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Cross dating (Th/U-^{14}C) of calcite covering prehistoric paintings in Borneo

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Abstract

We present the first application of cross-dating (Th/U measured by thermo-ionization mass spectrometry (TIMS) and ^{14}C measured by accelerator mass spectrometry (AMS)) of calcite covering prehistoric paintings. Th/U age estimates of cave drapery range from 9800 to 27,300 yr B.P. while conventional ^{14}C age is estimated between 9900 and 7610 yr B.P. depending on the dead carbon correction. The age discrepancy is attributed to a disturbance of Th/U and/or ^{14}C geochemical systems, showing the limits of the geochronological approach applied to this kind of material. For the Th/U system, the poor consistency of U data (U content, ^{234}U/^{238}U activity ratios) and apparent ages argue for open system conditions. For ^{14}C system, variation of the dead carbon fraction (dcf) and a possible mixing of successive generations of calcite could account for age discrepancy. Nevertheless, one sample shows concordant ages for the two methods. Compatible ages through corrections for open system conditions are assumed for other samples. Then, the cross-dating suggests 9900 yr as the minimum age of the piece of drapery; the underlying painting must be older. This study of rock art demonstrates the presence of a Pleistocene population before 9900 yr in the southeast of Borneo, whereas previously the only population in evidence in this area was of Austronesian type from ~5000 to 6000 yrs ago.

Keywords: Th/U dating; ^{14}C dating; Open system; Prehistoric paintings; Cave rock art; Borneo; Kalimantan

Introduction

Innovative applications to try to date prehistoric paintings remain the focus of archaeological interest. Direct radiocarbon dating by accelerator mass spectrometry (AMS) has been used on paintings that contain organic materials (Russ et al., 1990; Valladas et al., 1990, 1992; Clottes et al., 1995). Luminescence dating was used to date quartz grains contained in mud-wasp nests covering paintings (Roberts, 1997; Roberts et al., 1997). As these methods are not applicable in many cases, the measurement of Th/U disequilibrium represents an alternative. This method has already been used to date flowstones containing prehistoric remains (Falguères et al., 1992; Bischoff et al., 1994; Delagnes et al., 1999; Turney et al., 2001) as well as teeth or bones from palaeolithic sites (Bischoff et al., 1988; Grün et al., 1988; McDermott et al., 1993; Falguères et al., 1997).

Here, we present the first attempt of a cross-dating of Th/U measured by thermo-ionization mass spectrometry (TIMS) and ^{14}C measured by accelerator mass spectrometry to determine the age of calcite covering prehistoric cave paintings. Different types of calcite deposit may cover the walls or the ceiling of the caves such as (1) a homogeneous layer (calcite crust) covering the whole or a part of the painting, or (2) calcite deposits such as curtains or draperies. For the Gua Saleh cave (Mangkalihat peninsula, Borneo),

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we applied such cross-dating to a calcite drapery, the base of which covers one of the numerous stenciled handprints.

Archaeological question and site description

The first human settlements

Until 1994, Kalimantan (the Indonesian part of Borneo) was an area of southeast Asia (Fig. 1) considered by specialists as totally devoid of art-ornate prehistoric sites. A few archaeological studies of this island began at the end of 1940s but were restricted to Sarawak, one of the Malaysian provinces of Borneo in the north of the island (Harrisson, 1957, 1972; Bellwood, 1985; Bellwood et al., 1995). They describe a dense but discontinuous human presence from the upper Pleistocene to the end of the Holocene. Recent explorations (Chazine, 1995; Chazine and Fage, 1998, 1999; Fage and Chazine, 2001) uncovered several decorated caves in the extreme eastern part of Kalimantan, a region not yet subjected to any archaeological investigation. It was thought that cave rock art existed only on Sulawesi, the neighboring island, eastward of Makassar Straits and the Wallace Line. The recent discoveries of these Borneo prehistoric paintings are thus of prime importance in our understanding of the regional constitution of prehistoric cultures and population migrations.

