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Nuclear and non-nuclear analytical techniques applied to pre-colonial archaeological ceramics from the upper Madeira River/Brazil (940 to 760 B.P)

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ABSTRACT

This article presents the results of applying three archaeometric techniques in archaeological ceramics from the upper river Madeira region in Amazonia. The first consists of the nuclear technique, neutron activation analysis (INAA), to identify the trace elements of the clay sources used in the production of the vessels. The second, non-nuclear X-ray diffraction (XRD), was used to characterise the mineral phases of each clay source. The third technique, electron paramagnetic resonance spectroscopy (EPR), estimated burning temperature related to the production stage of the artefacts. This approach made it possible to understand technological choices linked to selecting specific clay sources. The results suggest that different indigenous groups that inhabited the region between 940 and 760 B.P selected three clay sources to produce their vessels.

Keywords: **INAA, EPR, DRX, Pottery, Amazon Archaeology, Arqueometric.**

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1. INTRODUCTION

Amazonia is considered the largest tropical forest in the world, and the multiple landscapes that it contains, since remote times until the present, are imbued with the history of its indigenous peoples. Archaeological work realized in Rondônia state, Brazil, has uncovered a sequence of continuous human occupation throughout the Holocene [1]. Recent research in this area, at the Monte Castelo archaeological site, located on the Guaporé river, presents the oldest ceramics in the southwestern Amazon, dating to 5,200 BP [2].

Around the year 1,000 BP, which corresponds to a period of population growth across all of Amazonia, archaeological sites with thick ceramic deposits are documented on the margins and fluvial islands of the upper river Madeira, near the city of Porto Velho. These sites present radiocarbon dates between 930 and 760 BP, and are associated with ceramic artefacts produced using distinct technologies [4]. Following archaeological investigations, these ceramics were classified as Santo Antônio ceramics, Dionísio ceramics, and ceramics of the Amazon Polychrome Tradition, which correspond to distinct cultural groups that cohabited the region during this period [5].

The current study applied nuclear and non-nuclear techniques to these ceramics with the objective of verifying the validity of the classifications, made based on typological classifications, paste and burn types, and decorative aspects. The physico-chemical techniques employed are important in trying to comprehend the ceramic materials in relation to technological processes.

The samples selected for this study come from the serven archaeological sites located along the upper Madeira River: 1) Ilha de Santo Antônio, 2) Do Brejo, 3) Teotônio, 4) Ilha das Cobras, 5) Coração, 6) Ilha do Japó and 7) Ilha Dionísio. Corresponds to 30 whole and semi-whole vessels and 88 vessel fragments. Fragments of border (container top), base (container bottom), and container body were analyzed, the ones which presented painted and plastic decorations. It also used one sample of clay residue, interpreted as raw material in the production of the ceramic artefact.

The present study integrates different analytical techniques in order to generate quantitative and qualitative data on the chemical composition of pottery samples from the upper Madeira River, southwestern Amazon, and to contribute to the discussion on the process of raw material choice (clay sourcing) and firing techniques used for the production of archaeological vessels.

To contribute towards the classification and differentiation of pottery making practices within the region, neutron activation analysis (INAA) was carried out to determine the chemical composition of the ceramics related to the mass fractions of Na, K, La, Sm, Yb, Sc, Cr, Fe, Cs, Ce, Eu, Hf, and Th.

The data set was analysed by two statistical methods: cluster and discriminant analysis. Next, the samples were analysed by X-ray diffraction (XRD) to determine the mineralogical composition of the clays belonging to each group. With this technique, phengite, quartz, alunogen and schaurteite were refined.

EPR spectroscopy was applied to identify the firing temperature and determine whether there were similarities or differences in the firing processes between each group. The results indicated a temperature range between 450°C and 550°C, suggesting that firing was done in an oxidising atmosphere, similar to that identified in Bacabal pottery, also from southwest Amazonia [6].

The dates used in this study are all radiocarbon dates obtained from materials associated with the same archaeological layers from which the ceramics were collected. These analytical techniques have allowed us to characterise the clay sources used by the makers of these pottery traditions in the upper Madeira River.

The interface between archaeometric data and archaeological analyses complement previous archaeological interpretations about the decision-making processes behind pottery manufacture for each ceramic community in the past. In particular, it has enables the exploration of more varied elements of artefact production linked to types of paste (clay mixed with other organic and non-organic elements) and firing.

