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Computed Radiography, PIXE and XRF analysis of pre-colonial pottery from Maranhão, Brazil

Renato A. Ikeoka [a], Carlos R. Appoloni [a], Marcia A. Rizzutto [b,*], Arkley M. Bandeira [c]

[a] Departamento de Física/CCE, Universidade Estadual de Londrina – UEL, Caixa Postal 10.011, CEP 86057-970 Londrina, PR, Brazil
[b] Instituto de Física da Universidade de São Paulo, Laboratório de Análise de Materiais por Feixes Iônicos (LAMFI), Rua do Matão Travessa R Nr.187, CEP 05508-090, Cidade Universitária, São Paulo, SP, Brazil
[c] Museu de Arqueologia e Etnologia, Universidade de São Paulo, Av. Prof. Almeida Prado, 1466, CEP 05508-900, Cidade Universitária, São Paulo, SP, Brazil

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ABSTRACT

This work concerns the archaeometric analysis of ceramic fragments from archaeological excavations carried out in "Sambaqui do Bacanga", "Sambaqui do Panaquatira" and "Rabo de Porco", located in the São Luiz city area, at Brazilian northeast. Ancient civilizations that inhabited that territory were characterized as fishing, catchers, hunters and ceramist populations. Dates obtained by thermoluminescence ranged from 6600 to 127 BP. The studied samples were sixty three representative pottery fragments selected of stratigraphic levels from the surface up to 170 cm deep for Sambaquis do Bacanga and Panaquatira and from surface up to 105 cm depth for Rabo de Porco. The three analytical methods employed were Computed Radiography, PIXE and XRF. Sixteen elements were measured with good statistics in the different ceramic samples through EDXRF and PIXE analysis: Al, Si, P, S, Cl, K, Ca, Ti, Mn, Fe, Cu, Zn, Rb, Sr, Y and Zr. Multivariate statistical analysis, Hierarchical Cluster Analysis (HCA) and Principal Component Analysis (PCA), of the fragments elemental composition were performed to separate and correlate the groups of the samples. Fragments of the three archaeological sites grouped into two clusters, the first is composed of samples from "Sambaquis do Bacanga and Panaquatira", and the second consists of the "Rabo de Porco" samples. This result indicates that each cluster of fragments was manufactured with different clays, and "Sambaquis do Bacanga and Panaquatira" fragments are derived from the same source or same kind of clay. The internal structure of the ceramic fragments observed by Computed Radiography revealed the presence of various sizes and types of anti-plastics in the sherds.

1. Introduction

In the State of Maranhão, where there is a lack of sufficient archaeological information to compose a minimum framework on the pre-colonial occupations in its territory, three archaeological sites are being excavated, namely: Sambaqui do Bacanga, Sambaqui da Panaquatira and Rabo de Porco. Several types of material traces were evidenced in these archaeological excavations, such as remains of fire, human skeletons, remains of animal bones, remains of fish, shells and ceramic objects [1].

Among all this material culture, ceramics are of great archaeological value, since they are extremely resistant to weather and the surrounding media conditions [2]. This also represents an integral part of a society, presenting sensitive singularities at a material level, which characterize the technological system and the way of life of the peoples studied [3,4].

Archaeometry is a physical-chemical research area studying problems related to cultural inheritance. It is based on the obtainment of information regarding the origin and history of the findings, material analysis related to the chemical structure and changes, as well as dating techniques [5].

Among the archaeometric techniques, Energy Dispersive X-Ray Fluorescence (EDXRF), Particle Induced X-ray Emission (PIXE), Mössbauer Spectroscopy, X-Ray diffraction, Raman spectroscopy and digital radiography are common. These techniques are becoming more and more important to the study of art objects, archaeological artifacts and cultural heritage in general [6–16].