Site description

At least 22 decorated caves were discovered in Kalimantan Timur province (Mangkalihat peninsula), a very mountainous karstic area, the more beautiful ones being found during the late surveys (Chazine and Fage, 1998, 1999; Fage and Chazine, 2001). Limestone outcrops are generally located between 300 and 700 m above sea level, but several peaks reach more than 1000 m. The poor knowledge of this karstic area is due to the difficulties of exploration of this island, still covered by primary forests with steep and almost inaccessible karstic outcrops. Most of the painted caves are located in the upper levels of these outcrops (Chazine and Fage, 1998; http://www.kalimanthrope.com/). They contain numerous paintings remarkable for their number, variety and pictorial content. The most beautiful decorated caves (Gua Saleh, Gua Ham, Gua Tewet, and Gua Masri) exhibit a very large number of paintings, mostly negative handprints particularly well preserved. Gua Saleh Cave is a system of three large chambers developed along an axis parallel to the cliff, each one with a large opening on the valley. This cave contains about 200 figures including more than 140 stenciled handprints, anthropomorphic figures, and zoomorphic representations such as a bovine and two big mammals without heads exceeding 1 m. Paintings are located upon the walls of the cave, from 1 to more than 10 m above the present ground level. Depending on location, some paintings are covered by different kinds of calcite layers of variable thickness.

Sampling and methods

Sampling

The Gua Saleh calcite drapery chosen for dating is about 15 cm high (Fig. 2a). It partly overlays a stenciled handprint and a pigmented blob which is likely to be a second handprint. They are considered contemporaneous because they are made of the same red pigment. This pigment is pure hematite and does not contain datable carbon (analyzed by Laboratoire des Musées de France in 1995). A piece of the drapery was sampled between the stenciled handprints (Fig. 2b). The sample was chosen to contain the first calcite layers that overlap the painting and from a position a few centimeters away from it to avoid any damage. The time elapsed between the execution of painting and the calcite deposition cannot be determined, but the drapery definitely postdates the painting.

Microscopic observation of a transverse section of the drapery shows laminar growth and a compact external calcite layer visible in the part closest to the roof. By contrast, the outermost zone of the drapery is characterized by porous calcite and the absence of the compact external calcite layer. This difference of structure suggests an alteration, particularly for the outermost part of the sample, due to the circulation of water along the swell that constitutes the edge of the drapery. In such a case, the Th/U geochemical system could have been opened and the uranium of the deposit leached by seepage. So, as the conditions of applicability of the Th/U method were not guaranteed, ^{14}C dating of the same samples was also undertaken.

The location of the five samples collected for Th/U and ^{14}C analyses are indicated in Figure 2d. The drapery growth follows the water flow direction along the slope of the cave roof and walls (Fig. 2c). A series of samples located nearest to the upper handprint and also closest to the roof were collected in order to date the painting as accurately as possible. First, three small adjacent samples (Bor7A, 7B, and 7C) were collected above the pigmented blob, Bor7A being the closest to the roof and samples Bor7B and 7C located below Bor7A in the direction of drapery's growth. Then, Bor5 was collected below the pigmented blob and below the set of Bor7 samples in the direction of water flow and above the handprint. Bor2 was collected in the outermost part of our piece of drapery, just above the final swell. According to the drapery growth, the age of the calcite is expected to decrease from the cave top (Bor7A, 7B, and 7C) to the bottom walls (Bor5), and from the inner (close to the cave walls, Bor7 and Bor5) to the outer part of the drapery (Bor2).