2. MATERIALS AND METHODS

2.1 Instrumental Neutron Activation Analysis (INAA)

The first step of sample extraction involves cleaning the surface of the ceramic with a rotating tungsten carbide file adapted to a variable speed drill to avoid any contamination. For most ceramic samples, it was necessary to drill holes in the side of the ceramic with a tungsten carbide drill (diameters of 1.5 and 2.2 mm) without drilling the respective wall. About 500 mg of the powder was collected and dried in an oven at 104°C for 24h [7].

To perform the analysis, about 100 mg of dry powder of each ceramic sample were weighed and packed into polyethylene wrappings sealed in a sealpack. Each envelope was wrapped in aluminium foil. Then, a set of 8 samples was assembled with approximately 100 mg of Standard Reference Material NIST-SRM 1633b (Constituent Elements in Coal Fly Ash) and the Sediment candidate certified reference material (RM), from Wageningen University, the Netherlands. After grouping the samples in parallel to receive the same neutron flux, they were irradiated for 8h in the IEA-R1 reactor of IPEN-CNEN / SP, under a thermal neutron flux in the order of 10^{12} cm^{-2} s^{-1} [8].

The measurements were performed in two stages, one after 7 days of decay to determine Na, K, La, Sm, Yb, Lu and U mass fractions elements concentrations, and the other after 25 to 30 days to determine Sc, Cr, Fe, Co, Zn, Rb, Cs, Ce, Eu, Fe, Hf, Ta and Th. All measurements were performed using the Ge hyperpure detector, model GX 1925 from Canberra, with a resolution of 1.90 keV at the 1332.49 keV gamma peak of ^{60}Co, with S-100 MCA of Canberra with 8192 channels. The Genie-2000 Gamma Acquisition & Analysis software, v. 3.1.a, developed by Canberra, analysed the gamma-ray spectra [8].

2.2 Statistcal Methods

The elementary concentrations of each sample were normalized by the transformed $\log_{10}$, a common procedure in archaeometric studies, whose function is to compensate for the large differences in magnitude between elements present at high and trace levels [9].

Subsequently, the average was calculated, alongside the standard deviation and maximum and minimum values for each ceramic fragment. After removing the discrepant samples using the Mahalanobis distance and Lambda Wilks criteria, the results of the elementary concentrations of 135 samples were subjected to statistical analysis. First, cluster analysis was applied to search for the presence of homogeneous groups of samples [10], adopting the criteria of similarity of quadratic Euclidean distance and as a Ward clustering algorithm, thus generating a dendrogram. Next, the discriminant analysis was performed to confirm the number of groups defined by cluster analysis.

The function of the discriminant analysis is to maximize the differences between two or more groups, ex-tracting new variables from the original variables [11].

2.3. X-ray Diffraction Analysis (XRD)

For XRD analyses, 6 samples of ceramic fragments were selected from the 3 groups formed after the discriminant clustering results. Approximately 100 mg of ceramic fragment powder was used in the sample holder. Grains with particle sizes smaller than 80 micrometres were selected. The sample was placed in the centre of a glass sample holder with dimensions of 15 x 10 x 2.0 mm, and a swab was carried out to remove excess material and obtain a uniform and smooth distribution. Then, the sample holder was coupled to the goniometer of the diffractometer. The XRD measurements were made in a RIGAKU X-ray diffractometer, model SmartLab SE. The X-rays used in the measurements were from the Kα band of copper with wavelengths (λ) of 1.540593 Å. With a 0.01° step and 1.0s of reading for each step, starting at 10° and 60º ending. The diffractogram data were analysed using the HighScore Plus software, version 4.9 (2019).

XRD is a technique used for characterizing crystalline materials. The incident X-ray beam on the sample may be deflected by the atoms, detected, and plotted as a curve using this approach (diffractogram). The position of peaks in a diffractogram is an exact representation of how these atoms are arranged within a crystal. The position of the peaks (qualitative analysis), peak breadth (size and form of the crystal), and peak intensity will affect the analysis and interpretation of the diffractogram (quantitative analysis). The X-ray diffraction technique allows for the separation of crystalline compounds with the same chemical content but distinct atomic configurations [12].

Knowing the position, distance, and intensity allows you to create a diffractogram. Bragg's law may then be used to determine the peak location and interplanar distance [13]:

$$2d\sin\theta(hkl) = n\lambda \tag{3}$$

d: distance between the crystalline planes;

θ: angle of incidence of the X-ray beam;

n: order of diffraction;

hkl: Miller indices;
λ: wavelength.

According to the structural factor, the intensity of the diffracted X-ray beam is connected to the type and position of the atoms [13]:

$$F_{calc,hkl} = \sum_{atomos j} f_j \left(\frac{\sin\theta}{\lambda}\right) e^{-B_j \left(\frac{sin\theta}{\lambda}\right)^2} e^{2\pi i\left(hx_j+ky_j+lz_j\right)} \tag{4}$$

xj, yj, and zj are the coordinates of atom j in the unit cell;
fj (senθ/λ) the shape factor of atom j;
Bj the thermal agitation factor.

