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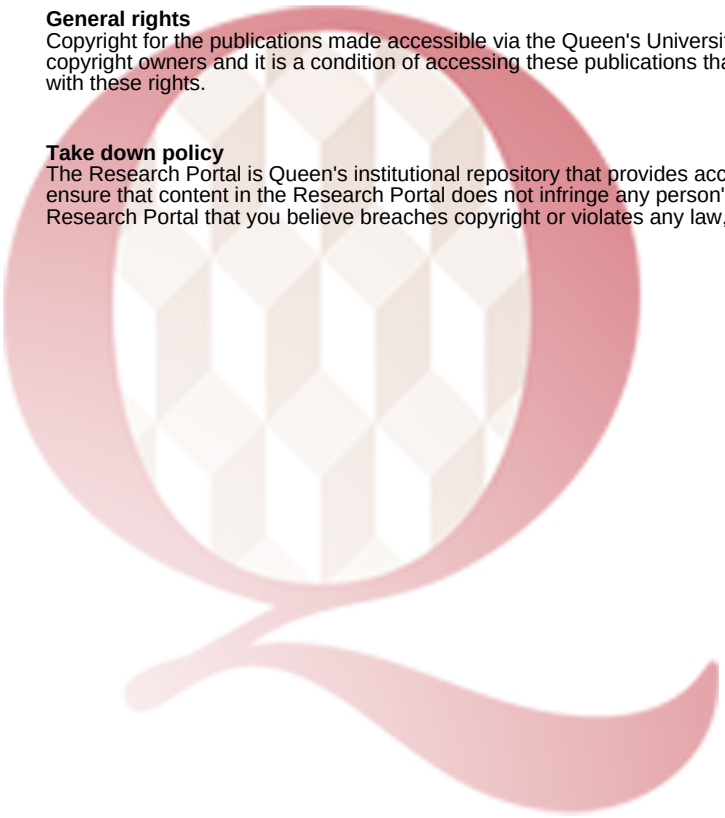

'Old wood' effect in radiocarbon dating of prehistoric cremated bones?

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- Radiocarbon dating of a cremated human bone is compared with the precise dendrochronological age of an associated oak coffin.
- The cremated bone shows an age discrepancy of 73 ± 26 ^{14}C years older than the dendrochronological age.
- The age discrepancy is best accounted for by the so called ‘old wood’ effect from the wood used in the cremation pyre.

'Old wood' effect in radiocarbon dating of prehistoric cremated bones?

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Abstract

Numerous reports of successful radiocarbon dating of cremated bones have emerged during the last decade. The success of radiocarbon dating cremated bones depends on the temperature during burning and the degree of recrystallisation of the inorganic bone matrix. During cremation bones undergo major morphological and mineralogical changes which have raised some interesting questions and discussion on the origin of the carbon source in archaeologically cremated bones. Recent laboratory experiments reveal that the properties of the combustion atmosphere play a significant role regarding the source carbon in cremated bones. Thus radiocarbon dating cremated bones is potentially dating the wood used for the cremation fire. Here we compare a high precision radiocarbon dated human bone with an associated dendrochronological age from an oak coffin. We find that the age discrepancy between the dendrochronological age and the cremated bone of 73 ± 26 ^{14}C yr is best accounted for by the so called 'old wood' effect.

Introduction

Radiocarbon dating of collagen in well-preserved human bone has routinely been carried out for decades, but cremated bone samples were always excluded because cremation destroys the bone collagen. However, within the last decade successful ^{14}C dating of cremated bones has frequently been reported (e.g. De Mulder, et al., 2009, De Mulder, et al., 2007, Lanting, et al., 2001, Olsen, et al., 2011). Furthermore, uniform results of radiocarbon dating of cremated bones have been proven in laboratory intercomparison tests (Naysmith, et al., 2007). The intercomparison test was designed to test the dating protocol, i.e. using the same method laboratories get similar ages on the same material within measurement error. Hence problems related to whether or not ^{14}C dating cremated bone yields an estimate of the true calendar age were not tested. Here we present new information on a previously published cremated bone sample found in an oak coffin which has been dated by dendrochronology (Olsen et al., 2008). Our updated results will be discussed in light of new laboratory studies suggesting that ^{14}C dating of cremated bones reflects the burning atmosphere of the cremation fire (e.g. Hüls et al., 2010, Van Strydonck et al., 2010). We believe that our case study may represent an archaeological example supporting the recent laboratory conclusions.

