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# AMS $^{14}\text{C}$ dating of the hominin archaeological site Chuandong Cave in Guizhou Province, southwestern China

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## ABSTRACT

This study presents detailed AMS  $^{14}\text{C}$  dating of charcoals, burned and unburned bones, and teeth from the hominin archaeological site Chuandong Cave, located in Puding County, Guizhou Province, southwestern China. The charcoal samples were pretreated with either the acid–base–acid (ABA) or ORAU–XR method, the unburned bone and teeth samples with ABA–collagen pretreatment, and the burned bone samples with the ORAU–CB method. The AMS  $^{14}\text{C}$  ages of the charcoal samples provide the most reliable results in this study. The AMS  $^{14}\text{C}$  dates of the bone samples are generally younger, possibly due to posterior amino acid contamination. Based on the AMS  $^{14}\text{C}$  ages, we propose the following chronology for the site: Layers 3 to 5 formed between 11.5–12.5 ka BP (all  $^{14}\text{C}$  ages reported in this study are calibrated ages unless stated otherwise. A BP = years before 1950 CE), a period corresponding to the Younger Dryas; Layers 6 to 7 formed between 14–24 ka BP, a period including the Last Glacial Maximum (LGM); and Layers 8–9 likely formed before 34 ka BP corresponding to the late Pleistocene. According to the above chronology, the Chuandong humans (*modern Homo sapiens*) were present at 12 ka BP, i.e., the late Pleistocene. This conclusion differs from the previous estimate at ~9 ka BP, i.e., the early Holocene. Furthermore, the Chuandong Culture likely began as early as 34 ka BP and survived in the region throughout the cold and dry LGM. Detailed studies on the hominin archaeological sites in Guizhou, including Chuandong Cave and Maomaodong Cave, are warranted to better understand hominin evolution in Asia.

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## 1. Introduction

Chuandong Cave (28°18'N, 105°45'E) is located in Houzhai village of Puding County, Anshun City, Guizhou Province in southwestern China (Fig. 1). After the archaeological site Chuandong Cave was found in 1978, three major excavations were organized between 1978 and 1982. The site contained abundant archaeological materials, including two modern *Homo sapiens* skulls, more than ten thousand stone artefacts, more than one thousand horn and bone implements and a large number of animal fossils (Wu and Cao, 1983; Zhang, 1995). All the recoveries are curated by the Guizhou museum. Many archaeological studies on these items have

been performed (Yu et al., 1983; Zhang, 1983; Yu, 1984; Huang, 1989; Mao and Cao, 2012). The fourth layer of the Chuandong site contains hominin fossils. Fire residues including charcoals and burned bones preserved in several layers of the archaeological site were caused by human activities (Wu and Cao, 1983; Yu, 1984; Huang, 1989; Zhang, 1995). Conventional radiocarbon dating ( $^{14}\text{C}$  beta decay measured via the liquid scintillation counting method) of bone samples in mid-1980s yielded  $^{14}\text{C}$  ages for the third to fifth layers of 8000–9000 a BP (a BP = years before 1950 CE. All  $^{14}\text{C}$  ages in this paper are calibrated ages) (Li et al., 1987) (Table 1). Thus, the hominin fossils were considered to belong to the early Holocene, when the climate was warm and humid. For the lower part of the Chuandong site, the dating work was rather poor. One conventional  $^{14}\text{C}$  age from an animal bone from the sixth layer was  $9610 \pm 100$  a BP (Li et al., 1987), and another  $^{14}\text{C}$  age of an animal bone from Layers 8–9 was 16,000 years (Zhang, 1988) (Table 1). According to

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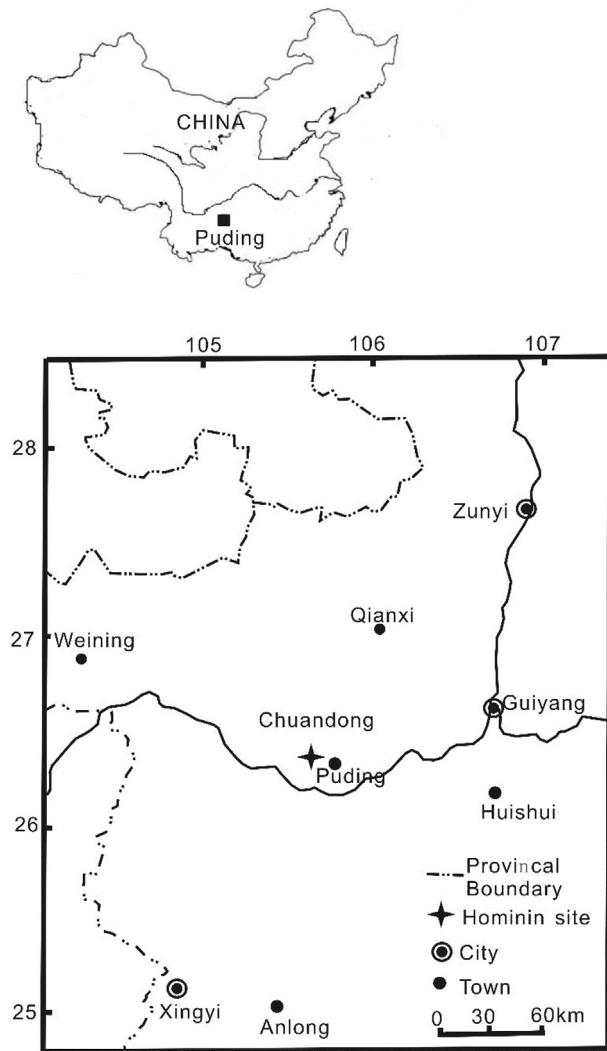


Fig. 1. The location map of the Chuandong archaeological site in China (Modified from Zhang, 1995).

these previously published ages, Huang (1989) suggested that the upper strata (Layers 3–5) of the Chuandong hominin site belonged to the Neolithic period and that the lower strata (Layers 8–10) was close to the end of the late Palaeolithic period. Furthermore, the morphological features of the hominin skulls from the site were generally modern morphologies but mixed with some archaic features. Based on the previous chronology and fossil features, the Chuandong humans have been classified as a modern type of hominin (Wu and Cao, 1983; Yu, 1984; Huang, 1989; Wu and Wu, 1989).

