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Preliminary Results of Speleothem Dating from Tabon Cave, Palawan, Philippines: Moisture increase at the Last Glacial Maximum

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Abstract

Preliminary study of a thick speleothem layer at Tabon Cave, Palawan, demonstrates the potential such layers hold for dating and for interpretation of local and regional climatic variations during archaeological periods. A layer of gypsum speleothem, previously suggested to be travertine, has produced uranium series dates generally correlating with bracketing radiocarbon dates on charcoal, confirming an increase in moisture around the time of the Last Glacial Maximum. Dating and study of additional speleothems in Tabon and other caves in Island Southeast Asia is recommended as an approach with great potential for archaeological studies and for reconstructing regional climate history in the Pleistocene and Holocene.

Introduction & Background

Tabon Cave (Philippines National Museum site code IV-200-T) is one of over 200 cave sites located on Lipuun Point, a limestone peninsula forming the northwestern end of Malanut Bay in the Quezon region of central Palawan,

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Philippines (Figure 1). The site is famous for producing the earliest human remains in the Philippines (Fox 1970), with uranium series (U-series) dates on modern human specimens from c. 16,300-47,000 yr. BP (Détroit *et al.* 2002; 2004; Dizon 2000; 2003; Dizon *et al.* 2001); this makes the site regionally important regarding migration into Southeast Asia and beyond, and where and how Pleistocene people lived in the landscape (Barker *et al.* 2002; 2003; 2007; Bellwood 1997; 2005; Brothwell 1960; Kennedy 1977; O'Connor 2007). Tabon Cave contains Late Palaeolithic occupation deposits, including artefact scatters and evidence for food preparation (burnt animal bones), and Metal Age burial deposits (Fox 1970). The Lipuun Point cave complex as a whole contains additional later Palaeolithic occupation sites, and Neolithic and later cemeteries (Fox 1970), which are broadly similar to those found in other caves in the region (e.g. Fox 1970; B. Harrisson 1967; T. Harrisson 1975). The location is thus also important in other regional archaeological issues include the impact of mid-Holocene sea level rise and environmental change, the beginnings of agriculture (Barker *et al.* 2002; 2003; Doherty *et al.* 2000; Spriggs 1989), the dating of and social mechanisms involved in the spread of 'Austronesian' cultures (Bellwood 1997; 2005; Blust 1976; Kayser *et al.* 2000; Meacham 1988; 1995; Terrell 2004; Tsang 1995), and the Nusantao Maritime Trade and Communication network (Solheim 1988; 2006). In this paper we briefly discuss another important aspect of these cave sites: the palaeoenvironmental record held in speleothem deposits that can inform on local, regional and potentially even global changes in climate during the Pleistocene and Holocene.

Current models of prehistoric occupation of the Tabon caves and their landscape generally fit with Anderson's (1997) understanding of cave sites in Southeast Asia. Early cave occupation of a sporadic or temporary nature by modern humans seems indicated up into the early Holocene. In the earlier Holocene several sites show more intensive or frequent occupation, local people appear to have been strongly focused on land-based, riverine and estuarine resources, and in many cases the sea is known to have been many kilometers away from the cave sites (Anderson 1997; Barker *et al.* 2007; Lewis *et al.* in press; Reynolds 1993). Although Tabon Cave is just a couple of minutes' walk from the sea today, the lack of marine shells from early cultural deposits in this cave is taken to support the idea that there was a substantial land shelf around the time of the Last Glacial Maximum (LGM – c. 21,000-19,000 yr. BP, after Bowen *et al.* 2002; Yokoyama *et al.* 2000), when estimates place global sea levels at 116 metres below present or possibly lower (Fairbanks 1989; Lambeck *et al.* 2002). Fox (1970) suggests that no 'habitation' deposits at Tabon Cave

