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RADIOCARBON DATING OF DUTCH MORTARS MADE FROM BURNED SHELLS

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ABSTRACT. AMS-based radiocarbon dating was applied to Medieval lime mortars made from burned shells and aggregate including both shore sediments and neovolcanic rocks. Three mortar samples from the city of Leiden near Amsterdam were prepared using the same kind of acid hydrolysis technique as has been earlier used for dating mortars made from burned marble and limestone. Five consecutive CO_2 fractions were collected from each sample to form age profiles as functions of the dissolution progress index. One of the samples, from a brick wall of known age, was taken as a reference from the Pieterskerk church. Two other samples were taken from the Burcht circular stronghold on a former island on the Rhine River. The age of Burcht is less well known; thus, the presented results are a contribution to an ongoing discussion on its history.

INTRODUCTION

Lime mortars made of burned limestone and marble have been successfully dated using accelerator mass spectrometry (AMS)-based ^{14}C analyses since the 1990s (e.g. Van Strydonck et al. 1992; Ringbom and Remmer 1995, 2000, 2005; Heinemeier et al. 1997, 2010; Hale et al. 2003; Ringbom et al. 2006; Ringbom 2011; Lindroos et al. 2007, 2012; Nawrocka et al. 2009; Al-Bashaireh and Hodgins 2011, 2012; Langley et al. 2011; Marzaioli et al. 2013; Ortega et al. 2012). Mortars containing hydraulic components like weathered vitric volcanics have also been dated, but with many complications and a lower success rate (Ringbom et al. 2006, 2011; Hodgins et al. 2011; Lindroos et al. 2011a).

The sampling in Leiden described in this article was aimed to test whether mortars made from burned shells are suitable for ^{14}C dating and to see what kind of ^{14}C profiles they would yield when processed in the same way as mortars made from limestone or marble. The analyzed samples may contain basaltic-basanitic volcanic material (van Balen et al. 2003) from the Rhine Graben Tertiary volcanic system (e.g. Ritter et al. 2001). They were taken from two sites: the Pieterskerk church and the Burcht circular stronghold. From the Pieterskerk, we dated one sample collected from a brick wall of a known age (den Hartog and Veerman 2011). From the Burcht, we dated two samples chiseled from between tuff blocks in loopholes near the present ground level. The expected age of the samples was early 13th century AD, but the exact chronology is not known. Some of the results have been published in an archaeological context earlier (Orsel 2012a,b). This article thus describes the main results coming from the sample characterizations and evaluates them as a material for ^{14}C dating. The obtained ^{14}C results will also be framed within the main open question regarding the attribution of the Burcht stronghold manufacture to the Dutch dukes of the 13th century AD.

MATERIALS AND METHODS

Sampling and Sample Pretreatment

The Burcht of Leiden (Figure 1A) is a circular stronghold made of mortared tuff stones and bricks (Orsel 2012a,b). It is situated on a former island on the Rhine River. The circular curtain wall, 18.8 m in diameter, was built on an older motte (Janssen et al. 1996). Three samples were taken, and after microscopic screening, two were selected for dating. The two samples dated were taken from the inside of the fourth loophole to the left of the entrance (Figure 1B). Three reference samples, also made from burned shells and with a fairly well-known age, were taken from the Pieterskerk in

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Leiden from the south wall of the choir (Figure 2). After microscopic screening, two of them were further analyzed and one was dated. According to dendrochronological studies (den Hartog and Veerman 2011), the age of the wall should date to AD 1390–1415.

