Radiocarbon, Vol 65, Nr 2, 2023, p 323–334 DOI:10.1017/RDC.2023.20

RADIOCARBON DATING OF STRAW FRAGMENTS IN THE PLASTERS OF ST. PHILIP CHURCH IN ARCHAEOLOGICAL SITE HIERAPOLIS OF PHRYGIA (DENIZLI, TURKEY)

Sara Calandra[1,2] • Serena Barone[3,4]* • Emma Cantisani[5] • Maria Piera Caggia[6] • Lucia Liccioli[3] • Silvia Vettori[5] • Mariaelena Fedi[3]

[1]Department of Earth Sciences, University of Florence, Florence, Italy
[2]Department of Chemistry "Ugo Schiff", University of Florence, Sesto Fiorentino (Fi), Italy
[3]National Institute of Nuclear Physics, Unit of Florence, Sesto Fiorentino (Fi), Italy
[4]Department of Physics and Astronomy, University of Florence, Sesto Fiorentino (Fi), Italy
[5]Institute of Heritage Science – National Research Council of Italy, Sesto Fiorentino (Fi), Italy
[6]Institute of Heritage Science – National Research Council, Lecce, Italy

ABSTRACT. Absolute dating of plasters and mortars clearly represents a key information to study important structures and buildings that may have undergone a difficult story starting from their construction. This is for instance the case of the architectures in the archaeological site of Hierapolis (Denizli, Turkey). However, when discussing about the possibility to apply radiocarbon (^{14}C) dating, in this site the presence of different sources of contaminants, due to the geological and geochemical conditions and to the used raw materials, prevents the binder dating. As an alternative, we thus decided to focus on the small fragments of straw that had been used as additives in the mortar/plaster matrices. The fragments were identified, selected and dated using a ^{14}C experimental set-up specifically optimized for microgram-sized samples. The obtained results were satisfying, even though the measured ^{14}C ages also pointed out some possible criticalities in dating such small samples collected from a carbonaceous matrix.

KEYWORDS: Hierapolis archaeological site, mortar, radiocarbon AMS dating, small samples, straw fragments.

INTRODUCTION

As many studies have by now pointed out (Folk and Valastro 1976; Hayen et al. 2017; Lindroos et al. 2020), radiocarbon (^{14}C) dating of aerial mortars or plasters may present many issues, which are ascribable to the vast heterogeneity of this kind of material. Despite the relatively simple dating principle (the anthropogenic calcite $CaCO_3$ resulting from the hardening process of the slacked lime can be collected and dated), the possible sources of contamination can be many, such as:

- aggregates typically added to the slacked lime can contain carbon and can be so fine-grained that their complete separation from the binder can be really challenging;
- some residues from the unburnt original $CaCO_3$ can be present;
- dissolution and recrystallization processes can have altered the original calcite content;
- carbonation can have proceeded over periods longer than typical ^{14}C uncertainties.

A complete characterization of the mortar before ^{14}C dating is thus mandatory to identify the type of mortar and then an efficient procedure that allows us to extract the only carbon deriving from the moment in which the mortar hardens (Toffolo et al. 2020; Cantisani et al. 2021; Daugbjerg et al. 2021). For instance, petrographic analysis gives us information about each component of the mortar (binder, aggregates, and inorganic additives) and about the

*Corresponding author. Email: barone@fi.infn.it

Figure 1 Aerial photo of the St. Philip Church.

identification of raw materials and technologies used for their production (Franzini et al. 2000; Riccardi et al. 2007; Rampazzi et al. 2016; Cantisani et al. 2022). Spectroscopic methods can support us to discriminate between the anthropogenic calcite to be dated and the geogenic calcite (Toffolo et al. 2019; Calandra et al. 2022). Other experimental methods focus on the possibility to separate the two components thanks to their different behavior as a consequence of increasing temperature (Barrett et al. 2021) or acidification (Lindroos et al. 2017). A possible advantage is represented by the presence of lime lumps, which—when formed during carbonation—can be considered as pure binder and can thus be isolated from the rest of the matrix (Al-Bashaireh 2013; Addis et al. 2019; Michalska 2019; Sironić 2019).