The crystalline phase of a sample can be determined and quantified using several refining procedures. This study used, the Rietveld refining approach, wich is commonly employed in diffractograms with very close and overlapping peaks. The theoretical basis for this approach is to compare the diffractogram derived from the sample model to the simulated model. The experimental parameters are adjusted so that the simulated diffractogram is close to the experimental diffractogram for this comparison. The mathematical approach of least squares is used to adjust the refining settings. The quality of refinement evaluation was achieved in this study by comparing multiple values for the weighted reliability factor Rwp [14].

2.4. Electron Paramagnetic Resonance spectroscopy (EPR)

The EPR analyses 6 samples of ceramic fragments selected from the 3 groups formed after the discriminant clustering results. Approximately 11 aliquots for each pottery fragment sample were prepared. 25 mg of ceramic powder with a granulometry smaller than 100 micrometres was used for each aliquot. Then, each sample's aliquots were transferred to porcelain crucibles and fired in a muffle furnace from 350°C, with increments of 50°C to 1000°C for 30min. The g-factor of Fe^{3+} of the ceramic fragment powder was measured using a Bruker MiniScope MS5000 EPR spectrometer. Measurements were performed with 25 mg of ceramic powder aliquots placed in quartz tubes of 4.3 cm diameter. For EPR measurements, the following spectrometer experimental parameters were used: magnetic field modulation frequency of 0.1 mT, microwave power of 3.991 mW, scanning range of 150 to 550 mT, and scan time of the 60s.

Electron paramagnetic resonance (EPR) is a spectroscopic method of chemical analysis that recognizes free radicals and paramagnetic species as structural flaws. A magnético and microwave fiedl are applied to the sample at the same time. To generate transitions, these fields interact with unpaired electrons in the sample. The spin of the paramagnetic centre may be identified by its position in the magnetic field (g-tensor) and the spacing of the lines [15].

The spin levels are separated by an external magnetic field H_o, and a photon of energy hv is absorbed by a second magnetic field perpendicular to the first (resonance condition) [16].

$$h\nu = \Delta E = g\mu B H_o \tag{5}$$

g = g-factor

μB = Bohr magneton

H_o = magnetic field value at resonance condition

The firing temperature of the prehistoric pottery fragments was determined using the EPR technique. The EPR signal is connected to the paramagnetic species Fe^{3+} while examining the firing temperature of ceramic shards [17].

The firing temperature of the archaeological pottery pieces was estimated using the assumption that when the g-factor reaches the temperature at which the ceramic fragment was fired, it suffers a perturbation and its value changes. This variation in the g-factor value is caused by variations in the symmetry of Fe^{3+} [18].

3. RESULTS AND DISCUSSION

Eighteen samples of the reference material were analysed in this research to determine the mass fractions of the following elements: Na, K, La, Sm, Yb, Lu, Sc, Cr, Fe, Co, Zn, Rb, Cs, Ce, Eu, Hf, Ta and Th. This analysis aimed to assess the quality control of the analytical method. In order to examine the homogeneity of the observations RM material, the r max and r min criteria were utilised [19, 20]. These experimental values were compared with the values tabulated for a significance level of 0.05 and with n-2 degrees of freedom [19]. After verifying the precision for each element, the results showed that most elements presented an RSD $\leq$ 10%. Elements such as Lu and Ta were removed because the precision was > 10%.

The determination of Zn was not reliable due to substantial gamma-ray interference by ^{46}Sc and ^{182}Ta. Although Co has an RSD of less than 10%, it was not included in the data set because the determination can be affected by tungsten carbides files. Thus, the elements used to interpret the data set were Na, K, La, Sm, Yb, Sc, Cr, Fe, Cs, Ce, Eu, Hf, and Th. After choosing the elements, the concentrations were normalised using transformed $\log_{10}$, a standard procedure used in archaeometry studies to compensate for significant differences in magnitude between high- and trace-level elements [20]. Subsequently, the average, standard deviation, maximum and minimum values were calculated for each ceramic fragment. After removing the discrepant samples using the *Mahalanobis* distance and the Lambda Wilks criteria, the elementary concentrations of 118 samples were subjected to statistical analysis.