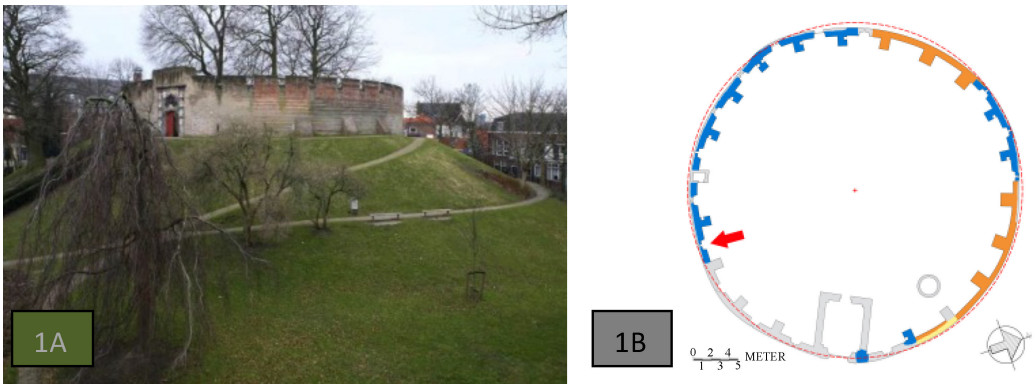

Figure 1 (A) The Burcht today. (B) Sketch of the sampling site in the Burcht: Medieval (blue), 18th century (orange), 19th century (yellow), and 20th century (gray). The red arrow indicates the place where the samples were taken (from Orsel 2012b).

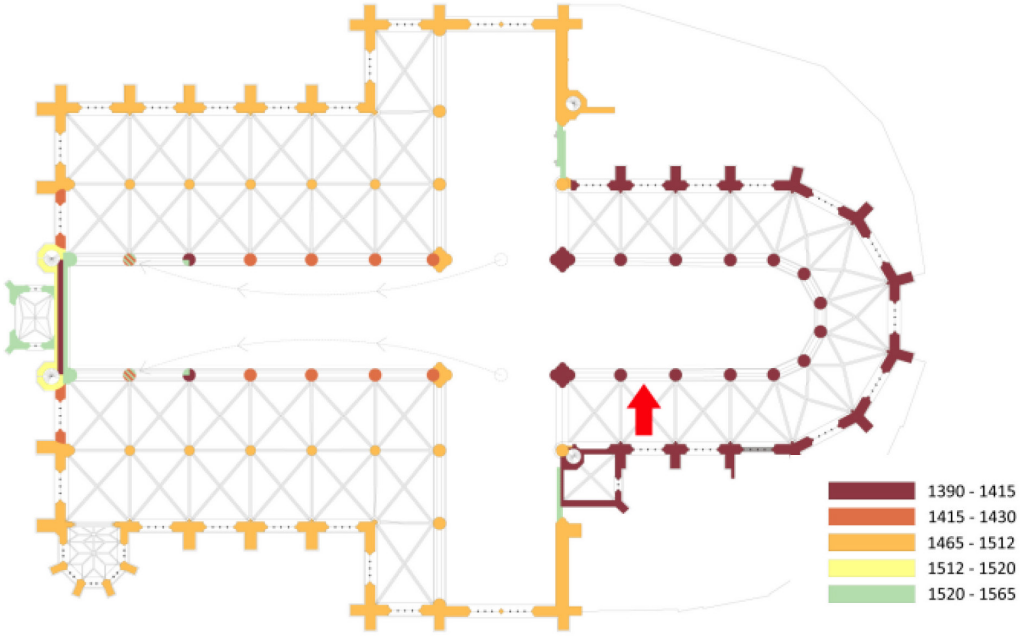

Figure 2 Plan view and building stratigraphy of the Pieterskerk church (modified from den Hartog and Veerman 2011) with the sampling point denoted with a red arrow.

The shells for the lime production were probably collected from postglacial shell banks along the North Sea coast (van Balen et al. 2003). The aggregate in the mortars is also, at least partly, from shore sediments because shells of Baltic clam (*Macoma balthica*) can be identified in the mortars.

