grande	0,20	0,50	4,20	2,80	41,90	12,50	60,50	6,00	14,50	3,30	1,50	80,10	3,90
L. Grande	0,20	0,50	4,80	3,40	46,40	15,40	64,80	11,20	15,80	4,50	1,60	88,30	6,00
L. Grande	0,60	0,60	4,80	3,70	42,41	15,50	77,00	11,00	18,20	3,60	1,50	111,70	6,80
L. Grande	0,05	0,40	4,50	2,60	44,50	15,00	62,60	11,60	13,70	2,90	1,00	78,70	6,70
L. Grande	0,40	0,40	3,40	2,60	33,50	12,10	68,20	6,10	14,70	4,60	0,90	62,50	5,60
L. Grande	0,30	0,40	3,30	2,40	37,80	12,10	53,50	13,70	13,70	2,90	1,20	71,70	4,50
L. Grande	0,40	0,40	3,60	2,60	46,30	14,40	69,20	13,50	16,90	3,30	1,50	91,10	5,20
L. Grande	0,20	0,30	2,60	2,40	33,70	11,30	46,70	11,00	12,20	3,10	1,00	70,60	4,90
L. Grande	0,03	0,30	2,70	1,50	35,50	14,00	53,10	12,90	12,50	2,20	0,80	62,40	5,00
L. Grande	0,42	0,50	4,80	3,30	39,40	13,30	73,50	9,50	14,30	3,20	1,60	76,80	10,80
L. Grande	0,20	0,50	5,20	3,60	58,50	14,90	83,10	12,30	17,40	4,10	2,00	110,30	5,20
L. Grande	0,10	0,50	3,30	3,20	53,10	15,10	71,00	9,20	16,60	3,50	1,70	95,60	6,30
L. Grande	0,06	0,60	4,00	4,00	59,20	17,30	89,90	2,90	21,30	6,00	1,80	111,50	9,10