These five samples were dated by the Th/U method, while Bor5, 2, and 7A (and 7A*, a duplicate of 7A) were

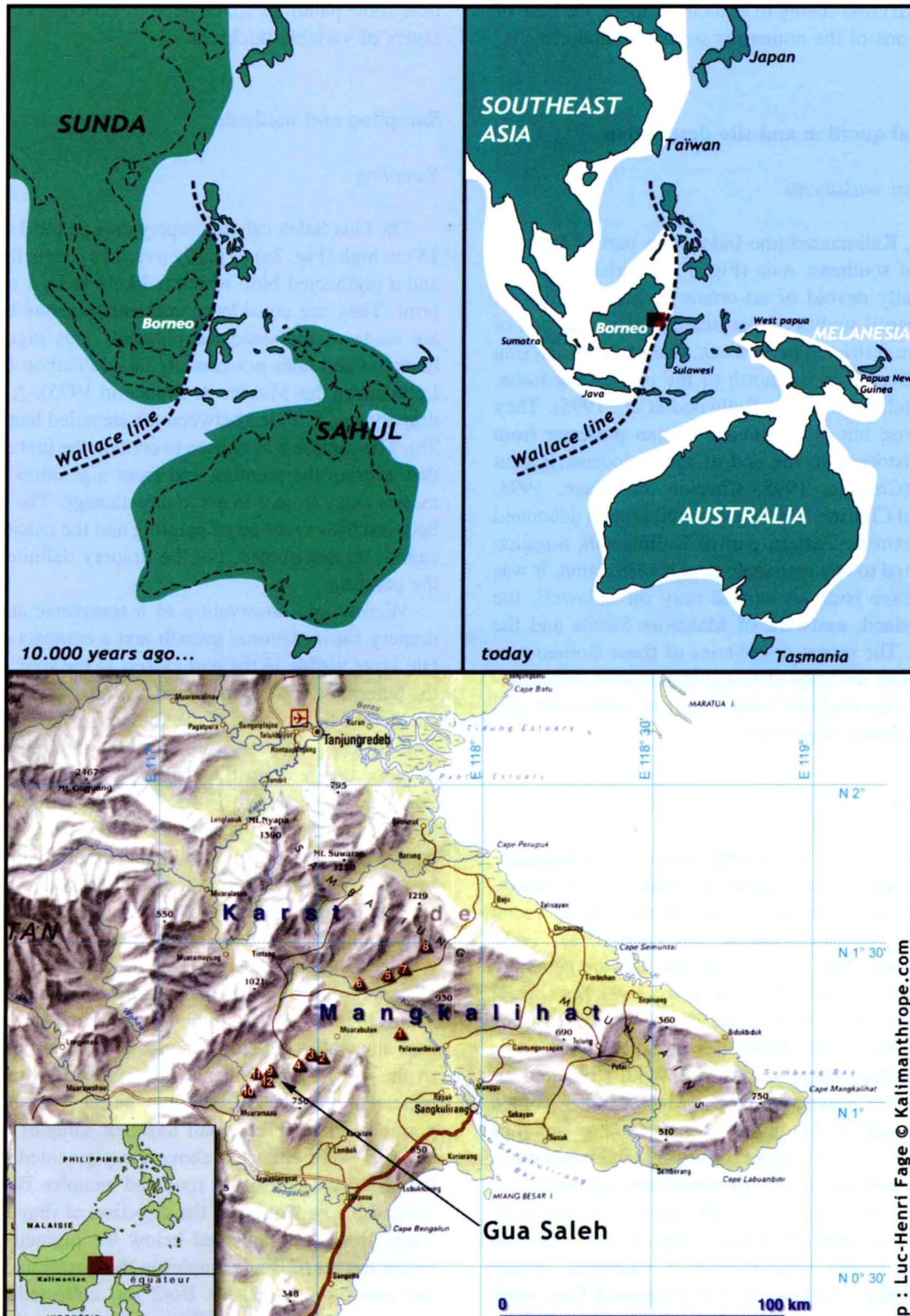

Fig. 1. Map of southeast Asia, showing the extension of emerged lands during the maximum of the last glaciation, 18,000 yr ago. At this time Borneo, Java, and Sumatra were connected to the Asiatic continent called Sunda. This continent was separated from the Australian continent named Sahul (connecting Australia, New Guinea, and Tasmania) by an arm of deep sea called the Wallace Line. This line is known to separate two continents characterized by different types of fauna and flora. But the role of this line with regard to human population movement is still unknown. Triangles show the location of the known archaeological sites and decorated caves discovered in the extreme east part of Kalimantan.

