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THE ROMAN AMPHITHEATRE IN MÉRIDA, SPAIN -AUGUSTAN OR FLAVIAN? RADIOCARBON DATING RESULTS ON MORTAR CARBONATE

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Abstract
Four lime mortar samples from the Mérida amphitheatre in Spain were dated in 2001 and re-dated in 2019 with refined dating methods and focus on carbon dioxide that was released in late CO_2 fractions when dissolved in phosphoric acid. The samples were difficult to date because they contained highly soluble, young carbonate contamination that dominated the carbon dioxide from the early stages of the reaction with the acid in the hydrolysis process. They were also rather hydraulic and rich in magnesium, which could have caused delayed hardening. However, there was very little dead carbon contamination so that late carbon dioxide fraction gave uniform ^{14}C ages, pointing to a late 1st c. AD Flavian, or later age of the amphitheatre.

Keywords
Mortar dating, hydraulic mortar, sequential dissolution, CO2 fractions.

1. Introduction

The Roman amphitheatre in *Emerita Augusta* or present day Mérida, was sampled in July 2000 at an early stage of the development of radiocarbon dating methods for lime mortars using partial dissolution and AMS. The goal at this stage was to test the suitability of different samples for radiocarbon dating. The city had been founded by Emperor Augustus in 25 BC for retired soldiers (Ortiz *et al*., 2014), but the consensus was that the amphitheatre would be Flavian, *i.e*. late 1st c. AD (Mateo Cruz, director of the Museum, oral communication at the sampling site). The samples collected were different in many ways compared with what we had encountered earlier including Roman samples from Rome and Torre de Palma, Eastern Portugal (Ringbom *et al*., 2006, 2011; Lindroos *et al*., 2011; Langley *et al*., 2011). The samples were remarkably hard, but they did not classify as hydraulic based on the chemical composition according to a classification used by Van Strydonck *et al*., (1986) and because the carbon yield was similar to that of many lime mortars, between 5.6% and 8.0%. Apparently, however, the hardness seems to be linked to poor permeability for carbon dioxide (CO_2) leading to delayed hardening of the mortars, as all four samples had readily soluble carbonate that yielded unreasonably young ages. One of the samples (Mérida 003) was dated by sequential dissolution (Lindroos *et al*., 2019a) in many CO_2 fractions: The first time in only two CO_2 fractions within 13 min and the second time with total dissolution in 13 hours and six consecutive CO_2 fractions. It turned out that after about 10 min of dissolution with 85% phosphoric acid (H_3PO_4) the effervescing CO_2 started yielding 1st c. AD ages quite consistently. The result from this one sample was published (Hale *et al*., 2003; Ringbom *et al*., 2006; Lindroos et al. 2020. According to the interpretation we made, combining the results from CO_2 fractions 3, 4 and 5 the age was AD 5-90 with 86.9% probability at 95.4% confidence level or AD 100-123 with 8.5% probability (C^2-Test: df 2, T=3.2 (5% 6.0)).

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A comprehensive description of the Roman mortars in Mérida was made recently (Mota-Lopes *et al*., 2018) and an Augustan, early AD 1st c. age was claimed. We therefore decided to revisit the samples in order to test whether the other samples would also yield 1st c. AD ages later on in the hydrolysis process and if it would be possible to get a better resolution for the chronology. Because there were unspecified carbonates causing young ages early in the dissolution progress, we also tested if it was possible to get rid of some of them by heating the sample before hydrolysis.

2. Material and methods

Mérida in SW Spain was the capital of the Roman province *Lusitania.* It was founded as *Emerita Augusta* by Emperor Augustus in 25 BC as a retirement place for merited soldiers from the Cantabrian wars (Ortiz *et al*., 2014). Of the main public buildings, the theatre and amphitheatre are preserved and stand next to each other. They were declared the UNESCO World Heritage site *Emerita Augusta* in 1993 (UNESCO 2015). The amphitheatre is an oval construction 126.3 by 102.7 m with a central arena of 64.5 by 41.2 m (Capello and Galán 1995). The buildings and the geological building materials are described in detail by Mota-Lopes *et al*., (2018). In 2000 we took four samples from the amphitheatre (Fig. 1):

Sample Mérida 001 from the main portal towards the theatre, the south side of an arched passageway, ca 2 m above ground level.

Sample Mérida 002, from the main portal leading to the theatre, arched passage, south wall, ca 1.5 m above ground level.

Sample Mérida 003, from the top floor, passage towards the north east, very thin surface piece, high up on the eastern side of passageway.

Sample Mérida 004, from the top floor, passage towards the north east, surface, high up on the wall.