In this paper, EDXRF, PIXE and Computed Radiography were used to study the ceramic fragments collected in the archaeological sites Sambaqui do Bacanga, Sambaqui da Panaquatira and Rabo de Porco, located in the island of São Luis, state of Maranhão, Brazil, with the following objectives:

i. Measurement of the elemental composition of the ceramic bulk of the fragments.
ii. Using a multi-varied analysis to check the similarity among the

* Corresponding author.
E-mail address: rizzutto@if.usp.br (M.A. Rizzutto).

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chemical elements identified in the fragments from the same and different stratigraphies, enabling the grouping of fragments that were manufactured with the same clay source. Studying their origin, using a multi-varied analysis.

iii. Analyze the internal structure of the sherds.

2. Materials and methods

2.1. Sambaqui do Bacanga

The building of knowledge regarding ancient populations, from the study of their settlements, usually marked by shellmounds (or Sambaquis, as called in Brazil), allows not only the understanding of the dynamics of the individuals and environmental characteristics, but also the discussion of the social complexity of these populations. Archaeological sites of the shellmound type are located in low altitude environments. They were structured from human occupations that left traces of their permanence.

Shellmounds are generally conceived as mounds of shells, whose denomination derives from the Tupi language, being *Tamba*/*Samba* denominating shells and *Ki* denominating heap. Despite the strong visual appeal caused by the large number of shells that create mounds that stand out in the landscape, these sites present a much greater variability of other faunistic vestiges, such as fish bones, mammalian bones, crustacean shells, among others; associated with material culture finely elaborated in rocks, bones, teeth and shells, ceramic fragments, bonfires, burials and vestiges of dwellings [17].

The archaeological site Sambaqui do Bacanga is located in the Central-Western area of São Luís do Maranhão Island, in the region of the Bacanga River. Its course is protected by an environmental preservation unit known as Bacanga State Park. The coordinates to São Luís are: 02°31′ 47″ Latitude South and 44° 18′ 10″ W Longitude West, with average altitude of 4 m above sea level. These sites were established in the region around 6600 years before the present and continued inhabiting that settlement until the year 900 before the present [18,19].

2.2. Sambaqui da Panaquatira

The Sambaqui da Panaquatira is located in the city of São José de Ribamar, in the metropolitan region of São Luís, east of São Luís Island.

The coordinates to São José de Ribamar are: 02° 27′ 35″ 02° 42′ 11″ Latitude South and 44° 01′ 18″ e 44° 13′00″ Longitude West, with average altitude of 20 m above sea level.

The human occupation chronology in this site is of 5730 years up to 127 years before the present [17].

2.3. Rabo de Porco

Rabo de Porco was considered the oldest pre-historical occupation in the state of Maranhão, with 9200 years of existence [11]. This site was characterized as an open air multi-component area, with 950 m^2 located at the banks of Rabo de Porco stream, 2-km from the Itapecuru river, in the city of Bacabeira, 60 km from São Luís. The coordinates to Bacabeira are: 02° 56′ 04″ Latitude South and 44° 14′ 06″ Longitude West, with average altitude of 14 m above sea level. The human occupation chronology in this site is of 9200 years up to 4500 years before the present [17].

2.4. Stratified collection of fragments

Hundreds of ceramic fragments were collected in the excavations carried out in the three archaeological sites. For archaeometric analysis, the archaeologist carefully selected some fragments representative of each stratigraphy (depth) excavated. A total of 63 fragments were analyzed in this work that represents all the stratigraphy performed from the surface up to 170 cm depth (Table 1).

Table 1
Information about stratigraphy of the selected fragments.

Site	Quantity of fragments	Stratigraphic levels (cm)
Sambaqui da Panaquatira	26	Surface up to 170 cm[a]
Sambaqui do Bacanga	17	Surface up to 170 cm[b]
Rabo de Porco	20	Surface up to 105 cm[c]
Total	63	

[a] Selected fragments every 5 cm.
[b] Selected fragments every 5 cm (from surface up to 20 cm) and every 10 cm from 20 cm up to 170 cm depth.
[c] Selected fragments every 10 cm.