The mortar pieces were crushed at the laboratory with plastic-covered pliers. All the samples were relatively soft despite the suspected hydraulic aggregate material. The crushed material was dry-sieved in a sieve shaker for 20 min. The sieving was done gently so that the material was only vi-

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brating in the sieves. A 46–75 μm grain-size fraction was isolated and washed with deionized water to remove fine dust.

Characterization of the Samples

The 46–75 μm grain-size fractions were dried and inspected using a CITL 4 (Cambridge Image Technology Ltd) cold cathode cathodoluminescence (CL) device combined with a stereomicroscope and a camera (for CL see e.g. Marshall 1988). About 10 mg of sample powder was spread over a glass backing and irradiated in the low-vacuum chamber with a 7-keV, 240-μA electron beam and luminescent minerals were identified.

The chemistry and mineralogy of the dated samples and some other samples from the same sites were determined using particle-induced X-ray emission (PIXE; see e.g. Johansson et al. 1995) and X-ray diffraction (XRD) at Åbo Akademi University in Finland. The PIXE analyses were done using a millimeter-sized external proton beam (Lill 1999). Aliquots of the same sample powders (46–75 μm grain-size window) that were sent for ^{14}C dating were pressed into 13-mm powder pellets on spectrographically pure graphite backings and irradiated in air with a 3-MeV proton beam. The samples were scanned in front of the beam at a 45° angle for 10 min in 16 spots on the pellet surface in a 4 × 4 spot grid. One spot represents approximately 0.3 mg of sample depending on the sample matrix and element considered. Additionally, a Baltic clam (*Macoma balthica*) found in the sample Burcht 001 was analyzed by irradiating a polished part of the outer surface of the shell. X-rays from calcium and heavier elements were registered with an intrinsic germanium planar detector. The intense X-rays from Ca were suppressed by a filter consisting of a 3-mm-thick polycarbonate disk with a 0.5-mm hole.

XRD analysis is a routine method to identify minerals. Every mineral with an organized crystal structure diffracts X-rays in a pattern that is dependent on the geometric structure of the crystal lattice (Jenkins and Snyder 1996). The powder pellets prepared for PIXE analyses were analyzed with X-ray diffraction (XRD) using a Bruker D8 Discovery (Bruker-AXS, Karlsruhe, Germany) with CuKα radiation ($\lambda = 1.54184$ nm). The XRD spectra were collected in the 2θ range of 20–80°. The X-ray tube was operated at 40 kV and 40 mA. For phase identification, the PDF-2 Release 2010 database from the International Centre for Diffraction Data (ICDD) was used.

Radiocarbon Dating

The ^{14}C dating procedure for lime mortars has been described in some detail in Heinemeier et al. (2010) and Ringbom (2011). It can be described shortly as follows: 100–150 mg of the wet-sieved 46–75 μm grain-size fraction is hydrolyzed with 85% phosphoric acid under vacuum to produce carbon dioxide (CO_2) for graphitization and AMS analysis. The CO_2 from each sample is collected as several fractions as it evolves in the hydrolysis from the carbonates in the sample. The ^{14}C ages are then reported as a function of the dissolution progress to produce ^{14}C profiles. Usually, binder carbonates constituting the sample react violently with the acid, producing enough CO_2 for dating within seconds. The CO_2 production from any of the possible contaminants, like geological aggregate carbonate or limestone residues after incomplete calcination in the lime burning process, are much slower. It is quite common to have both these contaminants, and their contribution of dead CO_2 is usually significant already before half of the sample has dissolved (Lindroos et al. 2007). By collecting a CO_2 fraction as rapidly as possible, one can minimize the effect of the contaminants. In this way, it is possible to date samples even if they have many contaminating carbonates. A successful dating of contaminated samples then comprises several (at least three) ^{14}C profiles having similar ages for rapidly formed CO_2. Figure 3 shows how a ^{14}C profile from a mortar made of pure marble is constructed and how the contaminants affect the ^{14}C values when the dissolution proceeds.