The samples were inspected visually and with a stereo microscope. From sample 003 we made a thin section for petrographic microscopy. The sample preparation for ^{14}C measurements included:

1. Crushing with plastic covered pliers to <5 mm pieces and powder.
2. Sieving in a mechanical sieve vibrator for 20 min into grain-size fractions >500μm, 301-500μm, 151-300μm, 101-150μm, 76-100μm and ≤75μm. Then wet-sieving/washing of the finest fraction to 76-100μm, 46-75μm, 21-45μm and ≤20μm. The washing included spraying the grain-size fractions with de-ionized water in the sieves.
3. Checking the alkalinity of the sample. In 2000 the procedure included putting about 100 mg of the 301-500μm fraction in a small beaker with the addition of two drops of phenolphthalein solution (2g powder in 100g alcohol) on the sample powder and then filling the beaker up with 10 ml of distilled water. Alkaline samples would turn the water aniline red immediately. When we re-dated the samples in 2018 we also measured the alkalinity with pH strips after one minute and again after 5 min.
4. Choosing a grain-size fraction for dating and examining it with cathodoluminescence. In this study all the hydrolysed and dated sample aliquots were 46-75μm.
5. Analysing the chemical composition with ICP-OES at Geological Survey, Finland or at the Institute of Analytical Chemistry, Åbo Akademi University (reference samples Mérida 005-007). 100mg of the 150-300μm grain-size fraction was dissolved in 1M

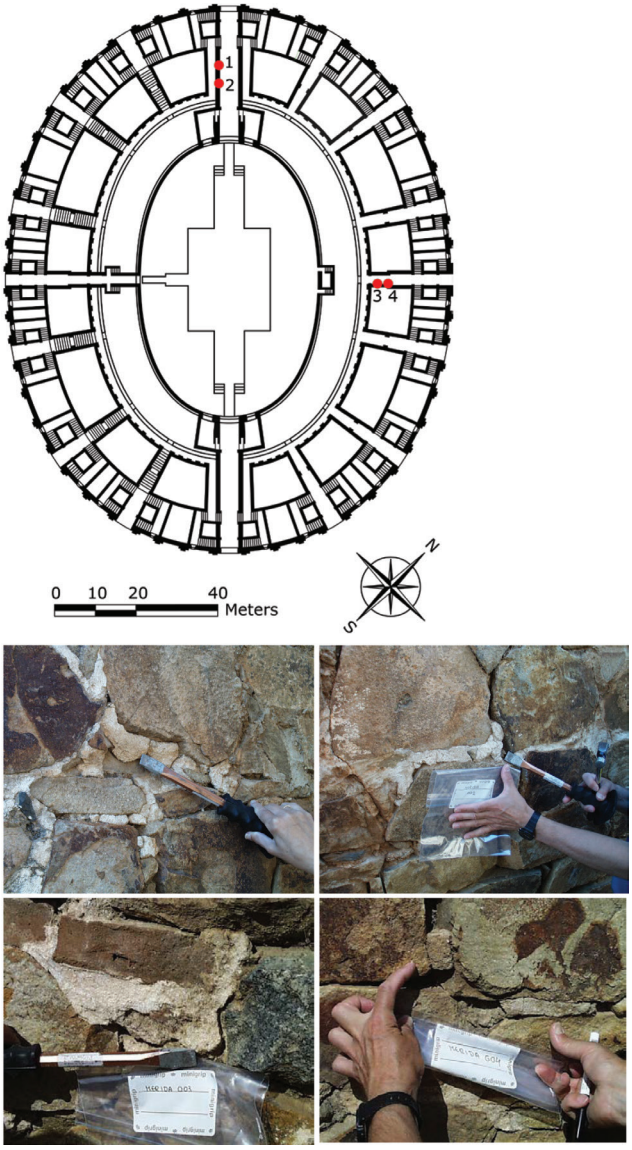

Fig 1. *Sample positions 1-4 in the amphitheatre. Sampling: Upper left, Mérida 001; Upper right, Mérida 002; Lower left, Mérida 003; Lower right, Mérida 004. See details in the text.*

HCl at room temperature for one hour and then diluted to 1/50 and analysed. All elements analysed were well over the quantification limits except for potassium.