Cluster analysis was applied to identify the presence of homogeneous groups of samples [21], adopting the criterion of similarity with the squared *Euclidean* distance and as a *Ward* clustering algorithm, it was possible to generate a dendrogram. Then, discriminant analysis was performed to confirm the number of groups defined by the cluster analysis. The function of the discriminant analysis is to maximise the differences between two or more groups and extract new variables from the original variables [22]. The results showed the presence of three compositional groups, indicating different clay sources of raw material in the manufacture of ceramic artefacts (Figure 1).

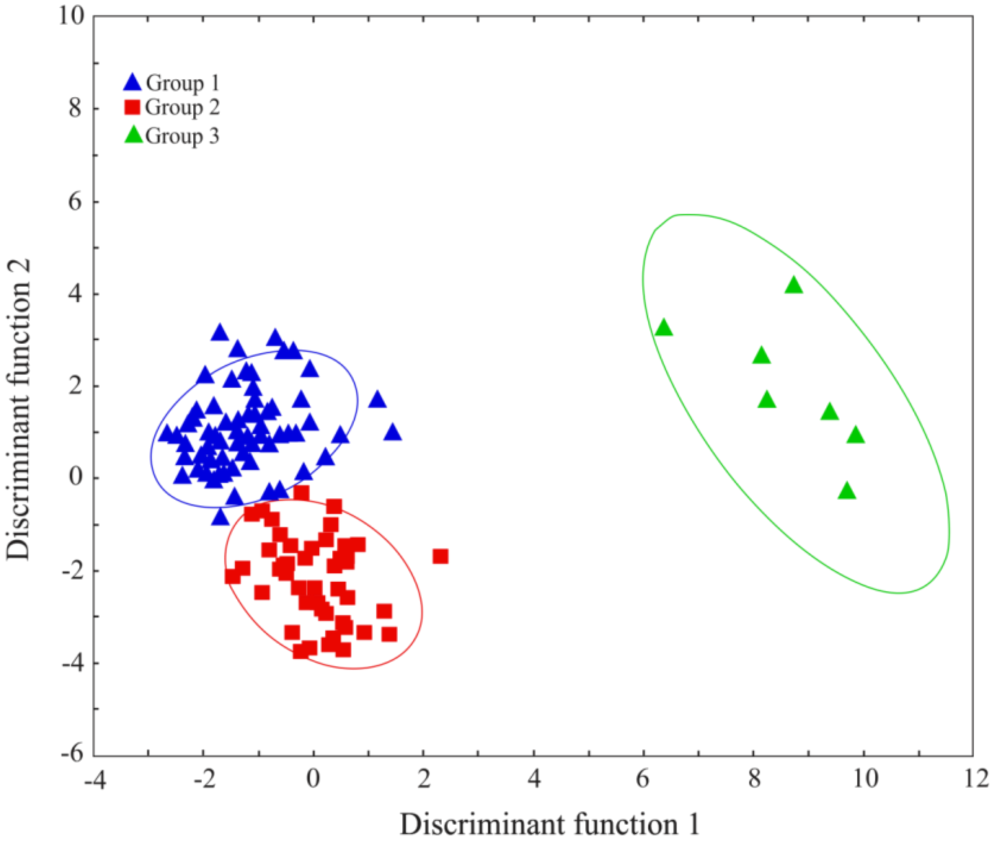

Figure 1: *Discriminant function 1 versus discriminant function 2. Ellipses represent a confidence level of 85%.*

Approximately 2 samples from each group identified in the discriminant analysis were selected to determine their mineralogical structure by XRD analysis. The criteria, in this case, were fragments from the archaeological layer dated by radiocarbon from the period between 940 and 760 B.P. We therefore selected fragments from the Santo Antônio ceramic assemblage collected at the Ilha de Santo Antônio (one funerary urn fragment and one fragment from the neck of a vessel used to serve liquids) and Do Brejo (possible funerary urn) archaeological sites; fragments from the Dionísio assemblage (a necked vessel used to store liquids, also used as a funerary urn) collected at the Ilha Dionísio site; and fragments from the Polychrome Tradition assemblage (a funerary urn lid) collected at the Coração site.

The XRD results show the presence of Mg-phengite phase, quartz, alunogen and schaurteite. The Mg-phengite phase is present in all groups. It is observed in all samples as a relative concentration of K, Fe and rare earth batteries (REEs) characteristic of the chemical composition of the Mg-phengite phase. Group 2 is characterised by the absence of this phase. Group 3 presents the highest concentration of Fe, K and REEs compared to the other groups, and this characteristic may be related to a more significant presence of Mg-phengite type 2 and 3 origin and well-crystallised lath forms (Figure 2).