However, mortars and plasters can also contain organic carbon that, in principle, can be collected and dated. Plant remains, such as charcoals and straw fragments, might be actually found among the aggregates as mixed with sand and/or powdered rocks. For example, according to a technique widely used in antiquity (Artioli et al. 2019; Maravelaki et al. 2021), straw fragments were added to the mortar mixture as inert to improve the mechanical properties of the material, such as its flexibility and lightness.

In particular, straw fragments can be good candidates for ^{14}C dating, since we can expect that the annual plants they originated from had been cut shortly before their use in the mortar mixture. Thanks to this, the straw ^{14}C concentration can be associated to the moment in which the mortar was set. Moreover, such a material is not likely to be affected by the oldwood problem, which, on the contrary, can influence charcoals (Schiffer 1986; Regev et al. 2011). The isolation and the following processing of such a material can be nevertheless particularly challenging, especially because of the extremely low masses that one may expect to collect. Anyway, organic remains can really represent the only possibility to date a mortar or a plaster in case any inorganic material is completely unsuitable for the measurement, according to what was mentioned above.

Here we discuss the case of the St. Philip Church (Figure 1), located in the archaeological site of Hierapolis of Phrygia, Denizli, Turkey. Hierapolis is one of the great Hellenistic, Roman and

https://doi.org/10.1017/RDC.2023.20 Published online by Cambridge University Press

Byzantine cities of southwestern Turkey, protected by UNESCO since 1988. It was founded in the 3rd century BCE, and it has survived for millennia (D'Andria 2016–2017; D'Andria 2017), even though the Denizli area has been always characterized by dangerous earthquakes and hydrothermal fluid circulation that clearly can affect the conservation of monuments. In the second half of the 6th century CE, a threenave church was built on the eastern hill of Hierapolis, in the area where the tomb of the Apostle Philip has been recently recognized by D'Andria. The church underwent important transformations during the 9th century CE. Then, in the 10th century CE, a catastrophic earthquake seriously damaged the building, which was no longer rebuilt but continued to be used for residential purposes during the Seljuk empire until the 14th century (Caggia 2016, 2022).

The reconstruction of the chronology of the site is not straightforward. Indeed, the presence of a faults system, which is still active today and which has caused—as mentioned—many earthquakes, have contributed to several subsequent collapses of walls and architectural elements over centuries, making the identification of the different archaeological phases of the construction difficult. In such a context, the possibility of dating the construction materials, i.e., mortars and plasters, would be thus important. Anyway, the environment is not favorable to mortar ^{14}C dating. The main critical aspects are the following:

- main raw materials used to produce mortars, also added as aggregates, were travertine and marble provided from local quarries (Caggia 2018; De Giorgi 2018);
- thermal waters, characterized by high concentration of dissolved CO_2, have been responsible for dissolution and corrosion processes and precipitation of various carbonate phases (Vettori et al. 2019a), which may have polluted the mortar mixture with geogenic carbon, thus apparently aging the mortar (Ricci et al. 2020).

As an alternative to mortar/plaster dating, in the following we will present the ^{14}C measurements on straw found in some of the plaster samples from the church.

MATERIALS AND METHODS

Analyzed Samples

Among all the studied materials from the site, seven samples from plasters of St. Philip Church were selected for the present study because they were characterized by the presence of tiny straw fragments in the matrix (see Figure 2 and left-hand side of Table 1): MSF04 and MSF15 were collected from pillars in the northern nave; MSF06 from west wall of northern nave; MSF33 from west wall of the southern nave; US543, US546 and US547 from west wall of the narthex. MSF-labeled samples are related to structures attributed to the early Byzantine period (second half of the 6th century), even though we are not certain that the plaster revetments belong to the original phase. Instead, the narthex can be dated to the middle Byzantine phase (since 9th century CE onwards).

Plasters Characterization and Collection of Samples for Radiocarbon Dating

The plaster samples from the St. Philip Church in Hierapolis were characterized at the Institute of Heritage Science – National Research Council of Italy, Florence (ISPC-CNR). Methods and results have been already published elsewhere (Cantisani et al. 2016; Vettori et al. 2019b) and here only that information that are related to the present discussion is briefly recalled.