6. Aliquots of the dated 46-75μm fractions were analysed with TGA (ThermoGravimetric Analysis) at the Institute of Inorganic Chemistry, Åbo Akademi University. About 10mg powder was heated gradually from 20-900°C, 10°/min in nitrogen atmosphere.
7. Hydrolysis with 85% H_3PO_4 at 0°C from an ice bath. In 2001 the hydrolysis was done at the Aarhus AMS Laboratory. A reactor vessel with a side arm containing the acid was used. When sufficient vacuum was reached in the preparation line, the vessel was tilted, so that the acid flowed onto the sample powder. The hydrolysis was done twice: First to determine the total CO_2 yield and the second time to collect two CO_2 fractions from the beginning of the acid reaction. The first fraction was collected within 14-104s and the second fraction in 3-14min. Samples Mérida 001 and 003 were re-dated. The former in a 10min first CO_2 fraction followed by a 37min second CO_2 fraction and the latter in six CO_2 fractions spanning from 30s to 13h. Re-dating in 2018 was done using the dedicated preparation line (Ringbom, 2014; Lindroos *et al*., 2018, 2019a) at Åbo Akademi University, Finland. Smaller CO_2 increments could now be collected and the resolution between carbonate phases with different ^{14}C ages and dissolution rates was improved. Since the samples had produced reasonable and consistent ages rather late in the hydrolysis progress the number of late CO_2 fractions was increased (see appendix) while some of the early CO_2 fractions were left undated. As it had turned out earlier that young carbonates was the major problem and dead carbon contamination only a minor problem, we also tested dating an aliquot that had been pre-heated to 620°C before hydrolysis with the intention to de-carbonate thermally less stable minerals than calcite.

AMS data is reported according to the convention of Stuiver and Polach (1977) and calibrations were done with the program OxCal 4.3 (Bronk-Ramsey (2017), using IntCal 9 data (Reimer *et al*., 2013). Old Aarhus AMS data come from the EN tandem accelerator and the sample preheating data was performed with the Aarhus HVE 1 MeV accelerator. Zürich measurements were carried out with the ETHZ MICADAS 200 keV accelerator (Synal *et al*., 2007). $\delta^{13}C$ values for old Aarhus data were measured on the GV Instruments Isoprime stable isotope mass spectrometer at the Science Institute in Reykjavik, Iceland on CO_2 splits before graphitization. For later Aarhus data, $\delta^{13}C$ values were similarly obtained from off-line stable isotope mass spectrometry at the AMS Centre. The Zürich data have δ values measured from graphite in the AMS.

3. Results

All samples passed the alkalinity test. pH strips gave values 6-7 and the phenolphthalein test gave a mild reaction, which was only slightly stronger for sample Mérida 002. The samples have a fine-grained aggregate composed of angular quartz and feldspar grains and pieces of mica schist. The angular shape suggests crushing of material rather than use of natural sand. There are only few small voids and some of them have secondary calcite along the walls. Lime lumps are small and scarce, mm-size and they are white to greyish. In CL the calcite binder appears red, indicating a high magnesium content (Marshall, 1988). However, in the 46-75μm grain-size fraction for dating the binder grains are dark. Only a few red grains appear (Fig. 2).

Chemically the soluble material of the samples is unique: *i.e*. completely different from hydraulic samples in Rome, and also quite different from medieval mortars in Mérida, except for sample Mérida 005 from Bishop Masonas Hospital in Mérida that resembles the amphitheatre samples (Table 1). Very low Al, Fe and Si values and high Ca and Mg values result in a very low hydraulic index, 0.02-0.03 defined as:

$$\frac{SiO_2 + Al_2O_3 + Fe_2O_3}{CaO + MgO}$$

A ratio >0.5 is considered to indicate hydraulicity. The chemical analyses verifies the high Mg content deduced from CL color of the binder.

When considering hydraulic properties as a ratio between carbonate CO_2 and the amount of OH^- bound to minerals as crystal water the situation is completely different. In TGA we consider the loss on ignition (LOI) between 550°-800° as CO_2 from carbonate and LOI 250°-550° as crystal water from hydraulic minerals and a ratio CO_2/OH^- (wt) <10 as hydraulic (Modified from Bakolas *et al*., 1998; Moropoulou *et al*., 2005). In that case, samples Mérida 001 and 002 are hydraulic but less so than samples Mérida 003 and 004 (Table 2). The CO_2 yield when the samples were hydrolysed for dating in 2018 is also listed.

Fig. 3 presents the actual TGA profiles

In the appendix there is detailed numerical hydrolysis and AMS data for the samples.

The ^{14}C data is presented below as functions of the dissolution progress variable F defining ^{14}C profiles (F is the ratio between the CO_2 produced at a certain time relative the total CO_2 yield). Sample Mérida 001 is presented in Fig. 4:

Sample Mérida 002 has the following profiles (Fig. 5).

The sample has similar profiles to the others, but the BP ages within the profile (2018) are not overlapping and, even in later fractions, the sample appears about a century younger than the other ones.