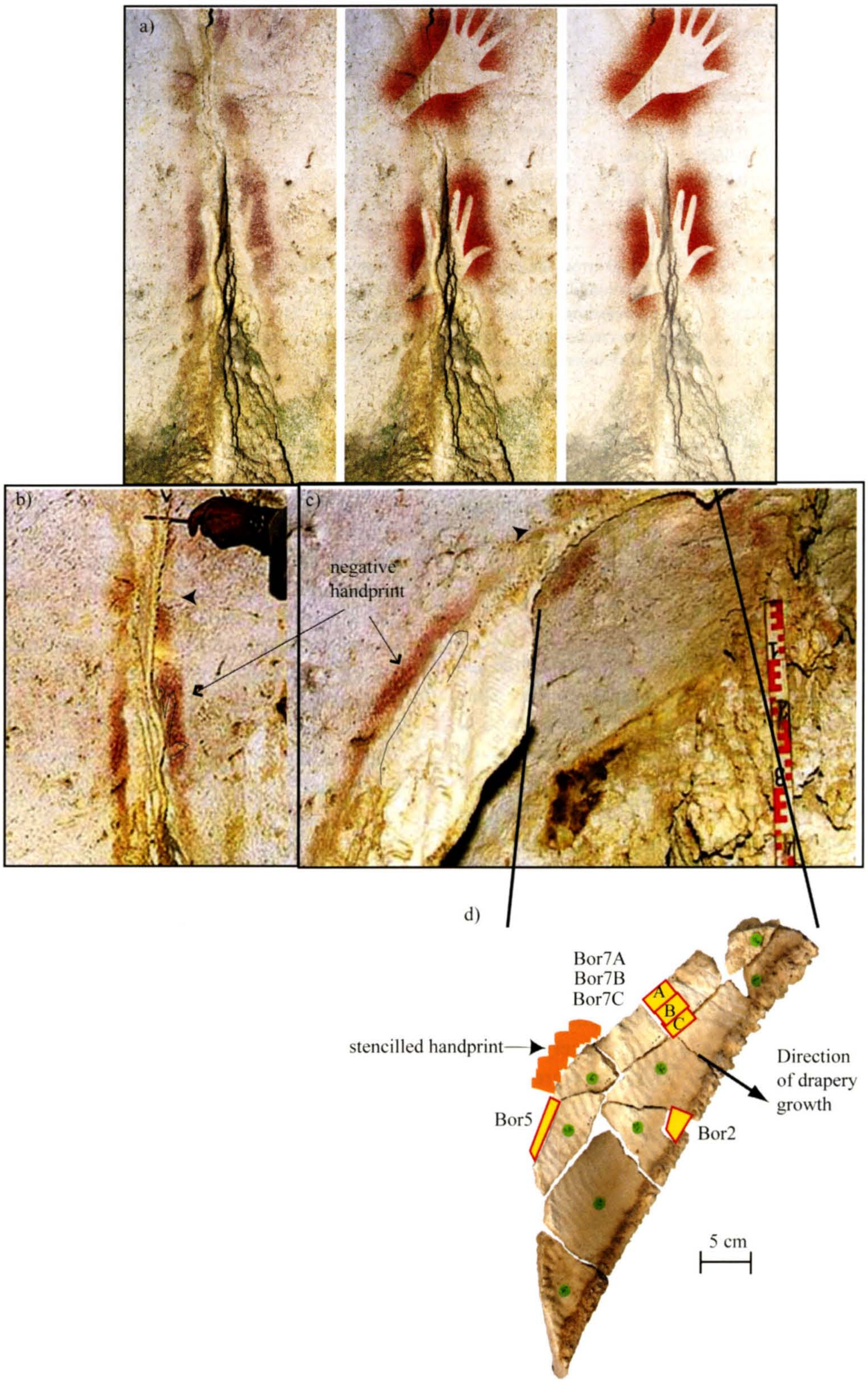

Fig. 2. (a) Location of the drapery with respect to the prehistoric paintings (negative stenciled handprints) in Gua Saleh Cave. (b and c) The drapery before and after sampling. (d) Location of the samples analyzed and direction of drapery growth.