Radiocarbon dating of bio-apatite is possible because of incorporation of carbonate ions into the inorganic bone matrix in living organisms. The carbonate ions originate from the energy production in cells and are substituted with phosphate ions in the bone matrix into the bio-apatite mineral-like bone structure (Krueger, 1991, Lee-Thorp and van der Merwe, 1991, Munro, et al., 2007, Newesely, 1988, Pate and Hutton, 1988, Posner, 1969, Saliège, et al., 1995, Sandford, 1993, Wright and Schwarcz, 1996).

Radiocarbon dating of the bio-apatite fraction has in general been abandoned decades ago due to incorrect ^{14}C results caused by contamination effects (Berger, et al., 1964, Hassan, et al., 1977, Stafford, et al., 1987). In fossil bones, exchange reactions with the bicarbonate ions dissolved in soil waters lead to ^{14}C contamination (Hassan, et al., 1977, Hedges and Millard, 1995, Surovell, 2000, Tamers and Pearson, 1965). Apparently, the exchange reaction with the dissolved bicarbonate ions does not occur for cremated bones and hence the bio-apatite fraction of cremated bone yields reliable ^{14}C results (Lanting, et al., 2001, Olsen, et al., 2008). This is because heating of

bones results in numerous microscopic and macroscopic changes which altogether yield a more robust and inert bio-apatite structure as a consequence, i.e. heating results in re-crystallization of the bio-apatite bone matrix into a more robust structure (Newesely, 1988, Stiner, et al., 1995, van Strydonck, et al., 2005). Crucial to radiocarbon dating of calcined or burned bones is assurance about the degree of bio-apatite re-crystallisation. As shown characterisation and subsequent careful selection of well cremated bones is essential for reliable ^{14}C age results (Olsen, et al., 2008, Van Strydonck, et al., 2009). To this end the cremated bones of humans should be characterised by visual inspection, IR spectroscopy (crystallinity index (CI) and the carbonate to phosphate ratio (C/P)), $\delta^{13}C$ of bio-apatite and the carbon weight percentage (Olsen, et al., 2008, Thompson, et al., 2009).

For radiocarbon dating knowledge of the carbon origin is in general of utmost importance because the carbon source defines the event being dated. The loss of structural carbon, the major morphological and mineralogical changes occurring during the cremation process has raised some interesting questions and discussion regarding the origin of the carbon source in archaeologically cremated bones (e.g. Hüls, et al., 2010, van Strydonck, et al., 2010, Zazzo, et al., 2009). Put simply, it all boils down to one plain question: What are you dating when radiocarbon dating cremated bones? It is remarkable that the $\delta^{13}C$ of charred and unburned bone apatite change from c. -15‰ to $\delta^{13}C$ values around -23‰ for cremated bones (Lanting, et al., 2001, Olsen, et al., 2008, van Strydonck, et al., 2005). This has lead to considerations about kinetic fractionation to explain the very depleted $\delta^{13}C$ values of cremated bones as favoured by Zazzo et al. (2009). On the other hand, carbon exchange processes during the fire may potentially explain the remarkable carbon isotope signature of cremated bones. Carbon from atmospheric CO_2, from bone organic matter (collagen) or from CO_2 evolving during combustion may all contribute even in tandem with kinetic isotope fractionation. Recent laboratory experiments by Hüls et al. (2010) and Van Strydonck et al. (2010) has demonstrated that the properties of the burning atmosphere plays a significant role as a carbon source in cremated bones. They found that the exchange processes between produced CO_2 during combustion and bio-apatite control the stable carbon isotope ($\delta^{13}C$) signature and radiocarbon age of cremated bones. Hüls et al. (2010) further argue that kinetic isotope fractionation is needed to fully explain

their results, but this process is much less significant than exchange reactions with the burning atmosphere. Thus radiocarbon dating cremated bones is potentially equivalent to dating the wood used for the cremation fire. Despite similar ^{14}C ages has been demonstrated of paired samples of associated context material (mostly pitch and charcoal) and cremated bone samples (Lanting, et al., 2001, Olsen, et al., 2008, van Strydonck, et al., 2005), this opens the possibility of the 'old wood' effect when radiocarbon dating cremated bones.