2.5. Energy Dispersion X-Ray Fluorescence (EDXRF)

The Shimadzu EDX-720 bench EDXRF system includes a Rh target, 5–50 kV X-ray tube with a tube current operating range of1–1000 μA, a 3 mm diameter collimator and a Si(Li) type solid state detector.

The system is optimized to measure both high and low atomic (Z) number elements. The low Z group (Sodium to Scandium) was measured with tube setting of 15 kV and 470 μA, while the high Z group (Titanium to Uranium) was measured with tube setting of 50 kV and 24 μA. No filters were used in the measurements. The instrument was operated in air.

An average of three measurements on the ceramic bulk (flat regions), each one with 1000 s acquisition time, was performed for each fragment. The ceramic fragments have an average thickness of 0.8 cm. The fragments were positioned vertically in the region delimited by the equipment with the help of a specially designed foam holder and the system camera. The measurements were performed using the "Easy Quali-Quantitative" model, which is already programmed in the equipment system and the values results of the average net areas of the elements were used. The system is calibrated with a standard metal material and certified materials are used to test the reproducibility of the measurement. Each element area above three uncertain is considered as present in the sample. Elements above Al are detected. Table 2 contains the values of the average net areas of each element identified in a representative fragment from each site.

2.6. Particle Induced X-ray Emission (PIXE)

The PIXE measurements were performed using a electrostatic Pelletron-tandem accelerator from NEC (National Eletronic

Table 2
Values of the average net areas of the elements by EDXRF of a representative fragment of each archaeological site.

Element	SP sample intensity (cps/μA)	SB sample intensity (cps/μA)	RP sample intensity (cps/μA)
Si	0.0546(10)	0.0787(40)	0.0109(10)
Fe	17.056(11)	51.175(25)	18.952(13)
Al	0.0050(10)	0.0078(20)	0.0064(10)
Ti	0.9608(10)	1.7559 (45)	1.3076(35)
Ca	1.7156(20)	1.2159(16)	0.0239(15)
K	0.0518(10)	0.0571(40)	0.2035(10)
P	0.0242(10)	0.0198(10)	–
Zr	1.1543(34)	2.1170(53)	2.2250(46)
Sr	0.6620(29)	0.7044(45)	0.1729(40)
Mn	0.5634(31)	0.9693(44)	24.000(45)
Zn	0.1670(12)	0.2634(37)	0.0762(16)
Rb	–	–	0.2721(36)
S	0.0029(10)	0.0055(20)	0.0049(10)
Cr	0.0300(14)	0.0752(20)	0.0391(13)
Y	0.2058(31)	0.2355(49)	0.1102(42)
Cu	0.0205(10)	0.0896(16)	–
Nb	0.0302(34)	0.1101(55)	0.0968(45)