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The *x* axis in Figure 3 describes the dissolution progress index F (0→1). F = 1 corresponds to total carbon yield during the hydrolysis. Usually, the dissolution process is aborted when there is no longer visible effervescence of CO_2 from the sample after 4–16 hr. The bars along the *x* axis denote the size and span of each analyzed CO_2 fraction as well as their position in the dissolution progress F.

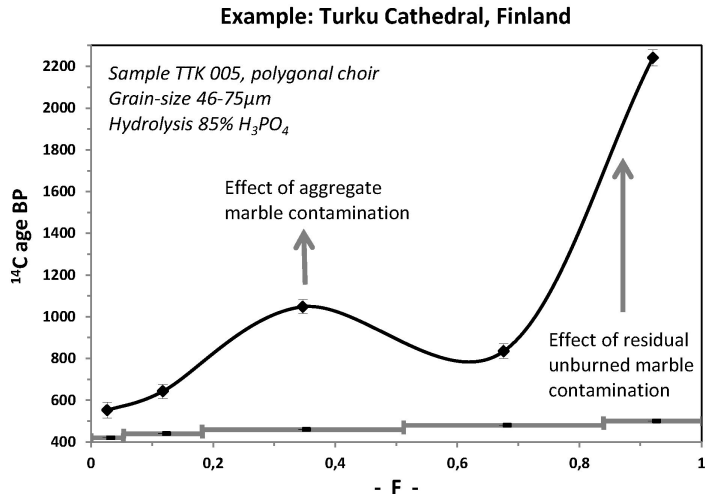

Figure 3 Typical ^{14}C profile displaying contamination from both aggregate marble and marble residues from incomplete calcination. The dated mortar is from the pentagonal choir in Turku Cathedral in SW Finland. It was made from very pure, burned marble and inert quartz-feldspar sand-gravel aggregate with some marble splinter. The first CO_2 fraction to the left has a ^{14}C age reflecting the 14th century AD age of the construction unit (from Lindroos et al. 2011b, 2012).

The bump in the profile is caused by CO_2 from marble grains in the aggregate and the rising end of the profile is caused by slowly dissolving marble residues from incomplete calcination. In this case, the curved line in the plot is only connecting the points. The ^{14}C profiles from Burcht and Pieterskerk will be presented according to this scheme. The curved line in the plot is only connecting the points. For actual modeling of contamination effects, see Lindroos et al. (2007).

Sample hydrolysis, collection of the CO_2 fractions, graphitization, and the ^{14}C analyses were done at the Århus AMS lab, Denmark. The calibration of the ^{14}C ages to calendar years has been done using the IntCal09 calibration curve (Reimer et al. 2009) and the OxCal v 3.10 program (Bronk Ramsey 2009). Part of the CO_2 gas was used for $\delta^{13}C$ and $\delta^{18}O$ 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).

RESULTS

The binder carbonate in the Pieterskerk sample has no luminescence; only a few luminescent aggregate grains of calcite and aragonite were visible (Figure 4A). The luminescence of the binder in the Burcht samples was also very weak, but there were also lots of grains with a dull red luminescence (Figure 4B).

The results of the PIXE analyses are presented in Table 1. Sample Burcht 003 had a low Ca concentration and high Mn, Fe, and Zn concentrations due to a high amount of aggregate material in the sample. For the other four samples, the Sr/Ca ratio was in the range of (40.5–54.9) × 10^{-4}. The clam

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has a similar ratio, 46.9×10^{-4}, which is near the average of 47.6×10^{-4} for the Dutch samples. The marble-based mortars and the limestone standard have much lower ratios (as do mortars in general except for Roman Pozzolana mortars; our unpublished data). The aggregate-rich Burcht 003 sample has the highest Sr/Ca ratio, which together with high Mn, Fe, and Zn values, is in agreement with the idea that basaltic rocks from the Eiffel area were used as aggregate in the mortars.