Limited by dating techniques, many fossils discovered in the last century, especially in earlier studies, lack precise dating results (Bonsall et al., 2015). Consequently, our understanding of the origin of modern humans in East Asia is limited. For instance, the  $^{14}\text{C}$  dating of the Chuandong site used the conventional beta-decay liquid scintillation counting method on bone samples. On one hand,  $^{14}\text{C}$  dating on bone sample is rather complicated due to multiple carbon compounds and their preservations in the deposited bones. Evidence suggests that many late Pleistocene bone dates (perhaps ~70% or more) published in the 1980s–1990s are liable to be underestimates of the true ages (Higham, 2011; Marom et al., 2012). In general, archaeologists prefer direct

dating on bone samples because the dating provides clear physical meaning (Keates et al., 2012). However, the  $^{14}\text{C}$  dates of the bone samples from the Chuandong site in the previous studies of Li et al. (1987) may bear large age uncertainties. Fortunately, charcoal samples can be found within the same cultural layer as the bone samples. Based on previous archaeological studies the charcoal grains were from fire use of the Chuandong human. As the site is 26 m higher than the surrounding ground, it was impossible for the ancient people to move large trees (woods) into the cave for the fire. Therefore, the charcoal ages should have good estimation of the cultural layers. Hence, these age constraints of the Chuandong site based on the conventional radiocarbon dating on bones could be improved by obtaining charcoal samples from the site. On the other hand, accelerator mass spectrometry (AMS)  $^{14}\text{C}$  dating is now a mature technique and widely used in archaeology (Kutschera, 2005). The AMS  $^{14}\text{C}$  dating technique can be used to date 1 mg of C, thereby improving the feasibility of using charcoal samples for dating. For these reasons, we collected new materials including charcoals, unburned and burned bones, and teeth from the Chuandong site in 2015 and conducted AMS  $^{14}\text{C}$  dating on the samples. The new dating results can be used to determine whether the Chuandong humans were active during the early Holocene or the late Pleistocene. The new chronology of the hominin site in Chuandong Cave will contribute to our understanding of the evolution of hominins in South China during the late Pleistocene as well as their relationship with hominins in other regions.

## 2. Materials and methods

### 2.1. Site description and sampling

Chuandong Cave is approximately 5 km southwest of the central township of Puding County. The cave is ~30 m in length, 13 m in width and 13 m in height and oriented in a northeast direction (Fig. 2a). The site experiences a humid subtropical monsoon climate, with an annual precipitation of 1393 mm and a mean air temperature of 15.1 °C based on the meteorological data of Anshun City during 1950–2012. With an elevation of 1264 m a.s.l., the site is located on an isolated karst peak that is 26 m higher than the surrounding ground. The deposits of the site were partly dug out for fertilizer by local villagers. However, a part of the fossiliferous section at the entrance of the cave has been preserved. Our sampling work on the fossiliferous section was following the description of the previous excavations. The archaeological section can be divided into 10 layers from top to bottom (Mao and Cao, 2012; Zhang, 1995) (Fig. 2b).

Layer 1 (L1): disturbed grey soil, including polished stone axes, approximately 0.2–0.4 m thick.

Layer 2 (L2): brownish yellow clayey deposits with dolomite gravels, rich in stone and bone artefacts, 0.4–0.6 m thick.

Layer 3 (L3): fine and dense brown clayey deposits with a few small gravels and charcoal grains, but rich in archaeological remains, approximately 0.5–0.7 m thick.

Layer 4 (L4): dark brown clayey deposits, containing a number of stone and bone artefacts. Human fossils were excavated from this layer, approximately 0.4–0.6 m thick.

Layer 5 (L5): sandy clay, with a thin flowstone sublayer at the bottom and containing ash aggregations, a number of burned bones, and a few stone and bone artefacts, approximately 0.4–0.6 m thick.

Layers 6 and 7 (L6 and L7): red and yellow sandy clay, containing big limestone gravels and a thin flowstone sublayer between the sixth and seventh layers. The sediment composition, particle size and colour are the same in both layers, so they can be merged into

**Table 1**

Details of the  $^{14}\text{C}$  dating of the samples collected from Chuandong site. ABA denotes the acid-base-acid treatment. ABA-collagen is used for total bone organic carbon of collagen in the unburned bones and teeth. ORAU-XR and ORAU-CB are the Oxford AMS Lab pretreatment protocols for old charcoal and burned bone samples, respectively. The error of the  $^{14}\text{C}$  age is  $1\sigma$  uncertainty.