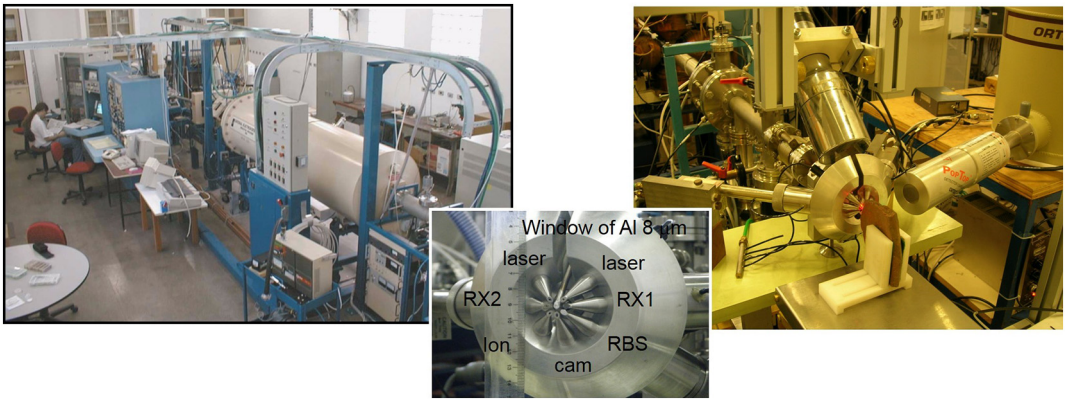

Fig. 1. Left: LAMFI-USP (Ion Beam Materials Analysis Laboratory of the University of São Paulo). Right: External beam setup at LAMFI. Center: Detailed view of the external beam array system and indications of the elements associated.

Corportation), type 5SDH, and an external multi-use analyses station, which allowed analyze of objects of different shapes and sizes in air. This system belongs to the Laboratory of Ion Beam Material Analysis (LAMFI-USP) at the University of São Paulo (Fig. 1). The external beam setup uses ~2.4 MeV proton beam and ~10 nA current to analyze the samples.

Three X-ray detectors were used, two Si-PIN detectors (RX1 and RX2 in Fig. 1) and one HPGe with standard spectrometry electronics; each spectrum was taken for 600 s. To perform normalization between the measurements a gamma-ray scintillation detector (NaI(Tl)) was used to determine the ion beam charge. The beam exit window with an 8 mm of aluminum foil is also used to monitor beam charge by gamma rays of the (p, γ) reaction by an external NaI detector.

The geometry and materials of the assembly is such to shield the Si-PIN detectors from the X-ray emissions from the exit beam window as well as reduce the detection of Ar K_α from the air path, due to the narrow field of view of the X-ray detectors.

2.7. Computed Radiography

The Computed Radiography consists of the X-ray tube Philips MG 450 and scanner CR30-X AGFA. The conditions used in the fragments with thicknesses lower than 1.0 cm were 50 kV of tension, 3 mA of current and 10s of irradiation in a distance of 3.5 m. Small piece of aluminum material with calibrated thicknesses were radiographed with the ceramic fragments. The calibrated thicknesses material varied between 0.2 and 1.0 cm.

2.8. Principal Component Analysis (PCA) and Hierarchical Cluster Analysis (HCA)

Multivariate statistical analysis techniques, such as Principal Component Analysis (PCA) and Hierarchical Cluster Analysis (HCA) are used to observe the formation of natural grouping of the samples based on their similarities and correlation. PCA reduces data dimensionality, preserving the relevant information, allowing the visualization in two-dimensional spaces. For HCA, all this is done in a hierarchical structure in which the samples are placed in a system of groups and subgroups depending on the degree of similarity [20,21].

3. Results and discussion

3.1. EDXRF and PIXE

Sixteen elements were measured with good statistics in the different ceramic samples through EDXRF and PIXE analysis: Al, Si, P, S, Cl, K, Ca,