Table 1. Continued

Site	Na(%)	Lu	U	Yb	La	Th	Cr	Cs	Sc	Fe(%)	Eu	Ce	Hf
L. Grande	0,15	0,50	4,30	3,10	44,10	15,50	69,90	10,70	15,80	3,90	1,40	86,60	7,70
L. Grande	0,03	0,40	3,40	2,50	39,50	12,60	62,00	6,30	14,20	3,20	0,90	70,80	8,30
L. Grande	0,05	0,50	3,90	2,70	54,80	14,60	56,40	7,80	11,00	3,10	1,60	81,30	9,70
L. Grande	0,04	0,50	3,00	2,70	33,50	15,00	51,00	8,00	10,20	2,80	0,90	57,00	9,60
L. Grande	0,60	0,60	2,80	3,80	45,40	14,60	63,70	13,70	16,10	4,80	1,70	86,50	7,00
L. Grande	0,06	0,40	3,50	2,20	35,60	15,40	59,80	16,00	12,20	1,90	0,80	65,00	11,90
L. Grande	0,25	0,40	5,60	3,20	44,60	14,60	65,50	7,90	16,30	2,80	1,30	82,70	5,00
L. Grande	0,11	0,50	4,10	3,40	42,80	14,40	72,90	10,90	15,50	4,10	1,40	78,90	8,40
L. Grande	0,30	0,30	2,40	2,00	31,00	9,40	52,00	9,30	11,80	3,10	0,90	57,50	3,40
L. Grande	0,04	0,30	2,80	2,20	30,00	7,60	38,60	6,80	9,50	2,20	0,80	57,60	4,00
L. Grande	0,40	0,40	3,80	3,10	43,00	13,10	68,50	8,00	16,80	3,90	1,40	79,50	5,20
L. Grande	0,30	0,40	2,60	2,80	38,00	12,00	57,00	8,00	14,50	3,40	1,40	73,50	3,90
L. Grande	0,20	0,50	5,20	3,00	44,60	13,50	66,50	3,90	17,10	4,70	1,30	83,00	4,70
L. Grande	0,30	0,60	4,40	3,70	52,30	15,50	74,10	1,30	19,00	4,50	1,70	98,30	5,70
L. Grande	0,05	0,30	2,60	2,10	30,10	11,20	48,70	13,10	11,40	2,20	0,80	54,30	4,10
L. Grande	0,20	0,50	3,90	3,20	41,60	14,90	70,00	9,10	16,20	3,80	1,30	81,30	7,20
L. Grande	0,50	0,50	3,80	3,30	43,30	14,40	65,30	8,50	16,60	3,50	1,60	87,40	5,80
L. Grande	0,10	0,70	3,70	3,70	61,00	16,70	80,80	1,50	17,80	4,10	2,00	113,50	7,10
L. Grande	0,20	0,50	4,30	3,30	43,60	15,70	73,10	9,90	18,00	4,80	1,40	91,70	9,00
L. Grande	0,30	0,40	3,20	2,60	38,10	14,60	71,80	12,90	16,90	4,00	1,10	79,30	4,80
L. Grande	0,05	0,40	3,70	2,00	34,10	14,20	59,30	13,70	13,60	4,20	0,80	64,00	9,00
L. Grande	0,04	0,50	3,00	3,20	42,00	13,70	59,40	11,00	11,70	1,70	1,20	115,70	11,80
L. Grande	0,30	0,60	4,20	3,30	46,80	14,30	72,10	7,00	17,60	3,90	1,50	88,60	4,80
L. Grande	0,11	0,50	3,00	2,80	35,10	11,80	54,30	6,70	11,70	2,80	1,10	61,20	7,40
L. Grande	0,13	0,50	4,00	3,20	45,80	11,50	64,00	9,50	11,40	3,40	1,40	81,60	4,50
L. Grande	0,21	0,60	4,70	4,00	53,40	18,60	84,80	16,20	18,30	4,70	1,60	97,00	11,20
L. Grande	0,14	0,50	3,70	3,80	57,40	16,70	87,70	4,60	19,80	3,90	1,80	114,00	6,40
L. Grande	0,20	0,60	4,70	3,50	41,30	14,40	69,80	7,60	15,60	4,10	1,30	77,50	8,00
L. Grande	0,30	0,50	3,50	2,80	35,50	12,30	58,40	12,30	12,90	2,90	1,02	65,00	9,10
Oswaldo	0,09	0,70	3,70	4,70	86,00	20,20	33,20	3,50	10,50	2,70	2,80	148,40	13,00
Oswaldo	0,20	0,50	3,50	2,90	42,10	13,80	63,10	6,10	14,60	3,50	1,40	76,90	6,40
Oswaldo	0,30	0,40	5,40	2,70	40,20	13,80	62,50	10,00	15,40	3,80	1,30	78,30	4,90
Oswaldo	0,04	0,50	7,40	2,30	21,40	11,90	61,80	8,80	11,80	3,70	0,60	41,80	8,20
Oswaldo	0,30	0,40	4,60	3,00	40,50	13,40	65,90	11,30	15,30	3,90	1,30	76,70	5,20
Oswaldo	0,20	0,40	4,50	3,10	44,80	14,10	63,50	8,60	15,20	3,40	1,50	87,40	5,90
Oswaldo	0,10	0,70	4,00	3,40	41,60	15,40	66,10	7,50	14,40	3,70	1,20	77,90	9,80
Oswaldo	0,09	0,60	3,90	3,40	36,90	15,00	62,20	11,10	13,50	4,00	1,04	64,90	12,50
Oswaldo	0,14	0,51	4,18	3,05	20,80	10,30	55,80	12,00	10,70	2,90	0,80	49,10	5,80
Oswaldo	0,30	0,50	3,20	2,70	34,20	12,20	58,00	11,10	13,50	3,20	1,03	62,20	6,40
Oswaldo	0,30	0,40	2,90	2,50	35,20	12,30	58,70	8,30	12,90	3,40	1,10	63,00	5,60
Oswaldo	0,20	0,50	2,90	2,80	44,70	11,30	56,90	11,00	12,00	2,60	1,20	87,30	6,30

using the lambda Wilk's as critical value to identify the outliers[14]. Two samples from Açutuba and Lago Grande were considered outliers; then, the four samples were eliminated in subsequent data interpretation.

Since differences in chemical composition are typically interpreted as evidence for distinct production location, so the similarities among the samples of the three sites were studied by means of bivariate plots and discriminant analysis. As shown in Figure 1 and 2 the plots of Sc vs Fe and Eu vs U reveal one chemical group very well defined. With the purpose to confirm the last assumption the data were submitted to discriminant analysis. The plot of the discriminant function 1 vs. discriminant function 2 obtained is presented in Figure 3. The plot shows the groups very clearly. Figure 3 show clearly that the three sites form only one chemically homogeneous group.

4. CONCLUSION

The compositional of data suggests that people from the each archaeological site contributed with population moving into all the area. Should future study confirm that assumption within the region then it could also suggest that people moved in household units rather than whole village groups, which opens interesting questions about the degree of social integration of these communities.

