EDX, Raman, Optical Microscopy and Multivariate Statistics Analysis of Archaeological Pottery from São Luiz, MA, Brazil

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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 a hundred thirty-three representative pottery fragments selected of stratigraphic levels from the surface to 170 cm deep for all sites. The three analytical methods employed were EDX, Raman Spectroscopy and optical microscopy. EDX measurements were performed with a Shimadzu Energy Dispersive X-Ray Fluorescence Spectrometer (EDX – 720), which consists of an X-ray tube (Rh target) to excite the samples, a Si(Li) detector and 3mm collimator. Raman spectroscopy was performed with an Inspector Raman DeltaNu, equipped with a 785 nm diode laser for excitation, which has a maximum output power of 120mW, 8 cm⁻¹ resolution and spectral range from 200 to 2000 cm⁻¹. Optical microscopy was performed with an Olympus BX-51 trinocular light microscope. The internal structure of the ceramic pieces observed by optical microscopy revealed the presence of shells fragments as anti-plastics in the sherds, and quartz grains with prevailing dimensions between 0.1 and 0.05 mm. The careful analysis of the Raman spectra revealed the presence of Hematite, Quartz, Wollastonite and Albite, and indicated the presence of Magnetite, Rutile phase of TiO₂, Ilmenite and Apatite. The presence of Wollastonite indicates crystalline phase formed between 600 and 800 ºC and the presence of Albite indicates that the firing temperature was below 950ºC. This implies a range for the burning temperature from 600 to 950°C, in an oxidant firing atmosphere, indicated by the presence of hematite. In addition to these informations inferred on the ceramic samples, it was also possible to indicate the hardness of the samples by the detected minerals. Hematite has hardness in the range 5.5 to 6.5 Mohs and quartz has hardness of 7 Mohs. Therefore, the hardness of the ceramic samples analyzed should be in the range from 5.5 to 7 Mohs. Eighteen elements were measured with good statistics in the different ceramic samples through EDXRF analysis: Al, Si, P, S, K, Ca, Ti, Cr, Mn, Fe, Cu, Zn, Rb, Sr, Y, Br, Zr and Nb. 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.