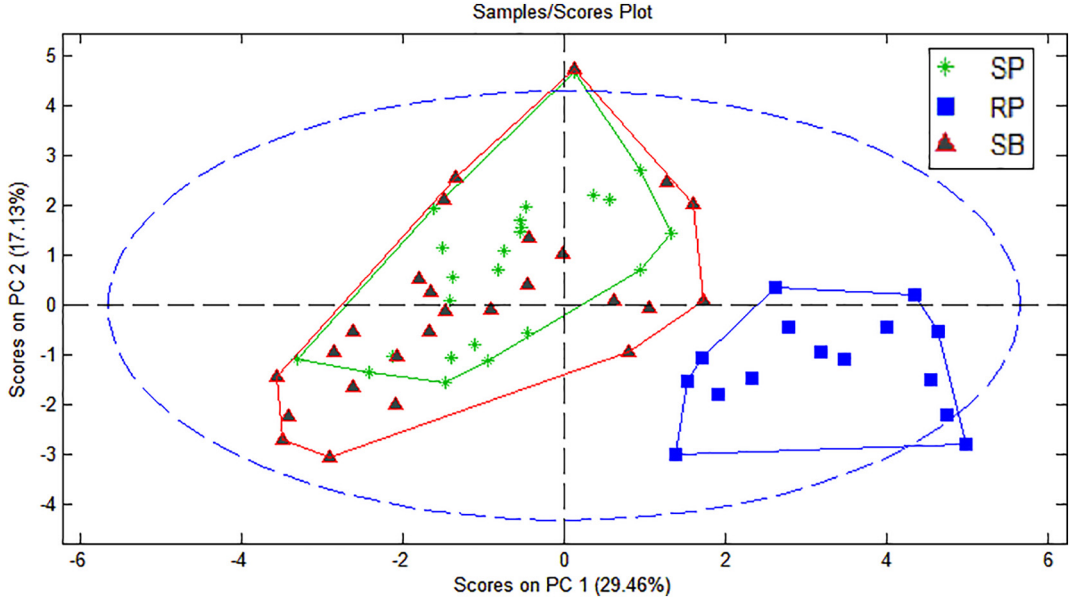

Fig. 2. Principal Component Analysis (PCA) with pottery fragments excavated in three archaeological sites, Sambaqui do Bacanga (SB), Sambaqui da Panaquatira (SP) and Rabo de Porco (RP) using the EDXRF data.

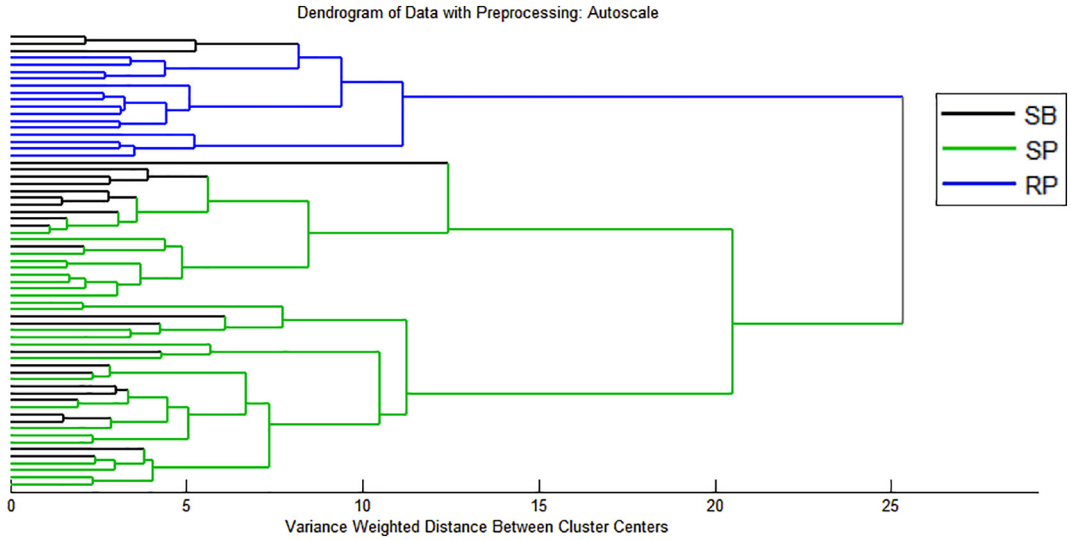

Fig. 3. Hierarchical Cluster Analysis (HCA) with pottery fragments excavated in three archaeological sites, Sambaqui do Bacanga (SB), Sambaqui da Panaquatira (SP) and Rabo de Porco (RP) using the EDXRF data.

Ti, Mn, Fe, Cu, Zn, Rb, Sr, Y and Zr, indicating that these elements are present in the composition of clay used in the manufacturing of those pieces of ceramic. For the grouping analysis, the net area averages of the chemical elements obtained in the ceramic bulk of all analyzed fragments were used, since the ceramic bulk characterizes the source of the clay used for manufacturing the artifacts and allows for identification of possible groupings of the fragments that were manufactured with the same clay source. In order to build the PCA and the HCA, the MATLAB® 2009a [22] software was used.