dated by ^{14}C. For Th/U analyses, the samples consist of the whole thickness of the deposit (thickness less than 1 cm). The external surfaces were mechanically abraded with a diamond saw in order to eliminate the outer calcite layer (which is assumed to be the youngest) and all impurities. Because of known low U content (0.057–0.180 ppm), the

Table 1
TIMS U-Th results and age estimates

	[U] (ppm)	$^{234}U/^{238}U$ (activity ratio)	$^{230}Th/^{234}U$ (activity ratio)	Age A_0 (yr)	$^{230}Th/^{232}Th$ (activity ratio)	Age A_1/yr 2000 (yr)	± (2σ)
Bor5	0.1797 ± 0.0001	0.9654 ± 0.0030	0.0897 ± 0.0001	10,222	30.58	**9,870**	60
Bor7A	0.0686 ± 0.0001	0.9669 ± 0.0042	0.1153 ± 0.0014	13,329	17.20	**12,560**	130
Bor7B	0.0736 ± 0.0001	0.9654 ± 0.0038	0.1149 ± 0.0016	13,287	16.53	**12,490**	160
Bor7C	0.0738 ± 0.0001	0.9622 ± 0.0031	0.1247 ± 0.0010	14,501	10.97	**13,200**	90
Bor2	0.0572 ± 0.0000	0.9668 ± 0.0029	0.2331 ± 0.0019	28,913	15.13	**27,320**	210

Note. The age A_0 is the apparent $^{230}Th/^{234}U$ age; all errors are calculated by error propagation and given at 2σ level. $^{230}Th/^{234}U$ ratios are activity ratios calibrated to HU1 (uraninite) assumed to be at secular equilibrium. Repeated analysis ($N = 10$) of the HU1 uraninite standard yields a mean $^{234}U/^{238}U$ activity ratio of 1.0026 with a reproducibility (2 standard deviations) of 0.0050 and a mean $^{230}Th/^{234}U$ activity ratio of 1.0015 with a reproducibility of 0.0066. $^{230}Th/^{232}Th$ activity ratios are small and require a correction for ^{230}Th excess, due to detrital material mixed with carbonate. Ages A_1 are corrected ages, assuming an initial $^{230}Th/^{232}Th = 1$ (see text). Decay constants used: $\lambda^{230}Th = 9.1953 \times 10^{-6}$, $\lambda^{234}U = 2.8338 \times 10^{-6}$, $\lambda^{238}U = 1.55125 \times 10^{-10}$.

highest possible masses (2–4 g) were taken. For ^{14}C analyses, four samples of about 10 mg were taken from the inner part of the samples used for Th/U analyses and are considered to represent the oldest calcite.

Th/U dating

Samples were heated at 900°C for 1 h to oxidize all organic and mineral components present in the speleothem matrix. Next, the samples were dissolved by 6 N HCl in Teflon beakers containing a known amount of mixed ^{233}U–^{236}U–^{229}Th spike. The sample-spike mixture (with carrier $FeCl_3$) was left on a hot plate overnight to ensure complete ionic equilibration with the spike solution. Subsequent coprecipitation with NH_4OH (at pH 7) separated the uranium and thorium from most of the calcium. The development of the precipitates was allowed to continue overnight. U and Th were separated using Dowex anion-exchange resin (1X8) conditioned with 6 N HCl. Next, U and Th were purified using Eichrom resins (Uteva and Teva, respectively) conditioned by 3 N HNO_3. U and Th fractions were loaded onto preoutgassed single rhenium filaments with a graphite coating and the isotope ratios were measured on a Finnigan 262 mass spectrometer (Plagnes et al., 2002).