Lab code	Sample ID	Sample description	C14 Counts	pMC (%)	$\Delta^{14}\text{C}$ (‰)	Age (a BP)	Calib. Age (a BP)	Previous age (a BP)
NTUAMS-1946	PCD-1	Charcoal in L3 (ABA)	21292	28.86 ± 0.27	-711.4 ± 6.5	9983 ± 92	11510 ± 180	8080 ± 100 (Bone)
NTUAMS-1946-A	PCD-1	Charcoal in L3 (ORAU-XR)	28263	28.11 ± 0.17	-718.9 ± 4.3	10193 ± 61	11875 ± 160	(Li et al., 1987)
NTUAMS-1947	PCD-4	Charcoal in L4 (ABA)	20441	27.97 ± 0.26	-720.3 ± 6.7	10235 ± 95	11995 ± 255	8670 ± 100 (Bone)
NTUAMS-2043	PCD-5	Burned bone in L4 (ORAU-CB)	35941	30.11 ± 0.16	-698.9 ± 3.7	9643 ± 51	11005 ± 140	(Li et al., 1987)
NTUAMS-2044	PCD-6	Bone residue in L4 (ABA-collagen)	11528	54.881 ± 0.51	-451.21 ± 4.2	4820 ± 45	5550 ± 50	
NTUAMS-2455	PCD-8	Charcoal in L4 (very small amount) (ORAU-XR)	9105	29.68 ± 0.49	-703.2 ± 11.6	9756 ± 162	11135 ± 270	
NTUAMS-1948	PCD-7	Charcoal in L5 (ABA)	22103	26.98 ± 0.25	-730.2 ± 6.6	10524 ± 96	12435 ± 200	8540 ± 100 (Bone)
NTUAMS-2456	PCD-9	Bone residue in L5 (ABA-collagen)	9632	29.56 ± 0.48	-704.4 ± 11.5	9790 ± 160	11210 ± 300	(Li et al., 1987)
NTUAMS-2459	PCD-10	Burned bone in L6 (ORAU-CB)	7394	23.34 ± 0.40	-766.6 ± 13.3	11687 ± 202	13600 ± 250	9610 ± 100 (Bone) (Li et al., 1987)
NTUAMS-2461	PCD-11	Burned bone in L7 (ORAU-CB)	10030	14.47 ± 0.24	-855.3 ± 13.9	15529 ± 252	18700 ± 400	
NTUAMS-2460	PCD-12	Tooth in L7 (ABA-collagen)	4480	8.89 ± 0.18	-911.1 ± 17.9	19442 ± 383	23285 ± 505	
NTUAMS-2457	PCD-13	Charcoal in L8 (ORAU-XR)	2911	2.58 ± 0.06	-974.2 ± 22.0	29394 ± 662	33640 ± 590	16000 (Bone)
NTUAMS-2462	PCD-14	Burned bone in L8 (ORAU-CB)	2910	4.51 ± 0.10	-954.9 ± 21.5	24890 ± 561	29655 ± 700	(Zhang, 1988)
NTUAMS-2476	PCD-15	Charcoal in L9 (ORAU-XR)	811	3.64 ± 0.14	-963.6 ± 36.0	26614 ± 995	31310 ± 865	
NTUAMS-2477	PCD-16	Burned bone in L9 (ORAU-CB)	2034	17.81 ± 0.55	-821.9 ± 25.5	13860 ± 430	16805 ± 720	

one layer. Few remains were excavated from the two layers (Fig. 3). Approximately 0.4–0.5 m thick.

Layer 8 (L8): clayey sand with small weathered dolomite pebbles, containing a few small stone artefacts and an ash aggregation upon the thin flowstone sublayer at the bottom. Approximately 0.25–0.35 m thick.

Layer 9 (L9): red and grey clayey deposits intercalated by six thin flowstone sublayers, containing a few charcoal grains and burned bones. Approximately 0.4 m thick.

Layer 10 (L10): Clayey sand with a few fossils and small stone artefacts. An exposed section ca. 0.3 m thick, total thickness unknown.

In this study, fragments of charcoal, bones, burned bones and teeth were collected from L3 to L9 for AMS  $^{14}\text{C}$  dating. Charcoal grains caused by the fire uses of the Chuandong man were remained in the culture layers. We collected the charcoal samples with forceps and separated them from the soil as much as possible at the site. The burned and unburned bone samples and teeth collected from the site were animal remains.

## 2.2. Methods

The pretreatment, carbon extraction,  $\text{CO}_2$  purification, graphitization and AMS measurement were all performed in the



Fig. 2. Pictures of the Chuandong archaeological site and sampling section.





Fig. 3. Pictures of the animal teeth from Layers 6–7.

Accelerator Mass Spectrometer Radiocarbon Dating Laboratory of the Geosciences Department at the National Taiwan University (the NTUAMS Lab). All the samples were initially surface cleaned using ultrapure water (Milli-Q) to remove dust and soil. The samples (charcoal, bone and teeth) were sequentially treated with a standard acid-base-acid (ABA) treatment (Olsson, 1986). In the ABA treatment, the initial acid wash of 0.5 M hydrochloric acid (3 rinses and over 18 h) removes any contaminating carbonate and fulvic acid. The sample is then treated repeatedly with 0.1 M sodium hydroxide (half an hour) to remove humic material, until the base solution does not show brown colour and remains clear. Then, another acid wash is performed using 0.5 M hydrochloric acid to remove any atmospheric CO<sub>2</sub> absorbed during the alkaline wash. Then, the sample is washed with Milli-Q water to remove any chloride until the solution has a neutral pH. The samples are then placed in a rack with ventilated caps and put in an oven to dry at 50 °C.