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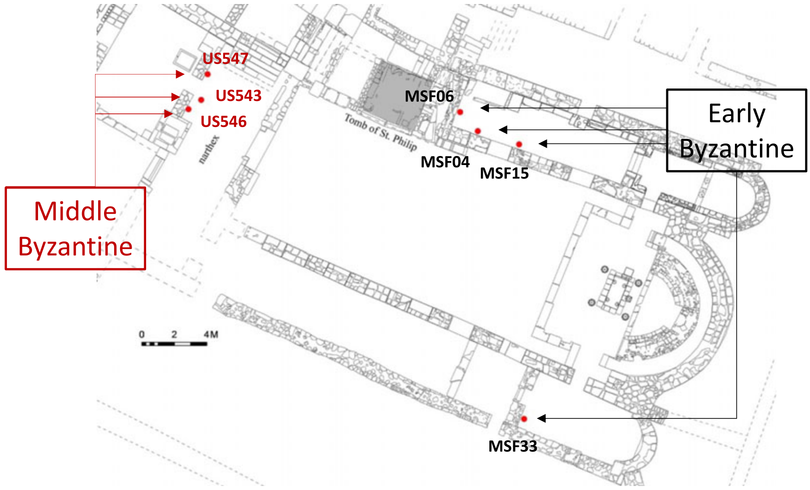

Figure 2 Plan of the church with localization of the plaster fragments containing straw traces analyzed in this paper.

The samples were first observed under a stereomicroscope, with a dedicated camera, and then petrographic analyses on thin sections (thickness 30 μm) were performed with a polarized light microscope (PLM). The mineralogical composition of the bulk samples was studied through X-ray powder diffractometry (XRPD) using a powder X-ray diffractometer (Cu anode, Kα line 1.54 Å).

While under optical investigation, straw fragments were identified as aggregates in the plaster matrices. The mechanical separation of these fragments was performed using a stereomicroscope (Figure 3a), as mentioned above. The separation of the fragments was highly difficult: the bulk samples, which the straw fragments were extracted from, were of the order of 2–3 cm long, while the fibers were visible almost exclusively under the microscope (Figure 3b). The collected materials had extremely thin stems and a length of about 1–2 mm. The presence of some mortar granules still in the separated samples makes estimating the straw mass obtained by the mechanical separation process difficult.

Radiocarbon Measurements: Sample Preparation and AMS

^{14}C-AMS measurements were performed at LABEC in Florence, one of the laboratories of CHNet, the INFN (Istituto Nazionale di Fisica Nucleare) network dedicated to Cultural Heritage.

The first step in sample preparation was clearly focused on pretreatment to remove possible contaminants. In the case of our straw fragments, the most important source of possible exogenous carbon was represented by carbonates coming from the plaster matrix. In addition, considering that we could not have any information about whether the straws might have come into contact with some organic contaminations before being embedded into the plaster, we decided not to completely neglect the possibility of an interference due to such substances. We thus applied an ABA (acid-base-acid) pretreatment, choosing temperature and duration of each of the steps as a balance between the expected cleaning efficiency and the mass preservation of the samples. Straw samples were treated as follows:

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Table 1 Samples from the plasters of the St. Philip Church where straw fragments were identified: location, XRD data and petrographic characteristics (PLM observation) are reported. Minerals are indicated according to (Whitney and Evans 2010). In addition to calcite in all samples, considering the aggregate composition, the prevailing presence of quartz and plagioclase and minor amounts of phyllosilicates (mica-like minerals and chlorites) are evidenced. Gypsum is due to alteration phenomena caused by the sulphation of carbonate binder.

Sample ID	Type	Location	XRD composition*	OM observation
MSF04	External layers of plaster	Third pillar in the northern nave (US 372)	Cal, gp, traces of qz	Air lime binder nonhomogeneous with lumps, addition of straw, only binder, porosity: very high
MSF15	External layers of plaster	Second pillar in the northern nave (US 373)	Cal	Air lime binder nonhomogeneous with lumps, addition of straw, only binder, porosity: very high
MSF06	External layers of plaster	Wall (US 66)	Cal, arg, traces of qz and gp	Air lime binder nonhomogeneous with lumps, addition of straw, B/A: 3/1 –4/1, porosity: medium/high
MSF33	External layers of plaster	Wall of southern nave (US 389)	Cal, qz, traces of ms and chl	Air lime binder nonhomogeneous with lumps, addition of straw, B/A: 3/1– 4/1, porosity: very high
US543	Inner layer of painted plasters	Narthex (plaster of 544 and 545)	Cal, traces of qz	Air lime binder homogeneous, addition of straw, only binder, porosity: very high
US546	Inner layer of painted plasters	Narthex (plaster of 544 wall)	Cal, qz, traces arg	Air lime binder nonhomogeneous with lumps, addition of straw, B/A: 3/1– 4/1, porosity: very high
US547	Inner layer of painted plasters	Narthex (plaster of 545 wall)	Cal, qz, traces pl	Air lime binder nonhomogeneous with lumps, addition of straw, B/A: 3/1– 4/1, porosity: very high

*Cal = calcite; gp = gypsum; qz = quartz; arg = aragonite; ms = muscovite; chl = chlorite; pl = plagioclase.

