

## **CHRONOLOGICAL STUDY OF ARCHAEOLOGICAL SITES FROM CANINDÉ DE SÃO FRANCISCO, SERGIPE STATE, BRAZIL, BY THERMOLUMINESCENCE TECHNIQUE.**

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### **ABSTRACT**

The ceramic vestiges rescued in the archaeological excavations have been considered important sources of chronological information because they constitute direct fossil of certain cultural periods. The Thermoluminescence Method for dating of inorganics materials has made the determination of ancient ceramics ages possible with adequate precision and accuracy. The intensity of the thermoluminescence emission is proportional to the energy deposited in the crystals, present in the pottery, by means of natural radioisotope material irradiation (U, Th, K and Rb) and cosmic rays. With the objective of determining the chronological profile of archaeological sites from “Xingó”, situated in the Canindé do São Francisco city, Sergipe State, Brazilian Northeast, the ages of 4 pottery samples from Justino, São José, Saco da Onça and Curituba sites were determined in this work. The ceramic collection has great expression because many ceramics are associated to mortuary rites. The acquisition of the site chronology studied in this work is a contribution to studies of ancient communities that lived in the region. The additive dose method was used to determine of the paleodose in the samples, and the TL response of 320 °C peak as function of the added dose was linear. The annual dose rate was determined by means of the instrumental neutron activation analysis, using the U, Th, K and RB concentration from soil and own sample, which yielded an average annual dose rate of  $2.89 \pm 0.33$  mGy.yr<sup>-1</sup>. Using of the paleodose and annual dose rate, the ages were determined which remained between 457 – 3716 BP, value which matches with the results obtained by carbon – 14 method in the sample from burned charcoal in Justino site.

### **1. INTRODUCTION**

The luminescence stimulated thermally (thermoluminescence – TL) has been applied in several archaeological studies to dating of inorganic materials (Wintle, 1997). The TL

emission is result of the light releases charge from light – sensitive traps in crystals of minerals such as quartz and feldspar. The amount of the light released is proportional to the energy deposited by natural radiation from natural radioisotopes ( $^{235}\text{U}$ ,  $^{238}\text{U}$ ,  $^{232}\text{Th}$  and  $^{40}\text{K}$ ) present in the soil and own sample, and also from cosmic radiation (Guibert et al, 1998). To establishing of the pottery samples TL age are necessary the absorbed dose of ionizing radiation (paleodose) and the annual dose rate.

In this work the TL method was used for dating the pottery samples from 4 archaeological sites: Justino, São José, Saco da Onça and Curitiba. The archaeological sites are situated in Canindé do São Francisco, a city in the area of São Francisco River, about 150 km from Aracaju, capital of Sergipe State, Brazil.

The fragments used for dating in this work are from vessel excavated during a rescue archaeological project. This rescue occurred when a large alluvial terrain on the margins of São Francisco River, in the area of Canindé do São Francisco, Brazilian Northeast, was flooded due to the construction of the Hydroelectric Xingó Dam.

Archeological studies accomplished in the area have shown that the potteries of the sites studied have the same technical profile (Luna, 1997). Recent studies carried out in the area have shown the existence of an independent ceramist group, with no relation to the ceramist group well established in the Brazilian Northeast, called Tupiguarani and Aratu (Martin, 2000). Dating obtained by means of carbon-14 from skeletons has indicated that in the “Xingó” region there is evidence of the human occupations 9,000 years BP.

For this reason, the chronology study done in this work by thermoluminescence method aims to contribute to studies related to the establishment of “new traditions”.

## 2. EXPERIMENTAL METHODOLOGY

The entire surfaces of the ceramics were polished and they were then gently crushed. After sieving, grains of diameter between 75 – 150  $\mu\text{m}$  were chemically treated. Carbonate phases were removed by HCl (50%) for 2 h and organic matter was removed by washing for 4 h in NaOH (6M). During multiple rinsing in distilled water, the clayed fraction was consecutively removed. An additional treatment by immersion in dilute HF (20%) for 1 h was also performed to dissolve remaining clay materials that coated quartz and to eliminate the contribution of the alpha radiation. After being immersed in HF the sample was treated in HCl to eliminate fluorides. Through of the procedure above the quartz fraction of the 75 to 150  $\mu\text{m}$  served as basic material for thermoluminescence measurements.