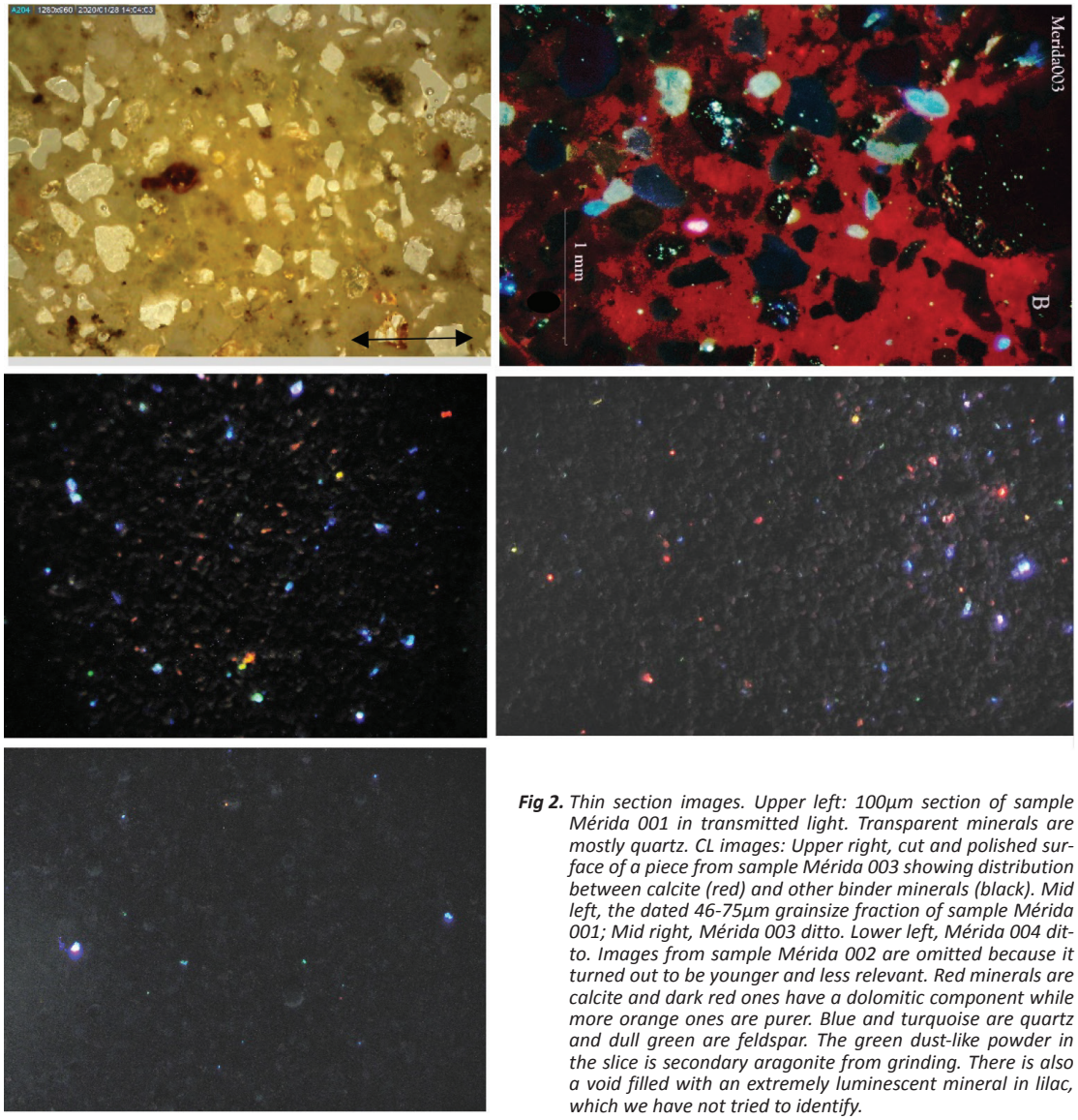

Fig 2. *Thin section images. Upper left: 100µm section of sample Mérida 001 in transmitted light. Transparent minerals are mostly quartz. CL images: Upper right, cut and polished surface of a piece from sample Mérida 003 showing distribution between calcite (red) and other binder minerals (black). Mid left, the dated 46-75µm grainsize fraction of sample Mérida 001; Mid right, Mérida 003 ditto. Lower left, Mérida 004 ditto. Images from sample Mérida 002 are omitted because it turned out to be younger and less relevant. Red minerals are calcite and dark red ones have a dolomitic component while more orange ones are purer. Blue and turquoise are quartz and dull green are feldspar. The green dust-like powder in the slice is secondary aragonite from grinding. There is also a void filled with an extremely luminescent mineral in lilac, which we have not tried to identify.*

