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Distinguishing burnt from partly bleached unburnt quartz pebbles of Pedra Furada, Brazil

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Abstract: Discussed in this article are the effects of light and heat on the TL emissions of quartz pebbles from Brazilian paleoindian site of Pedra Furada. The means used to distinguish specimens heated in prehistoric hearths and datable by TL from unheated and partly sunbleached will be discussed.

Introduction

Excavations of the Upper Pleistocene prehistoric site of *Toca do Boqueirão do Sítio da Pedra Furada* (Piaui, Brazil) have uncovered a series of occupation levels radiocarbon dated to between 5,000 and over 48,000 years BP (Guidon and Delibrias, 1986; Guidon *et al.*, 1994). Since the lower strata gave ages at the limit of C-14 dating it seemed advisable to try and date them by some other method. TL seemed appropriate since the ancient hearths at the site yielded transparent quartz pebbles showing such signs of past heating as surface reddening.

One of the problems encountered in trying to date them by TL was how to tell adequately heated dateable specimens from unheated ones that might have been partially sunbleached before burial, because preliminary measurements indicated that the traditional criteria whereby saturation of the TL signal is used to tell burnt quartz from unburnt were unsuitable for the quartz pebbles from Pedra Furada. The TL signal varied from sample to sample but increased in all instances after additional irradiation in the laboratory. Since these relatively translucent pebbles were protected from light after excavation one must assume that before being covered by sediment they remained exposed to sunlight for an unspecified period of time.

Effects of light exposure on quartz specimens

Numerous studies (Wintle, 1979; Aitken, 1985; Spooner *et al.*, 1988; Smith *et al.*, 1990; Zhou and Wintle, 1994; Feathers, 1997) have shown that when sedimentary quartz grains are exposed to sun light

their TL is partially bleached. To see if the BPF pebbles have indeed been bleached, several randomly-selected quartz pebbles from the site were crushed, sieved, and the 100-160 µm fraction subjected to bleaching experiments. The TL was measured in our TL oven equipped with a Thorn EMI 9635QB photomultiplier and a 2 mm thick MTO UV filter with a maximum of transmission at 325 nm which allowed TL measurements up to 600°C (Valladas and Valladas, 1979); the heating rate was 10°s⁻¹. The curves in all the figures of this article have been traced after the background emission has been substracted. Figures 1A-2A show the natural TL of samples BPF115 and BPF117 and the residual TL of the same two samples after they were exposed for varying periods of time (3, 6, 12, 24, and 30 hr) to a halogen lamp (OSRAM, HQI-T, 250 watt/D) placed 30 cm away. Figures 1B-2B show the relative decline of the peaks having maxima near 300, 400, and 500 - 600°C as a function of duration of exposure to light.

After about 5-10 hr of exposure the peaks in the vicinity of 300 and 400°C shrunk to about 10% of their original size and became practically undetectable after 20 hr. The 510°C peak of BPF115 took about 30 hr to decline to 10% of initial value, while after the same length of exposure the 550°C peak of BPF117 still retained about 80% of its value. After these samples were bleached the TL induced by artificial irradiation was similar in shape to the NTL (fig. 3). The other specimens exhibited similar behavior.



Figures 1 & 2.

Bleaching experiments on samples BPF115 (Figure 1), and 117 (Figure 2) A - NTL and TL emissions obtained after different durations of light exposure varying between 1 to 30 hours. B - Variation of the amplitude of the peaks near 300, 400, and 500-600°C as a function of length of exposure to light.

The results tend to strengthen our suspicion that TL emissions between 300 and 500°C of some of our quartz pebbles were probably partially bleached by exposure to sunlight before becoming covered by sediment. Unfortunately, such bleaching cannot be used to date unburnt pebbles by TL, since the residual TL is a function of opacity and pebble size, which in our work ranged from 100 to 800 g. Only specimens heated at 500°C or above were dateable, so it became paramount to establish viable criteria for recognizing such specimens.





The effects of heating on raw quartz pebbles

To see how the temperatures attained by such quartz pebbles might affect their TL properties, we collected six unburnt quartz pebbles at some distance from the Pedra Furada hearths and performed the following experiments on them.