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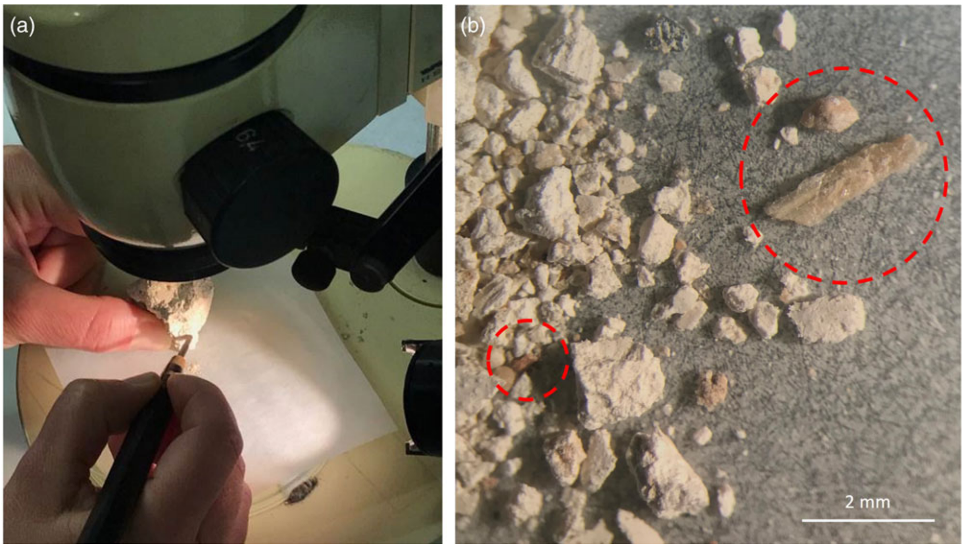

Figure 3 The mechanical separation of straw fragments: (a) using the stereomicroscope, (b) selecting straw from the bulk sample. Straw fragments are highlighted using dotted line red circles. (Please see online version for color figures.)

- 1 hour bath in 1M HCl at room temperature (instead of at 80°C as in our typical procedure), to remove possible carbonate residues from the fragments;
- a quick bath in 0.1M NaOH at room temperature, for up to few minutes, i.e., until the complete "whitening" of the fragments. Both lignin from the straw and possible humic acids contaminants are brownish or dark colored and are soluble in alkaline solutions. Thus, we expect that a lighter color of samples can be associated to a good efficiency of the treatment, suggesting us that we removed the possible organic contaminations;
- 1 hour bath in 1M HCl at room temperature (instead of, again, at 80°C as in our typical procedure).

Since the mass of the straw fragments after the pretreatment was clearly smaller than the initial collected mass, in the order of about a few hundreds of micrograms, graphitization was carried out using our so-called Lilliput graphitization line, specifically designed for microgram-sized samples (Fedi et al. 2020a, 2020b). In particular, this line is optimized for samples sizes corresponding to about 50 micrograms of carbon. Since the individual masses of the recovered straw samples were actually below the aforementioned limit, we decided to combine some of them together following archaeological evidence, such as chronological and functional criteria. Table 2 indicates how the samples were combined, their total masses before combustion and the extracted CO_2. As one can notice, the collected CO_2 from MSF04+MSF15 is lower than the carbon dioxide obtained from the combustion of the other samples, the masses being practically equal: this suggests that MSF04+MSF15 might have some components different from the original material, like for instance a possible residual contamination.

Table 2 Straw fragments samples as they were combined after pretreatment: total masses and collected CO_2 pressures are shown.