In the archaeological analysis, paste characterisation was carried out with a binocular loop microscope, with Group 1 being characterised by the presence of caraipé (tree bark) temper, which is high in silica, mixed with quartz, charcoal and – in some cases – feldspar and mica. The presence of phengite could be an effect of the presence of mica, which was identified in smaller amounts in the samples from Ilha de Santo Antônio and Do Brejo.

In the case of the Group 2 ceramic paste, the microscopic analysis identified temper of cauixi (freshwater sponge spicule), considered animal-derived silica, considered an animal-derived silica, that was mixed with quartz and possibly haematite in the sample from the Ilha Dionísio site. However, the XRD analysis detected a high concentration of phengite in relation to other argilominerals, which could be related to the presence of mica not detected with the binocular loop microscope. The absence of alugogen in this sample could be explained by the absence of feldspar.

The results for Group 3 demonstrated the presence of the same argilominerals as in Group 1, albeit with a more negligible contribution of alunogen, perhaps suggesting lower concentrations of feldspar in the utilised clay source.

The presence of some of the argilominerals detected by XRD analysis, such as feldspar, could result either from the formation history of the clay source, or from their addition by the ceramic maker at the paste preparation stage. Mica is unlikely to have been added, since it occurs naturally in most clay sources.

The clay collected for ceramic artefact production undergoes a phase whereby certain elements are added or eliminated according to the sociocultural norms of each indigenous group [23]. The inclusion of certain elements, such as caraipé or cauixi, is intended to reduce the plasticity of the clay, hence why they are termed antiplastics or tempers. By contrast, it is harder to determine the intentionality behind the inclusion of minerals.

