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Can calcined bones be used to date Final Palaeolithic and Mesolithic open-air sites? A case-study from the Scheldt basin (NW Belgium)

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ABSTRACT

This paper presents the results of an inter-comparative study in view of assessing the reliability of radiocarbon dates obtained on calcined bones from open-air Palaeolithic and Mesolithic sites. The results demonstrate that the success rate is largely dependent on site-taphonomy, in particular the speed of covering of the site. Sites quickly covered by aeolian, alluvial or marine sediments yield on average good dating results. At worst they can be affected by an wood-age offset, generally <100 years, caused by the uptake of carbon from the firewood. Sites which are uncovered or have been covered rather late suffer from contamination problems resulting in radiocarbon dates much younger than the reference dates. For these sites, which unfortunately represent the vast majority of open-air Palaeolithic and Mesolithic sites, calcined bones are not a valuable dating material for developing robust, decadal-to-centennial chronologies.

1. Introduction

Since the pioneering study by Lanting and Brindley (1998), calcined bones (CB) are increasingly used as sample material for radiocarbon dating archaeological contexts. Initially considered a fully reliable dating material, at least for contexts <5000 ^{14}C BP, several intercomparison and experimental studies have revealed frequent inconsistencies, which warns us against too much optimism. Besides contamination with secondary carbonates (Van Strydonck et al., 2009), one of the main causes is carbon exchange from the firewood during the burning process, which often leads to a wood-age offset or old-wood effect (Hüls et al., 2010; Rose et al., 2020; Snoeck et al., 2014; Van Strydonck, 2016; Van Strydonck et al., 2010; Zazzo et al., 2012). However, so far comparative research using paired samples has almost exclusively focused on human cremations from the Late Neolithic onwards (Annaert et al., 2020; De Mulder et al., 2007; 2009; Lanting and Brindley, 1998; Olsen et al., 2008; 2013; Snoeck et al., 2018). Much less attention has been paid to prehistoric contexts, yielding calcined animal bones from domestic hearths. One exception is the extensive study by Zazzo et al. (2013) focusing on Late Palaeolithic Aurignacian and Gravettian layers from the French cave site of Abri Pataud. In this study 24 dates obtained on CB from five different levels are compared with collagen dates from unburnt bones. The results seem to indicate fairly good correspondence at least for those samples not pretreated with sulfix. Less conclusive results, however, were obtained in a series of smaller intercomparison studies focusing on Final Palaeolithic (Lauwerier and Deeben, 2011; van der Plicht, 2012) and Mesolithic open-air sites in NW Europe (Crombé et al., 2013). In these studies dates from CB are compared to dates on "associated" charred plant remains, mostly charcoal. None of these studies, however, yielded agreements for all dated CB samples; besides some positive correlations, lots of dates turned out to be either younger or older than the associated plant dates. Despite these inconsistent results, CB is increasingly being used as dating material for Final Palaeolithic (Deeben et al., 2000) and Mesolithic sites (Cooper and Jarvis, 2017; Souffi et al., 2018; Niekus et al., 2016), in particular those lacking other reliable dating materials. Although in most cases the obtained dates broadly fit with the expected time-range, deduced from tool-typology, the lack of cross-reference does not allow to fully assess the validity of CB dates.

This paper presents the results of an extensive intercomparison study conducted on four open-air prehistoric sites situated in NW Belgium. Except for the Final Palaeolithic site of Verrebroek-Dok 2, the studied sites (Verrebroek-Dok 1, Doel sector B, Doel sector M) belong to different stages of the Mesolithic. These sites were selected because they are all securely dated by means of mostly single-entity radiocarbon dates on short-lived charred plant remains with no or neglectable in-built or

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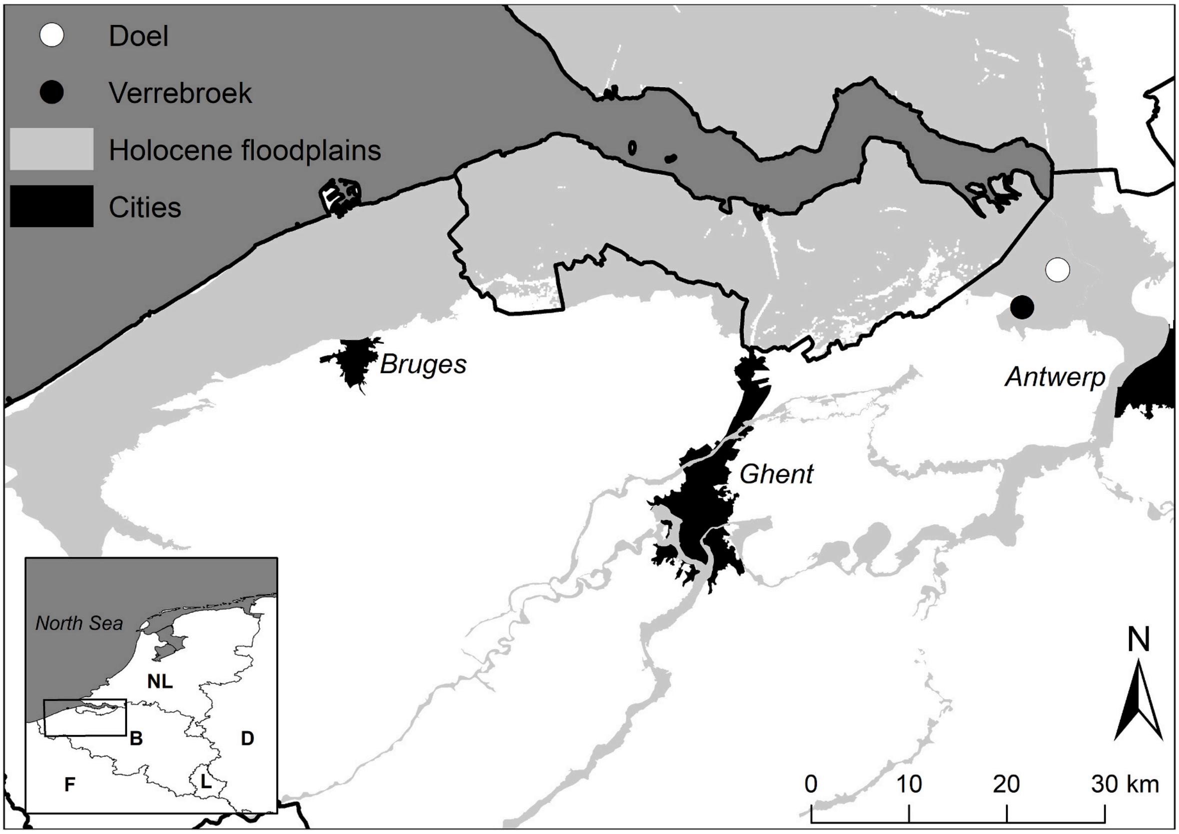

Fig. 1. Map of the Scheldt basin with indication of the studied sites of Verrebroek and Doel.

intrinsic age, providing us reliable reference dates. Earlier attempts in dating CB from these sites, conducted 10–15 years ago shortly after the pioneering study of Lanting and Brindley (1998), failed or were inconclusive. However, recent progress in dating CB samples stimulated us to organize a new inter-comparative study to test the reliability of CB dates.

2. Material and methods

2.1. Site selection

All sampled sites are situated in the lower-Scheldt valley, and are well-preserved on a stratigraphical and spatial level due to covering with aeolian sands (Verrebroek-Dok 2), peat and/or (peri-)marine sediments (Verrebroek-Dok 1 and Doel-sites) (Fig. 1). Organic preservation on the other hand is bad as only carbonized remains, consisting mainly of charred plant (charcoal, seeds, nut shells, etc.) and animal bone fragments (mammals, fish, reptiles, etc.), are present.

2.1.1. Verrebroek-Dok 2

Verrebroek-Dok 2 is a very small Final Palaeolithic site (542 lithic artefacts), which was situated on top of a humiferous soil of 0.10/0.15m thickness, representing the base of a Lateglacial shallow freshwater pond. This soil was covered with ca. 1m of redistributed Lateglacial aeolian sands followed by Holocene deposits (Crombé et al., 1999). The lithics were found stratigraphically associated with a charcoal patch, mainly consisting of charcoal from poplar (*Populus* sp.) (Bastiaens et al., 2005). The humiferous soil was dated by means of pollen (Deforce et al., 2005) and radiocarbon dates (Van Strydonck, 2005) to the Older Dryas and/or Early Allerød, indicating that the lithic industry most likely can be attributed to the *Federmesser* Culture. This is further confirmed by a date obtained on a bark fragment from the charcoal patch, which yielded an age of 11,900 ± 90 ^{14}C BP (Van Strydonck and Crombé, 2005). A small cluster of 81 CB, among which two fragments of wild boar, was found stratigraphically associated with the lithic and charcoal finds (Van Neer et al., 2005).

