LETTERS TO THE EDITOR

THERMOLUMINESCENCE DATING OF BURNT CLAY FROM AN UPPER PALEOLITHIC OCCUPATION LEVEL AT A CAVE SITE IN SOUTH INDIA

K. S. V. NAMBI
Health Physics Division
Bhabha Atomic Research Centre
Bombay 400 085, India
AND
M. L. K. MURTY
Department of Archaeology
Deccan College, PG and Research Institute
Poona 411 006, India

The Archaeological Site and the Samples

BURNT clay samples were found in a fire place belonging to an Upper Palaeolithic occupation level in a test excavation of a streamside cave complex site known as Muchchatla Chintamanu Gavi (15° 25′ N ; 78° 8′ E) in the Karnool district of Andhra Pradesh.

A seemingly fire place structure was noticed at a depth between 1·50–1·80 m ; it consisted of limestone boulders in a horse-shoe shaped fashion measuring 1·6 m at its maximum length and 0·70 m along the interior breadth. Within the confines of this fire place occur burnt bone fragments that have been completely lithified as a result of long association with the carbonate rich cave sediments, burnt chunks of limestone and clay and nuclei and nodules of green coloured chert and a few large chert blades. In all likelihood, this fire place was used for roasting meat and also for heat-treating of chert nodules to produce artifacts.

The burnt clay samples taken up for thermoluminescence (TL) dating come from 1·65 m–1·80 m level, the bottommost of the present excavation ; and the deposit all around the sample comprised of the same clay, either burnt fully or partly.

TL Dating Results

The principles of TL dating are well recognised[1] ; the 'fine grain technique' was employed in the present study and the procedural particulars and instruments used are the same as detailed in an earlier report on TL dating of ancient potteries[2].

The TL glow curves from the samples were typical of limestones[3] ; the natural thermoluminescence peaks were at about 280° C and 350° C while artificially produced thermoluminescence, *i.e.*, by laboratory irradiation, showed additional peaks at lower temperatures of about 110° C and 230° C. A preheat of the sample upto 250° C was done in the TL Reader, to evaluate accurately the relative build-up of 280° C and 350° C peaks for artificial irradiations. The well-known 'plateau test' revealed a stability within ± 15% in the temperature region of 280–350° C. No "anomalous fading" could be detected in artificially irradiated samples after one month storage at room temperature.

The "calibration" of the NTL in terms of "absorbed gamma dose" was done by the "additive dose" technique and a value of 3255 rads was obtained as the paleo dose equivalent (PDE). The TL sensitivity of these samples for α radiations was found to be 34% as compared to γ radiations (*i.e*, $k = 0{\cdot}34$ in TL dating parlance). The radioactivity of the sample was measured in terms of surface alpha emission rate and potassium content ; the values were respectively 0·87 cph cm^{-2} and 0·003%. Significant radon/thoron gas emanations could be detected from the sample and hence the gross alpha count rate was corrected for these 'gas losses' from gas cell measurements[4]. The annual dose rates to the samples were estimated from these radioactivity determinations and using the well-known dose conversion constants. The saturated water uptake by the sample was measured to be 11·2% by weight and the 'wetness corrections' on the dose rates were applied on the assumption that the annual weighted average for the water uptake by the sample was 27% ± 25% of the saturated uptake. (This was based on observations of 100% saturated water uptake during 3 monsoon months, 6% of saturated water uptake during 4 winter-spring months and complete dryness during summer months.) The final dose rate ($\dot{D}$) values are : 416·7, 19·5 and 21·0 mrad yr^{-1} for α, β and γ components respectively. The cosmic dose rate was assumed to be 5·0 mrad yr^{-1} a value generally found applicable inside caves. The TL age is given by the equation,

$$\text{TL age, yrs. (Before Present)} = \frac{\text{(PDE)}}{k.\dot{D}_\alpha + \dot{D}_\beta + \dot{D}_\gamma + \dot{D}_{cos}}$$

The age obtained is 17390 yrs. B.P. with a predicted error of ± 10%.

Conclusions

Earlier excavations of an adjacent cave site have yielded Upper Palaeolithic cultural materials, and these were ascribed to Late Pleistocene period by virtue of their association with the Late Pleistocene fauna[5].

The TL date of 17390 ± 10% B.P. presently obtained provides an absolute date for the Upper Palaeolithic in the Kurnool cave areas, known from both cave sites and surficial open-air occurrences of blade tool assemblages. It is also significant in that it corroborates the known ^{14}C dates of the Upper Palaeolithic elsewhere in the country[6].