Table 1. *ICP-OES analyses of the soluble phase of the samples when dissolved for 1h in 1M HCl at room T. For comparison, 3 samples from the Medieval Bishop Masonas Hospital (Mérida 005, 006 and 007) and 3 samples from the Colosseum are included. Major elements are converted to oxides.*

Sample ID	Al_2O_3	Ba	CaO	FeO	K_2O	MgO	Mn	Na_2O	SiO_2	Sr	Hydraulic
	%	mg/kg	%	%	%	%	mg/kg	%	%	mg/kg	index
Mérida 001	0.08	70.8	30.2	0.21	<0.05	3.12	253	0.09	0.51	578	**0,02**
Mérida 002	0.08	61.9	27.3	0.21	<0.05	4.22	295	0.58	0.68	552	**0,03**
Mérida 003	0.11	60.7	17.9	0.13	<0.05	2.59	133	0.04	0.34	376	**0,03**
Merida 005	0.14	56.9	19.0	0.19	0.02	1.18	225	0.05	0.25	378	**0,03**
Mérida 006	1.69	169	15.3	0.25	0.09	0.71	73.3	0.02	0.36	51	**0,17**
Mérida 007	1.54	184	15.6	0.21	0.12	0.82	61.2	0.01	1.79	85	**0.13**
Colosseum001	5.61	519	15.8	0.54	2.04	0.28	312	1.49	2.65	490	**0,55**
Colosseum002	5.29	621	20.7	0.55	1.85	0.35	165	1.26	3.17	588	**0,43**
Colosseum003	8.01	1660	6.66	0.76	1.87	0.93	355	1.28	3.83	690	1,66

Table 2. *Numerical TGA data and the CO_2 yield in H_3PO_4 hydrolysis for aliquots of the same sample material. For TGA about 10mg of the 46-75µm was heated and in the hydrolysis about 50mg was reacted with 85% H_3PO_4. The acid was taken from an ice bath but it reached room T before the reaction ceased after about 1h.*

Sample	LOI 550	LOI 850	CO_2 yield	Residue	LOI(850°/550°)
ID	(%)	(%)	H_3PO_4 (%)	(%)	ratio
Mérida 001	3.6	26	26	69	7.2 hydraulic
Mérida 002	4.3	27	16	66	6.3 hydraulic
Mérida 003	4.0	19	16	74	4.8 hydraulic
Mérida 004	4.8	22	25	71	4.6 hydraulic
Colosseum 003	2.7	7.6	3.7	81	2.8 hydraulic

Sample Mérida 003 was the subject of four different dating attempts. Fig. 6 shows profiles comparable with those of Mérida 001 and 002. A calibration (AD 25-85; at 58.8% probability and AD 100-120 at 9.4% ditto), based on the boxes 3, 4 and 5 3, 4 and 5 defining a plateau in the profile is presented in Hale *et al*., (2003) and Ringbom *et al*., (2006). In 2018 we tested the storage stability of the sample. The 46-75μm sample powder had been in a non-airtight container for 17 years, however, re-dating, did not reveal significant modern carbon absorption, at least not among readily soluble carbonates.

We tested heating the samples before doing the hydrolysis. In our first experiment we applied pre-heating at 620°C only. Fig. 7 shows the result.

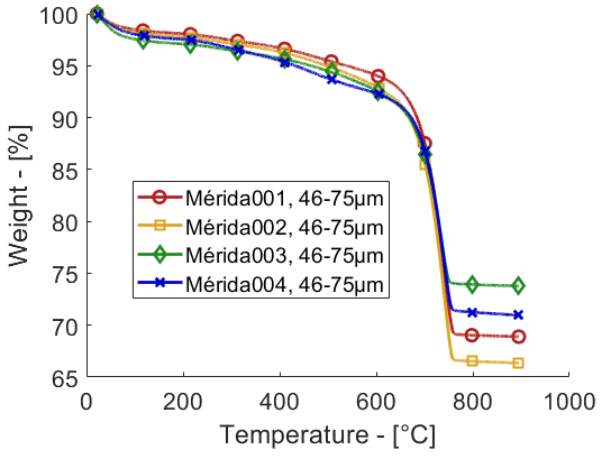

Fig 3. *TGA profiles of the four samples from Mérida. The profiles are similar, but the most hydraulic samples Mérida 003 and 004 have lower CO_2 yields and consequently more residue.*