^{14}C dating

The conventional treatment was applied to the calcite samples, which were finely crushed, and the grains, were washed in distilled water using an ultrasonic bath. The surface of these grains was cleaned using 0.01 N HNO_3 and then rinsed with distilled water. Under vacuum, the calcite was reacted with phosphoric acid and the evolved CO_2 was reduced to get graphite targets prepared following the method described by Tisnerat-Labordes et al. (2001). Analyses were performed using Gif AMS facilities (UMS T2004, Gif-sur-Yvette, France). The sample 7A* was dated by β-Analytic INC (Miami, FL).

Results

Results are presented in Tables 1 and 2. The uranium content of these samples is low, ranging between 0.0572 and 0.1797 ppm. $^{234}U/^{238}U$ activity ratios are notably constant, around 0.966. All samples contain small amounts of ^{232}Th, meaning addition of detrital material (clays) to pure carbonate. Typically, $^{230}Th/^{232}Th$ activity ratios of detrital material originating from volcanic and nonsedimentary rocks exhibit values close to 1 (Gaven, 1975; Gill et al., 1992). This value is taken for the correction for ^{230}Th

Table 2
AMS ^{14}C ages and correction for dead carbon fraction (dcf)

	Analysis GifA	Conv. age, ^{14}C yr B.P.	dcf-corrected age, ^{14}C yr B.P.			Calibrated age relative to A.D. 2000 (cal yr B.P. + 50)		
			dcf5	dcf10	dcf20	dcf5	dcf10	dcf20
Bor5	100 792	9,010 ± 90	8600	8160	7210	9900 → 9480	9430 → 8770	8190 → 7840
Bor7A	100 793	8,675 ± 90	8270	7830	6880	9430 → 9060	8760 → 8470	7790 → 7610
Bor7A*								
Bor2	100 791	9,010 ± 100	8600	8160	7210	9900 → 9480	9430 → 8770	8190 → 7840

Note. Three ^{14}C ages were determined at the Gif AMS facilities and one sample was determined by βAnalytic INC (Bor7A*). The age of Bor7A is the mean age of the two ^{14}C analyses. A correction is made for the dcf carried out from the carbonate host rock. Calibration of ^{14}C ages has been done with the INTCAL 98 radiocarbon age calibration (Stuiver et al., 1998). The age, normalized to the year 2000, has been calculated in order to compare Th/U and ^{14}C ages.

excess due to detrital material (Causse and Vincent, 1989). Corrected ages range between 9870 and 27,320 yr. ^{14}C yr B.P. ages ranging between 8670 ± 90 to 9010 ± 100 yr need to be corrected for a dead carbon fraction (dcf) coming from the dissolution of the carbonate host rock. The dcf is generally considered as stable, ranging from 5 to 20% of total carbon for contemporaneous carbonate deposits of the same site (Ford and Williams, 1989; Genty and Massault, 1997; Genty et al., 1999, 2001; Beck et al., 2001). The dcf being unknown in our case, we used three dcf corrections of 5, 10, and 20%, respectively. The dcf of 20% gives the minimum ^{14}C ages of calcite. The ^{14}C ages are given in ages with respect to the year 2000 in order to facilitate comparison with Th/U ages (Table 2). Depending on the dcf value used, calibrated ^{14}C ages (relative to A.D. 2000) vary between 9900 and 7610 yr B.P.

Th/U apparent ages range from 9870 to 27,320 yr and they correspond to a decrease in U content from 0.18 to 0.06 ppm. Though Bor2 gives the lowest U content (0.057 ppm) and the oldest Th/U age of 27,320 ± 210 yr, the age is not consistent with its stratigraphic location in the growth of the drapery. The Bor2 age should be younger than the ages of the samples collected close to the cave roof (Bor5 and 7). In addition, the ^{14}C age of Bor2 of 9900–7840 cal yr (with a dcf of 5 and 20%, respectively, and relative to A.D. 2000) is not consistent with the Th/U age, thus demonstrating an open Th/U geochemical system.