2.1.2. Verrebroek-Dok 1

A few hundred meters further east of Verrebroek-Dok 2 on the same (cover)sand ridge, an extensive Mesolithic site was excavated yielding over fifty artefact clusters (Crombé et al., 2003, 2006) (Fig. 2). Contrary to the former site, Verrebroek-Dok 1 is situated in the podzolised top of the Pleistocene coversands, sealed by up to 0.75m of peat. The start of the peat growth was dated by pollen (Deforce et al., 2005) and a radiocarbon date (4690 ± 30 ^{14}C BP; Van Strydonck, 2005) to the beginning of the Sub-boreal. A series of 54 radiocarbon dates on charred hazelnut shells, retrieved from latent surface-hearths, situate the main occupation of the site in the Early Mesolithic, between ca. 8740 and 7560 cal BC (95% probability range). Three younger hazelnut dates indicate that the site was also visited during the Late Mesolithic, however on an ephemeral basis (Van Strydonk and Crombé, 2005). Spatially associated with the hazelnut shells, numerous CB fragments were collected from the latent surface-hearths (Sergant et al., 2006a) (Fig. 3). In 2005 a first attempt to date one of these CB turned out negative, yielding an age more than a millennium younger compared to the associated hazelnut shell (Van Strydonck and Crombé, 2005). Hence at that moment it was decided not to continue the dating of CB.

2.1.3. Doel-Deurganckdok: sector B

During the construction of a dock, called the "Deurganckdok" at Doel, two sealed coversand ridges yielded evidence of prehistoric occupations (Sergant et al., 2006b). The site situated in sector B was occupied during the Final Palaeolithic (*Federmesser* Culture) and Late Mesolithic/Early Neolithic (Swifterbant Culture) (Crombé et al., 2000). In this paper only the Swifterbant Culture site will be discussed (Crombé et al., 2002), since no CB were collected at the Final Palaeolithic site.

The Swifterbant site yielded several latent surface-hearths and

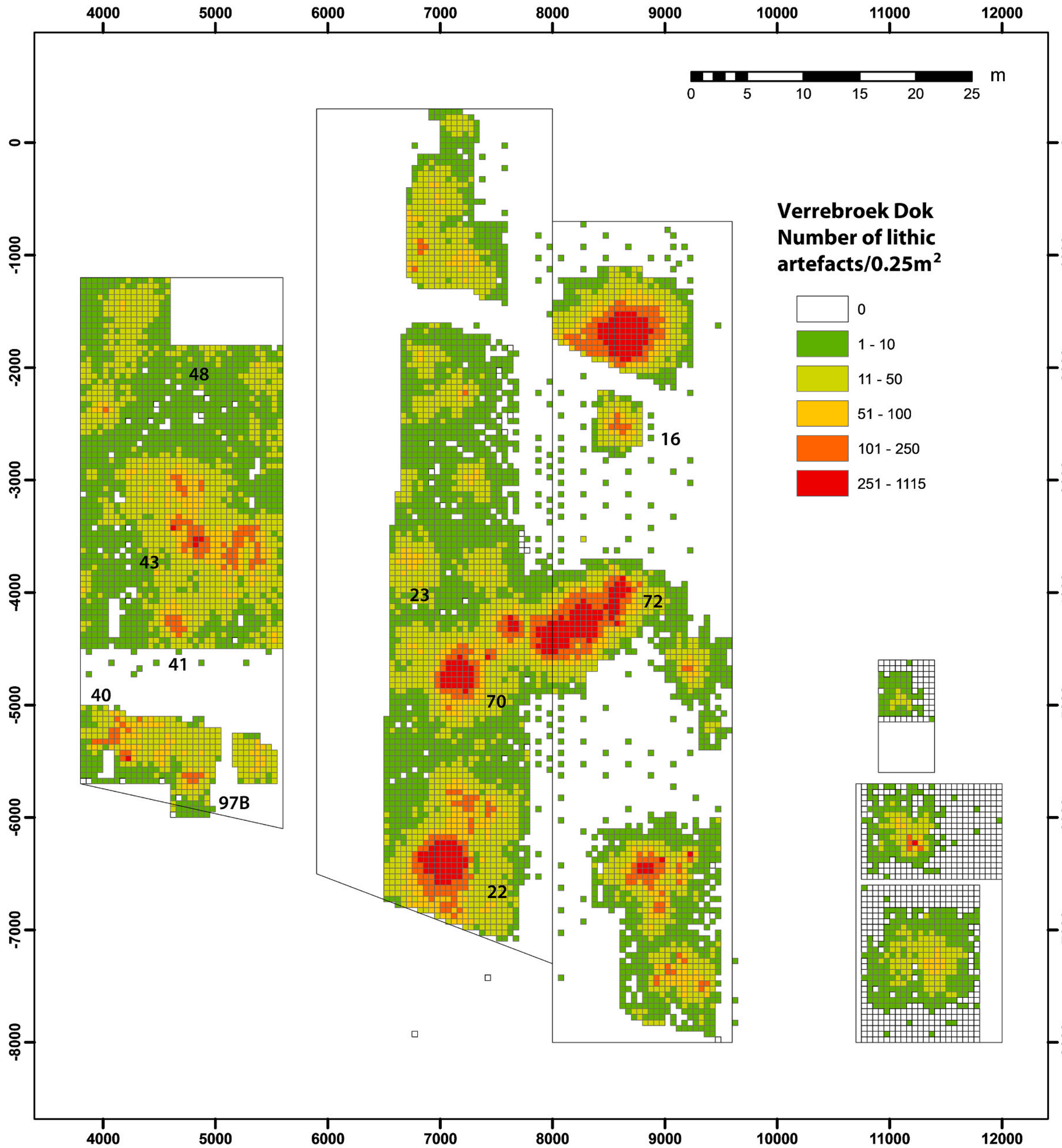

Fig. 2. Density map of the lithic artefacts excavated at the Early Mesolithic site of Verrebroek-Dok 1. The numbers refer to the artefact loci sampled for CB dating in this study.

hearth-dumps, containing charred plant remains (hazelnut shells, seeds, charcoal, …) and numerous calcined animal bones, including both mammals (wild boar, red deer, ..) and freshwater fish, mainly cyprinids (Van Neer et al., 2005).

The occupation was radiocarbon dated on charred plant remains to the second half of the 5th millennium cal BC, with an emphasis during the third quarter (Van Strydonck and Crombé, 2005). Shortly after this occupation, the sand ridge became inaccessible for occupation due to peat growth. The latter was radiocarbon dated (5050 ± 55 ^{14}C BP) to the Atlantic-Subboreal transition (Van Strydonck, 2005). In addition 9 CB, including six from mammals and two from freshwater fish, were also dated using the standard HCL pretreatment, however with variable results (Boudin et al., 2009; Crombé et al., 2013).

2.1.4. Doel-Deurganckdok: sector M

The second sand ridge in the "Deurganckdok" situated in sector M yielded remains of occupations from the Early Mesolithic and the Late Mesolithic/Early Neolithic Swifterbant Culture phase (Sergant et al., 2006b). The latter were found concentrated in a ca. 20m wide strip on the top of the sand ridge, indicating that the slopes were already too wet for occupation by that time. A Bayesian modelling of the radiocarbon dates from the peat and intercalated tidal sediments points to a covering of the sand ridge ultimately between 4090 and 3770 cal BC (Verhegge et al., 2014), so shortly after the Swifterbant occupation. The latter was dated on short-lived charred plant remains in the second half of the 5th millennium cal BC, however, with an emphasis during the last quarter (Boudin et al., 2009; Crombé et al., 2013). Attempts to date six CB samples using an acetic acid pre-treatment yielded inconsistent results between the mammal and fish bones, the former being much older than the latter (Crombé et al., 2013).