Charcoal samples are pretreated in two ways: one involves that standard ABA pretreatment described above, and the other is applied to older charcoal samples and is called ORAU-XR. This method removes contaminants more efficiently than the standard ABA pretreatment, but sample sizes of 50–100 mg or more are required (Brock et al., 2010) because significant sample amounts are lost during this rigorous chemical pretreatment. In this process, 50–100 mg of charcoal is placed in a pre-combusted (at 400 °C) 50 ml beaker and treated with 6 M HCl at room temperature for 1 h. Then, 1 M NaOH is added at room temperature, and the solution is left to sit for 30 min. The samples are then washed 3 times with Milli-Q water. Then, the sample is left to react with 0.1 M potassium dichromate (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>) and 2 M sulfuric acid in a 9 mm sealed tube at 60 °C for 20 h. The remaining material is washed 3 times with ultrapure water at 35 °C for 5 min and then freeze dried (Brock et al., 2010). The charcoal samples pretreated with the ABA and ORAU-XR procedures are then used for CO<sub>2</sub> extraction, purification and graphitization following the organic carbon process described below.

The total bone organic carbon (TBOC) of collagen from the bones and teeth is also used for AMS <sup>14</sup>C dating. The dried bone and teeth samples were crushed and sequentially treated with the ABA method described above. The crude collagen of these ABA treated samples was gelatinized in a solution with pH of 3 at 75 °C for more than 20 h. After pouring the supernatant, the resultant gelatin solution was then washed with ultrapure water before being freeze-

dried. Then, the bone organic carbon of the collagen in the samples was extracted for the AMS <sup>14</sup>C dating. The details of collagen extraction procedure are discussed by Brock et al. (2010).

All pretreated organic carbon samples (charcoal and collagen from bones and teeth) were weighed and placed in clean 9 mm quartz tubes with CuO (pre-combusted at 500 °C) and a small piece of silver. The quartz tubes were placed under vacuum on line, sealed under 10<sup>-3</sup> mbar, and combusted in a Muffle furnace at 850 °C for 8 h. The yields of CO<sub>2</sub> were quantified using an absolute pressure gauge on a vacuum extraction line, and the CO<sub>2</sub> was used to make graphite.

Burned bones experienced the loss of organic carbon during the burning process. According to the study of Shipman et al. (1984), pale yellow and brown colour of burned animal bones indicated a temperature less than 285 °C, black meant 645 °C (cremated bone), and white or light blue-grey meant 940 °C (cremated bone). In this study, some burned bones have black colour (cremated bone), e.g., PCD-14, but some burned bones have light yellow colour such as PCD-16. Therefore, we use “burned bone” instead of “cremated bone”. A special protocol is used for burned bones to extract original carbonate (total inorganic carbon, TIC) formed in the bones when the animal was alive. The procedure is known as ORAU-CB (Brock et al., 2010). Burned bones were surface cleaned using the ABA method to remove carbonate deposits (allogenic input). The dried bones were crushed, and organic material was removed via several rinses with a 1.5% sodium chlorite solution at pH 3 over 48 h at room temperature. Adsorbed carbonates were then removed via several washes with 1 M acetic acid over 24 h at room temperature (Lanting and Brindley, 1998; Lanting et al., 2001). The treated bone fragments were washed with ultrapure water and then freeze dried. The dried bone samples were then reacted with 100% phosphoric acid in a 2-armed Pyrex<sup>®</sup> reaction vessel under vacuum, and any produced CO<sub>2</sub> was transferred to a glass tube and sealed for graphitization as described below.

The purified CO<sub>2</sub> from charcoal, bone and tooth samples was introduced to the graphitization line under a vacuum of 10<sup>-3</sup> mbar. Using approximately 400 mg of Zn at 450 °C for catalysis, the CO<sub>2</sub> was reduced to graphite in the presence of Fe (Fe:C = 3.5:1) at 550 °C for 6–8 h. The graphite samples were pressed into targets, and the <sup>14</sup>C/<sup>12</sup>C and <sup>13</sup>C/<sup>12</sup>C ratios were measured together with the targets made from oxalic acid standards (OXII, 4900C) and back-grounds (NTUB is a pure limestone sampled from the upper Devonian stratum in Guilin, China and BKG is an anthracite

purchased from US National Institute of Standards and Technology–NIST) in the NTUAMS Lab with a 1.0 MV Tandemron Model 4110 BO-AMS made by High Voltage Engineering Europa B.V. (HVE). Since the 1.0 MV AMS has strong  $2\text{Li}^+$  interference with  $^{14}\text{C}^{2+}$ , we measure  $^{14}\text{C}^{3+}$  to avoid the Lithium interference so that the transmission rate of  $^{14}\text{C}$  beam is reduced from ~50% (for  $^{14}\text{C}^{2+}$  mode) to ~18% (for  $^{14}\text{C}^{3+}$  mode). Consequently, the  $^{14}\text{C}$  counting rate of the AMS is relatively low. The measured  $^{14}\text{C}/^{12}\text{C}$  and  $^{13}\text{C}/^{12}\text{C}$  ratios were used to calculate  $\Delta^{14}\text{C}$  values and conventional  $^{14}\text{C}$  ages with a  $^{14}\text{C}$  half-life of 5568 years after correction for carbon isotopic fractionation using the  $\delta^{13}\text{C}$  values of the samples, which were measured by the AMS (Stuiver and Polach, 1977). The conventional  $^{14}\text{C}$  ages were converted to calibrated calendar ages (a BP = years before 1950 CE) using the CalPal Online Radiocarbon Calibration of  $^{14}\text{C}$  (<http://www.calpal-online.de/>) (Stuiver and Reimer, 1986, 1993). The AMS  $^{14}\text{C}$  counting statistical error for OXII is generally less than 5‰ (>40,000  $^{14}\text{C}$  counts).