The set of three adjacent Bor7 samples exhibit similar U content and $^{234}U/^{238}U$ activity ratios, arguing for closed system conditions. Bor7A and Bor7B give the same Th/U age (12,560 ± 130 and 12,490 ± 160 yr, respectively) while Bor7C gives an older age of 13,200 ± 60 yr. This small difference between the ages of Bor7C and Bor7A-7B could be caused by a tiny difference in the level of abrasion of the recent outer calcite layer of these samples before dating. On the other hand, the ^{14}C measurements of Bor7A and its duplicate 7A* give identical ^{14}C ages (8610 ± 90 and 8740 ± 40 ^{14}C yr B.P.) with a mean value of 8670 ± 90 ^{14}C yr B.P. Therefore, the ^{14}C ages are 3100 to 4900 cal yr younger than the Bor7A Th/U age, depending on the dcf correction used (5 and 20%, respectively), showing a disturbance of either one or both of the geochemical systems (U-Th and/or ^{14}C).

Bor5 presents identical Th/U and ^{14}C ages with a dcf of 5%: 9870 ± 60 and 9900 to 9480 yr, respectively. But the age difference between the two methods reaches 2000 yr with a dcf of 20%. The Bor5 Th/U age is younger than the Th/U age of Bor7, in accordance with its stratigraphic location with respect to the direction of growth of the drapery, while this difference of age is not observed for the ^{14}C ages.

Discussion

The Th/U and ^{14}C ages do not match except for the sample Bor5 (9870 yr). This age discrepancy is discussed in terms of opening and/or closing of the geochemical systems since the time of calcite deposition. For ^{14}C, the opening of the geochemical system would imply some calcite dissolution and recrystallisation. This would either result in a different carbon isotopic composition or would cause an open Th/U system or both.

Uranium seems to be immobilized within the calcite structure for millions of years, implying that the local coordination of uranium in natural calcite is structurally stable. Sturchio et al. (1998) showed this to be true for U (IV) in natural calcite, where the U^{4+} ion occupies the Ca position with minimal distortion. Reeder et al. (2001) showed that a disruption of the local coordination around UO_2^{2+} is expected for U(VI) in natural calcite. These observations make possible a selective loss of soluble U in the case of the opening of the system by leaching, as thorium is much more insoluble. If such is the case, this process involves uranium solubility without necessarily implying dissolution and recrystallization of calcite and without breaking down the crystal lattice. If we make the hypothesis that any other process has affected the carbonate fraction, we can consider the geochemical system as closed for ^{14}C.

A closed Th/U geochemical system?

One hypothesis to explain the age discrepancy is to assume an opening of the Th/U geochemical system after calcite deposition. The circulation of water on and through the deposit is generally the source of this system opening, which yields a leaching of U and results in an age overestimated. This certainly appears to be the case for the sample Bor2 which exhibits an anomalously old Th/U age (27,320 ± 210 yr) and the lowest U content (0.0572 ppm) even though it is the youngest sample stratigraphically. The probable leaching of U is supported by the observation, in thin section and at macroscopic scale, of porous calcite. Consequently, the Th/U age of Bor2 is considered unreliable and invalid.

By contrast, the series of samples collected nearest to the handprints (Bor7A-7B-7C and Bor5) gave a reasonably consistent set of $^{234}U/^{238}U$ activity ratios and apparent Th/U ages consistent with their location along the drapery growth direction (Bor5 is younger stratigraphically than the set Bor7). Generally, the consistency of data such as homogeneous U content and $^{234}U/^{238}U$ ratios for a few samples belonging to the same environment, as well as equivalent measured ages for contemporaneous samples or ages varying according to stratigraphy, is a good criterion for a closed Th/U geochemical system since the time of crystallization. However, the U content of Bor7 (~0.07 ppm) is less than half that of Bor5 (0.18 ppm), arguing for possible U leaching. As noted, Th/U and ^{14}C give different ages for sample Bor7A whatever the dcf may be: ^{14}C age is younger by 3100 to 4900 cal yr than the Bor7A Th/U age with a dcf of 5 and 20%. This discrepancy can be attributed to U leaching though it is obviously less than that for Bor2.