Method

Sample preparation follows procedures described in Olsen et al. 2008: Cremated bone samples (2 grams) are soaked in a 1.5% sodium hypochlorite solution to dissolve remaining organic material (48h, 20°C). The sample is then washed and submerged in 1M acetic acid to remove post-depositional carbonates as well as less crystalline, soluble fractions of bio-apatite (24h, 20°C). Next the sample is washed and dried (12 h, 80°C) with a bio-apatite yield of approximately 96%. The pre-treated sample is crushed and 1.5 g is treated with 100% de-hydrated phosphoric acid (8h, 25°C) to liberate CO_2 from which sulphur impurities are removed prior to conversion to graphite for AMS targets (Lanting, et al., 2001). Part of the resulting CO_2 gas was used for $\delta^{13}C$ analysis on a GV Instruments Isoprime stable isotope mass spectrometer to a precision of 0.15‰, while the rest was converted to graphite for AMS ^{14}C measurements via reduction with H_2 using cobalt as a catalyst (Vogel, et al., 1984). The AMS ^{14}C measurements were carried out using the EN tandem accelerator at Aarhus University (Denmark). The dating results are reported as conventional ^{14}C dates in ^{14}C yr BP based on the measured $^{14}C/^{13}C$ ratio corrected for the natural isotopic fractionation by normalising the result to the standard $\delta^{13}C$ value of –25‰ PDB (Andersen, et al., 1989).

The samples have been visually inspected for surface and interior colour and burn cracks and IR-spectroscopy was performed on powdered pretreated sample material, i.e. bio-apatite. The sample material was mixed with KBr and hydraulically pressed into pellets prior to measurement of infrared spectra with a Perkin Elmer FTIR spectrometer (PARAGON 1000). The spectrum of KBr was automatically subtracted by an online computer. IR spectra on the bio-apatite bone fraction provide information on

the crystallinity index (CI) and carbon to phosphor ratio (C/P) (Garvie-Lok, et al., 2004, Olsen, et al., 2008).

Results and discussion

A well-preserved coffin from Egtved, Denmark, consisting of a hollowed-out oak trunk was excavated in 1921 by the Danish National Museum. It contained the famous Egtved girl, dressed in full costume covered with a woollen blanket and wrapped in a cow skin (Thomsen, 1929, Alexandersen et al., 1983, Aner and Kersten, 1990, No.4357A). The grave goods consisted of a belt-plate, a small bronze earring, two arm rings, an awl in a wooden handle, and a horn comb. The archaeological date is the Bronze Age, period II (1500 – 1300 BC, Randsborg, 2006). At her feet there was a bucket of bark, which contained residues from honey sweetened beer, and at her left leg a bundle of cloth with the cremated bones of a child. There was another bucket of bark at her head also with a few cremated bones, the mentioned awl and remains of a hair net (Figure 1). Consistent with the archaeological finds, the coffin has been dated to 1370 BC by dendrochronology (Christensen, 2006). The investigation carried out by Kjeld Christensen showed that the lower part as well as the lid was well preserved. 110 tree rings were preserved and 9 of these were sapwood rings. Moreover, the preserved bark ring consisted of early wood as well as a very narrow zone of latewood indicating that the tree presumably was felled in July or August prior to the end of the growth season (Christensen 2006). All Danish dendrochronological dates of oak coffins resulted in a master curve comprising 419 years, and this curve was anchored to a German reference chronology (Christensen 2006).

The human remains of the young (16-18 years old) woman in the coffin were rather poor due to the humid and acid peat bog environmental conditions from which she was retrieved. Only the woman's hair, brain, teeth, nails, and parts of her skin were preserved, but no bones at all (Thomsen 1929, Alexandersen et al., 1981, Aner and Kersten, 1990, No.4357A). In contrast, the cremated bones found at the young woman’s head and left leg appeared well preserved (Figure 1, Thomsen, 1929, Alexandersen et al., 1983, Hvass, 2000). The cremated bones are most likely from the same individual, as fragments from the two sets of bones proved to fit precisely and represent a 5-6 year old child. (Alexandersen et al., 1983).

Because of the age difference between the two individuals which excludes a mother-child relationship, it has without any evidence been suggested that the child was a sacrifice (Thomsen 1929, Alexandersen et al. 1983, Jensen 2002). It appears that the cremated bones correspond to regular cremated bone samples, i.e. colour, structure, fragmentation and form (Alexandersen et al.,1983, Olsen et al. 2008). One could imagine, in case of ritual deposition of the cremated bones (e.g. ancestral bones) that a number of years elapsed from cremation to deposition in the coffin. There are, however, remains of the funeral pyre among the cremated bones, i.e. bone dust, charcoal, sand, and ashes (Alexandersen et al., 1983). According to McKinley (2006), cremated bones may be curated and transported, but it is unlikely that pyre debris would that too. Following this argument, the presence of pyre debris suggests that the bones were deposited in the coffin shortly after the cremation.

