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Geochronology of cave deposits at Liang Bua and of adjacent river terraces in the Wae Racang valley, western Flores, Indonesia: a synthesis of age estimates for the type locality of *Homo floresiensis*

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A B S T R A C T

A robust timeframe for the extant cave deposits at Liang Bua, and for the river terraces in the adjoining Wae Racang valley, is essential to constrain the period of existence and time of extinction of *Homo floresiensis* and other biota that have been excavated at this hominin type locality. Reliable age control is also required for the variety of artifacts excavated from these deposits, and to assist in environmental reconstructions for this river valley and for the region more broadly. In this paper, we summarize the available geochronological information for Liang Bua and its immediate environs, obtained using seven numerical-age methods: radiocarbon, thermoluminescence, optically- and infrared-stimulated luminescence (collectively known as optical dating), uranium-series, electron spin resonance, and coupled electron spin resonance/uranium-series. We synthesize the large number of numerical age determinations reported previously and present additional age estimates germane to questions of hominin evolution and extinction.

Introduction

Archaeologists and physical anthropologists commonly require numerical age determinations for artifacts, human remains, and associated deposits to arrange the "target" objects or events in their correct temporal sequence. Numerical-age methods (following the terminology of Colman et al., 1987) are entirely or mostly self-contained, and produce quantitative age estimates that can be placed on a standard timescale. The resultant ages are typically expressed in years before present, with an error term that indicates the degree of uncertainty associated with the age estimate. Recent reviews are provided in Walker (2005), Wintle (2007), and Roberts and Jacobs (2008).

The excavations of the cave deposits at Liang Bua from 2001 to 2004 involved the collection of samples for dating by a variety of numerical-age methods, including radiocarbon (^{14}C), luminescence – a collective term for the related techniques of thermoluminescence (TL), optically-stimulated luminescence (OSL), and infrared-stimulated luminescence (IRSL) – uranium-series (U-series, specifically $^{230}Th/^{234}U$), and electron spin resonance (ESR), the latter coupled with U-series. We applied this range of dating methods which are (based on different physical principles) to diverse materials (of different origin) so that problems with any particular method or material could be identified before the final

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ages were determined for the objects and events of interest. Such quality-assurance measures are necessary for the construction of accurate chronologies, which became of paramount importance with the discovery of the holotype of *Homo floresiensis* (LB1) in September 2003. Robust numerical ages are also needed for the associated stone artifacts and other faunal remains recovered from these excavations, to provide chronological control for the Liang Bua stratigraphy and for reconstructions of the prevailing environmental conditions in and around this cave.

This paper reviews the geochronological investigations conducted since 2001 on the cave deposits at Liang Bua and on the nearby river terraces in the Wae Racang valley. We summarize all numerical age determinations, including those reported previously as well as several unpublished age estimates (see Table 1 for a complete listing), which are of relevance to the archaeology, palaeontology, and palaeoenvironmental context of the discoveries made at Liang Bua. The stratigraphy and sedimentology of the cave deposits are described by Westaway et al. (2009a), and details of the river terrace deposits are given in Westaway et al. (2009b).

Study sites and sample collection

An extensive series of samples (85 in total) has been analyzed from Liang Bua and its environs; more have been collected but not analyzed. This total consists of 26 sediment samples for luminescence dating (several of which have been dated by more than one luminescence technique), 22 samples of speleothem for U-series dating, 25 charcoal samples for accelerator mass spectrometry (AMS) ^{14}C dating (some of which have been dated from more than one temperature fraction), and 12 teeth (*Stegodon* molars) for ESR dating (two of which have also been dated by U-series to obtain "coupled" ESR/U-series ages).

Samples for dating by all of the above methods have been collected from several excavated Sectors throughout the cave and from the conglomerate deposit at the rear of the cave (Figs. 1 and 2) to constrain the ages of the buried artifacts and faunal remains, especially those of *Homo floresiensis*. Samples for TL and OSL dating have also been collected from the river terraces T3, T4, and T5, located outside the cave entrance in the Wae Racang valley; site details are given in Westaway et al. (2009b). U-series samples U21 and U22 were collected from the top and bottom of a stalagmite (SPI-1) formed in a small cave situated ~90 m above, and connected to, Liang Bua; this remnant cave is referred to as "Outcrop I" in Westaway et al. (2009b).