Sample ID	Mass (mg)	CO_2 pressure (mbar)
MSF04 + MSF15	0.40 ± 0.05	50 ± 5
MSF06 + MSF33	0.40 ± 0.05	90 ± 5
US543 + US546 + US547	0.35 ± 0.05	80 ± 5

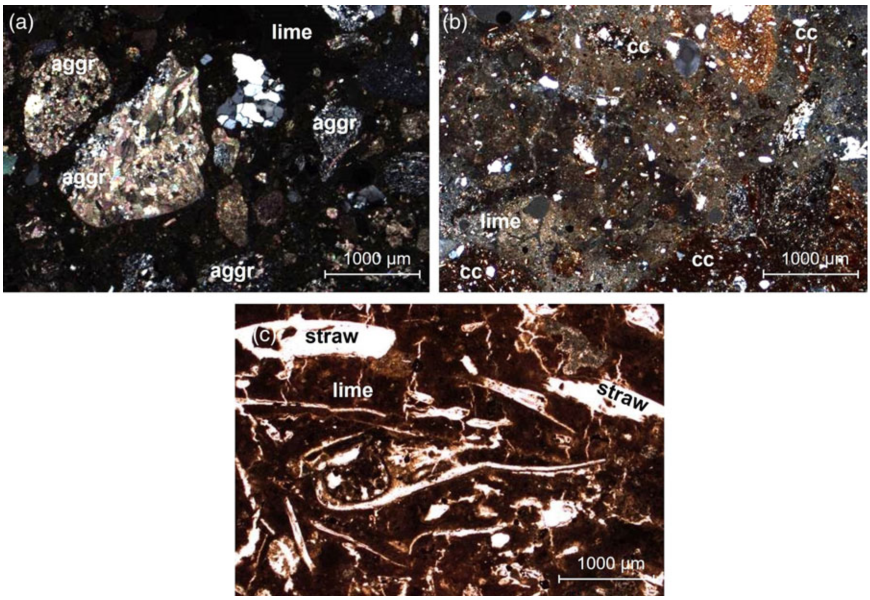

Figure 4 Microphotographs of thin sections of representative types of mortar and plaster in the St. Philip church: (a) mortar with air hardening calcic lime (lime) with a very abundant amount of aggregate (aggr); (b) mortars with air hardening calcic lime with fragments of crushed ceramic (cc); (c) plasters produced by lime with straw and very low amount of aggregate. (by PLM: (a), (b) crossed nicols, (c) parallel nicols).

^{14}C AMS measurements were performed at the AMS beam line of the 3MV Tandem accelerator installed at LABEC (Fedi et al. 2007; Chiari et al. 2021). Routine procedures provide for both $^{14}C/^{12}C$ and $^{13}C/^{12}C$ isotopic ratio measurements during the AMS run. $^{14}C/^{12}C$ ratios of the unknown samples are corrected for background counts, as evaluated measuring blank samples, and for isotopic fractionation (thanks to measured $^{13}C/^{12}C$ ratios). Isotopic ratios are also normalized to the ratios measured during the same run in a set of standard samples with a certified ^{14}C concentration (NIST 4990C, Oxalic Acid II). Samples produced from other standard reference material (IAEA C-7) are also measured as a check of accuracy. Normalized and corrected ^{14}C concentrations are expressed in pMC, according to Stenström et al. (2011). To convert conventional ^{14}C ages to calibrated ages using the software OxCal v 4.4.4 (Bronk Ramsey 2009; Bronk Ramsey and Lee 2013), the IntCal20 curve (Reimer et al. 2020) is employed.

Table 3 Measured ^{14}C concentrations, corresponding conventional ^{14}C ages, and calibrated ages time intervals (when more than one possible range are identified, time intervals are reported considering the lower and the upper extreme, respectively).

Sample ID	^{14}C concentration (pMC)	t_{rc} (BP)	Calibrated age (68% level of probability)	Calibrated age (95% level of probability)
MSF04 + MSF15	75.30 ± 1.4	2278 ± 144	715–120 BCE	775 BCE–5 CE
MSF06 + MSF33	87.20 ± 1.1	1100 ± 105	770–1035 CE	675–1160 CE
US543 + US546 + US547	88.50 ± 1.0	979 ± 88	990–1170 CE	885–1260 CE

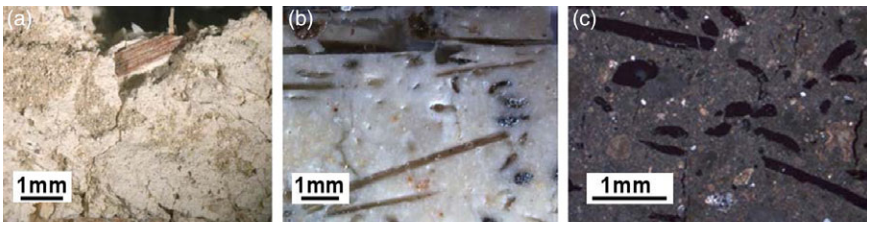

Figure 5 Straw fragments: (a) macroscopic aspect, (b) stereomicroscope image, and (c) microphotograph of thin section of plaster with straw fragments (by PLM: crossed nicols).