A fragment of the cremated jaw was radiocarbon dated and published by Olsen et al. (2008) yielding an age of 3128 ±28 ^{14}C yrs BP. This result was compared with the dendrochronological age 1370 BC by converting the calendar age into a ^{14}C age by applying the radiocarbon calibration curve (IntCal04, Reimer, et al., 2004). The resulting age difference was calculated to 74 ±32 ^{14}C yr (see Olsen, et al., 2008), the bone being the older of the two. Hence the two samples almost agree within 2σ (standard deviations). In order to test this age discrepancy another fragment of the jaw was radiocarbon dated resulting in an age of 3126 ±29 ^{14}C yrs BP. Combining this new date with the previous date yields a combined ^{14}C date of 3127 ±20 ^{14}C yrs BP (Table 1). The dendrochronological date 1370 BC is converted into a ^{14}C age of 3054 ±16 ^{14}C yrs BP via the radiocarbon calibration curve (IntCal09, Reimer, et al., 2009). Testing the converted dendrochronological date against the combined cremated bone ^{14}C date results in an age difference of 73 ±26 ^{14}C yr (Figure 2). This result deviate more than 2.8σ from the expected 0 year difference or in other words there is only a 0.7% chance that the results represent the same age. Hence, beyond doubt the two samples are incompatible. How can this significant age difference be explained?

First of all, to ensure that the age deviation is not due to a low burning temperature and thus possible diagenetic alterations it is necessary to evaluate the quality of the cremated bone sample. The previous published CI of AAR-8789 indicated the possibility of low temperature burning (CI=2.9) whereas all other parameters such as

187 $\delta^{13}C$, C/P and C wt% suggested high temperature burning and re-crystallization of the
188 bone matrix (Table 1, Olsen et al., 2008). However, a re-evaluation of the IR spectra of
189 AAR-8789 yields a CI of 5.3 (Figure 3). Thus the age discrepancy is not likely to be due
190 to diagenetic effects, i.e. all parameters points towards high temperature burning.
191 One crucial difference between the controlled laboratory experiments conducted
192 by Hüls et al. (2010) and van Strydonck et al.(2010) is that the laboratory combustions
193 occurred in closed furnaces which likely resulted in larger CO_2 concentration than may
194 be expected for cremation in open fires as carried out by prehistoric people. It
195 therefore remains an open question whether their results can be directly transferred
196 to prehistoric cremated bones. As argued by Zazzo et al. (2009) three potential carbon
197 sources are available for exchange reactions with bio-apatite bone structure during
198 cremation 1) carbon from bone organic matter (collagen), 2) atmospheric CO_2 and 3)
199 CO_2 evolving during combustion (flesh, bone and wood).
200 The age discrepancy lead Olsen et al. (2008) to speculate if possible marine or
201 freshwater diets might influence the ^{14}C age of cremated bones as is commonly known
202 from radiocarbon dating on the collagen fraction of human bones (e.g. Arneborg, et
203 al., 1999, Cook, et al., 2001, DeNiro and Epstein, 1978, Fischer, et al., 2007, Olsen, et
204 al., 2010, Richards and Hedges, 1999). Using a marine reservoir age of 400 years the
205 age deviation amounts to a 18% marine diet. This is from a prehistoric diet perspective
206 not unreasonable. This may indicate that the CO_2 originating from the burning of flesh
207 and bone collagen may exchange with structural bone carbonate during the
208 combustion. However, unless kinetic fractionation effects significantly alter the stable
209 carbon isotope signature of the bio-apatite the uniform $\delta^{13}C$ values of cremated bones
210 speaks against this possibility. Most prehistoric cremated bones show- remarkable
211 uniform $\delta^{13}C$ values, e.g. as the -24 ± 3‰ (n=39) reported by Lanting et al. (2001) and
212 -23 ± 2‰ (n=33) (Olsen, et al., 2008, Olsen, et al., 2011). The laboratory results by Hüls
213 et al. (2010) show that kinetic fractionation only partly account for observed changes
214 in ^{14}C content and $\delta^{13}C$ values. Hence believing kinetic fractionation to play an
215 insignificant role and assuming that the carbon exchange between flesh and structural
216 carbonate is the major carbon source in the cremated bio-apatite, then the age
217 discrepancy reported here may derive from a predominantly terrestrial diet (c. -21‰)
218 combined with a minor fraction of freshwater derived food (typically around c. -21‰

or lower). However, again the uniformity of the $\delta^{13}C$ values of prehistoric cremated bones speaks against this possibility because numerous tests on paired samples of cremated bones and associated context materials has resulted in insignificant ^{14}C age differences, all with $\delta^{13}C$ values similar to the Egtved sample (Lanting, et al., 2001, Olsen, et al., 2008, van Strydonck, et al., 2005). Exchange with flesh carbon (c. 5‰ lower than collagen) during cremation is therefore not a likely dominant carbon source for prehistoric structural carbonate in cremated bones. With prehistoric carbon dioxide $\delta^{13}C$ values around -6.4‰ (Elsig et al., 2009) also exchange with atmospheric CO_2 seems unlikely (unless kinetic fractionation processes dominate).