### 3. Results

All AMS  $^{14}\text{C}$  dating results are listed in Table 1. Because different samples involved different pretreatment methods (i.e., ABA, ORAU–XR, ABA–collagen and ORAU–CB) and different carbon compounds were used for  $^{14}\text{C}$  dating (e.g., TBOC of collagen in pretreated unburned bones and teeth, and TIC of pretreated burned bones), we evaluate the dating quality below. Owing to the small sample size especially after the pretreatment of ORAU–XR and ORAU–CB as well as the  $^{14}\text{C}^{3+}$  measurement mode of the AMS, the  $^{14}\text{C}$  counts of the samples are not very high (Table 1). Therefore, the age uncertainties of those  $^{14}\text{C}$  dates are relatively large though the uncertainties would not affect the data interpretation significantly.

First, NTUAMS-1946 and NTUAMS-1946-A are from the same charcoal sample but pretreated with the two different methods, ABA and ORAU–XR, respectively. Their ages are consistent within counting error (Table 1), demonstrating that the ages are reliable and that the ABA treatment is sufficient for the charcoal samples. In Layer 4, two charcoal samples, NTUAMS-1947 (11995 ± 255 a BP), which received the ABA pretreatment, and NTUAMS-2455 (11135 ± 270 a BP), which received the ORAU–XR pretreatment, gave consistent age results as their difference exceeds slightly  $1\sigma$  (Table 1). PCD-8 has small sample size, and its age may have been biased towards younger age. And, considering their large age uncertainties, these two ages do not show large difference. This comparison again illustrates that the AMS  $^{14}\text{C}$  ages of charcoal samples are reliable, regardless of whether the ABA pretreatment or the ORAU–XR pretreatment is used. The AMS  $^{14}\text{C}$  ages of charcoal samples from the lower strata (Layers 8 and 9) are significantly older than the ages of charcoal samples from the upper strata (Layers 3–5), in correct stratigraphic order. However, the  $^{14}\text{C}$  age of the charcoal sample (PCD-15) from Layer 9 seems younger than the one (PCD-13) from Layer 8, but the age difference is less than  $\pm 2\sigma$  counting error, so not sufficient to affirm an age stratigraphy reversal. Since both charcoal samples were subjected to the ORAU–XR pretreatment, contamination from groundwater and soil should be removed. Note that the  $^{14}\text{C}$  counts of PCD-15 are very low due to poor target current which turns to give younger (than expected) age for old samples (Table 1). Of course, stratigraphic disturbance of this layer or slightly unexpected contamination during the sample processing in the lab may be also possible reasons for the younger than expected age of PCD-15. Nevertheless, the age of PCD-15 should be older than 31310 ± 865 a BP.

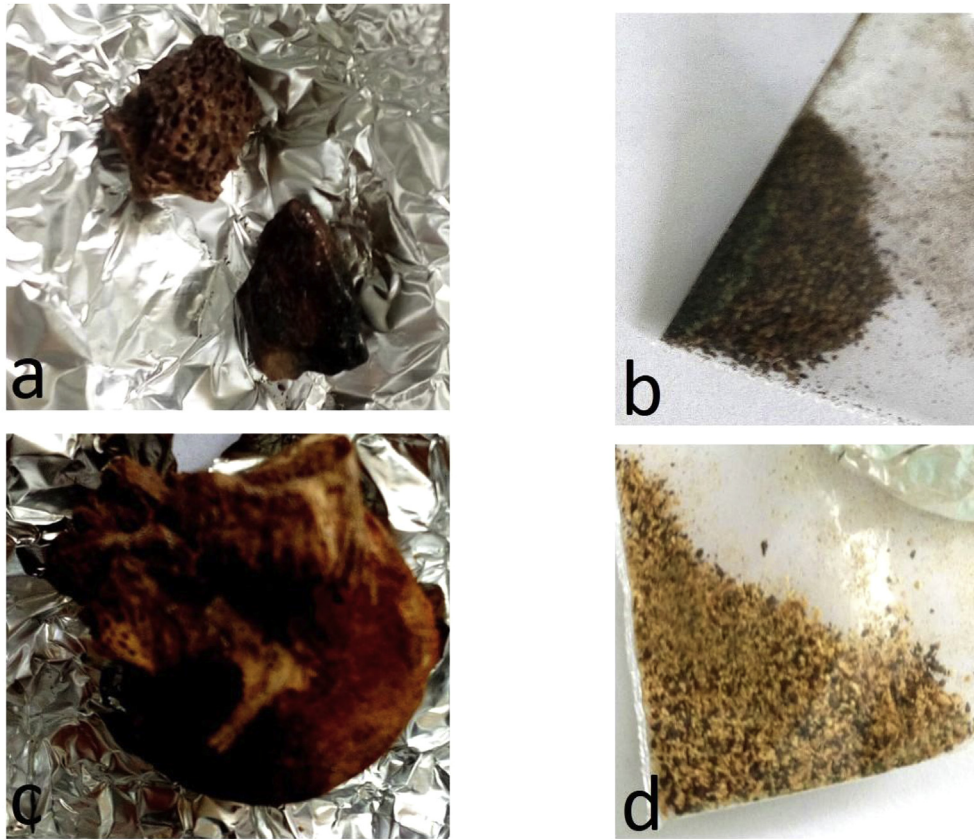
Secondly, although the AMS  $^{14}\text{C}$  ages of the bone samples are generally younger than that of the charcoal samples from the same layer, they are in good stratigraphic order (Table 1). PCD-5 (11005 ± 140 a BP) is a burned bone sample that received the