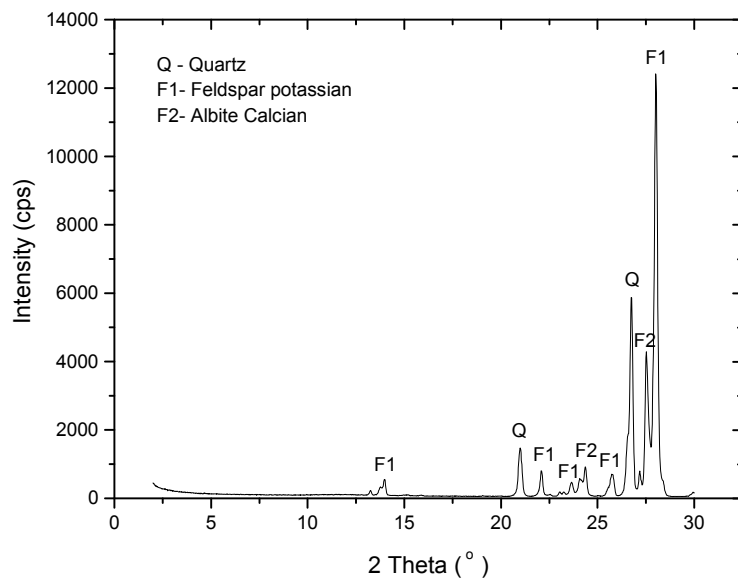
The additive dose method was used to determine the equivalent dose (paleodose) in the samples (Aitken, 1985). The TL curves were recorded at room temperature to 400°C using a heating rate 4  $^{\circ}\text{C}.\text{s}^{-1}$  in a home made TL reader, equipped with EMI photomultiplier, model 9789QB, with sample compartment and associated electronic. Prior to readout, the samples were preheated to 100 – 150  $^{\circ}\text{C}$  on the planchet, to eliminated low temperature TL from laboratory irradiated samples.

For irradiation, a  $^{90}\text{Sr}/^{90}\text{Y}$  source was used which delivered 0.415  $\text{Gy}.\text{min}^{-1}$  to the quartz sample in the standard.

The annual natural dose rate was determined by means of the Instrumental Neutron Activation Analysis (INAA) using the Uranium, Thorium and Potassium content in the soil and in the sample. To INAA, the sample was powered and dried at 105 °C for 24 h. The powder was weighed into a polyethylene bag, which was packed in aluminum foil and irradiated into the research reactor IEA-R1m with a thermal flux of about  $5.10^{12} \text{ n.cm}^{-2}.\text{s}^{-1}$  for 8 h. The gamma spectrometry was carried out using Ge (hyperpure) detector, with 1.90 keV resolution at the 1,332.49 keV gamma peak of  $^{60}\text{Co}$ , coupled with Canberra S – 100 MCA, constituted with 8192 channels. These measurements were divided in two, one realized after of 7 cooling days for K and U determination and after 30 cooling days for Th determination.

### 3.RESULTS AND DISCUSSION

After the sample preparation an X-ray diffractometry was performed to verify the crystallography obtained. The X - ray diffractogram in Figure 1 shows that the sample obtained was predominantly quartz and feldspar.



**Figure 1 – X – ray diffractogram of sample prepared for TL dating.**

TL glow curves of natural and natural plus  $^{90}\text{Sr}/^{90}\text{Y}$ -dose of 10, 20, 30 and 40 Gy were recorded. The Figure 2 provides a typical glow curve of the sample collected from Justino site. Peaks are seen at 200 and 320 °C. The plateau test not shown here indicates that 320 °C TL peak can be used for dating.

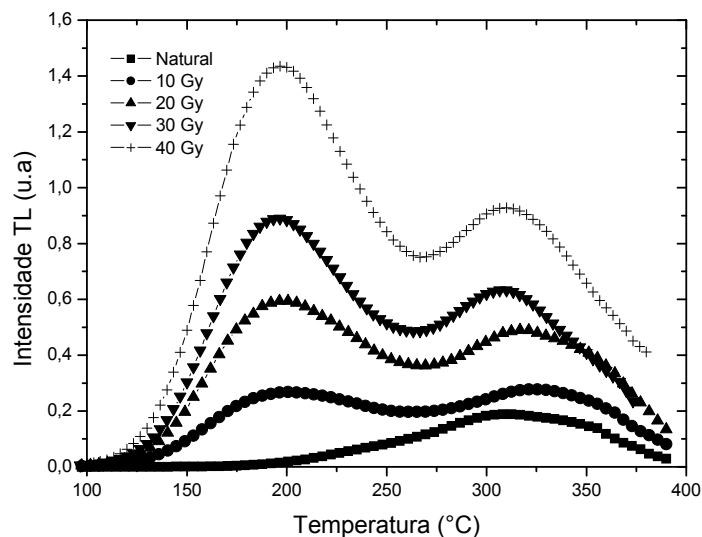
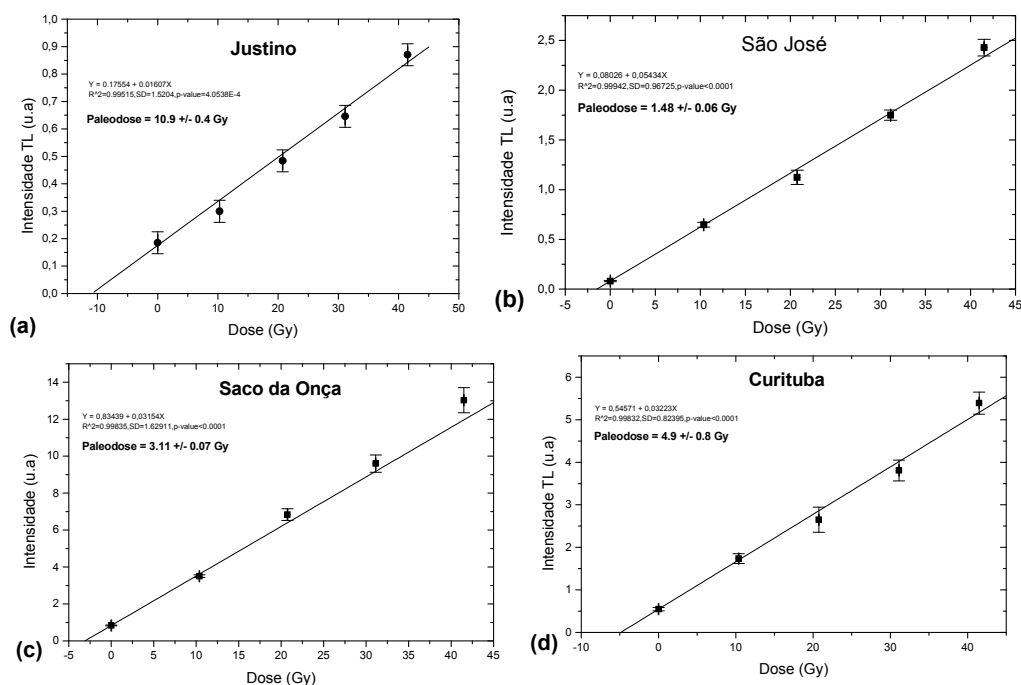