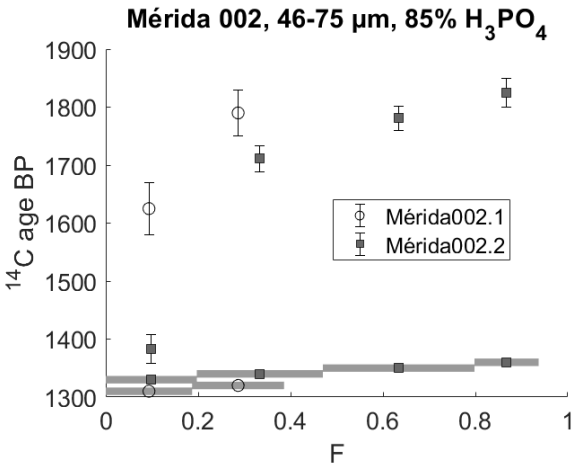

Fig 5. *^{14}C profiles of sample Mérida 002. Open circles present dating in 2001 and black boxes dating in 2018.*

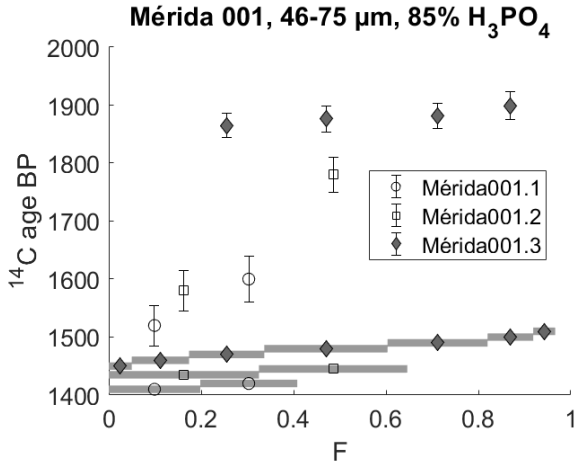

Fig 4. *^{14}C profiles from three different sample preparations of sample Mérida 001: Open circles are from the first dating attempt in 2001. Open boxes are from supplementary measurements later the same year and black diamonds from re-dating 2018. The grey bars along the abscissa denote the size of each CO_2 fraction relative to the total CO_2 yield. In all the following ^{14}C profile plots the size of the CO_2 fractions will be presented the same way without specific mention.*

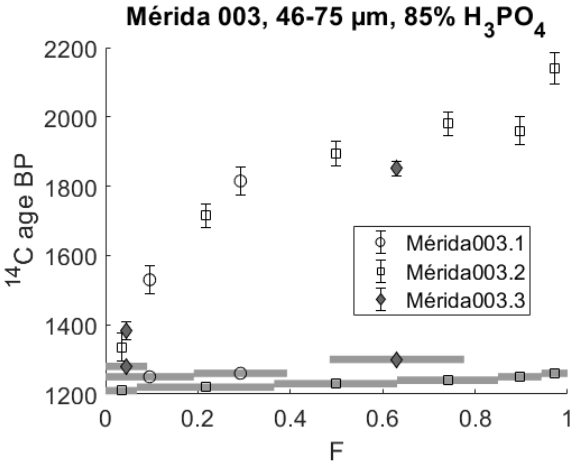

Fig 6. *^{14}C profiles of sample Mérida 003. Open circles denote dating in 2001 and open boxes a profile in six CO_2 fractions dated later the same year. Black diamonds represent re-dating in 2018. For this sample we used the same powder that had been in a non-airtight container for 17 years. The data point with low F value representing initially effervesced CO_2 was measured to ensure that the sample powder had not captured modern CO_2 and grown new calcite on the grains.*

Sample Mérida 004 was also dated both in 2001 and in 2018. Fig. 8 presents the results graphically.

The sample is similar to the others; with readily dissolved young carbonates and ages stabilizing near 1900 BP in later CO_2 fractions.

4. Discussion

Dating of the Mérida amphitheatre was attempted already in 2001 but the ^{14}C ages were not conclusive and only a small part of the data was published, and in graphical form only. It was clear that the samples were difficult to date because in hydrolysis the initially produced CO_2 yielded variable and unreasonably young ages. However, a profile with ^{14}C measurements from six CO_2 fractions yielded 1st c. AD ages for fractions 3, 4 and 5. We therefore also tested the other samples to see whether they would produce similar profiles. It turned out that they did and we now have seven new measurements from rather late CO_2 fractions in the hydrolysis and they all yield ^{14}C BP ages in the range 1850-1930 (Fig. 9). Furthermore the CO_2 of this age is the main component in the three relevant samples comprising >50% of the carbon inventory. One sample, Mérida 002, seems younger, but without a conclusive age.

A combined calibration of the five oldest CO_2 fractions mentioned was conducted (Fig. 10) and based on these samples, the amphitheatre is Flavian and not Augustan. Any post-Flavian age is not discussed here.