February 27, 1981

1. Aitken, M, J., *Archaeological Involvements of Physics*, Physics Reports 40 C(5), 1978, p. 277.
2. Nambi, K. S. V., Sasidharan, R. and Soman, S. D., *Thermoluminescence Dating of Ancient Potteries* excavated at Bhagwanpura and Mathura, Bhabha Atomic Research Centre (Bombay) Rept. BARC-1013, 1979, p. 42.
3. — and Mitra, S., "Thermoluminesscence investigations on old carborate sedimentary rocks," *N. Jb. Miner. Abh.*, 1978, **133**, 210.
4. Aitken, M. J., *Radon Loss Evaluation by Alpha Counting*, PACT (Council of Europe, Strasbourg), 1978b, **2**, 104.
5. Murthy, M. L. K., "A Late Pleistocene cave site in Southern India," *Proc. Amer. Phil. Soc.*, 1974, **118**, 196.
6. —, "Recent research on the Upper Palaeolithic Phase in India," *Journal of Field Archaeology*, 1979, **6**, 301.

PRELIMINARY REPORT OF PRODUCTION OF A NEW TOXIN BY *PENICILLIUM CYCLOPIUM*

N. Ramani and E. R. B. Shanmugasundaram

University Biochemical Laboratories

A.C. College Campus, Madras 600 025, India

Penicillium cyclopium has been reported to be a common food contaminant[1-3]. In our attempts to screen the toxin(s) produced by a strain of *P. cyclopium* isolated by us from a sample of rice, a yellow pigment was secreted into the medium and a study of this compound is presented here.

P. cyclopium was grown on Raulin-Thom medium of the following composition in g/l : glucose, 50 g ; tartaric acid, 2·6 g ; ammonium tartrate, 2·6 g ; ammonium phosphate, 0·4 g ; magnesium carbonate, 0·2 g ; ferrous sulphate, 0·05 g and zinc sulphate, 0·05 g. The pH was adjusted to 6·4 and incubated at 28–30° C. After 14 days of growth, the mycelia were removed and the culture filtrate was acidified to pH 2·0 with conc. hydrochloric acid. To isolate the yellow pigment, lipid materials from the culture were first removed by extracting with *n*-hexane. Then the yellow pigment was repeatedly extracted using ethyl acetate. The ethyl acetate extract was concentrated to about 50 ml under reduced pressure in a flash evaporator at 40° C; dried by anhydrous Na_2SO_4 and finally concentrated to near dryness in vacuum. The pigment in ethyl acetate was passed through a silicagel-60 column in benzene and eluted with ethyl acetate. The eluate was dried and purified by TLC on silica gel.

The plate was developed in toluene : ethyl acetate : formic acid (5 : 4 : 1) solvent system and the yellow pigment exhibiting a dark absorbance in uv was scrapped and eluted with ethyl acetate. The pigment obtained was crystallised using ethyl acetate and alcohol.

The purified yellow pigment was found to be soluble in polar solvents like ethyl acetate,, dioxan, dimethyl formamide, ether and partially soluble in chloroform and benzene. The uv absorption spectrum of yellow pigment in absolute alcohol indicated that it has maximum absorption at 220 and 256 nm. In TLC, it had an Rf value of 0·78 and gave a reddish brown colour with phenyl hydrazine indicating that it has a carbonyl group. It reacted with Folin-phenol reagent to produce blue colour showing the presence of a phenolic-OH group.

To test the toxicity, 10 mg of the pure yellow pigment was dissolved in 20 ml of water and 1·0 ml of the solution (containing 500 mcg) was fed orally on alternate days to a dozen one day old chicks, while the control birds received the same amount of water. 80–90% mortality was observed at the end of sixth dose of administration of the compound. Histopathological examination of liver, kidney and intestine showed evidence of cell damage with inflammatory cellular infiltration in liver and kidney. In the case of intestine, inflammatory cellular infiltration in the laminar portion was seen.

This is the first time a toxic yellow pigment has been observed to be produced by *Penicillium cyclopium* and detailed work on biochemical aspects of its toxicity is being worked out.

The authors thank Dr. K. Muragesan, Professor of Pathology, Kilpauk Medical College, Madras, and Dr. A. Kamalam, Mycologist, Madras Medical College, Madras, for their help during the histopathological examination of the tissues. The financial assistance rendered by the CSIR to NR is gratefully acknowledged.

January 27, 1981

1. Wilson, B. J., Wilson, C. H. and Hayes, A. W., *Nature*, 1968, **220**, 77.
2. Scott, De B. *Mycopathol. Mycol. Appl.*, 1965, **25**, 213.
3. Tsunoda, T., Kishi, K., Okubo, K. and Ohtsubo, K., *Proc. Jap. Assoc. Mycotoxicol.*, 1977, Nos. 5/6, p. 32.