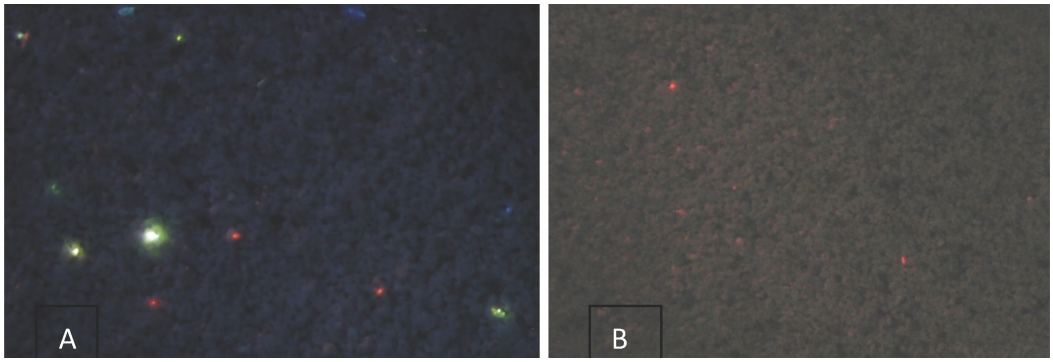

Figure 4 CL micrographs of aliquots of the dated 46–75 µm grain-size fractions: (A) Pieterskerk 002; (B) Burcht 002. Binder calcite grains are dark, unburned, and poorly burned calcite grains are red, aragonite is green, and quartz is blue. In (B), some reflected light was let in through the vacuum chamber window to enable distinction of the binder grains.

Table 1 Results of PIXE analyses of sample powders from the Burcht circular stronghold and Pieterskerk church and a clam from within sample Burcht 001. Samples of mortars made from lime of burned marble (TTK 006 and TTK 023 from the same building unit as sample TTK 005 in Figure 3) are included for comparison. The method was evaluated by analyzing SRM 1d from NIST. Limits of detection (LOD) are found in the rightmost column (nc = not certified).

	Burcht			Pieterskerk church			Turku Cathedral		Limestone		
Sample:	Burcht 001	Burcht 002	Burcht 003	Pieterskerk 001	Pieterskerk 002	*M. balthica* shell	TTK 006	TTK 023	SRM 1d		
Fraction size:	46–75 µm			46–75 µm			46–75 µm		meas.	cert.	LOD
Ca (%)	36.7	36.4	7.82	32.8	33.1	39.1	37.4	37.6	37.78	37.75	0.127
Mn (ppm)	150	220	1380	230	160	<94	460	460	195	209	94
Fe (ppm)	2640	3460	15,030	3430	3680	1475	3340	4530	2234	2173	53
Cu (ppm)	13	18	15	17	<15	21	<15	<15	<15	nc	15
Zn (ppm)	9	17	125	14	17	<6	21	31	18	22	6
Br (ppm)	15	9	5	34	<5	<5	<5	<5	<5	nc	5
Rb (ppm)	15	16	155	13	16	<6	<6	15	<6	6	6
Sr (ppm)	1488	1550	767	1802	1728	1835	189	212	254	256	6
Sr/Ca ($\times 10^4$)	40.54	42.58	98.08	54.94	52.21	46.93	5.05	5.64	6.72	6.78	—

Only calcite was identified with XRD in the Dutch samples, except for Burcht 003, where also the zeolite mineral chabazite was identified. The mineral is common in voids in neovolcanic, basaltic rocks (Deer et al. 1992). The few aragonite grains identified by CL were not enough to yield an aragonite signal. Figure 5 shows the diffractograms and the inset shows the peaks from chabazite more clearly. The results of sample hydrolysis, collection of the CO_2 fractions, and the ^{14}C and stable isotope analyses are shown in Table 2.