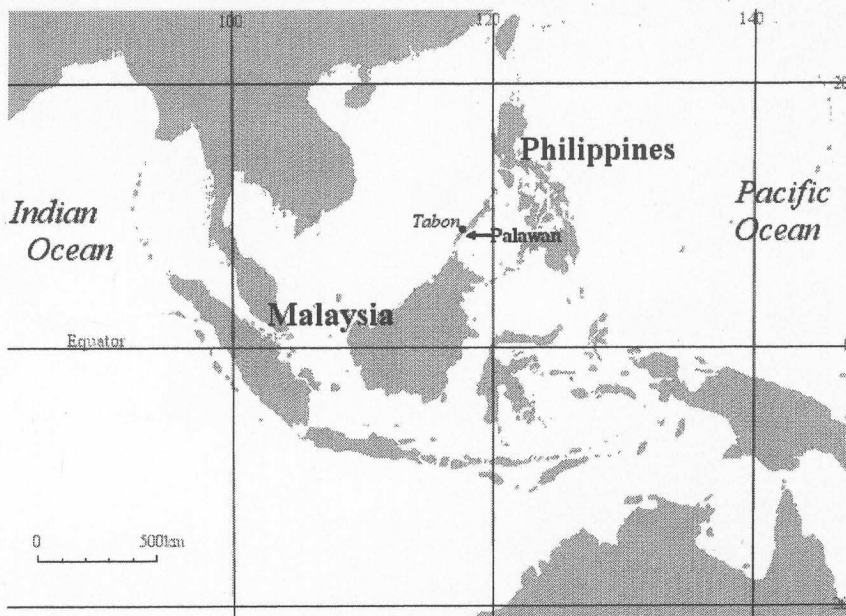


Figure 1. Location of Tabon Cave

post-date c. 8000-9000 yr. BP (all later remains are Early Metal Age cemetery deposits, c. 200-500 B.C. - Victor Paz and Wilfredo Ronquillo, 2005, pers. comm.). In the local area the sea appears to have reached 1-3 metres above present level c. 6000-7000 yr. B. P. (Maeda *et al.* 2004; Omura *et al.* 2004). The appearance of marine shells in middens in other caves on Lipuun Point from c. 7,000 yr. BP, and especially in later periods, suggests increasing focus on marine resources in the area in general; the abandonment of Tabon Cave as an occupation site just prior to this time is suggested to be related to sea level rise (Fox 1970).

The dating of materials from Tabon Cave has been an aim of archaeologists for decades, starting from Fox's (1970) radiocarbon-dated charcoal sequence and inferences from sediment build-up, up to the more recent U-series dates on human bones, which have pushed the age range back to c. 47,000 years ago (Détroit *et al.* 2002; 2004; Dizon 2000; 2003). The general stratigraphy of Tabon cave as described through excavations (Dizon *et al.* 2001; Fox 1970; Orogo 2000; 2001) is presented in Table 1; recent work on the

| Depth cm below Fox 1970 datum | Layer | Sediment sequence | Archaeology, cave and landscape paleoenvironment sequences | Dating (after Fox unless indicated) |
|-------------------------------|----------------------------|--|--|--|
| Surface (50-60 cm BD) | EI, OI, DA or B; D1-2 | Disturbed, compact gritty grey-brown soft mudstone (F); sandy loam, dull brown (O) | Disturbed; Early Metal Age jar burials, beads, bones, human bones, possible preparation, lithics from c. 20 cm, human bones (D). | Est. 2,200-2,500 years ago |
| 50-80 | OII; D2-3 | Sandy loam, dull yellow orange (O) | Deer becomes extinct Sea level est. 3 m above present | Est. c. 4,000 years ago Est. 6,000-7,000 years ago |
| 60-110 | EII; DA or B; D3-4 | Soft, fine light grey to dark grey deposit | Upper Palaeolithic: riverine shell, bones, teeth, charcoal | Est. 8,500-9,500 years ago or younger |
| 50-75 | FIII; D6; D3-4; OIII or IV | Soft, fine grey brown to brown deposit (F); clay loam, dull yellow orange (O) | Upper Palaeolithic: flake assemblage I-A (surface to c. 25 cm depth), riverine shell, bones, teeth, charcoal | C-14 9,250±/250 years BP (charcoal) |
| 50-70 | F(I) | Hard, gritty brown deposit (F) | Upper Palaeolithic: flake assemblage I-B (20-70 cm depth below surface), 'brut' fragmentation; land shell, sea level est. 30 m below present (F) | Est. 10-20,000 years ago Dizon et al. 16,500±/2,000 years BP (U-series, frontal bone) |
| 110-150 | F (II); D5; DC; O7IV | Soft, fine light brown deposit (F); clay loam, dull yellow orange (O) | Upper Palaeolithic: flake assemblage II, bones, human and animal teeth, charcoal, frequent chert debitage; 'long habitation area' with unclear stratigraphy, 'Tabon Man' bones in disturbed area, either FA II or III (F). U-series dates put Tabon Man frontal into FA II (D) | C-14 > 21,000 years BP (charcoal from base) |