2.2. Sample selection

On all sites CB were selected from surface-hearths (Figs. 2 and 3)

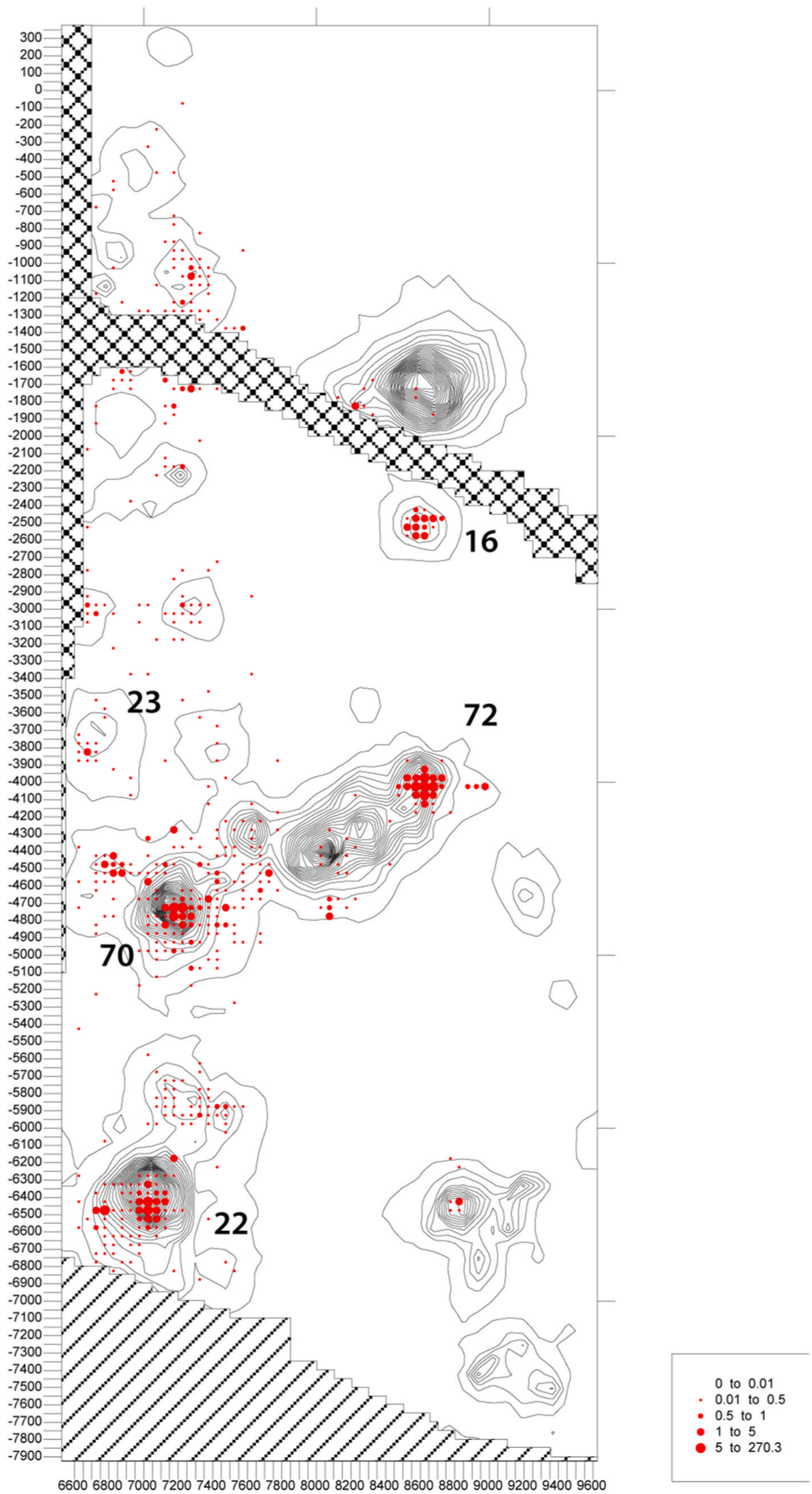

Fig. 3. Density map of the CB (red dots; expressed in gr.) against the background of the artefact density (contour lines) at the Early Mesolithic site of Verrebroek-Dok 1. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

Table 1
Overview of the dated CB samples; dates obtained earlier (cf. 2.1) have been excluded.

	Method 1 (bone fragment)	Method 2 (bone powder)	Method 3 (bone powder)	Total
	AA	HCL + AA	AA	
Verrebroek-Dok 2	1		1	2
Verrebroek-Dok 1	7	3	14	24
Doel-sector B		8	6	14
Doel-sector M		9	12	21
TOTAL	8	20	33	61

which were already dated earlier on charred plant remains, the latter providing us reliable reference dates (see 2.1). As much as possible, the same excavation squares as the charred hazelnut shells, consisting of 0.25 m^2 units, were chosen. In case it was not possible to select samples from the same square, e.g. due to insufficient bone material, the excavation square closest to the original square was chosen, albeit always within the limits of the same surface-hearth. By doing this we tried to reduce the risk of contamination and mixing as much as possible. As we are dealing with unstratified open-air sites which have been re-occupied several times, except for Verrebroek-Dok 2, admixture of settlement waste from different occupation events cannot be fully excluded (cf. principle of spatial and cumulative palimpsests according to Bailey, 2007). This particularly holds for the site of Doel M, for which two surface-hearths (n°1 and 3; Table 5) yielded incompatible reference dates. This is probably related to the very restricted occupation surface at this site in contrast to the other studied sites, situated on larger dunes (cf. 2.1).

Only bone fragments presenting a white color both at the surface and in the interior were selected for radiocarbon dating. Earlier research using Fourier-transform infrared spectroscopy (FTIR) and/or x-ray diffractometry (XRD) (Van Strydonk et al., 2005; Olsen et al., 2008; Hüls et al., 2010; Zazzo et al., 2013; Agerskov Rose et al., 2019; Minami et al., 2019) has clearly demonstrated that completely white bones have been exposed to >600 °C and can thus be termed calcined bones. In contrast to charred bones that turned grey or black, indicating exposure to lower temperatures and thus lower bone crystallinity, white bones are the best candidates for yielding reliable radiocarbon dates. Unfortunately FTIR and XRD could not be applied on the dated CB samples due their too small size.

2.3. Sample preparation

Based on the experience of our former attempts to date CB (cf. 2.1) and recent experimental research (cf. Introduction) we tested different pre-treatment procedures (Table 1):

- method 1: a bone fragment was deposited for 24 h in acetic acid (CH_3–COOH, 1% = 0.17 M; Agerskov Rose et al., 2019). Then it was rinsed with demineralized water and dried;
- method 2: a bone fragment was pre-treated with hydrogen chloride (8% HCl), which dissolved ca. 30% of the outer part of the bone. Then the fragment was washed with demineralized water, dried and grinded. This was followed by an immersion in 1% acetic acid for 24 h and subsequent rinsing and drying (Wojcieszak et al., 2020);
- method 3: similar to method 2, but without the HCL pre-treatment.

CO2 was extracted from the sample with 85% phosphoric acid (90 °C). To remove any sulfur compounds, the CO2 was heated together with Ag for 30 min at 1000 °C. The purified CO2 was reduced using H2 and Fe as catalyst and then pressed into targets for AMS measurements (Van Strydonck et al., 2009).

Radiocarbon dating was applied on 61 CB samples, mainly consisting of unidentified mammal fragments and to a lesser extent of fish remains (Doel M). The latter was done in view of estimating the possible reservoir age of freshwater and marine fish, as assumed on the basis of former research on foodcrusts attached on contemporaneous pottery (Boudin et al., 2009, 2010; Teetaert et al., 2017).

For the calibration we used the OxCal software package (Bronk Ramsey, 2009; OxCal 4.4) and atmospheric calibration curve IntCal20 (Reimer et al., 2020). If multiple reference dates were available per individual hearth, the R_Combine function available in OxCal was used to calculate the weighted mean age. Contemporaneity between reference dates and CB dates from the same hearth was determined using a X^2-test (R_Combine function), the age-differences being expressed as differences between the conventional radiocarbon ages before calibration (radiocarbon years BP). In the rare cases of incompatible reference

Table 2
List of reference dates and CB dates from the Final Palaeolithic site of Verrebroek-Dok 2.

Context	Dating material	lab. refe rence	date BP	Lab. reference	Sampled unit	date BP	Lab. reference	Sampled unit	date BP	X^2-test	Difference
	Reference dates			**Calcined bone Method 1**			**Calcined bone Method 3**				
charcoal patch	Charcoal fragment	UtC-9434	11900 ± 90	RICH-27353.1.1	354	5659 ± 31	RICH-27524.1.1	354	11671 ± 39	T = 5.557 failed	−229
top humiferous layer	Peat sample	UtC-11021	11740 ± 60							T = 0.9	−69

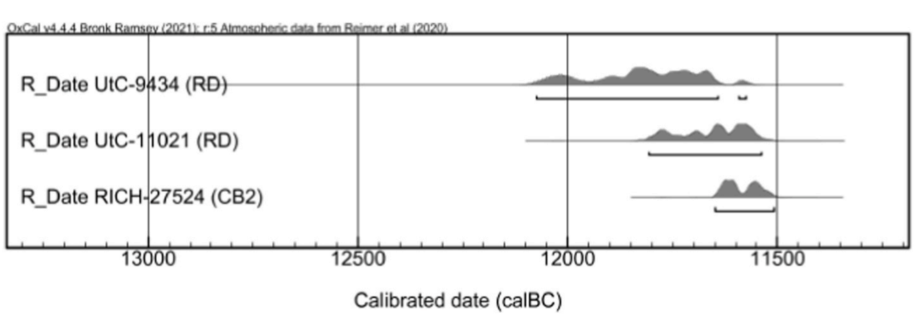

Fig. 4. Calibrated dates from the Final Palaeolithic site of Verrebroek-Dok 2. Key: RD = Reference Date; CB1 = Calcined Bone Method 1; CB2 = Calcined Bone Method 2; CB3 = Calcined Bone Method 3.