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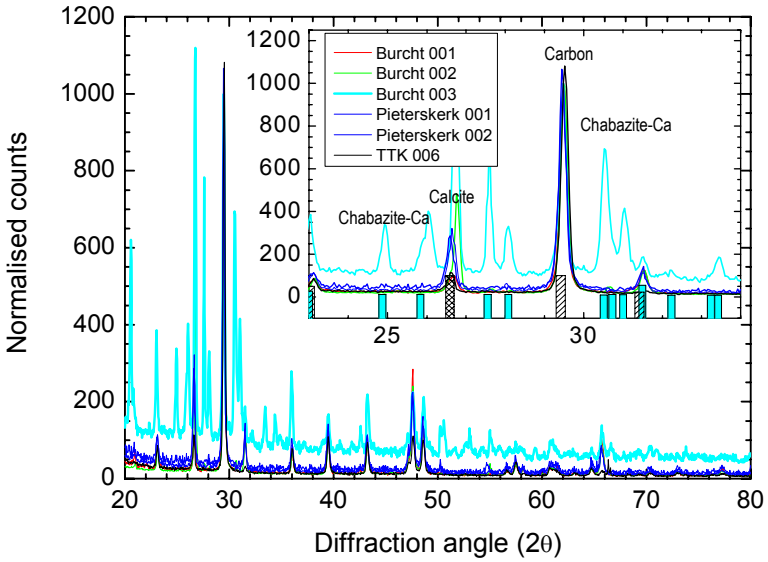

Figure 5 Diffractograms for the Dutch samples compared with a marble-derived lime mortar from SW Finland (TTK 006). The locations of the three identified phases are marked as bars in the inset. The carbon is due to the graphite used as backing material in the pressed pellets.

Table 2 ^{14}C data from hydrolysis with 85% H_3PO_4 and ^{13}C and ^{18}O measurements. $\delta^{18}O$ values are not directly comparable with limestone values from standard measuring procedures using dehydrated acid. They are, however, comparable with mortar values from Heinemeier et al. (2010) and Lindroos et al. (2012).

Sample C yield (%)	CO_2 fraction (%)	^{14}C age yr BP	±	$\delta^{13}C$ (‰)	$\delta^{18}O$ (‰)	Lab nr
Pieterskerk 002	0–8.9	497	35	–21.32	–23.3	AAR-12318,1
8.6	8.9–28	798	34	–6.42	–15.89	AAR-12318,2
	28–48	1181	34	–11.36	–14.89	AAR-12318,3
	48–68	1285	38	–11.42	–14.91	AAR-12318,4
	68–100	1661	45	–10.95	–11.45	AAR-12318,5
Burcht 001	0–9.5	739	35	–18.24	–21.5	AAR-12316,1
9.0	9.5–29	1259	37	–7.67	–14.41	AAR-12316,2
	29–48	1710	38	–9.83	–12.02	AAR-12316,3
	48–57	1811	40	–9.26	–10.94	AAR-12316,4
	57–100	2077	35	–8.99	–9.24	AAR-12316,5
Burcht 002	0–10	736	38	–18.87	–21.54	AAR-12317,1
8.6	10–30	1302	34	–9.36	–15.28	AAR-12317,2
	30–49	1766	39	–10.39	–12.12	AAR-12317,3
	49–68	1665	35	–10.38	–13.07	AAR-12317,4
	68–100	2191	34	–9.74	–9.74	AAR-12317,5

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Figure 6 shows the ^{14}C profile of the Pieterskerk sample with the known date of AD 1390–1415. The profile indicates heavy contamination and shows that the relative activity of the contaminants increases as the dissolution proceeds. The $\delta^{13}C$ values in Table 2 and Figure 8B, apart from the first fractions, are similar to values from limestone-derived mortars. The second CO_2 fractions are characterized by the highest value, which is also common among mortars in general (e.g. Lindroos et al. 2007; Heinemeier et al. 2010). The calibrated age AD 1320–1460 (92.1% probability) at the 2σ confidence level and 1412–1440 at the 1σ confidence level are not statistically different, at 5% of significance, from the presumed age of AD 1390–1415 (Figure 7) of the analyzed sample.