Table 1. Overview of the published Tabon Cave sequence (after Lewis 2007). Sediments are oriented on a gradient, and layer numbers arise from four main excavation sequences: F1-VII and F1-III) (Fox 1970 = F), DA-F and D1-12 (Dizon et al. 2001 = D) and O1-XI (Orogo 2000 and 2001 = O). Equivalences are approximate

| Depth cm below Fox 1970 datum | Layer | Sediment sequence | Archaeology: cave and landscape paleoenvironment sequences | Dating (after Fox unless indicated) |
|-------------------------------|----------------------------|--|---|--|
| 110-150 | OV; DD | Speleothem deposit (interpreted as 'travertine' by Fox) and stalagmites | Possible pluvial period, or wet cave conditions (F) | |
| 110-135 | FV; DE; D6; OVII | Soft, fine ivory or grey-ivory deposit (F) | | |
| 140-170 | FV, F(III); DF; D7; O7VIII | Dark red-brown gritty deposit, red pan at base; consolidated or soft, fine flesh deposit (F); light reddish brown sandy loam (O) | Upper Palaeolithic flake assemblage III, extensive habitation, animal bones, charcoal, frequent human remains (F). A mandible from another human specimen found by Fox was dated to 31,000 years BP | C-14 $522,000$ years BP (2 dates); C-14 $23,200 \pm 1,000$ years BP (charcoal); Dizon (2003) $31,000 \pm 5,000$ years BP (U-series, jaw) |
| 150-190 | FVI; D8; O7VIII | Soft to hard brown to red-brown deposit (F); light reddish brown sandy loam (O) | Upper Palaeolithic flake assemblage IV (c. 170 cm below datum) (F) | C-14 $30,500 \pm 1,100$ years BP (charcoal); Dizon (2003) $47,000 \pm 11,000$ years BP (U-series, tibia) |
| 160-250 | FVII | Hard dark brown to soft black deposit (F) | Upper Palaeolithic flake assemblage V (F) | Age-depth equation est. 45,000-50,000 years ago |
| 200 cm | D9-12; OIX-XI | IX brown loamy sand; X weak red loamy sand; XI pinkish grey loamy sand (O) | Fox probe - 'sterile' | |

Table 1. (cont.)

site (Jago-on *et al.* 2007) is expected to produce a revision of this outline, but this will not affect the part of the sequence discussed here. Fox (1970) identified five chert flake assemblages, which, although occurring in limited horizontal space in the cave, were suggested to make up a sequence based on context and depth of occurrence, ranging from >30,000 yr. BP to c. 8500 yr. BP backed by six radiocarbon dates.