The laboratory experiments clearly demonstrate that exchange of carbon between bone apatite carbonate and CO_2 in the combustion gases depend on both temperature and CO_2 concentrations. Hence CO_2 derived from woods from the cremation fires is likely substituted into the bone bio-apatite fraction explaining the remarkable similarity of $\delta^{13}C$ values of cremated bones (Lanting, et al., 2001, Olsen, et al., 2008, Olsen, et al.,2011). The old wood effect therefore provides a more likely explanation for the age discrepancy between the cremated bone sample (AAR-8789, -13967, Table 1) and the associated dendrochronologically dated oak coffin. However, it should be pointed out that in the case of a normal ritual cremation, the difference in ^{14}C content of the cremated body and the fuel will in most cases be minimal. Hence a possible carbon exchange is probably difficult to recognize as demonstrated by the numerous tests on paired samples of cremated bones and associated context materials (Lanting, et al., 2001, Olsen, et al., 2008, van Strydonck, et al., 2005).

Conclusion

The bones of a cremated 5 – 6 year old child found in an oak coffin have been radiocarbon dated to 3127 ± 20 ^{14}C yrs BP. The oak coffin is dendrochronologically dated to 1370 BC. From the dendrochronlogical date converted into a ^{14}C age using the radiocarbon calibration curve (IntCal09, Reimer, et al., 2009) the age difference between the two samples is calculated to 73 ± 26 ^{14}C yr. The cremated bone is thus significantly older than the coffin. Recently laboratory experiments revealed that the exchange processes between the CO_2 produced during combustion and the bio-apatite control the stable carbon isotope ($\delta^{13}C$) signature and radiocarbon age of cremated

bones (Hüls, et al., 2010, van Strydonck, et al., 2010). However, one crucial difference between the controlled laboratory experiments is that the laboratory combustions occurred in closed furnaces which likely resulted in larger CO_2 concentration than may be expected for cremation in open fires as carried out by prehistoric people. In the case of the cremated bones sample presented here we find that the age discrepancy is best described by the 'old wood' effect. Hence radiocarbon dating of cremated bones may potentially result in too high radiocarbon ages, similar to the effects seen when dating charcoal. Nevertheless, the difference between the ^{14}C content of the cremated bone and the wooden fuel is probably minimal in most cases. The possible effect of using old wood in the cremation fires is probably limited and not easily recognized as also demonstrated by the numerous tests on paired samples of cremated bones and associated context materials (Lanting, et al., 2001, Olsen, et al., 2008, van Strydonck, et al., 2005).

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Figure 1

To the Left a drawing of the Egtved coffin is shown (Aner & Kersten 1990, 40, Abb. 19). The placements of cremated bones are marked by arrows. Shown to the right are the Egtved cremated bone samples.

Figure 2

IntCal09 radiocarbon calibration curve with $\pm 1\sigma$ uncertainty lines (Reimer, et al., 2009) and the calibrated age probability density function of the combined ^{14}C date of AAR-8789 and AAR-13976 determined by OxCal 4.10 (Ramsey, 2009). Using the radiocarbon calibration curve the dendrochronological coffin date 1370 BC is converted to the corresponding ^{14}C age of 3054 ± 16 BP.

Figure 3

IR absorption spectrum of AAR-8987. IR spectra of the bio-apatite bone fraction are represented by vibration bands of mainly CO_3 and PO_4 giving absorption peaks at 710, 874 and 1415 cm-^{1} and 565, 603 and 1035 cm^{-1} of CO_3 and PO_4 respectively (Garvie-Lok et al., 2004). The crystallinity is a function of the extent of splitting of the two absorption bands at 603 and 565 cm^{-1}. Arrows shows the splitting in the IR spectrum indicating high crystallinity.

Table 1: ^{14}C dating of the Egtved cremated bones

Lab. No.	Material	Prep. Yield	C wt%	C/P	CI	$\delta^{13}C$ ‰ VPDB	^{14}C age BP	Colour		Visible burn cracks
								surface	interior	
AAR-8789 (Olsen et al. 2008)	Bone	98.6%	0.09	0.09	5.3	-23.05	3128±28	Yellow	White	No
AAR-13976 (this study)	Bone	96.9%	0.12	n/a	n/a	-23.69	3126±29	Yellow	White	No
Combined (AAR-8789, -13976)						-23.37	3127±20	0.0≤3.8