Table 3
List of reference dates and CB dates from the Early Mesolithic site of Verrebroek-Dok 1.

Artefact locus	feature ref.	Sampled unit	lab. refe rence	date BP	Lab. reference	Sampled Unit	date BP	X^2-test	difference	Lab. reference	Sampled unit	date BP	X^2-test	difference	Lab. reference	Sampled unit	date BP	X^2-test	difference
		Charred hazelnut shell Reference dates			**Calcined bone Method 1**					Calcined bone Method 2					**Calcined bone Method 3**				
72	SH	O88/Z41 (1A)	UtC-7252	8750 ± 40	RICH-27343.1.1	088/Z41 (1A, D)	8707 ± 38	T = 0.6	−43						RICH-27438.1.1	088/Z41 (4A)	8796 ± 32	T = 0.8	46
70	SH	O72/Z48 (2B)	UtC-9223	9080 ± 60	RICH-27342.1.1	O72/Z48 (2A, B)	8517 ± 32	T = 71.183 failed	−563						RICH-27513.1.1	072/Z48 (2B)	9021 ± 31	T = 0.8	−59
68	SH	O68/Z46 (2B)	UtC-9222	8400 ± 60											RICH-27857.1.1	O68/Z45 (2B)	8652 ± 31	T = 13.670 failed	252
16	SH	O88/Z25 (3B)	UtC-7117	8850 ± 40	RICH-27355.1.1	O88/Z25 (3B)	8619 ± 33	T = 19.946 failed	−231						RICH-27437.1.1	087/Z25 (2A, B)	8739 ± 30	T = 4.947 failed	−111
22	SH1	O71/Z64 (3B)	UtC-8393	9210 ± 40	RICH-27345.1.1	O71/Z65 (4A)	9083 ± 40	T = 5.040 failed	−127						RICH-27512.1.1	071/Z65 (4B)	9.051 ± 32	T = 9.675 failed	−159
23	SH1	O67/Z37 (4A)	UtC-9228	9020 ± 60	RICH-27344.2.1	067/Z39 (1A)	9067 ± 32	T = 0.5	47						RICH-27606.1.1	067/Z39 (1A, B)	8578 ± 45	T = 35.230 failed	−442
23	SH2	O73/Z12 (1A)	UtC-9433	8800 ± 80	RICH-27354.1.1	O73/Z11 (2B-D)	8376 ± 36	T = 24.178failed	−424						RICH-27607.1.1	073/Z11 (2B-E, 1B)	8674 ± 41	T = 2.0	−126
40	SH1	O41/Z53 (4)	NZA-11009	8660 ± 60	RICH-27338.3.1	O40/Z53 (1)	8267 ± 35	T = 32.767 failed	−393						RICH-27510.1.1	O40/Z53 (I B)	8663 ± 36	T = 0.0	−3
40	SH2	O40/Z55 (1)	RICH-27641.1.1	8624 ± 37						RICH-27339.1.1	O40/Z55 (1B)	8563 ± 35	T = 1.4	−61	RICH-27439.1.1	040 Z55 (1A)	8727 ± 31	T = 4.543 failed	103
41	SH	O46/Z44 (4A)	NZA-11012	9180 ± 60						RICH-27337.1.1	O44/Z43 (2A)	8810 ± 37	T = 28.107 failed	−370	RICH-27523.1.1	044/Z43 (2A bis)	8858 ± 32	T = 22.920 failed	−322
	SH	O44/Z43 (2A)	RICH-27640.1.1	9200 ± 40															
43	SH									RICH-27340.1.1	O44/Z40 (3B)	8643 ± 35			RICH-27511.1.1	044/Z40 (3B bis)	8713 ± 32		
97B	SH	O47/Z57 (3A)	NZA-11024	9160 ± 60											RICH-27848.1.1	O47/Z56 (2, 2A)	8963 ± 34	T = 8.262 failed	−197
															RICH-27855.1.1	O47/Z57 (3, 3A)	8717 ± 32	T = 43.730 failed	−443
48		O44/Z19 (1A)	RICH-27831.1.1	6154 ± 31											RICH-27856.1.1	O44/Z19 (1)	6151 ± 29	T = 0.0	−3

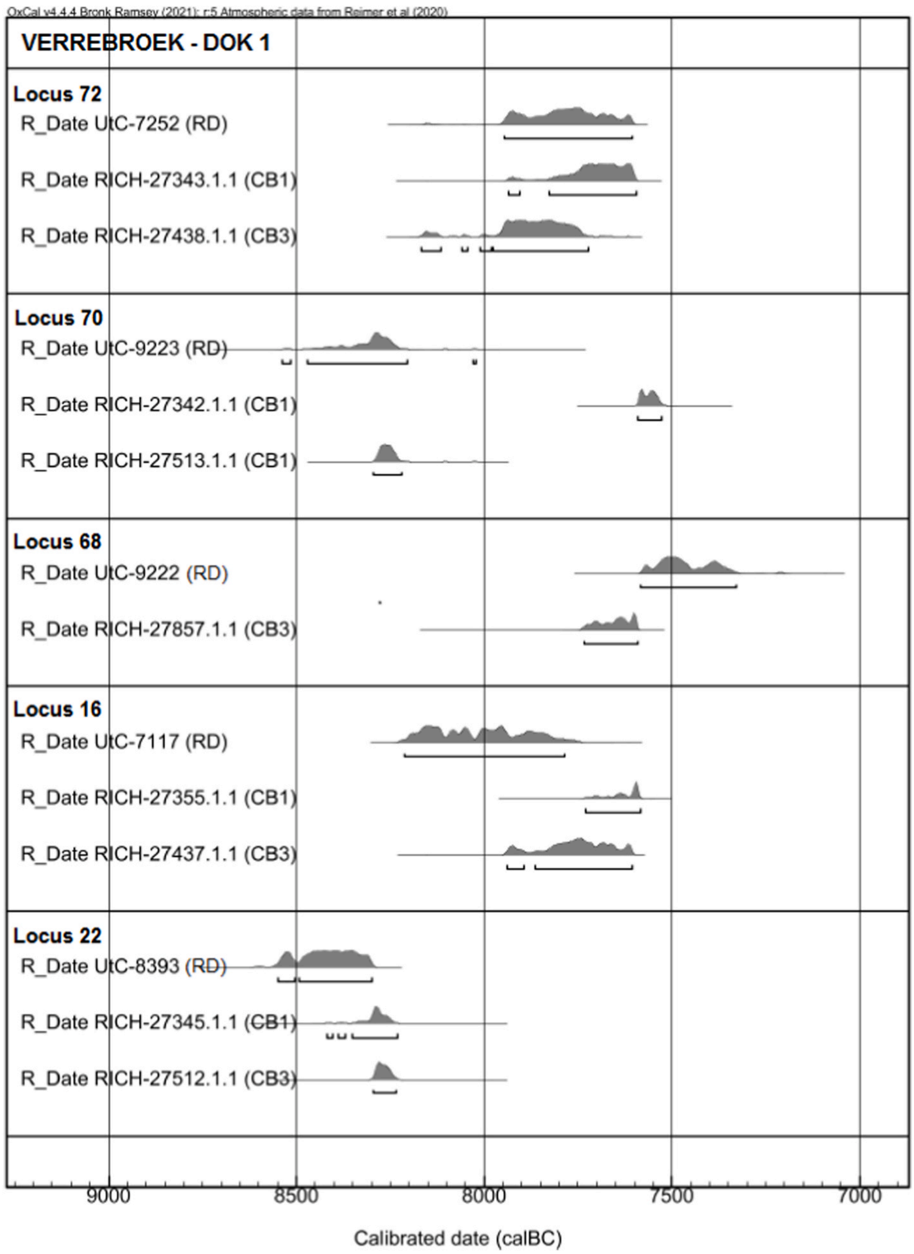

Fig. 5. a–c: Calibrated dates from the Early Mesolithic site of Verrebroek-Dok 1. Key: cf. Fig. 4.

dates (e.g. at Doel M; cf. 2.2), comparison was done using the reference date chronologically closest to the CB dates.