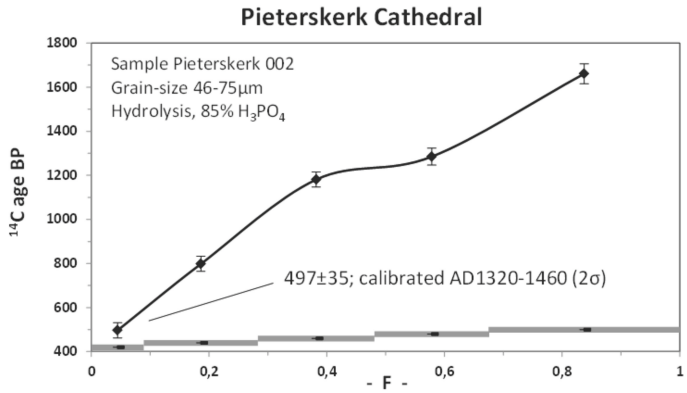

Figure 6 ^{14}C profile of a test sample with the known age AD 1390–1415

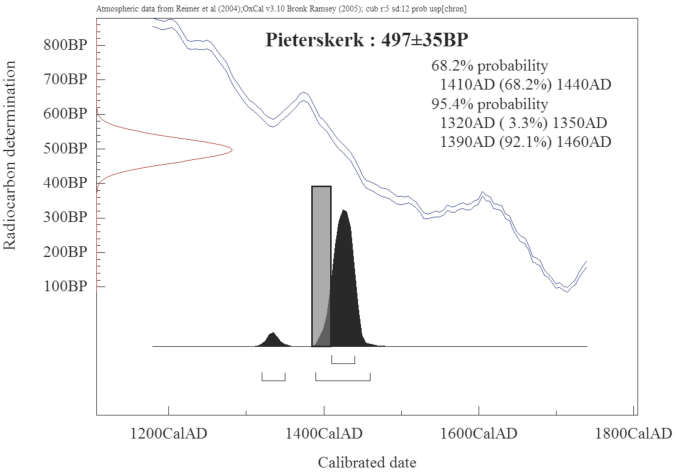

Figure 7 Calibrated results from the Pieterskerk 002 sample with the known age inserted as a gray bar.

The two samples from Burcht yield similar ^{14}C and $\delta^{13}C$ profiles as the one from Pieterskerk, but they seem to reflect an older age (Figure 8). The calibrated ages for the first fractions are AD 1215–1300 and 1210–1390 for Burcht 001 and Burcht 002, respectively, for the 2σ confidence level. The combined calibration of the ages for the first fractions yield the age AD 1225–1295 at the 2σ confidence level (Figure 9).

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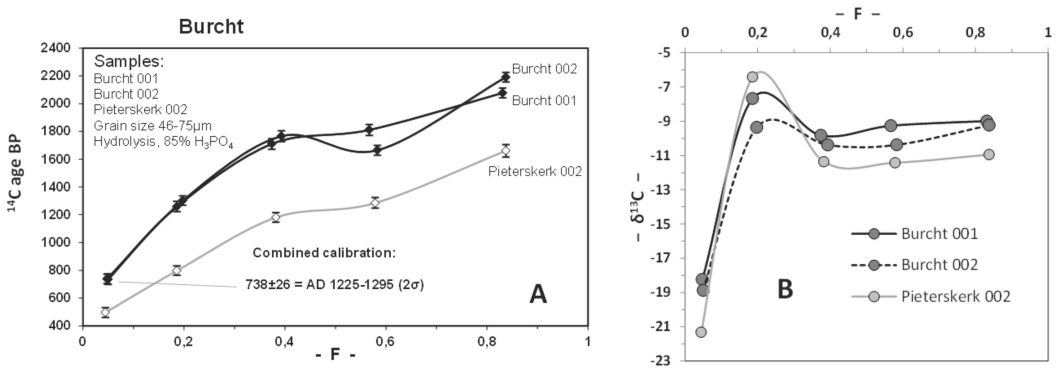