Of particular interest for this paper is a thick speleothem layer, thought originally to be travertine, which separates later and earlier soft sediments and archaeological assemblages. Fox (1970) dated charcoal on top of this deposit to >21,000 yr. BP maximum age, and charcoal underneath it to c. 23,000 yr. BP. The speleothem layer appears to cover the entire cave floor area, lying directly in between Fox's earlier culture layers ('6'-'7', pre-22,000 yr. BP) and cultural deposit 'C' (c. 21-10,000 yr. BP) (Dizon *et al.* 2001; Fox 1970). The main issues surrounding this layer are its chemical nature, its date (i.e. whether or not it formed as a speleothem at the time period discussed, or later on as an authigenic formation within soft sediments), and its possible relationship to a phase of local or regional wetness around the time of the LGM, as proposed by Fox (1970). This potential relationship has also been attributed to increased incidence of rockfall activity elsewhere in archaeological Southeast Asian caves (e.g. Anderson 1997), but in no case have the deposits in question seen further study or absolute dating.

Sampling from Tabon in 2006

The work discussed here carries on from the study of five soil micromorphology block samples described from cultural deposits at Tabon Cave (Lewis 2003; 2005; 2007). Questions raised through that study of particular cultural sediments (Lewis 2007) led to new sampling for dating of deposits through U-series and optical stimulated luminescence (OSL) methods, and for granulometric and mineralogical assessment (Table 2; Figure 2). Final results for all of these studies remain forthcoming, and will be the subject of future papers, but the preliminary findings of the U-series test study of the Tabon Cave 'travertine' already demonstrate the importance of further research in this area.

Sampling for U-series dating was undertaken from the thick, hard greyish-white layer recorded by Fox and later authors as 'travertine' (Figure 2), discussed above. The upper part of this deposit is hard, layered speleothem, while the lower part is soft loose silt; in thin section the lower part of the layer was seen to comprise mainly unidentified authigenic mineral crystals, micrite

and guano (Lewis 2007). Only the upper hard layers were sampled for U-series study, with a block (TBG1) collected from an exposed section. The aim of studying this sample was to check the radiocarbon dates, and study the origin and significance of this deposit for local and regional climatic conditions. Sample TBG1 was U-series dated at the University of Oxford Earth Sciences Department (Johnson and Lewis, in prep.).

Uranium series dating methods and application

Understanding the precise timing of speleothem growth is essential to their use in constraining the ages of archaeological deposits, in addition to their use as palaeoclimate archives. U-series dating is the dominant dating technique applied to calcite speleothems (Richards and Dorale 2003), and is based on the chemical fractionation of the parent U isotopes (^{238}U , ^{235}U , ^{234}U) from their long-lived daughters (^{231}Pa , ^{230}Th) in natural waters due to the high solubility of U (as UO_2^+) and the insolubility of Th (thorium) and Pa (protactinium). Calcite speleothems, which are precipitated from natural groundwaters, typically form with relatively high U concentrations (ppb to ppm range) and very little Th or Pa. Thus, by precisely measuring the ingrowth of radiogenic Th or Pa, through mass spectrometer measurements, the ages of speleothem formation can be calculated.

However, examination of the speleothem sample from Tabon Cave showed it to be composed mainly of gypsum, not calcium carbonate. This means that the layer should no longer be referred to as 'travertine', and has implications for dating. The sample was initially determined not to be calcite because the drilled U-Th samples did not dissolve in 7.5N HNO_3 . We therefore tested a powdered aliquot of the speleothem in the Oxford SEM, which revealed the major elemental composition to be Ca, S, and O, consistent with gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$). In principal, the same technique used for calcite speleothems can be applied to gypsum deposits, though efforts have previously been hampered by low U concentrations, the difficult dissolution of gypsum, and open-system behavior. To test this method on the gypsum deposit from Tabon Cave, we drilled four approximately 200 milligram solid wedges (U-Th1-4) from the sample using the 'moat and spall' technique (Figure 3). U/Th samples were dissolved in 7.5N HNO_3 with ~500 millilitres of concentrated HCl added, spiked with ^{229}Th - ^{236}U , dried down, redissolved in concentrated HNO_3 , centrifuged to remove a small amount of remaining solids, dried down again, and brought back up in 7.5N HNO_3 . U and Th were then purified using ion exchange chemistry and analysed by multi-collector ICP-MS at the University of Oxford following techniques modified from Robinson and colleagues (2002).