3. Results

3.1. Verrebroek-Dok 2

From the two CB dates (Table 2; Fig. 4), only the one obtained on a powered sample (method 3) yielded a Final Palaeolithic age. However, the X^2 test indicates that it does not match with the reference date provided by an associated charcoal fragment. On the other hand the CB date matches perfectly with the date of the top of the humiferous soil on which the Final Palaeolithic site was situated. The second CB date from the same context, conducted on a fragment pretreated according to method 1 (Table 2), is totally aberrant, as it dates to the Neolithic. Given the thick sedimentary cover, part of which dates to the Lateglacial (cf. 2.1), the dated bone cannot be considered intrusive, but rather seems to be contaminated with younger carbonates which were not fully dissolved during the pre-treatment.

3.2. Verrebroek-Dok 1

X^2-tests reveal that just 8 CB dates (= ca. 36%) on a total of 22 dates statistically fit with the reference dates, which were all conducted on charred hazelnut shells (Table 3; Fig. 5 a-c). Except for two specimens, all deviating CB dates turned out to be younger than the reference dates. The deviations of the failed CB dates range from ca. 100 to several hundreds of years, up to almost 600 ^{14}C years, with a mean of ca. 295 ^{14}C years. There seems to be no real difference in success rate between dates obtained on samples treated with method 1 or 3. However, the latter yielded slightly more corresponding dates, resp. 2 on 7 dates versus 5 on 13 dates, but given the low numbers this is not statistically robust. In addition the deviations on bone fragments (method 1) are generally somewhat higher compared to those on bone powder (method

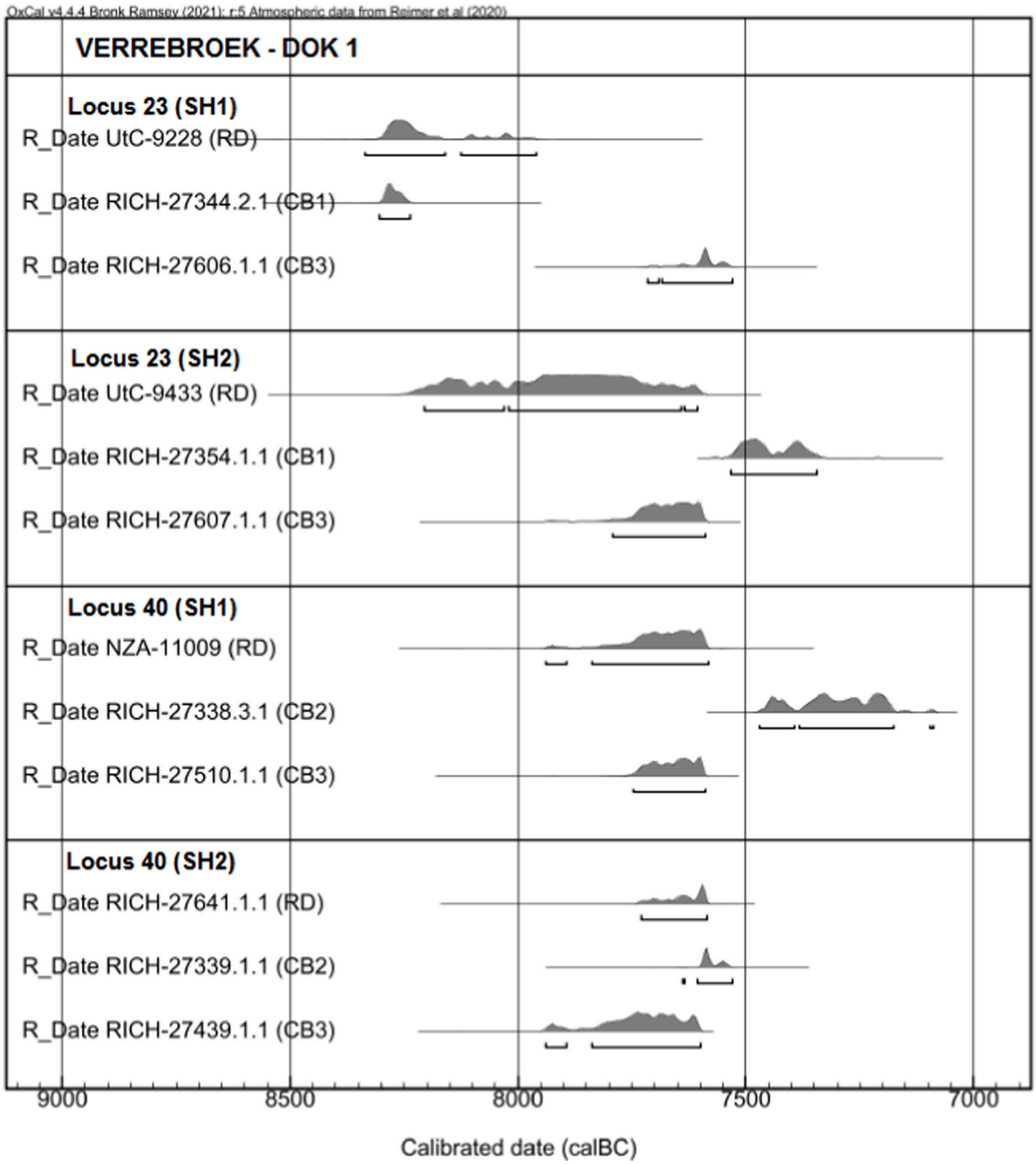

Fig. 5. (*continued*).

3), the mean deviation being respectively ca. 347 and 253 ^{14}C years.

3.3. Doel-Deurganckdok: sector B and M

According to the X^2-tests, CB dating at Doel B (Table 4; Fig. 6) and M (Table 5; Fig. 7 a-b) resulted in respectively 11 (ca. 79%) and just 5 (ca. 24%) dates in full agreement with the reference dates. The latter drops even further to just 2 positive dates if the incompatible reference date is also considered (cf. 2.2 and 2.3). This marked difference in success rate between both sites will be further discussed below (cf. 4). Compared to Verrebroek-Dok 1, two major differences need to be highlighted. First, nearly all deviating CB dates at Doel B (2 on 3 failed dates) and Doel M (15 on 16 failed dates) yielded older dates compared to the reference dates, which is the opposite of Verrebroek-Dok 1. Secondly, the deviations from the reference dates at both Doel-sites are considerably smaller than at Verrebroek-Dok 1. The mean deviation at Doel B and M accounts to respectively ca. 32 and 108^{14}C years or ca. 81 and 132 ^{14}C years if only the failed CB dates are considered. Excluding the outlier RICH-26694.3.1 (with 401 years difference), the mean deviation at Doel M drops to ca. 93 or ca. 114 ^{14}C years, the latter if only the failed CB dates are considered, but remains nevertheless higher than at Doel B. Another interesting observation is the close resemblance between the CB dates on mammal and fish remains at Doel M. Comparing the CB dates on freshwater fish (*Cyprinidae* or cyprinids) and marine fish (*Alosa* or shad), it seems that the deviations in the former are slightly smaller than in the latter. One date on cyprinid remains even belongs to the few CB dates matching with the reference dates.

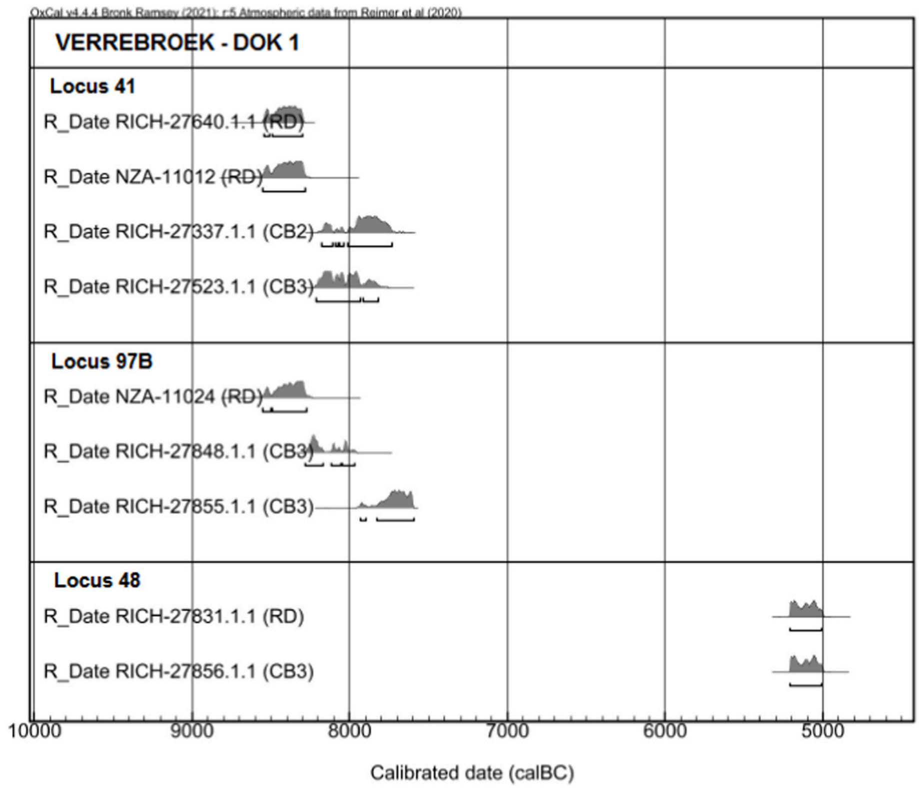

Fig. 5. (*continued*).