Figure 8 (A) ^{14}C profiles from the Burcht circular stronghold with black lines and filled dots and from Pieterskerk with the gray line and open dots. The bars denoting the fraction sizes are omitted because they are very similar to those presented for Pieterskerk in Figure 6. Numerical values for the fraction sizes can be found in Table 2. (B) Evolution of $\delta^{13}C$ values as a function of the dissolution progress variable F.

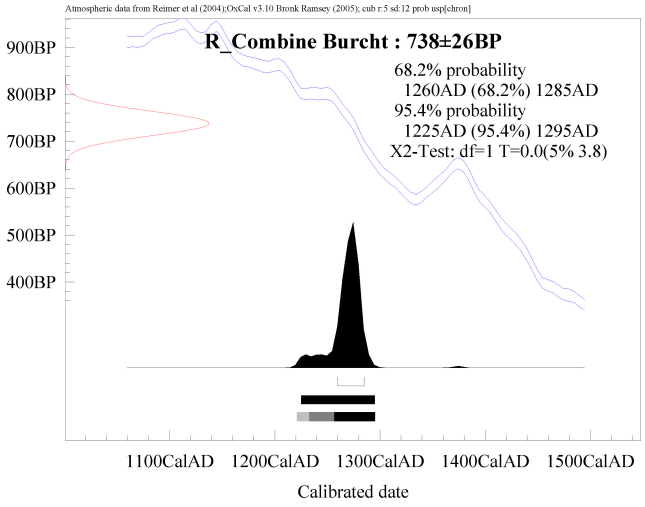

Figure 9 Combined calibration of the two primary CO_2 fractions (Burcht) in Figure 8. The horizontal gray-black bar under the 95.4% confidence level bracket denotes the reigns of three dukes referred to in the discussion: light gray: Floris IV (AD 1222–1234); dark gray: William II (AD 1234–1256); and black: Floris V (AD 1256–1296).

DISCUSSION

The dating attempt on the Burcht samples yielded similar ^{14}C profiles as the successful dating of the Pieterskerk test sample. Heinemeier et al. (2010) presented criteria for good mortar dating based on ^{14}C profiles. According to this classification, the Burcht samples would be classified in the category III: Two initial CO_2 fractions yield similar results. The second best, category II dating, would have required three initial fractions giving similar results. Category I requires less contaminant activity, giving flatter ^{14}C profiles.

Orsel (2012a,b) discusses the historical context of the ^{14}C ages: Which one of the 13th century Dutch dukes took the initiative to build Burcht: Floris IV (AD 1222–1234), William II (AD 1234–1256), or Floris V (AD 1256–1296)? The presented ^{14}C analyses do not significantly rule out any of the

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dukes, but give a higher probability in favor of Floris V. Optimized hydrolysis and a few more samples would provide a more reliable result.

CONCLUSIONS

The chemistry and mineralogy of the studied samples is in agreement with the presumption that the mortar limes were made from burned shells and a volcanic aggregate from the Rhine Graben system was used. Shells originally contain aragonite with a much higher Sr concentration than calcite in limestone or marble. The aragonite structure is destroyed in the burning process and replaced by calcite in the mortar, but Sr prevails in the bulk composition. The aggregate in the mortars seems to originate from two sources: a shore deposit with shells, shell fragments, and a volcanic source enriched in Mn, Fe, Zn, and the zeolite mineral chabazite.

Mortars made from burned shell behave in a similar way as mortar made from burned marble and limestone in our ^{14}C dating procedure. CO_2 fractions from the very beginning of the acid hydrolysis reflect the right historical age without significant contribution of dead carbon from unburned carbonates. There is, however, an abundant dead carbon contamination in the slowly dissolving part of the samples.

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