ORAU–CB pretreatment. This sample is 990 years younger than the charcoal sample of PCD-4 from the same layer. In Layer 5, the bone sample PCD-9 (ABA–collagen pretreatment) has an age of 11210 ± 300 a BP that is 1225 years younger than that of the charcoal sample (PCD-7) from the same layer. These age comparisons indicate that, after the pretreatments, some bone samples may provide reasonable  $^{14}\text{C}$  ages. Those underestimated bone ages are able to provide us a minimal age of each layer. Thus, although no charcoal samples were found in Layers 6 and 7, the AMS  $^{14}\text{C}$  ages of the bone and teeth samples in these layers can be used. Similarly, we retain most of the AMS  $^{14}\text{C}$  ages of the bone samples even though they are somewhat younger than the  $^{14}\text{C}$  ages of the charcoal samples from the same layers.

Radiocarbon dating of bone samples is much complicated than that of charcoal samples (Hedges and van Klinken, 1992; Van Klinken, 1999). Fresh bone tissue contains an organic component (~22%), an inorganic component consisting of bone minerals (~69%), and bone fluid (~9%) associated with the organics and the minerals (Cazalbou et al., 2004). The organic carbon in bones exists in a number of different compounds, including bone collagen, humic acid, fulvic acid, humin and others. Although the quantity of humic acid, fulvic acid and humin in bone samples can be reduced via the ABA–collagen pretreatment, bone collagen itself may be contaminated by foreign organic carbon (Chen, 1990; McCullagh et al., 2010). Bone collagen includes various polypeptide chain organic compounds, such as glycine, proline, hydroxyproline and alanine. After samples are buried, organic acids, including amino acids generated by organisms or plants, can enter the bones. Those amino acids can potentially become integrate into or exchange carbon with bone collagen. Once carbon exchange occurs, it is difficult to remove the contamination via chemical methods (McCullagh et al., 2010). Previous studies have shown that the AMS  $^{14}\text{C}$  ages of bone collagen are liable to be younger than their true ages (Gillespie et al., 1986; Gowlett and Hedges, 1986; Chen, 1990). Some researchers have proposed that the AMS  $^{14}\text{C}$  dating of proline and hydroxyproline might produce more accurate ages (McCullagh et al., 2010). However, the requisite extraction process is very complex and results in the loss of considerable organic carbon. Therefore, for some bone samples contaminated by free amino acids, the ABA–collagen pretreatment may not be able to remove the contamination (Van Klinken, 1999), and the bone collagen may yield a younger  $^{14}\text{C}$  age than the true age. A bone residue sample (PCD-6) from Layer 4 is one such example, and we have excluded this sample from further analysis.

For burned bones, as organic carbon is influenced by the burning (loss of organic carbon and/or contaminated by the carbon from fire material at different ages), the original carbonate in burned bones is considered for AMS  $^{14}\text{C}$  dating (Saliège et al., 1995; Hüls et al., 2010; Van Strydonck et al., 2010; Zazzo et al., 2012; Fiedel et al., 2013; Snoeck et al., 2014; Bonsall et al., 2015). The inorganic carbon of bone samples is commonly accepted to be a mixture of original carbonates formed in the bone during the life of the animal and contaminating carbonates that have accumulated since the bone was buried (Chen, 1990). Hassan et al. (1977) demonstrated that the  $^{14}\text{C}$  ages of inorganic carbon from bone samples were commonly younger than the true ages. The ORAU–CB pretreatment proposed by the Oxford Group aims to remove deposited and absorbed carbonates and extract original carbonates for AMS  $^{14}\text{C}$  dating of burned bone samples (Brock et al., 2010). Theoretically, the  $\text{H}_3\text{PO}_4$  acidification method cannot always remove contaminating carbonate from original carbonate. If the contaminating carbonates are in the surface layer of the bone, the ORAU–CB pretreatment can effectively remove the contamination. However, if the contaminating carbonates enter the inner part of the bone tissue or combine with the original carbonates, the ORAU–





**Fig. 4.** Pictures of burned animal bones from Layers 8 and 9. (a) Burned bone (PCD-14) from Layer 8. (b) Powder of PCD-14. (c) Burned bone (PCD-16) from Layer 9. (d) Powder of PCD-16.

CB pretreatment may not work well. A burned bone sample (PCD-16) from Layer 9 may have this problem. In general, after bone is buried, bone apatite bone apatite [ $\text{Ca}_5(\text{PO}_4)_3(\text{F},\text{OH},\text{Cl})$ ] (Wopenka and Pasteris, 2005) or a more complicated formula given by Cazalbou et al. (2004) and Pasteris et al. (2008)] in the bone becomes crystal hydroxyapatite under the influence of groundwater. The original bone apatite in the bone is replaced by  $\text{CaCO}_3$  or new  $\text{Ca}_3(\text{PO}_4)_2$  that precipitates in the bone tissue. Therefore, the influence of groundwater is a major problem for the preservation of bone samples. Layer 9 is the lowest level in the sampling section. Samples in this layer might have been more affected by rainwater and soil water. Sample PCD-16 is unlike the burned bone samples from the other layers (e.g., PCD-14 in Layer 8) (Fig. 4). PCD-14 has black colour whereas PCD-16 has light yellow colour, which indicates that the burning temperature of PCD-16 is lower than  $300^\circ\text{C}$  (Shipman et al., 1984). Therefore, PCD-14 is considered as a cremated bone, but PCD-16 is not a cremated bone. With lower burning temperature, the bone tissue may be subjected more exchange with ambient carbon after deposition. Hence, we suspect that PCD-16 contains significant contaminated carbonates. In order to examine the preservation of the bone samples, we measured X-ray Diffraction (XRD) on PCD-14 and PCD-16 (Fig. 5). The XRD results show clearly that PCD-14 contains large crystal hydroxyapatite and non-detectable carbonate minerals. In contrast, PCD-16 contains large amount of calcite and very low crystal hydroxyapatite (Fig. 5). The XRD results indicate that the burning temperature PCD-14 should be  $> 600^\circ\text{C}$  (Shipman et al., 1984) without foreign carbonate contamination, whereas PCD-16 is an opposite case. Thus, we have therefore excluded the age of PCD-16 from further interpretation.