1) The central core of each pebble was crushed, sieved, and the 100-160 μ m grains saved.

2) Each powder sample was divided into five aliquots, which were placed in an electric furnace, heated at a rate of 25° C/min to a maximum of 200, 300, 400, 500, and 600°C, respectively, and allowed to cool slowly after staying at the peak temperature for 5 minutes. Each aliquot then received 40 Gy of gamma radiation from a ¹³⁷Cs source.

3) The natural (geological) and artificial TL of all six pebbles were measured using a MTO 380 nm optical filter which selected the blue component of the spectrum.

Some of the glow curves obtained for specimens BPF103, 78, and 71, are shown in Figures 4 and 5 (ab). The geological TL of unheated pebbles, curve 1 in each figure, shows a maximum at about 400°C (heating rate : $5^{\circ}s^{-1}$). As one can see by comparing curves 1 and 2 in Fig. 4, the TL signal of quartz grains irradiated after being heated at temperatures inferior to 400°C has a shape similar to geological TL. The behavior of BPF103 is typical of the other similarly treated samples. When the heating temperature attains or exceeds 500°C the artificial TL signal (curve 3 in Fig. 4 and curve 2 in Fig. 5a or b) shows a 400°C peak accompanied by a new one at about 300°C. The sensitivity of the latter increases with temperature (curve 4 in Fig. 4 obtained after a 600°C heating), a phenomenon already noted by (David et al., 1977).

These experiments indicated that when Pedra Furada quartz is heated at 500°C and irradiated it exhibits TL peaks at 300 and 400°C. On the other hand, if the same sample is reheated at a temperature of 500°C, or lower, the TL obtained after same irradiation remains unchanged (Fig.6).



Figure 4.

Effect of heating temperature on the TL emission of the raw quartz pebble BPF103

Curves 1 ——> NTL (geological TL)

Curves 2, 3, and 4 \longrightarrow TL emissions obtained after this sample has been heated respectively at 300 (curve 2), 500 (curve 3) and 600°C (curve 4) and irradiated with an artificial dose of 40 Gy

Thus one can see that exposure to temperatures of 500°C or higher left an indelible mark on the TL signal of Pedra Furada quartz. The glow curves obtained after laboratory irradiation exhibited a distinct maximum near 300°C which was absent in curves of similarly treated but unheated specimens. The shape of the TL signal in the 300-400°C range allows one to select specimens that have been exposed to high enough temperatures to be dateable.





Fig. 5, a- b 1 — Natural 2 — 500°C + artificial dose

Figure 5.

Comparison between the geological TL of samples BPF78, and 71 and the TL glow curves obtained after these samples have been heated at 500°C and irradiated with an artificial dose of 40 Gy

How this criterion was used to select for dating sample BPF53, collected near a hearth, is shown by the data in Figure 7. Since the natural TL (curve 1) exhibits a distinct peak at about 300°C, in addition to the one near 400°C, the specimen must have been heated at a temperature no lower than 500°C and is consequently dateable. Moreover, as in the case of experimental sample BPF103 (Fig.6), one can see



Figure 6.

Effect of two successive heatings at 500°C on the TL emission of sample BPF103

Curve 1 : TL emission induced by an artificial dose of 40 Gy obtained after the sample has been heated at $500^{\circ}C$

Curve 2 : TL emission induced by an artificial dose of 40 Gy obtained after the sample has been heated twice at $500^{\circ}C$

In Figure 8 are plotted the natural and natural + artificial TL of BPF50 for artificial doses of 30, 60, and 90 Gy. The paleodose was determined by the *Normalization Method* (Valladas and Gillot, 1978; Mercier *et al.*, 1992) using the second TL growth curve (regeneration curve). The paleodose of 28 ± 3 Gy was constant between 350 and 430°C (plateau test), so the specimen was suitable for dating.

The age estimates for the samples discussed above will be published after we have obtained all the relevant dosimetric data.

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NTL of sample BPF53 and TL emission induced by an artificial dose of 40 Gy after the sample has been heated at $500^{\circ}C$



Figure 8.

Paleodose measurement on sample BPF50 A - NTL and artificial TL obtained after successive irradiations of 30, 60 and 90 Gy

B - Plateau test : Paleodose as a function of temperature

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