Table 4
List of reference dates and CB dates from the Late Mesolithic/Early Neolithic site of Doel B.

Sampled unit	lab. reference	Sample composition	Date BP	Sampled unit	lab. reference	Date BP	X^2-test	Difference	Sampled unit	lab. reference	Date BP	X^2-test	Difference
Reference dates				**Calcined mammal bone Method 2**					**Calcined mammal bone Method 3**				
HEARTH 2													
W19/Z1 (4)	KIA-17997	Hedera helix seeds	5550 ± 35	W18/Z1 (2)	RICH-27859.1.1	5584 ± 30	T = 0.1	11	W18/Z1 (1)	RICH-27860.5.1	5574 ± 29	T = 0.0	1
W19/Z1 (4)	KIA-17996	Quercus charcoal	5595 ± 35	W18/Z1 (1)	RICH-27860.1.1	5652 ± 29	T = 4.883 failed	79	W18/Z1 (2)	RICH-27859.4.1	5598 ± 29	T = 0.5	25
W20/Z2 (1)	KIA-17994	Cornus charcoal	5575 ± 35	W19/Z1 (1)	RICH-27861.1.2	5591 ± 30	T = 0.2	18	W19/Z1 (2)	RICH-27863.5.1	5599 ± 29	T = 0.5	26
X2-Test	T = 0.8		5573 ± 21	W19/Z1 (2)	RICH-27863.1.	5649 ± 29	T = 4.519 failed	76					
				W19/Z2 (2)	RICH-27864.1.1	5556 ± 28	T = 0.2	−17					
HEARTH 6													
W16/Z14 (4)	RICH-27829.1.1	charred hazelnut shell	5558 ± 31	W16/Z14 (2)	RICH-27858.1.	5513 ± 29	T = 0.1	−12	W16/Z14 (2)	RICH-27858.4.1	5438 ± 29	T = 5.7 failed	−87
W17/Z14 (2)	RICH-27830.1.1	charred hazelnut shell	5493 ± 30	W16/Z14 (3)	RICH-27862.3.1	5541 ± 25	T = 0.2	16	W16/Z14 (3)	RICH-27862.5.1	5559 ± 28	T = 0.9	34
X2-Test	T = 2.3		5525 ± 22	W16/Z14 (4)	RICH-27865.1.1	5549 ± 26	T = 0.5	24	W16/Z14 (4)	RICH-27865.3.1	5497 ± 29	T = 0.6	−28

4. Discussion

Comparison of the results demonstrates that there are no marked differences between the three pre-treatment methods with 1% (0.17M) acetic acid applied to the CB samples. However, the case-study of Verrebroek-Dok 1 and Dok 2 clearly indicates that dating bone fragments (method 1) on average yields dates with substantially larger deviations from the reference dates than dates on powdered samples. This fits with observations during earlier attempts in dating CB fragments on the same sites (cf. 2.1). Hence future dating should preferably be conducted on powdered CB (method 2 and 3). However, it is worth investigating whether dating of CB fragments (method 1) can be improved by using 1M instead of 0.17M acetic acid (Brock et al., 2010).

Both Doel cases seem to indicate no real difference in the results between methods 2 and 3, suggesting that pretreatment with hydrogen chloride does not yield much advantage at least in sandy soils. This is interesting as it implies that also small samples (e.g. fish remains), which are too small for applying the hydrogen chloride treatment, can also be dated securely. In more calcareous soils with a higher risk of carbon exchange, method 2 might be preferable since acetic acid only removes secondary carbonates (depositary calcium carbonate).

However, the most important observation from this intercomparison

Table 5
List of reference dates and CB dates from the Late Mesolithic/Early Neolithic site of Doel M (dates in italic among the reference dates have not been considered in the X^2-tests).

Sampled unit	lab. reference	Sample composition	Date BP	Sampled unit	lab. reference	Date BP	X^2-test	Difference	Sampled unit	lab. reference	Date BP	X^2-test	Difference	Sampled unit	lab. reference	Date BP	Sample composition	X^2-test	Difference
Reference dates				**Calcined mammal bone Method 2**					**Calcined mammal bone Method 3**					**Calcined fish bone Method 3**					
HEARTH 1																			
vak 4	KIA-35774	Hedera helix charcoal (twig)	5700 ± 35	Vak 4	RICH-26492.1.1	5728 ± 30	T = 0.4	28	Vak 4	RICH-26492.2.1	5605 ± 32	T = 4.017 failed	−95						
vak 4	*KIA-35770*	*Viscum album charcoal (twig)*	*5490 ± 40*	Vak 4 bis	RICH-26479.1.1	5663 ± 32	T = 0.6	−37											
X2-test	*T = 15.552 failed*		*5611 ± 27*																
HEARTH 2																			
Vak 17	KIA-36257	Viburnum opulus seed	5385 ± 30	Vak 5	RICH-26695.1.1	5444 ± 27	T = 4.557 failed	80	Vak 5	RICH-26695.3.1	5446 ± 29	T = 4.437 failed	82	Vak 5	RICH-27601.1.1	5471 ± 31	Alosa	T = 7.010 failed	107
Vak 17	KIA-36231	charred hazelnut shell	5305 ± 50	Vak 6	RICH-26697.1.1	5484 ± 29	T = 9.507 failed	120	Vak 6	RICH-26697.3.1	5491 ± 29	T = 10.649 failed	127	Vak 5	RICH-27602.1.1	5456 ± 33	Cyprinidae	T = 4.808 failed	92
X2-test	T = 1.9		5364 ± 26	Vak 8b	RICH-26696.1.1	5472 ± 28	T = 7.996 failed	108	Vak 8b	RICH-26696.2.1	5508 ± 31	T = 12.705 failed	144	vak 8b	RICH-27603.1.1	5456 ± 32	Cyprinidae	T = 4.990 failed	92
				Vak 17	RICH-26475.1.1	5495 ± 29	T = 11.331 failed	131						vak 8b	RICH-27605.1.1	5479 ± 33	Alosa	T = 7.517 failed	115
				Vak 22	RICH-26694.1.1	5450 ± 31	T = 4.526 failed	86	Vak 22	RICH-26694.3.1	5765 ± 31	T = 98.987 failed	401	vak 22	RICH-27600.1.1	5403 ± 32	Cyprinidae	T = 0.9	39
														vak 22	RICH-27604.1.1	5489 ± 31	Alosa	T = 9.570 failed	125
HEARTH 3																			
Vak 9	KIA-35771	Hedera helix seeds	5490 ± 40	Vak 9	RICH-26477.1.1	5698 ± 28	T = 17.983 failed	208	Vak 9	RICH-26477.2.1	5525 ± 32	T = 0.5	35						
Vak 9	*KIA-35786*	*charred hazelnut shell*	*5280 ± 40*																
X2-test	*T = 13.777 failed*		*5387 ± 29*																
HEARTH 5																			
Vak 27	KIA-35804	Quercus sp. acorn	5570 ± 35	Vak 27	RICH-26483.1.1	5564 ± 31	T = 0.0	−6											