Pre-heating the sample Mérida 003 yields similar results but some of the contamination from young carbonates in

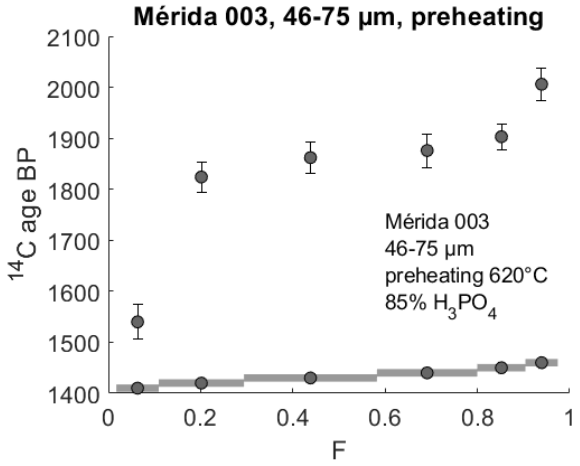

Fig 7. ^{14}C profile from sample Mérida 003 after heating the sample to 620˚C. Some of the young carbonates have been lost and the profile now increases more rapidly in the beginning compared with the profile from the non-heated original sample powder (Fig. 6).

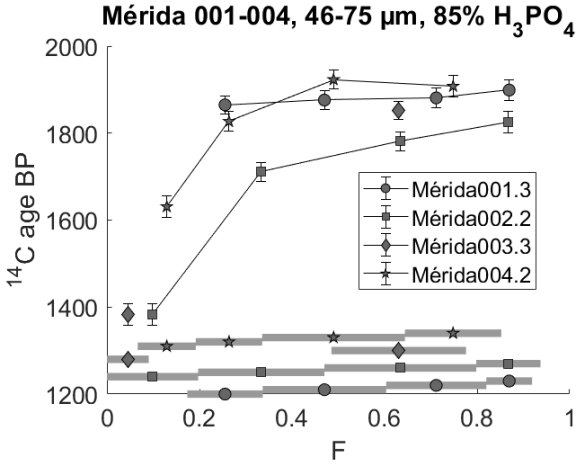

Fig 9. ^{14}C data from 2018. Data points for each sample are connected with lines for clarity. Samples Mérida 001, 003 and 004 yield similar ^{14}C ages after about 20% dissolution (F>0.2) whereas sample Mérida 002 appears younger in all fractions.

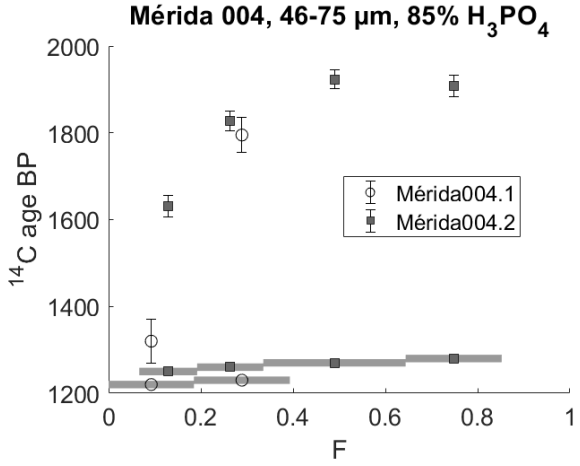

Fig 8. ^{14}C profiles from sample Mérida 004. Open circles, dating 2001. Black boxes, dating 2018.

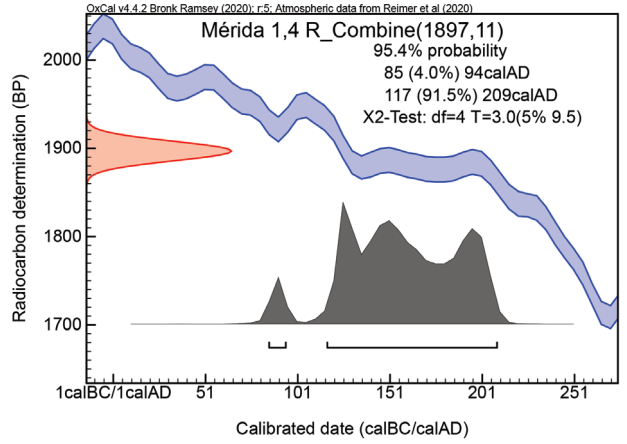

Fig 10. Combined calibration of 5 CO_2 fractions with similar ages. The calibration includes the 3 last fractions from sample Mérida 001 and the two last ones from Merida 004 in Fig 9.

early CO_2 fractions have disappeared. The role of the magnesium-rich binder is unclear. It probably slows down the hydrolysis and the Mg part should release CO_2 earlier than the Ca part when heated (Deer *et al*., 1992). Heating the sample before hydrolysis is yet an unexplored field, which may have potential in the future especially when dating both the thermally released CO_2 and the carbonate residue left for the hydrolysis (Lindroos *et al*., 2019b).