In summary, the charcoal samples from the Chuandong site have the most reliable AMS  $^{14}\text{C}$  ages, except PCD-15 from Layer 9, which has a  $^{14}\text{C}$  age that is younger than its true age, perhaps due to the strong influence of rainwater and soil water. The  $^{14}\text{C}$  ages of the bone collagen organic carbon from the ABA-collagen pretreated bones and teeth are generally reasonable, even though they are younger than their true ages. However, the ABA-collagen pretreatment process cannot remove the contamination of collagen by free amino acids. For these contaminated samples, AMS  $^{14}\text{C}$  dating of proline and hydroxyproline may obtain more accurate ages (McCullagh et al., 2010). The ORAU-CB pretreatment (Brock et al., 2010) can reduce the influence of contaminating carbonates in burned bones but may not work when the contamination is in the deep part of the bone. It is suggested that XRD analysis should be performed for burned bone samples. The AMS  $^{14}\text{C}$  ages of the carbonates in the burned bone samples after ORAU-CB pretreatment are generally underestimates of the true ages but may still be useful as reference ages.

#### 4. Discussion

In Table 1, the AMS  $^{14}\text{C}$  ages of both the charcoal and bone samples in our study are older than the ages of the previous study (Li et al., 1987). Based on our results, the age of Layers 3–5 is 11.5–12.5 ka BP, corresponding to the Younger Dryas period (12,900 to 11,700 calendar years ago). Therefore, the chronology of the hominin fossil in Layer 4 is 11.5–12.5 ka BP, not 8000–9000 a BP. This new chronology indicates that the Chuandong humans lived during the late Pleistocene under a relatively cold and dry climate instead of during the early Holocene under warmer and wetter