The PCA and HCA of the fragments of the three archaeological sites are shown in Figs. 2 to 5.

Analyzing the PCA results, the formation of two groups can be noted. The first one is composed of samples from the Bacanga and Panaquatira Shellmounds, and the second is composed by the Rabo de Porco samples. This result is also observed in the HCA.

These groupings indicate that RP group of fragments was manufactured with a different clay respect to the other two groups, and that the SB and SP fragments are from the same or similar clay source. These results make sense since SB and SP sites are located nearby at São Luiz and the RP site is located 60 km further away.

The differentiation of the two groups with the EDXRF data occurs due to the high Ca and Zn concentration of the SB and SP samples in relation to the RP samples, and also by the presence of Rb only in the RP samples. Table 2 shows the data of the average net areas of the elements of a representative fragment of each site studied.

In the case of PIXE, the differentiation occurs due to the high Si, Ti and Cu concentrations of the PR samples in relation to the SB and SP samples.

It may be said that the general agreement of the grouping results obtained by EDXRF and PIXE system are satisfactory, validating this type of potentiality of the two techniques.

3.2. Computed Radiography

The internal structure of the ceramic fragments observed by Computed Radiography revealed the presence of various sizes and types of

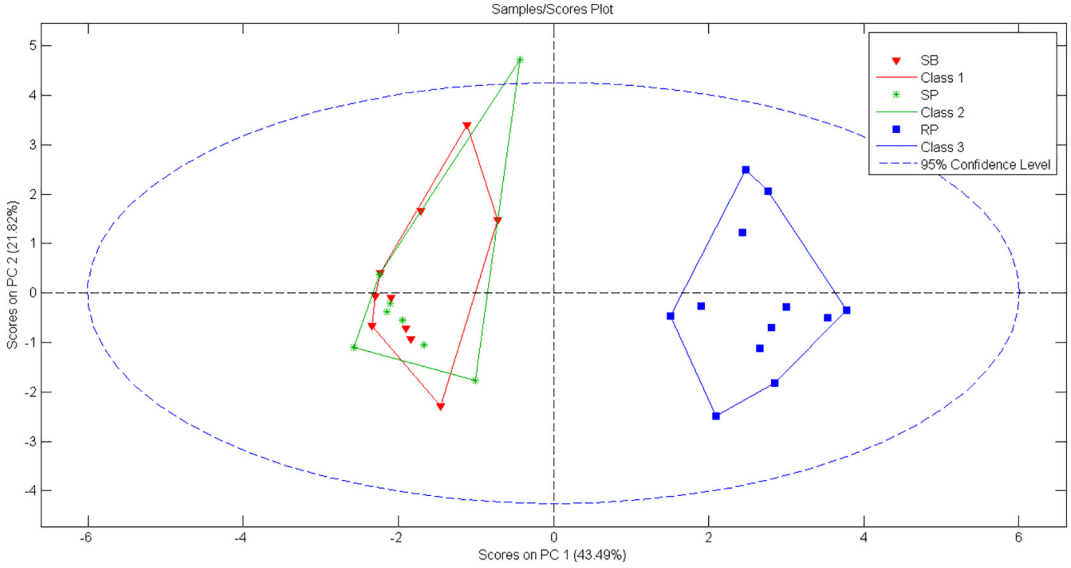

Fig. 4. Principal Component Analysis (PCA) with pottery fragments excavated in three archaeological sites, Sambaqui do Bacanga (SB), Sambaqui da Panaquatira (SP) and Rabo de Porco (RP) using the PIXE data.