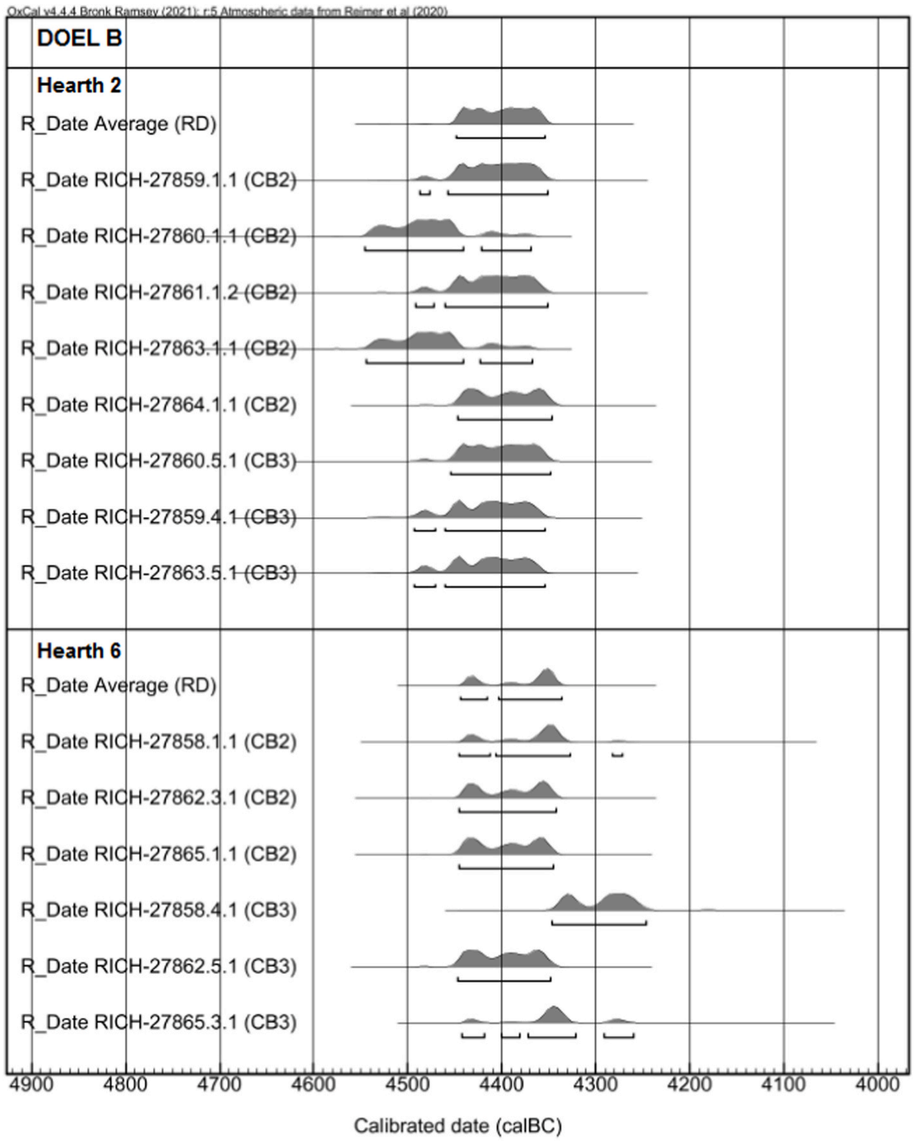

Fig. 6. Calibrated dates from the Late Mesolithic/Early Neolithic site of Doel B. Key: cf. Fig. 4.

study is the considerable inter-site variation in the obtained results. Highly contrasting results have been obtained for the four sites under study. While the vast majority of CB dates at the Early Mesolithic site of Verrebroek-Dok 1 are (much) too young compared to their reference dates, at both Late Mesolithic/Early Neolithic sites of Doel dating generated results that were overall slightly older. At the Final Palaeolithic site of Verrebroek-Dok 2 CB dating seems to confirm the reference date, based on the stratigraphical position of the site.

The somewhat older dates at both Doel-sites most likely can be explained as resulting from a wood-age offset or old-wood effect caused by the exchange of carbon from the fire fuel into the bones during the burning process (Rose et al., 2020). Earlier studies, based on cremation experiments (Hüls et al., 2010; Snoeck et al., 2016; Van Strydonck et al., 2010; Zazzo et al., 2009), have demonstrated a carbon exchange of between 40/53% to 91/95% from the fire wood. This is corroborated by the CB dates on marine and freshwater fish from Doel M. While some kind of reservoir effect could be expected, these fish dates do not show a real difference in age with the dates obtained on CB of mammals from the same site and contexts. This implies that radiocarbon dating of CB measures the carbon of the fire fuel rather than the carbon initially present in the bone, certainly in small fish bone fragments, in which the original bone apatite carbon was entirely replaced by carbon from the combustion fuel. Hence most dates from the Doel-sites should be corrected according to the inbuilt-age of the fire wood, which is variable according to different types of wood. Anthracological analysis at both Doel-sites (Deforce et al., 2013, 2014) has revealed the use of a mix of wood species in most hearths (Fig. 8). However, a clear preference for alder (*Alnus* sp.), ash (*Fraxinus excelsior*), oak (*Quercus* sp.) and in some hearths also mistletoe (*Viscum album*) could be observed. Most of these species can be considered short-lived taxa, with an average maximum age of ca. 150 years or less (Annaert et al., 2020, and references therein). Only oak is a long-lived species with an average maximum age of 500 and exceptionally 1000 years. It thus seems reasonable to assume that the mixed use of these wood species during firing at Doel might account for the observed wood-age offset of <100 years. The differences in offset between sites B and M remains difficult to explain, but might be caused by differences in used fire wood, e.g. more branches or twigs at B. Lacking detailed anthracological data, this unfortunately remains purely hypothetical. Alternatively the intersite difference might be caused by the differential importance of mistletoe as firewood. Clearly mistletoe was much more important at Doel M compared to Doel B, with a mean frequency of 12.7% versus 1.8%. Looking more in detail at the former site, it seems that the CB dates from the hearths with the largest amount of mistletoe charcoal (hearths 2 and 3) yielded the largest off-sets

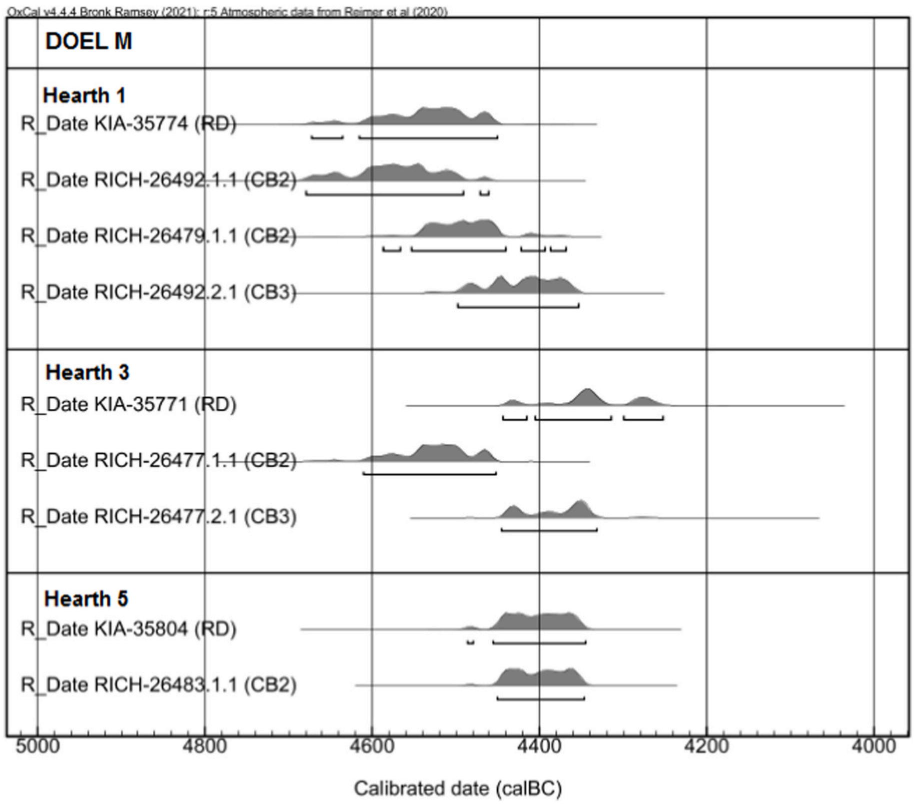

Fig. 7. a–b: Calibrated dates from the Late Mesolithic/Early Neolithic site of Doel M. . Key: cf. Fig. 4.

(Fig. 8). Knowing that mistletoe mainly occurs on larger diameter trees and old trees, e.g. older than 100–120 years (Lech et al., 2020), this could indirectly point to the fact that the firewood used at Doel M was mainly collected from older trees compared to Doel B.

Interestingly at the Final Palaeolithic site of Verrebroek-Dok 2 the CB dated on powder seems not to be affected by a wood-age offset when compared with the stratigraphical date. This might be due to the fact that the vast majority (96.1%) of the charcoal from the associated charcoal patch belongs to poplar (*Populus* sp.), a relatively short-lived species (Bastiaens et al., 2005). However, compared to the charcoal date, the CB date is much younger which reinforces the hypothesis that the formation of the charcoal feature occurred before the human occupation and related burning of the bones; it might even be linked to a purely natural event (forest fire).