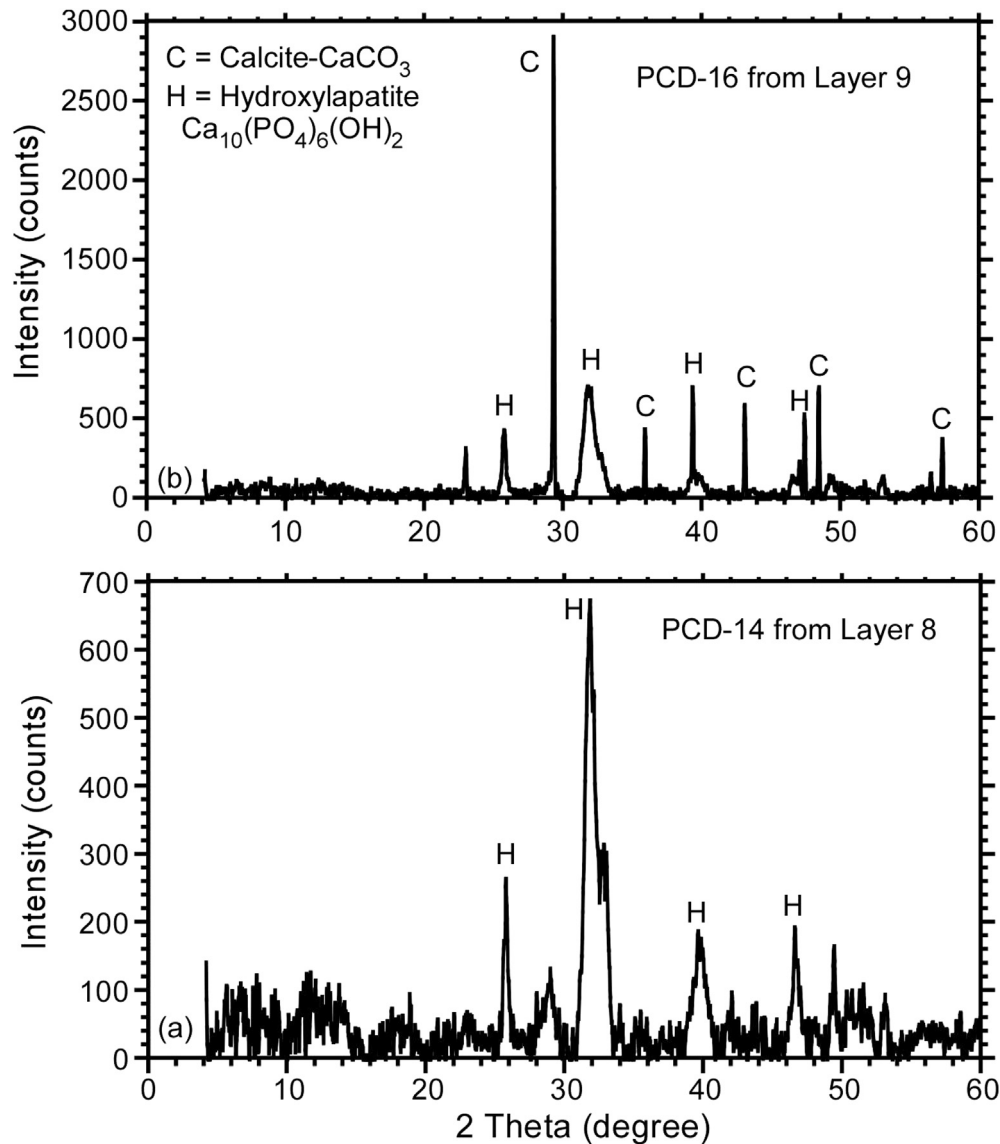


Fig. 5. The measured XRD results of PCD-14 and PCD-16 shown in Fig. 4.

conditions. The AMS  $^{14}\text{C}$  ages of bone and teeth samples from Layers 6–7 indicate that the animals in the Chuandong site lived during 14–24 ka BP, a period that included the Last Glacial Maximum (LGM). In the Puding area, the climatic conditions were cold and dry during the LGM and Younger Dryas (Zhao et al., 2015, 2016). Changes in external environments, such a transition to severe climatic conditions, have played an important role in controlling the evolution of human beings (Wolpoff, 1996; Liu and Ding, 1999). The extremely cold and dry conditions in the northern high latitudes during the LGM might have affected hominin evolution. However, in the Chuandong site, humans and animals were able to survive the cold and dry conditions of the LGM and Younger Dryas, implying that the climate conditions in South China were suitable for hominin evolution (Wang et al., 2001, 2008).

Although no consensus has been reached on many fundamental issues in the model of modern human evolution in east Asia, recent discoveries and re-dating of the sites have shown the great complexity of hominin evolution in late Pleistocene in southern China (Bae et al., 2014; Curnoe et al., 2012; Ji et al., 2016; Kaifu et al., 2015; Liu et al., 2015; 2016; Shen and Michel, 2007). The

Chuandong hominin lived in late-final Pleistocene period which is a critical stage for the formation and differentiation of modern population in East Asia. Human fossils from this period manifest great diversification in cranial as well as postcranial morphology (Cunningham and Wescott, 2002; Shang and Trinkaus, 2010; Cao et al., 2015). For example, the fossils from Longlin and Maludong Cave, which are one of the best studied and accurately dated late Pleistocene hominin fossils so far in south China, have shown mosaic of archaic and modern features in the skull (Curnoe et al., 2012; Ji et al., 2013) and strong resemblances to archaic humans in femur morphology (Curnoe et al., 2015a). Although still controversial, hybridization with archaic hominins is one of the explanations of its unusual morphology (Curnoe et al., 2015b). The contemporaneous and sympatric Chuandong fossils may serve as a useful window to infer details about the phylogenetic relationships between archaic and modern humans in the region.

Hundreds of bone implements and thousands of stone artefacts were discovered together with human fossils at Chuandong (Huang, 1989; Mao and Cao, 2012; Zhang, 1995). The charcoal and bone ages from Layers 8–9 illustrate that this section formed at

approximately 30–34 ka BP or earlier and is therefore much older than the previous  $^{14}\text{C}$  age of 16 ka BP. Our results show that the Chuandong artefacts appeared by at least 34 ka BP. Analysis of these artefacts will shed light on the change in subsistence strategies and human behavior during late Pleistocene, whereas accurate chronology of Chuandong established by this study is the basis for the intensive research in the future.

## 5. Conclusions

Charcoals, burned and unburned bones, and teeth were collected from the hominin site Chuandong Cave for AMS  $^{14}\text{C}$  dating. These samples were subjected to different pretreatments: ABA for charcoals; ABA-collagen for unburned bones and teeth; ORAU-XR for old charcoals; and ORAU-CB for burned bones. After the pretreatments, the organic carbon in the charcoal and collagen in the unburned bones and teeth was extracted by combustion, and the inorganic carbon in the burned bones was extracted via  $\text{H}_3\text{PO}_4$  acidification. The extracted carbon was then dated via the AMS  $^{14}\text{C}$  dating technique. The  $^{14}\text{C}$  ages of the charcoal samples are the most reliable. Although the  $^{14}\text{C}$  ages of the ABA-collagen pretreated bones and teeth are somewhat younger than the true ages, these ages still provide reasonable chronologies. For burned bone samples, XRD analysis will help in examining foreign carbonate contamination. The ORAU-CB pretreatment may not remove contaminating carbonate that entered the inner part of the bone tissue. Based on the results of this study, the hominin site Chuandong Cave can be described as follow: Layers 3–5 formed during 11.5–12.5 ka BP; Layers 6–7 formed during 14–24 ka BP; and Layers 8–9 formed at least during 30–34 ka BP. All of the ages in this chronology are older than the ages of the corresponding layers in previous chronologies. The results of our study demonstrate that the Chuandong humans, whose bones were excavated from the fourth layer, lived during 11.5–12.5 ka BP, i.e., the late Pleistocene, not the early Holocene. This period corresponded to the Younger Dryas, when cold and dry conditions prevailed. The morphological features of this group of hominins (completely modern type) were present the end of the late Palaeolithic period. The Chuandong Culture appeared by at least 34 ka BP and survived the cold and dry LGM. Our results will contribute to the understanding of hominin evolution in South China.

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