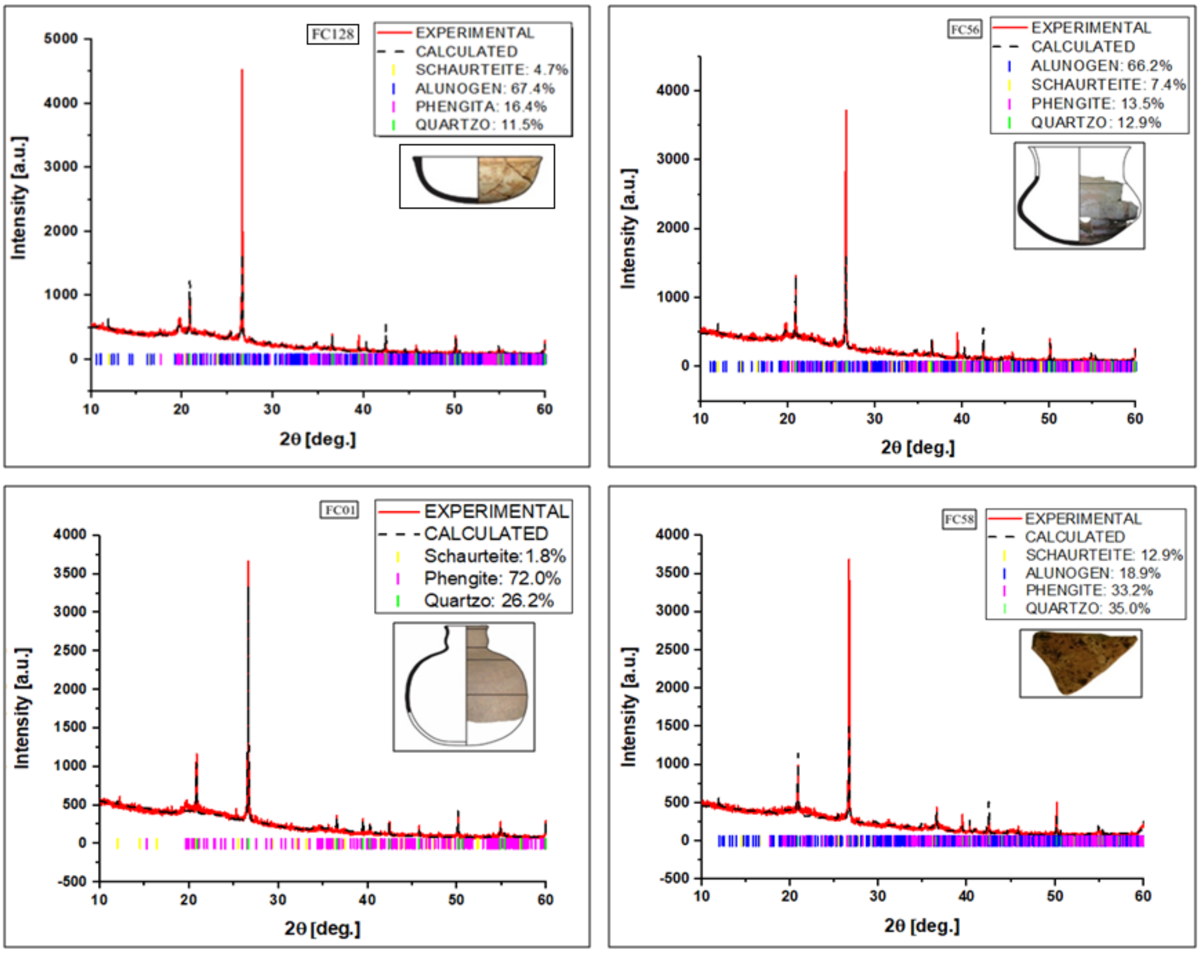

Figure 2: *Results of the DRX analysis of samples FC128 and FC56 from group 1, sample FC01 from group 2 and sample FC58 from group 3.*

The firing temperature of the ceramic piece was determined using variations in the electronic paramagnetic resonance (EPR) spectrum. Figure 3 shows the Fe^{3+} EPR spectra of ceramic powder. The paramagnetic species Fe^{3+} is heat sensitive and a variation in the value of the g-factor is seen after repeated heating [17, 18]. Equation 5 may be used to derive the g-factor of the Fe^{3+} EPR spectrum by substituting the value for the magneton of Bohr and the value of the experimental H_o.

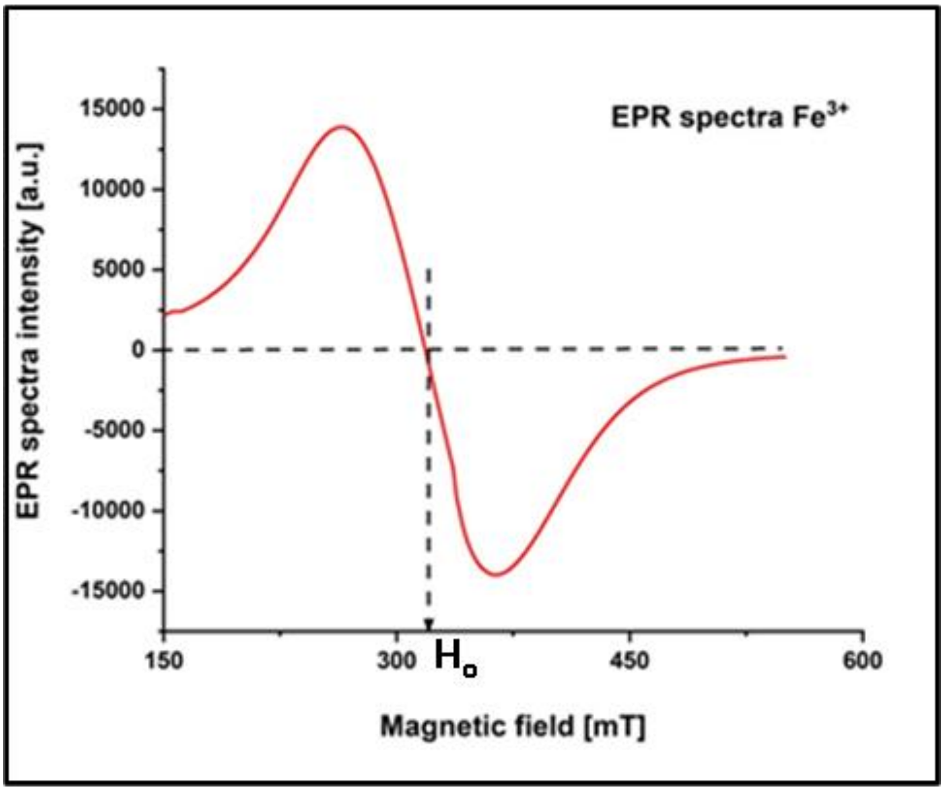

Figure 3: *The first derivative of the EPR spectra obtained from the clay.*

The sample was subjected to a series of heatings treatments ranging from 350 ºC to 1000 ºC. When the sample was annealed in temperature, there was a considerable shift in the EPR spectrum. Figure 4 depicts the EPR spectra of Fe^{3+} samples collected at ten different temperatures [17]. Figure 4 shows the minor displacement of H$_o$ generated by the experimental firing of the sample when it reaches the firing temperature.