RESULTS AND DISCUSSION

As mentioned above, the complete characterization of the mortar and plaster samples from the St. Philip Church has been already reported in Cantisani et al. (2016). Three different categories of mortars/plasters were identified: (1) air hardening calcic lime with a very abundant amount of carbonate and silicate aggregates (see Figure 4a), (2) air hardening calcic lime with crushed ceramic fragments (*cocciopesto*) (see Figure 4b), and (3) air hardening calcic lime with straw and very low amount of carbonate aggregates (see Figure 4c).

Table 1 (on the right-hand side) summarizes the analytical data acquired for those plaster samples belonging to this latter type of material, which were selected in this context because of the presence of sufficient straw fragments (Figure 5). Samples MSF06, MSF33, US546 and US547 are characterized by medium/high porosity and by a binder/aggregate ratio equal to 3/14/1 with a very low amount of aggregates, made of fragments of rocks (schists, quartzite, travertines, and marbles). Aragonite was also found (samples MSF06 and US546): in fact, aragonite was usually found in mortars associated with travertine, present here as aggregate. Samples MSF04, MSF15, and US543 were produced without aggregates and are characterized by very high porosity with shrinkage microcracks and elongated pores due to the loss of straw. The binder is typically not homogeneous and numerous lumps can be recognized, except for US543 that presents homogeneous binder and absence of lumps. The lumps are unburnt fragments and suggest the use of marble as the original stone to produce lime (this was indeed typical of ancient production

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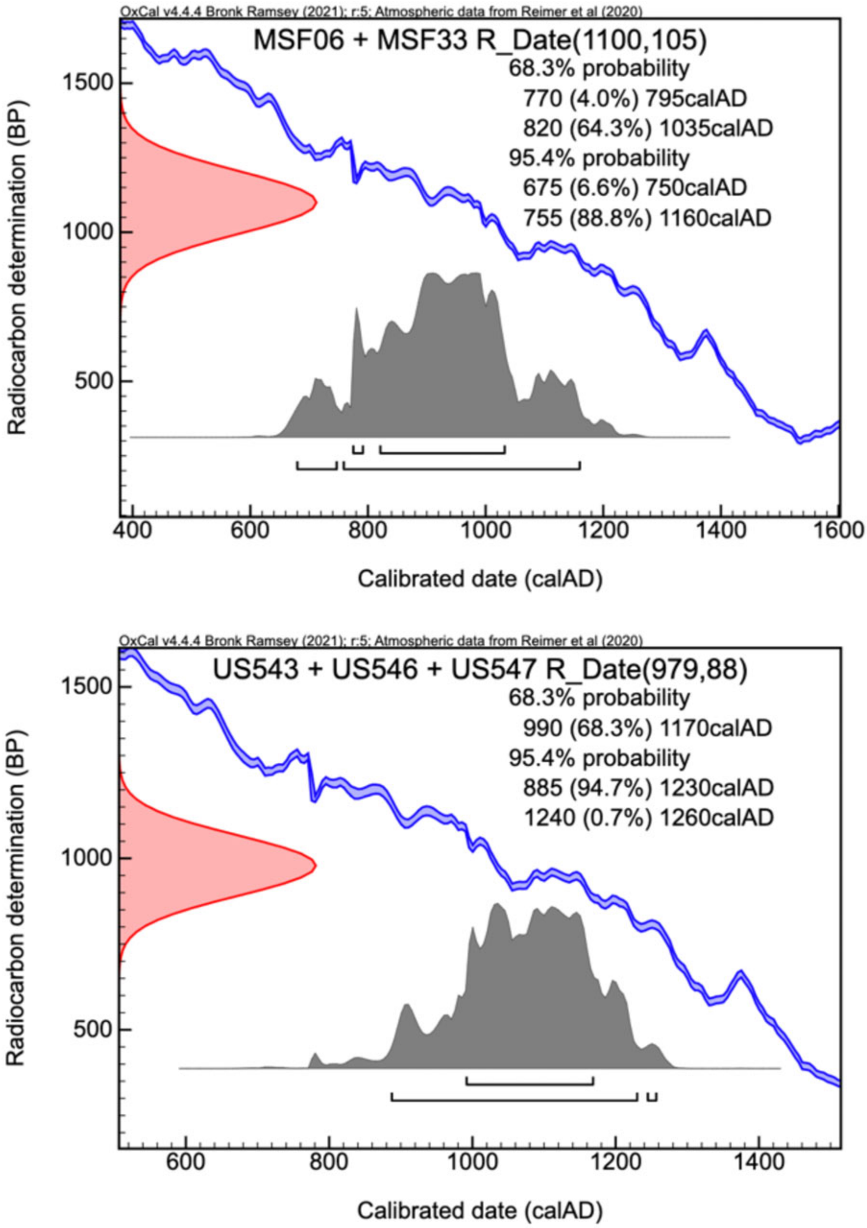