The remains of LB1 were found at a depth of 5.9 m in Sector VII, at the same level as TL/IRSL sample LBS7-42 (denoted as L13 in Fig. 2d) and ^{14}C samples ANUA-27116 and ANUA-27117 (shown as C17 and C18 in Fig. 2d). All of the samples reported here were collected during the course of the 2001–2004 excavations at Liang Bua, except for 9 radiocarbon samples (C3–10 and C12) collected from earlier excavations by R.P. Soejono (Morwood et al., 2009). The latter samples originate from the upper ~3 m of deposit in Sector IV, and are reported here to provide a complete account of all known numerical age determinations at Liang Bua.

Samples were collected using standard procedures for each dating method. For ^{14}C dating, the excavated cave deposits were placed in plastic bags and immediately sealed. Scattered charcoal fragments were subsequently hand-picked in the laboratory for analysis. These fragments were recovered from the material excavated from individual spits and retained on 2 mm sieve mesh (i.e., not directly from the cleaned walls or floor of an excavation), so their locations can be fixed vertically to within ± 10 cm (the thickness of a typical spit) and horizontally to within ± 25 cm (the planform dimensions of a typical bucket-load of deposit). We did not attempt direct ^{14}C dating of bone collagen from LB1, because this would have resulted in partial destruction of the holotype of a new hominin species with only a low chance of success, given that collagen is unlikely to have been preserved in the tropical weathering environment.

Sediment samples for luminescence dating were collected by hammering opaque plastic tubes into the cleaned section walls, and then removing and sealing the tubes to prevent light exposure and mixing of the sediments, and to retain their field moisture contents. The empty tube holes were then enlarged and a portable gamma-ray spectrometer inserted to obtain *in situ* measurements of the gamma dose rate at the sampling locations. Quartz and potassium-rich feldspar grains were extracted from the bulk sediments for TL/OSL and IRSL dating, respectively, under safelight conditions in the laboratory. Quartz and feldspar grains were also extracted from a burnt pebble found at a depth of 8.4 m in Sector VII (Morwood et al., 2005). When collected, the pebble was immediately wrapped in light-proof black plastic and the gamma dose rate was measured at the find spot (marked as L14 in Fig. 2d). At each location, an additional sample of sediment was collected and sealed in a plastic bag for laboratory determinations of radioactivity and moisture content. Full details are given in Westaway (2006).

Samples of calcite for U-series dating were collected by removing chunks of speleothem from subaerially-exposed and buried flowstones using a hammer and chisel, and then sealing them in plastic bags for later preparation and analysis in an ultra-clean laboratory environment.

Whole *Stegodon* molars and fragments of molar were collected for ESR dating. Four of these teeth were recovered directly from the deposit during excavation (denoted as E5, E7, E9, and E12 in Fig. 2c), so their locations are known to the nearest cm; field gamma-ray spectrometry measurements were made at each of these find spots. The other 8 teeth were collected from the material excavated from individual spits and retained on 2 mm sieve mesh, so their stratigraphic positions are constrained with the same precision as the ^{14}C samples. For the latter ESR samples, gamma dose rates were estimated from measurements made in the same stratigraphic layer, from as close as possible to the excavated spit. The teeth were deliberately not cleaned in the field, so that accurate estimates could be made of the external beta dose rate from the adhering sediment. U-series dating of the enamel and dentine was also conducted on three of the whole teeth – LB-JR-8A (a juvenile *Stegodon* molar with dimensions of 4 cm × 2 cm × 2 cm, plotted as E5 in Fig. 2c) and two adult molars (LB-JR-14B and LB-JR-15A) – to test for U loss and obtain combined (or "coupled") ESR/U-series ages for suitable samples (Grün et al., 1988; Rink, 1997; Grün, 2006, 2007). The field moisture content of the deposits surrounding each of the ESR samples was determined from the additional sediment samples collected for luminescence dating.

Dating techniques, equipment, and procedures

Radiocarbon dating

Eleven charcoal samples ("ANUA-" in Table 1) were prepared using acid–base wet oxidation (ABOX) ^{14}C procedures and then step-combusted at 330 °C, 650 °C, and either 850 °C or 910 °C in a vacuum line insulated from atmospheric contamination by a secondary backing vacuum (Bird et al., 1999). Previous studies have shown that most contamination is removed at the 330 °C combustion step, so that greatest confidence can be placed in ^{14}C ages obtained from the fractions combusted at temperatures of 850 °C and higher (e.g., Bird et al., 1999, 2002, 2003; Fifield et al., 2001; Turney et al., 2001; Bird, 2007). Graphite targets were