Figure 2 – Glow curve for natural and natural plus  $^{90}\text{Sr}/^{90}\text{Y}$  doses of 10, 20, 30 and 40.

The Figures 3 – 6 show the TL response of 320 °C peak as function of the added dose. The TL response to additive dose for all samples, seen in the Figures 3 – 6, could be fitted by linear equation. By extrapolating these curves to dose axis the paleodoses were obtained for the 4 pottery studies in this work. The results showed that the paleodoses are  $10.9 \pm 0.4$  Gy,  $1.48 \pm 0.06$  Gy,  $3.11 \pm 0.07$  and  $4.90 \pm 0.08$  Gy for Justino, São José, Saco da Onça and Curitiba, respectively.



Figures 3 – TL responses as function of additive dose for 320°C TL peaks for natural plus additional laboratory dose. The plots (a), (b), (c) and (d) correspond to Justino, São José, Saco da Onça and Curitiba, respectively

The INAA yielded an average of  $^{238}\text{U}$ ,  $^{232}\text{Th}$ , Rb, and  $^{40}\text{K}$  are  $3.71 \pm 0.48$  ppm,  $12.83 \pm 2.35$  ppm,  $77.82 \pm 12.68$  and  $2.45 \pm 0.29$ , respectively, where the error is a standard deviations. According to these values it can be observed that there is not a big difference in the values to determine of annual dose. From these values the annual dose rate was calculated using Ikeya's equation (Ikeya, 1993). Considering that the cosmic rays contribution is of  $250 \mu\text{Gy/yr}$ , an average annual dose rate of  $2.89 \pm 0.33 \text{ mGy.yr}^{-1}$  was obtained. The age estimation was done by means of paleodoses and dose rates cited above. The estimated ages are listed in the table 1.

**Table 1 – TL dating of pottery fragments from Xingó sites. Total uncertainty was obtained by propagation of uncertainty of the uncertainty of the paleodose and annual dose rate.**

Sample	Paleodose (Gy)	Estimated age (BP)
Justino (phase 15)	$10.9 \pm 0.4$	$3716 \pm 398$
São José (phase 08)	$1.48 \pm 0.06$	$457 \pm 35$
Saco da Onça (phase 06)	$3.11 \pm 0.07$	$1021 \pm 190$
Curituba (phase 09)	$4.90 \pm 0.08 \text{ Gy}$	$1640 \pm 140$

#### 4. CONCLUSIONS

In this work was seen that the  $320^\circ\text{C}$  TL peak of the quartz exhibited a response linear as function of additive dose. The ceramic age estimation was done from the estimated paleodose and annual dose rate. It was found that samples from Justino, São José, Saco da onça, and Curituba sites presented ages  $3716 \pm 398$ ,  $457 \pm 35$ ,  $1021 \pm 190$  and  $1640 \pm 140$ , respectively. The burned charcoal sample collected from Justino site (phase 15) and dated by carbon – 14 method indicates an age of about  $3270 \pm 135 \text{ BP}$  (phase 13) and agrees with the result obtained by TL method in this work. Therefore, the TL dating can contributed significantly for establishing the chronology of the ancient community of “Xingó” region.

#### ACKNOWLEDGMENTS

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