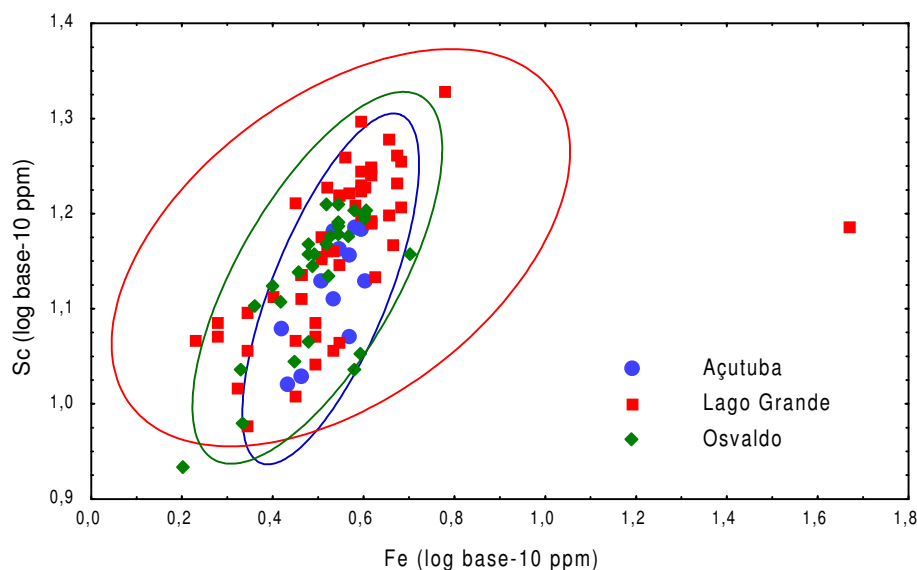


Figure 1. Bivariate plot of Sc vs. Fe base-10 logged concentrations in ceramic samples. Ellipses represent 95% confidence level for memberships in the groups.

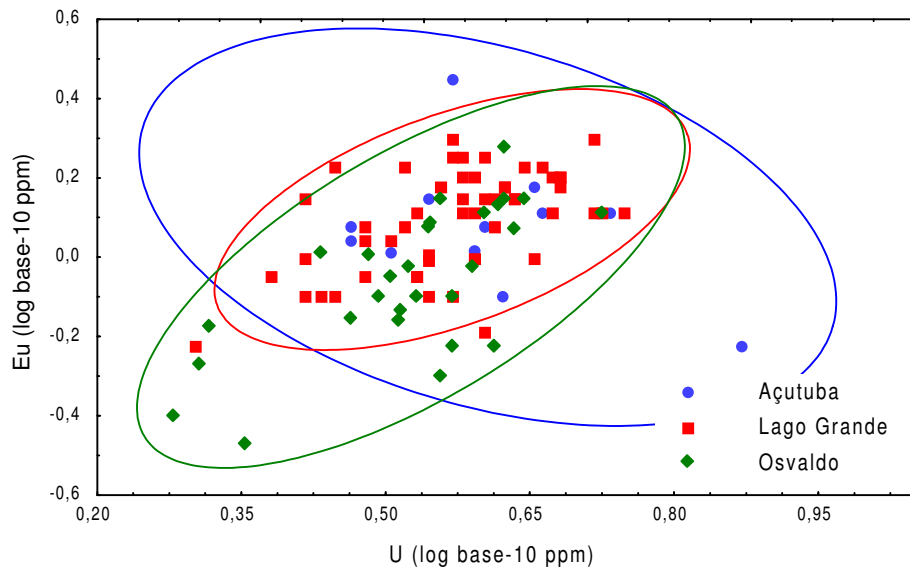


Figure 2. Bivariate plot of Eu vs. U concentrations in ceramic samples from the three sites. Ellipses represent 95% confidence level membership in the groups.

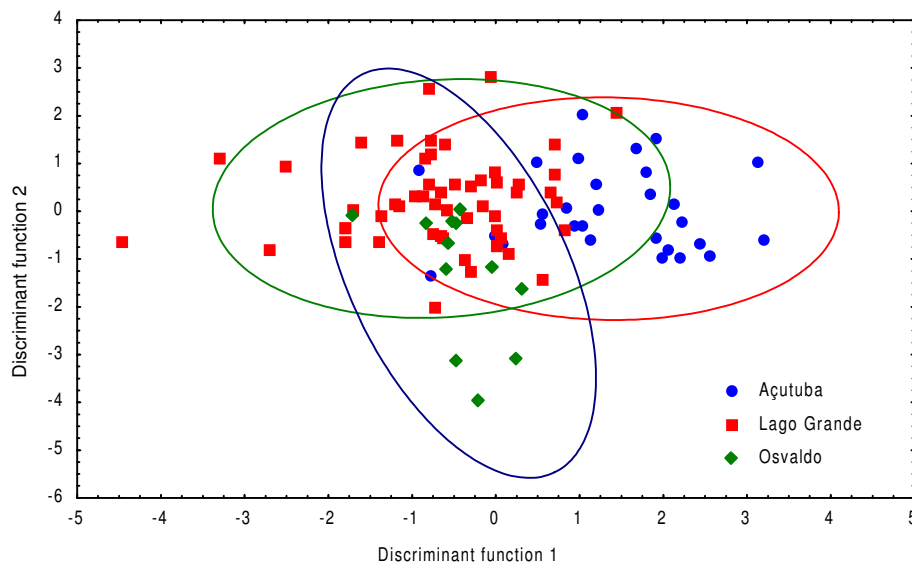


Figure 3. Discriminant functions for all samples studied. Ellipses represent 95% confidence level for samples inclusion into cluster.

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