Because ^{14}C and Th/U ages of Bor5 are in agreement, we can consider that this sample, which is characterized by the highest U content, has not been affected significantly by U leaching. So, we can assume that the Th/U geochemical system remained closed only for sample Bor5, while it has been opened to different degrees for the samples Bor7 and Bor2. Hence, the only Th/U age validated is the one of Bor5: 9870 ± 60 yr.

If Bor5 U content is assumed to represent the best approximation for initial U content, Bor2 would give, using this correction, a Th/U age of 8390 yr instead of 27,320 yr and Bor7A would give a Th/U age close to ~4900–5700 yr instead of 12,500 yr. These new Th/U ages calculations are more consistent with ^{14}C ages with a dcf correction of 15% for Bor2, while it should reach 30% for Bor7A. However, such differences of dcf correction are improbable for the same sample.

A closed ^{14}C geochemical system?

The three dated samples give ^{14}C ages converging between 7610 and 9900 cal yr before A.D. 2000, indicating that the growth phase would have been short. These convergent ^{14}C ages (compared the scattering of Th/U ages) constitute a good argument for a closed ^{14}C geochemical system. Another observation supports the hypothesis of a ^{14}C carbonate closed system: the constant $^{234}U/^{238}U$ activity ratio which is compatible with both a short period of calcite precipitation and a dissolution of uranium that does not induce isotopic fractionation and a fortiori calcite dissolution/recrystallization processes.

Considering that cross-dating validates the age of Bor5, we can assume that the dcf is about 5% and that this dcf has been quite stable during the whole deposition of the drapery. With such a dcf, the sample Bor7A is younger by few tens of years than Bor5, while it would have the same age with a lower dcf of about 3%. The three samples give nearly identical ^{14}C ages with a dcf of about 3–5%, indicating a very fast growth of the drapery between ~300 and 500 μm yr^{-1}. In such a case, all the ^{14}C ages can be considered valid and they correspond to a minimum age of 9400 yr for a single growth phase of the drapery.

The age difference between Bor7 and Bor5/Bor2, however, can also be considered to result from a disturbance of the ^{14}C carbonate geochemical system due to the deposition of successive generations of calcite in holes of porosity as part of the dissolution/recrystallization processes. If such a contamination exists, it would need less than 1% modern carbon contamination to get compatible ^{14}C ages. At this level, the system can be considered closed.

We cannot ensure that the single ^{14}C and Th/U age of Bor5 is strictly reliable, but it seems improbable that two altered geochemical systems (Th/U and ^{14}C) could give the same age for the same sample. Taking in account all the arguments listed above, 9870 yr probably represents the youngest age limit for the stand of the drapery and we can thus conclude that the painting which is covered by this calcite deposit was painted before that period.

The deposition of calcite in caves is mainly controlled by water flow through the overlying karstic system. In this case, the deposition of the drapery could be related to the abrupt increases of Asian monsoon intensity between 9000 and 10,000 yr ago as discussed by Rossignol-Strick (1983), Sirocko et al. (1993), Fontugne et al. (1994), Overpeck et al. (1996), and Wang et al. (1999). Such an independent chronological correlation corroborates clearly hypothesis about the age drapery.

Conclusion

The application of Th/U and ^{14}C chronometers appears to be critical in dating calcite coverings such as drapery because of a porous matrix and possible mixing of successive generations of calcite. For calcite drapery of Gua Saleh Cave, considering the variation of U content and comparison between U/Th and ^{14}C measurements, we proposed a minimal age of 9870 yr for painting covered by drapery. This is in agreement with archaeological hypothesis and interpretations (Chazine and Fage, 1998). This study confirms the presence of a population more than 9900 yr ago in southeastern Borneo already practicing rock art, while previously, the only population known in this area belonged to the Austronesian culture with evidence dating roughly to 6000 yr ago. The discovery of the existence of such advanced artistic activity at such an early period provides new insights into the regional presentation of prehistoric culture and migration of populations in this part of southeast Asia. This conclusion is of prime importance for the Pleistocene-Holocene period of this still poorly documented area.

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