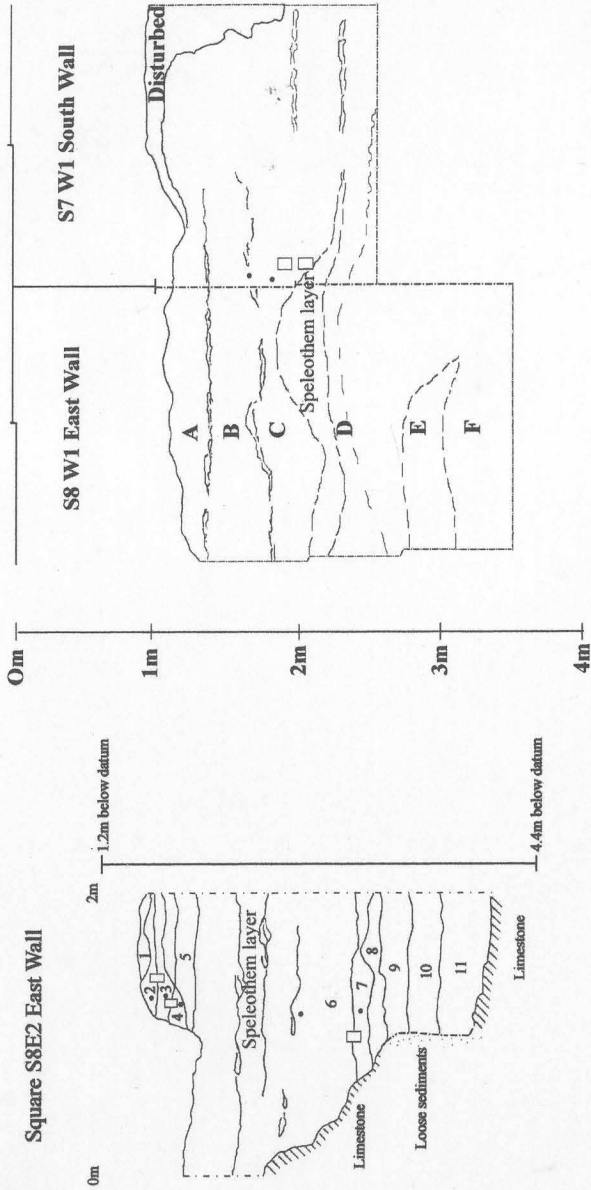


Figure 2. General stratigraphy of sampled areas at Tabon Cave (after Dizon et al. 2001). Dots indicate OSL and bulk samples from 2006; boxes are soil micromorphology samples (Lewis 2007). Speleothem sample TBG1 came from the uppermost, hard layers immediately under the equivalent of Layer C

| | OSL (J-L Schwenniger) | Granulometry | Mineralogy (S. Mentzer) | Other information |
|---------------------------------|--------------------------|--------------|----------------------------|--|
| Tabon 06/1 | OSL #1 & Moisture 1 | Gran #1 | Subsample #1 | Layer 2 (brown guano) |
| Tabon 06/2 | OSL #2 & Moisture 2 | Gran #2 | Subsample #2 | Layer 3 (red guano) SMM 2/1-2 |
| Tabon 06/3 | OSL #3 & Moisture 3 | Gran #3 | Subsample #3 | Red guano with white lumps (Layer 4?) SMM 2/1-2 |
| Tabon 06/4 | OSL #4 & Moisture 4 | Gran #4 | Subsample #4 | Layer 6 (under speleothem layer) |
| Tabon 06/5 | OSL #5 & Moisture 5 | Gran #5 | Subsample #5 | Layer 7 |
| Tabon 06/6 | OSL #6 & Moisture 6 | Gran #6 | Subsample #6 | Layer B/C1 (dark reddish-brown guano) |
| Tabon 06/7 | OSL #7 & Moisture 7 | Gran #7 | Subsample #7 | Layer C1/C2 (light orange brown guano) |
| Tabon 06 - entrance | n/a | n/a | n/a | 1 small block, sand on cave NE entrance wall (facing SW) |
| Tabon 06 - modern beach sand | n/a | Gran #8 | Subsample #8 | n/a |
| Tabon 06 - 'travertine' | n/a | n/a | n/a | 1 large lump (TBG1) to K. Johnson |