Figure 6 Calibration graphs of samples MSF06 + MSF33 and US543 + US546 + US547.

technologies). As far as ^{14}C dating is concerned, geogenic carbonate aggregates and unburnt fragments of marble are "extremely dangerous" dead carbon bearers. In addition, the medium/high binder porosity indicates the presence of secondary calcite, which is likely to have been caused by dissolution and recrystallization processes, due to rain and hydrothermal percolating waters.

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All things considered, as already anticipated, the reported data show that these plasters were not suitable for ^{14}C dating and therefore all the attention focused on the recovered straw fragments. Table 3 shows the results of the AMS measurements of the straw samples.

The sample MSF04 + MSF15 appears to be older than expected and in fact the calibrated age does not match the archaeological evidence. This, together with the low CO_2 collection yield after combustion, suggest that the sample could be still contaminated by residual "old" $CaCO_3$ from the plaster. Probably, some marble traces have not been fully removed during the pretreatment. In addition, complete combustion of marble by elemental analyzer is not easily achievable, thus the possible presence of $CaCO_3$ residues can explain the low carbon dioxide yield.

MSF06 + MSF33 and US543 + US546 + US547 samples are consistent with the expected age based on archaeological evidence (see also Figure 6). MSF06 + MSF33 sample appears to be slightly older than the other dated sample. In fact, considering that the church was built and later modified as the consequence of several earthquakes, two different construction phases can be expected at least in accordance with the time range during which the church was used. However, unfortunately, the experimental uncertainties on the measured ^{14}C concentrations do not allow us to statistically discriminate between them. After all, the higher uncertainties that were achieved in this case are consequence of the extremely small samples that were prepared and measured: in our set-up, smaller masses correspond to lower ion currents extracted and measured into the accelerator and thus to lower count rates.

CONCLUSIONS

^{14}C dating of mortar is very challenging. Depending on the context, the application of ^{14}C is not feasible, since many factors can interfere with the dating. This is just the case of the St. Philip Church, in the archaeological site of Hierapolis. Mortars and plasters are highly heterogeneous as for the binder composition and the aggregates. Moreover, the presence of carbonate aggregates, secondary calcite and hydraulic binder suggest that direct ^{14}C dating is not ideal. In this situation, the presence of straw fragments as organic inclusions drew our attention: straw fragments represent indeed a very good candidate for ^{14}C dating, since their ^{14}C concentration can be strictly linked to the moment in which the mortar was set.

Here, we have shown that even such small quantities of straw fragments can be identified and isolated from the matrix. Due to the extremely small masses that can be collected and recovered after the mandatory pretreatment, the measurement of the ^{14}C concentration is only possible using a set-up which is specifically optimized for microgram-sized samples. Obtained results of MSF06 + MSF33 and US543 + US546 + US547 are satisfying, since they are consistent at large with the expected archaeological dates. Nevertheless, our data also underline the possible limitation of using this kind of material: a milder pretreatment as the one we chose to preserve as much mass as possible could not be sufficient to remove all the possible contaminants.

Dating straw and, in general, organic inclusions in mortars and plasters can thus represent a valid alternative to the direct dating of the carbonate material, even though the critical aspects connected to sample preparation may suggest that this cannot be applied in routine operations.

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