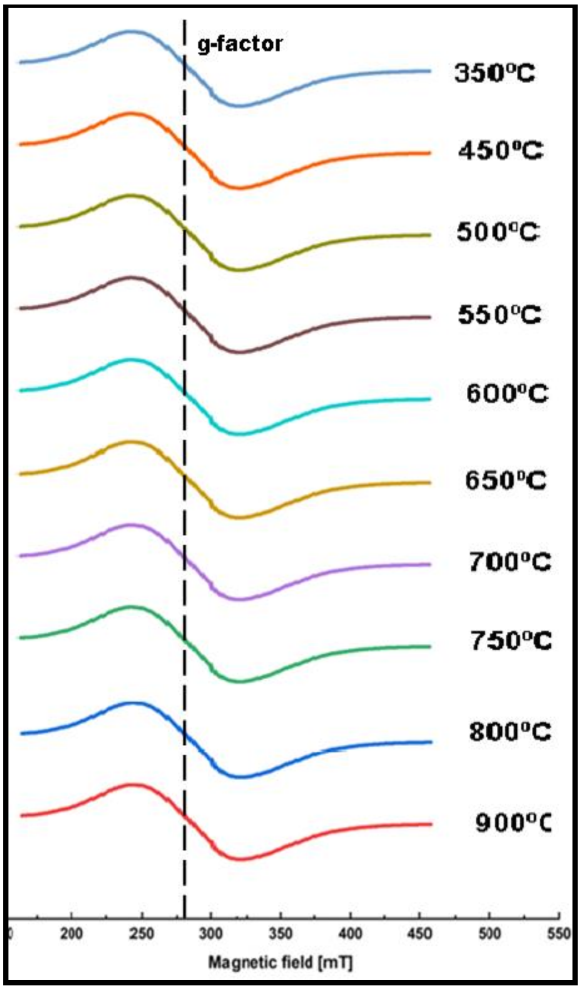

Figure 4: *The EPR spectra recorded for the raw clay and the thermally treated clay from 350 °C to 900 °C for 30min.*

Figure 5 depicts the firing temperature of ceramic pieces. Since the Fe^{3+} paramagnetic species, with g 2.15, shifted in this region after annealing [24], it is assumed that the firing temperature was less than 550 °C (Table 1). This suggests that the in question were manufactured in an open fire.