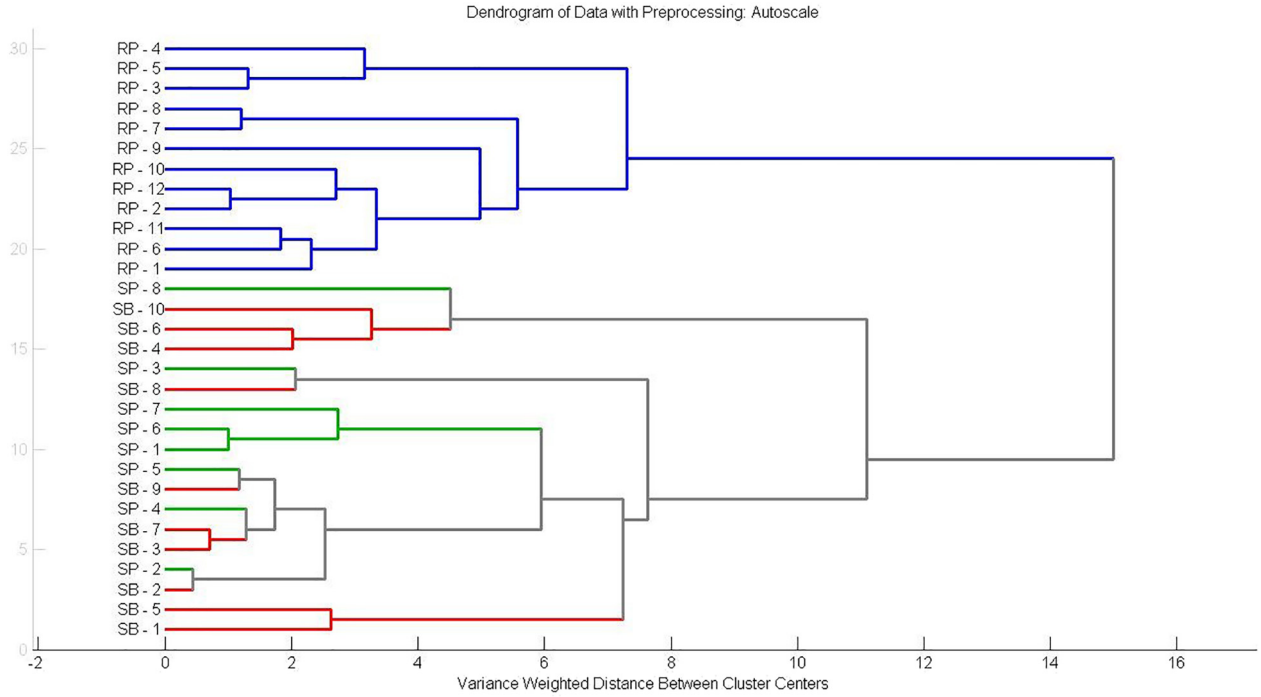

Fig. 5. Hierarchical Cluster Analysis (HCA) with pottery fragments excavated in three archaeological sites, Sambaqui do Bacanga (SB), Sambaqui da Panaquatira (SP) and Rabo de Porco (RP) using the PIXE data.

anti-plastics in the sherds. Fig. 6 shows the radiograph of three representative ceramic fragments, one from each site. The binarization of the image and the calculation of the percentage of anti-plastic in the samples were performed with ImageJ software.

The mean values of the anti-plastic percentage of the fragments of the Sambaqui do Bacanga (SB), Sambaqui da Panaquatira (SP) and Rabo de Porco (PR) fragments are 4.23%, 0.96% and 0.34%, respectively. It was observed that the SB samples had more seasoning than the other fragments, followed by the SP fragments and with a lower RP percentage. The anti-plastic concentration in the SB fragments is much larger than the fragments of the other sites. For this reason, two fragments of SB were analyzed by Optical microscopy.

3.3. Optical microscopy

The internal structure of the fragments from Sambaqui do Bacanga site was studied using Optical microscopy, performed with an Olympus BX-51 trinocular light microscope. Figs. 7 and 8 present the images of two fragments.