Carbon exchange from the fire wood clearly cannot explain the too young CB dates with respect to the hazelnut dates at Verrebroek-Dok 1. It is generally assumed that bio-apatite in calcined bones, in contrast to collagen in unburnt bones, is robust and inert due to re-crystallization during heating (Lanting et al., 2001; Olsen et al., 2008; Van Strydonck et al., 2009). The enhanced crystallinity of the bone matrix would form a mechanical barrier protecting the remaining structural carbonate. However, it does not stop the exchange process between the bio-apatite and exogenous carbon but rather slows it down (Van Strydonck, 2016; Van Strydonck et al., 2005). Hence, the most likely explanation for the significantly deviating results at Verrebroek is contamination with younger, exogenous carbon, which could not be fully extracted during any of the pre-treatment methods. If correct, the question arises why the CB dates at Doel and Verrebroek-Dok 2 are not affected despite the fact that all studied sites come from similar geological contexts, i.e. coversands covered by peat and (peri)marine sediments. Probably site taphonomy plays a very important role. Both Doel-sites as well as Verrebroek-Dok 2 are sites which were quickly covered by sediments, respectively peat and aeolian sands. Peat covering at Doel B and M followed almost immediately after the human occupation and even might have been the reason why human occupation ended on both sand dunes (Verhegge et al., 2014). Although the sands on top of the Final Palaeolithic site of Verrebroek-Dok 2 were not directly dated, there is indirect evidence which points to a deposition during the Allerød. Indeed, the sands immediately on top of the humiferous soil were deposited under water, so at the moment the pond was still existing (Louwagie and Langohr, 2005). Recently direct proof of intra-Allerød deflation has been collected at nearby locations in the Lower-Scheldt basin (Crombé et al., 2020). On the other hand the Early Mesolithic site of Verrebroek-Dok 1, although also covered by peat around the same time as the Doel-sites, has remained uncovered for at least 4 to 5 millennia. Hence artefacts and ecofacts, including CB, have been lying unprotected at the surface or shallow subsoil for a very long time-period. This is also observable in the general preservation of the CB, which is visually much poorer at Verrebroek-Dok 1 compared to the Doel-sites (Van Neer et al., 2005). The former present clear traces of weathering, such as edge-rounding, while the former have a more “fresh” appearance.

5. Conclusions

This extensive inter-comparative study has clearly demonstrated that radiocarbon dating of CB from Final Palaeolithic and Mesolithic open-air sites yields varying degrees of success, as it largely depends on the post-depositional history and taphonomy of the site. Apparently the best results can be expected on sites covered quickly after the formation and deposition of the CB, be it by aeolian (Verrebroek-Dok 2) or peat and alluvial/marine deposits (wetland sites of Doel). These contexts probably closely match the protected soil conditions of CB recovered from archaeological features, (e.g. cremation pits) and sheltered contexts (e.g. caves), which usually yield reliable radiocarbon dates. Dating CB from uncovered sites or sites covered only after a long time-period

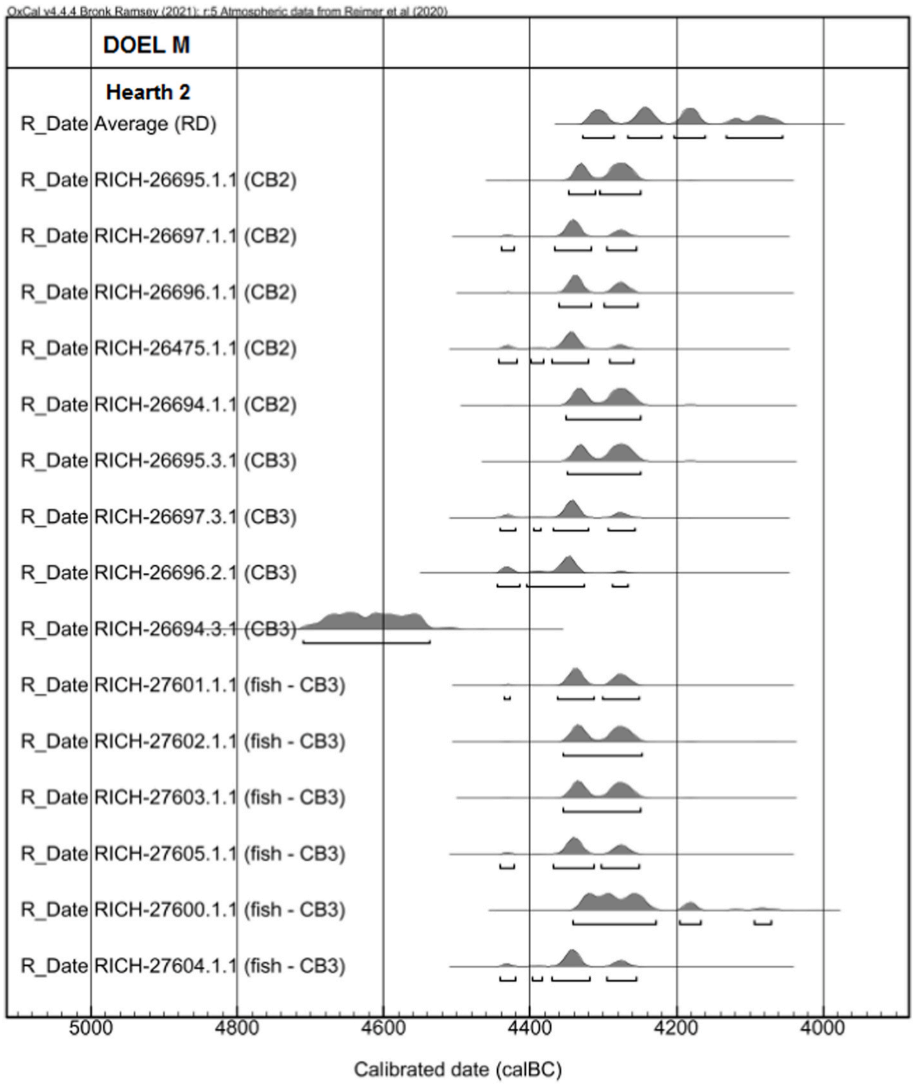

Fig. 7. (*continued*).

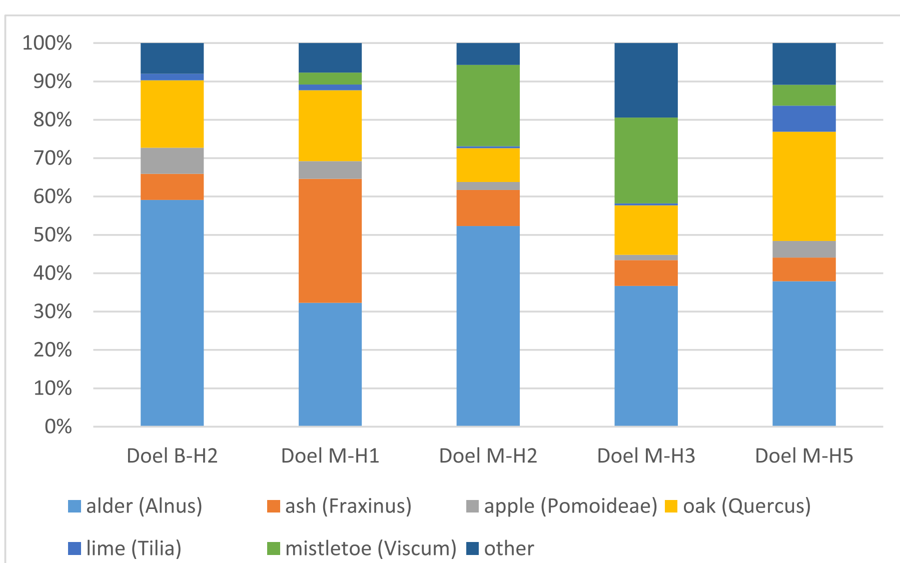

Fig. 8. Charcoal composition of surface-hearths at the Late Mesolithic/Early Neolithic sites of Doel B and Doel M (based on data from Deforce et al., 2013; 2014).

(Verrebroek-Dok 1) remains problematic and is absolutely to be discouraged in the future. However, even on quickly covered sites accurate dates, i.e. CB dates which actually date the occupation event precisely, are not always guaranteed as some may be affected by an old-wood or wood-age offset resulting from the uptake of carbon from the fire wood. However, the site of Doel B demonstrates that in certain cases off-sets are limited and almost negligible, while on other sites (Doel M) CB dates need to be corrected and should hence be considered as *termini post quem*. Correcting these dates is not straightforward since the deviations depend on the types and age of the wood used during the burning process. So in order to correctly asses CB dates one should possess detailed information on the firewood used to burn the fires, as demonstrated by the comparison between both Doel-sites. Unfortunately most Final Palaeolithic and Mesolithic open-air sites lack detailed anthracological information as charcoal associated with hearths is generally missing completely (e.g. Verrebroek-Dok 1), mainly as a result of a (too) late covering of the sites. In conclusion, CB dates are in general not appropriate for developing robust, decadal-to-centennial chronologies for Final Palaeolithic and Mesolithic open-air sites and "cultures", e.g. in the context of typo-chronological research or synchronization with other proxies, such as paleoclimate (Crombé, 2019).

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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