5. Summary

Some mortar samples are difficult to date and require many ^{14}C measurements before a conclusive age can be reached. The situation is especially difficult when the sample has readily soluble carbonate that yields erroneous ages or if the proper binder dissolves very slowly and has a low carbonate content. In the case of the Mérida amphitheatre, there is a relatively slowly dissolving hard binder with a high magnesium concentration and as yet unidentified carbonate minerals, of which at least part of them can be thermally decomposed at lower temperature than the proper binder calcite. The binder should, however, be the main carbonate component and multiple measurements should identify it and reveal its age. Three of the samples point at a Flavian age and none of them at an Augustan age.

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Appendix Merida

Site	Sample nr	Reaction	Carbon	Fraction	^{14}C	±	$\delta^{13}C$	Laboratory
location	run nr	time	yield	size	Age		‰	
	CO_2 fraction nr	(s) from t_0	(tot %)	(relative 1)	(BP)		VPDB	nr
Merida amphiteater	Merida 001.1.1	67	7.4	0-0.198	1520	35	-13.0	AAR-6721.1
Main entance	Merida 001.1.2	727		0.198-0.408	1600	40	-8.96	AAR-6721.2
	Merida 001.2.1	600	6.9	0-0.325	1580	35	-10.1	AAR-6721.2.1
	Merida 001.2.2	2820		0.325-0.646	1780	30	-8.40	AAR-6721.2.2
	Merida 001.3.3	20-115	7.2	0.174-0.337	1865	21	-8.3	ETH-87848
	Merida 001.3.4	540		0.337-0.604	1876	22	-10.2	ETH-87849
	Merida 001.3.5	1380		0.604-0.820	1881	22	-10.9	ETH-87850
	Merida 001.3.6	1980		0.820-0.919	1899	24	-9.3	ETH-89323
Main entrance	Merida 002.1.1	43	8.0	0-0.187	1626	45	-13.7	AAR-6722.1
	Merida 002.1.2	523		0.187-0.386	1790	40	-7.10	AAR-6722.2
	Merida 002.2.2	30-120	4.4	0.273-0.470	1711	22	-7.0	ETH-87851
	Merida 002.2.3	600		0.470-0.798	1781	21	-7.5	ETH-87852
	Merida 002.2.4	1180		0.798-0.937	1825	25	-5.3	ETH-87863
Passage towards NE	Merida 003.1.1	104	5.6	0-0.191	1530	40	-12.5	AAR-6723.1
	Merida 003.1.2	884		0.191-0.394	1815	40	-9.9	AAR-6723.2
	Merida 003.2.1	30	3.2	0-0.068	1335	40	-18.4	AAR-6723.2.1
	Merida 003.2.2	510		0.068-0.365	1715	35	-9.82	AAR-6723.2.2
	Merida 003.2.3	2490		0.365-0.631	1895	35	-9.66	AAR-6723.2.3
	Merida 003.2.4	8790		0.631-0.850	1980	35	-10.4	AAR-6723.2.4
	Merida 003.2.5	15990		0.850-0.943	1960	40	-11.8	AAR-6723.2.5
	Merida 003.2.6	63000		0.943-1.00	2140	45	-10.2	AAR-6723.2.6
	Merida 003.3.1	30	4.4	0-0.093	1383	25	-15.7	ETH-87864
	Merida 003.2.8	530-1950		0.487-0.777	1852	21	-12.2	ETH-87853
"Roasting"	Merida 003 Roa1.1	8	2.4	0-0.0174				
hydrolysis after	Merida 003 Roa1.2	25		0.0174-0.109	1539	34	-13	AAR-30431
heating to 620˚C	Merida 003 Roa1.3	100		0.109-0.294	1821	29	-6	
	Merida 003 Roa1.4	580		0.294-0.582	1859	30	-8	
	Merida 003 Roa1.5	1320		0.582-0.800	1873	33	-10	
Passage towards NE	Merida 004.1.1	14	6.3	0-0.185	1320	50	17.7	AAR-6724.1
	Merida 004.1.2	194		0.185-0.393	1795	40	-7.10	AAR-6724.2
	Merida 004.2.2	6-19	6.8	0.068-0.128	1631	25	-14.1	ETH-87854
	Merida 004.2.3	105		0.128-0.343	1827	23	-7.8	ETH-87855
	Merida 004.2.4	580		0.343-0.658	1923	22	-11.6	ETH-87856
	Merida 004.2.5	1440		0.658-0.870	1908	25	-10.6	ETH-89324