According to Fig. 7, it can be noticed that the composition and distribution of components are heterogeneous and with chaotic aspect. The matrix is composed of shell fragments, quartz grains with varied granularity (≤0.5 mm), mud nodes (pellets) with oriented internal structure and angled ceramic fragments. There are indications that the small elongated structures are thin straw mixed in the clay.

Fig. 8 shows an internal heterogeneous structure. The clay matrix is formed by shell fragments, quartz grains of varied sizes (from 0.5–0.8 mm to <0.05 mm), a few angled ceramic fragments and weak flow orientation in the sample body.

The SB fragments are characterized by using fragments of shells as anti-plastic. This peculiarity became a cultural marker very characteristic of the region, which resulted in the creation of a ceramist tradition denominated of Mina, in the decade of 1960. The use of the burnt and crushed shell as anti-plastic improves the handling of the clay. In this way, the shell can act in the increase of the resistance to the thermal shock and the impact of the ceramics [23].

4. Conclusions

Through the use of Multi-Varied Statistical analysis of EDXRF and PIXE measurements, the composition of fragments from the three archaeological sites were assigned to two distinct groups, the first one

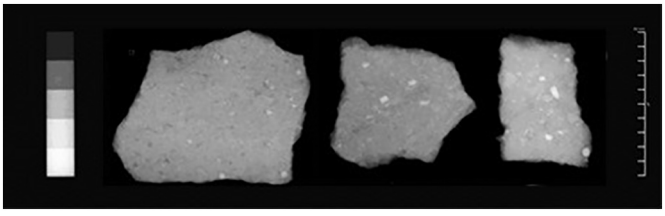

Fig. 6. Image obtained through Computed Radiography of the ceramic fragments of the Rabo de Porco, Sambaqui do Bacanga and Sambaqui da Panaquatira sites, respectively. Small piece of aluminum material with calibrated thicknesses were radiographed with the ceramic fragments. The calibrated thicknesses material varied between 0.2 and 1.0 cm.

Fig. 7. Image obtained of the ceramic fragment from 30 to 40 cm stratigraphic level of the Sambaqui do Bacana (SB) site.

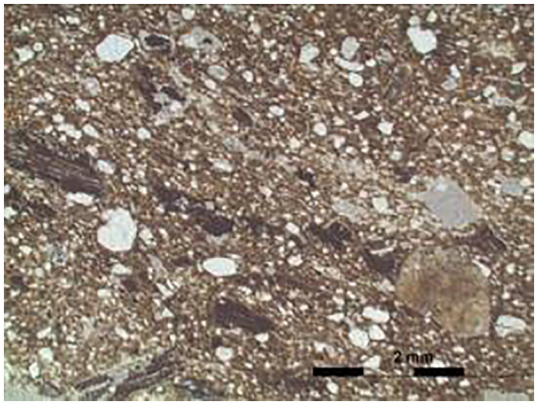

Fig. 8. Image obtained of the ceramic fragment from 150 cm stratigraphic level of the Sambaqui do Bacana (SB) site.

composed of samples from the Bacanga and Panaquatira Shellmounds, and the second composed only by the Rabo de Porco samples. This result indicates that each group of fragments was manufactured with different clays, and 'Sambaquis do Bacanga and Panaquatira' fragments are derived from the same source or same kind of clay. The differentiation of the two groups occurs due to the high Ca and Zn concentration of the SB and SP samples in relation to the RP samples, and also by the presence of Rb only in the RP samples.

The analysis of internal structure by Computed Radiography of the ceramic fragments indicated the presence of various sizes and types of anti-plastics added to the clay of the sherds.

Our study shows that PIXE, EDXRF and Computed Radiography, constitute powerful complementary approaches for ceramics characterization study and the grouping analysis, not least due to their representing non-destructive alternatives, an extremely important consideration for archaeologists, archaeometrists and local museum custodians alike.

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