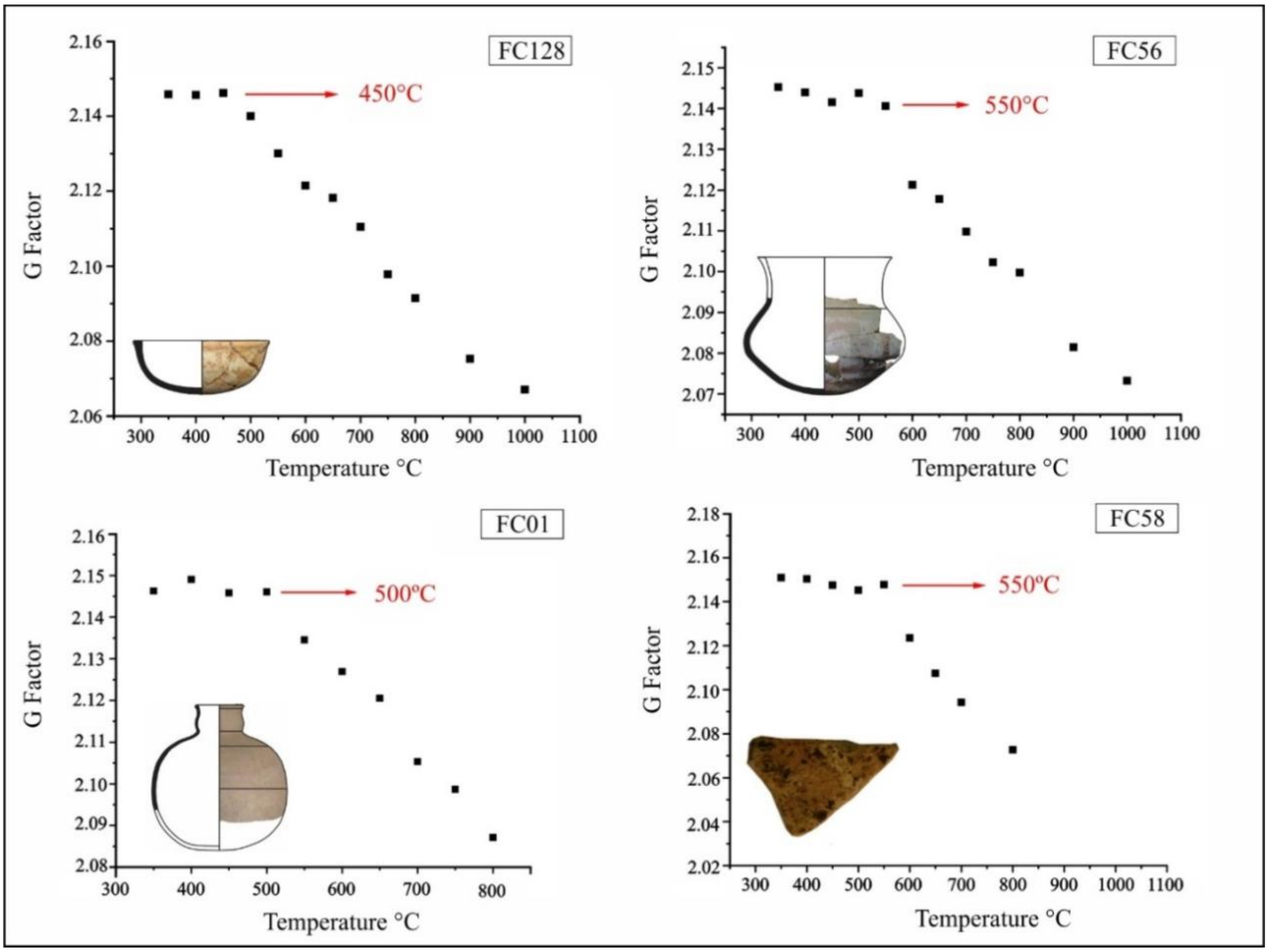

Figure 5: *Results of the EPR technique for samples FC128 and FC56 from group 1, sample FC01 from group 2, and sample FC58 from group 3.*

The same firing temperature range was observed in Bacabal ceramics from southern Rondônia [25]. Ceramic samples from the Amazonian Polychrome Tradition, associated with Group 1 (FC128), were fired at 450 ± 50 ºC. This result contrasts with the predictions of archaeological analysis, which generally identifies "oxidative burns" (in open environments) when the pottery is lighter or has shades of red, orange and brown, and "reductive burns" (at high temperatures in closed environments) for pottery with greyish tones [23, 26].

Table 1: Pottery firing temperature and their respective uncertainties (σ).

Site	Group	Samples	Pottery Firing Temperature ± σ
Coração	1	FC128	450 ± 50
Ilha de Santo Antônio	1	FC56	550 ± 50
Do Brejo	1	FC78	500 ± 50
Ilha Dionísio	2	FC01	500 ± 50
Ilha de Santo Antônio	3	FC58	550 ± 50

As can be seen in the burning temperature results by EPR analysis, the group 1 samples were all classified as having undergone reductive firing (identified in the breaks in the fragments). The Group 2 and 3 samples as having undergone oxidised firing. Ceramic samples from *Santo Antônio* (FC56 and FC58) and *Do Brejo* (FC78), assembled in Groups 1 and 3, are in the 500 to 550 ºC firing range.

The Dionísio ceramic sample FC01, Group 2, was fired at 500 ± 50 ºC. Sample FC02 from the Dionísio site showed a substantial variation in the value of the g-factor, making it impossible to determine the firing temperature.

4. CONCLUSION

The nuclear and non-nuclear analytical techniques used in this work showed that the indigenous communities that produced the Santo Antônio pottery and those that produced Amazon Polychrome Tradition pottery made their artefacts using mainly the Group 1 clay source. The firing temperature of this material varies between 450-550°C. In addition to the Group 1 clay source, the producers of Santo Antônio pottery also exploited a second clay source assembled in Group 3 rich in Fe^{3+}.

Geological studies conducted in the region report the presence of the River *Madeira* Formation, distributed along both margins of the homonymous river and in the interior of the Abunã river basin. The stratigraphic description of this formation includes inferior strata composed of very plastic clay with a grafite graphite to light grey colouration and contain rare quartz grains [27]. The present study hypothesises that the producers of *Santo Antônio* and Polychrome Tradition ceramics could have exploited clay sources linked to this formation, given that their firing temperature did not exceed 550 ºC.

Meanwhile, the indigenous group that produced *Dionísio* pottery and inhabited the Ilha *Dionísio* site [28] used a different clay source (Group 2), characterised by the absence of alunogen. The firing temperature of this material varies between 500-550 °C.

Our results demonstrate that the producers of Santo Antônio, Dionísio and Polychrome Tradition ceramics fired their artefacts at low temperatures in an oxidising environment. The characterisation of these materials through analytical techniques complements studies about each community's manufacturing processes and technological choices that produced these ceramics. INAA allowed the detection of raw material provenance, while XRD characterised the clay minerals phases presented in each clay source, and EPR offered a way of estimating firing temperature.

This approach has contributed to understanding the technological choices linked to selecting specific clay sources [29]. The results suggest that these different indigenous groups who inhabited the upper Madeira River region between 940-760 B.P selected three clay sources to produce their vessels.

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