Table 2. Samples from Tabon Cave 2006 (P-XIII-T)

Results of U-Th dating

Preliminary results on samples U-Th1 and U-Th2, both taken from the top layer of TBG1 (Figure 3), give ages of 20.2 ± 0.8 kyr and 19.5 ± 0.8 kyr respectively (Table 3). These ages agree well with previous ^{14}C ages obtained from charcoal samples that bracket the gypsum deposit (Fox 1970). This suggests that the Tabon Cave gypsum deposit can be successfully dated with U-series methods and that this method could be further utilised to constrain the timing of human activity on the site and in the wider region, as well as to date potential palaeoclimate proxy records contained within the deposits. It is possible that some U-Th fractionation occurred during the chemical separation, but this is not thought to be significant given the excellent agreement with the radiocarbon ages. To avoid this possibility, future work will involve development of methods utilising total sample dissolution for U-Th dating of gypsums, and $^{231}\text{Pa}/^{235}\text{U}$ dating as a means to check for open-system behavior. The detrital ^{232}Th component of the other two samples (U-Th3 and U-Th4) is high, meaning that usable ages are unlikely to be

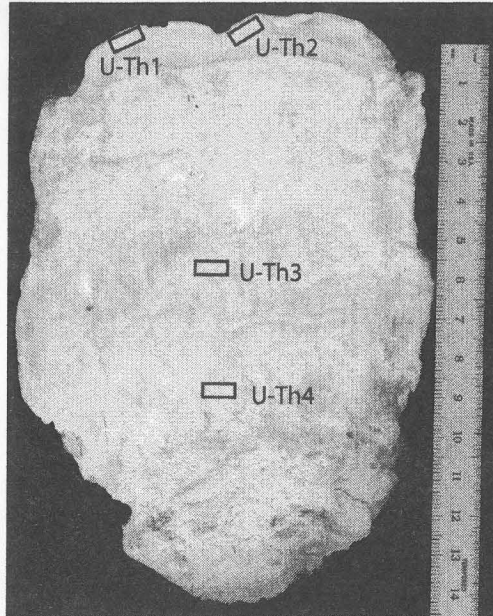


Figure 3. Section of the speleothem layer sample, TBG1, showing U/Th sample locations

forthcoming from them; however, we are awaiting results on these and the details of all will be reported separately (Johnson and Lewis, in prep.).

The confirmed deposition of the speleothem floor deposit around the time of the LGM indicates a significant hydrological change, which could be related to climatic changes at this time. Large gypsum deposits are relatively rare features of carbonate caves, and are largely thought to result from the circulation of sulfate rich water (Galdenzi and Maruoka 2003). The deposition of this deposit may reflect a regional increase in the water table level, possibly due to climatic or tectonic changes. It is not possible to determine the origin of this deposit, however, without further study. Further field and geochemical study is needed to evaluate the exact mechanism of gypsum formation, which could have important implications for regional climate, hydrology, and speleogenesis. To account for gypsum precipitation, we must determine both the source of sulfate ions and the mechanism(s) for gypsum deposition. Given the close association of the gypsum deposit with guano in Tabon Cave, a likely source of sulfate is leaching of bat guano. In particular, sulfuric acid is

thought to form in bat guano deposits through the action of sulfur oxidising bacteria (Hill and Forti 1997). Without further geological and S isotope investigation, however, we cannot rule out other potential sulfate sources, including sulfide oxidation (e.g., pyrite in the bedrock), sulfate bedrock (interbedded gypsum, for example), and hydrocarbon-derived hydrogen sulfide. In any case, a relatively wet cave floor is necessary to allow for gypsum deposition through a variety of mechanisms. Dating similar deposits in other caves in the region and studying their sulfur isotope composition could provide useful clues about their formation mechanism and hydrologic conditions before and during the LGM in this region. provide useful clues about their formation mechanism and hydrologic conditions before and during the LGM in this region.

| Sample | ^{238}U conc. (ppm) | ^{232}Th conc. (ppb) | $\delta^{234}\text{U}^a$ | $(^{230}\text{Th}/^{232}\text{Th})^b$ | $(^{230}\text{Th}/^{238}\text{U})^b$ | Age (raw) ^c (ka) | Age (corr.) ^d (ka) |
|--------|------------------------------|-------------------------------|--------------------------|---------------------------------------|--------------------------------------|-----------------------------|-------------------------------|
| U-Th1 | 0.0546 ± 0.0001 | 0.089 ± 0.003 | 9.0 ± 18.0 | 246.7 ± 12.4 | 0.1715 ± 0.0055 | 20.2 ± 0.8 | 20.2 ± 0.8 |
| U-Th2 | 0.0489 ± 0.0001 | 0.064 ± 0.004 | 40.8 ± 18.0 | 280.3 ± 18.2 | 0.1711 ± 0.0057 | 19.5 ± 0.8 | 19.4 ± 0.8 |

Table 3. U and Th concentrations and isotope ratios with calculated U-Th ages. All errors are 2 σ . ^a $\delta^{234}\text{U} = [(^{234}\text{U}/^{238}\text{U}) - 1] * 1000$ where $(^{234}\text{U}/^{238}\text{U})$ is the measured activity ratio. ^bRound brackets denote activity ratios. ^cAges are calculated using half lives from Cheng et al. (2000) and ISOPLOT software. Uncertainties incorporate mass spectrometric uncertainty, weighing uncertainty, and spike uncertainty. ^dRaw ages are corrected for initial ^{230}Th content using the measured ^{232}Th content and assuming the detritus has $(^{232}\text{U}/^{238}\text{U}) = 1.210 \pm 0.605$, $^{230}\text{Th}/^{238}\text{U} = 1 \pm 0.1$, and $(^{234}\text{U}/^{238}\text{U}) = 1 \pm 0.1$.

Conclusions

While the study of the sediments at Tabon is far from complete, the preliminary results of the analysis of the thick gypsum speleothem layer are significant in supporting the radiocarbon sequence obtained by Fox (1970). Assuming the age of the gypsum decreases from bottom to top, gypsum deposition ended sometime after 19.5-20.2 kyr. Thus far, no U-Th ages constrain the timing of the onset of gypsum deposition, however ^{14}C data from Fox (1970) suggest that it began no earlier than c. 23,000 yr. BP. The excellent agreement between U-series dates of the gypsum and ^{14}C dates of the bracketing charcoal confirm that the gypsum deposition and the surrounding deposits formed by vertical accretion according to the Law of Superposition and that the speleothem was not somehow formed authigenically within a pre-existing sedimentary sequence.

Speleothems of various sorts are widespread in Southeast Asian caves, and are often found within, between and growing around archaeological

deposits. Further U-series dating of gypsum and calcite speleothems could significantly improve the chronology of archaeological deposits in this region. In addition, speleothems are widely used for palaeoclimate reconstruction, because their chemistry is controlled by climatic conditions at the time of formation (Fairchild *et al.* 2006). Precise records of climate change could, therefore, be obtained and directly related to records of human occupation and to other palaeoenvironmental sequences (e.g. Bird *et al.* 2005; 2007) at these sites. The development of this type of palaeoclimate data on a regional scale will help answer questions about the role of the tropics in global climate and the complex relationships between El Niño, the intertropical convergence zone, and the Australasian monsoon.

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