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A FIELD GUIDE TO MORTAR SAMPLING FOR RADIOCARBON DATING**

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Radiocarbon dating of mortars is a method for absolute dating of historical mortared stone structures. Successful mortar dating studies have answered chronological questions, while other studies have revealed that mortar samples can have complications and contaminants. These can cause inconclusive results even with present state-of-the-art techniques. Previous

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research shows that adequate and proper sampling of mortar samples is of fundamental importance for a conclusive radiocarbon analysis. Therefore, this article thoroughly reviews the processes and environmental factors that may cause problems for successful radiocarbon dating of mortar samples, and presents best-practice sampling strategies for radiocarbon mortar dating.

KEYWORDS: RADIOCARBON DATING, MORTAR SAMPLING, DELAYED HARDENING, DEAD CARBON, RECRYSTALLIZATION

INTRODUCTION

An important task in archaeology is to establish the absolute chronology of a site under investigation. Often the radiocarbon method is employed to achieve this using a site's organic samples, such as charcoal, seeds, bones and wood (Bayliss 2009). However, organic material embedded in historical mortars can have an inherent age older than the associated masonry (see the Discussion section below). Moreover, sites dating to antiquity are often lacking in suitable organic materials for radiocarbon dating, in turn making it difficult to obtain absolute chronologies for these sites. Radiocarbon dating of lime mortar may offer a good alternative by basing the chronologies of the often well-preserved buildings on radiocarbon analysis of mortared stone constructions (Labeyrie and Delibrias 1964; Stuiver and Smith 1965; Baxter and Walton 1970). Radiocarbon dating of mortar dates the actual time of construction or renovation when the mortar hardened. Furthermore, mortar can be widely available throughout an archaeological site, covering different stages of construction and sections of the site itself (Heinemeier *et al*. 2010; Thomsen 2019). If successful, mortar dating can provide the building history of a site, and contribute to the answering of questions in classical and medieval archaeology (Nawrocka *et al*. 2009; Heinemeier *et al*. 2010; Hajdas *et al*. 2012; Ortega *et al*. 2012; Ringbom *et al*. 2014; Van Strydonck 2016).

Radiocarbon dating of mortars, however, does have certain complications, which can lead to inconclusive mortar dating results. This article reviews the complications associated with radiocarbon dating of mortars, and it reviews three commonly encountered types of mortar. Its purpose is to present sampling strategies to reduce the number of mortar samples affected by avoidable complications, and thereby increase the proportion of mortar samples with conclusive radiocarbon dating results. It also emphasizes the importance of multi-fraction dating (see the section Mortar dating studies below), without which complications can even lead to undetected errors (Stuiver and Smith 1965; Baxter and Walton 1970; Nonni *et al*. 2018; Ponce-Anton *et al*. 2018).

Other important aspects of mortar dating are characterization methods and preparation methods, which this article also discusses briefly, but not in depth, as its focus is on presenting strategies for the sampling of mortar for radiocarbon dating. The presented sampling strategies can increase the number of unproblematic samples, but they cannot guarantee straightforward results. Therefore, any mortar dating study should greatly consider characterization and preparation.

The principle of radiocarbon dating of lime mortars

The working principle of radiocarbon dating lime mortars relates to the production process of lime mortar (Fig. 1) (Labeyrie and Delibrias 1964; Stuiver and Smith 1965; Van Strydonck 2016). To produce lime mortar, limestone must be heated to $>$ 900°C to achieve complete thermal decomposition of its main mineral calcite ($CaCO_3$). The limestone then releases CO_2 and transforms to quicklime (CaO). The quicklime is mixed with water, where it reacts to form slaked lime or portlandite ($Ca(OH)_2$). Finally, the slaked lime is mixed with an aggregate, typically sand,

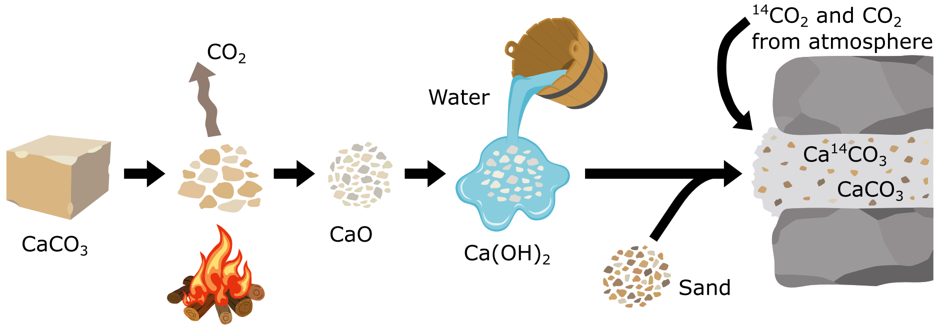

FIGURE 1 *Production and hardening of lime mortar. Lime mortar absorbs atmospheric CO_2 as it hardens, and stores a ^{14}C signal. Source: Redrawn from Hale et al. (2003).*

and wet mortar is ready for use in construction. As wet mortar hardens, the slaked lime reacts with atmospheric CO_2 and transforms back to calcite ($CaCO_3$), which is the binder of the lime mortar. In this way, the mortar binder captures the atmospheric ^{14}C signal at the time of hardening and stores it as calcium carbonate ($CaCO_3$), which can then be radiocarbon dated.

Mortar dating studies

Mortar dating studies have worked with different types of mortar and from a broad range of locations, for example, England, Finland, France, Greece, Israel, Italy, Jordan, the Netherlands, North Macedonia, Poland, Spain, Sweden, Switzerland and Syria (e.g., Baxter and Walton 1970; Folk and Valastro 1976; Van Strydonck *et al*. 1986; Zouridakis *et al*. 1987; Lindroos *et al*. 2007; Nawrocka *et al*. 2009; Heinemeier *et al*. 2010; Al-Bashaireh and Hodgins 2011; Ortega *et al*. 2012; Ringbom *et al*. 2014; Hajdas *et al*. 2012; Lichtenberger *et al*. 2015). Essential to the success of ^{14}C mortar dating is first and foremost to sample the correct material, then characterization to identify possible contaminants and lastly choosing the most appropriate preparation method. The sections Characterization methods and Preparation methods (see below) provide brief overviews of such methods.

Mortar ^{14}C preparation methods aim to separate the allochthonous carbonate (i.e., non-atmospheric carbon not originating from a hardening of the lime mortar) from the binder's autochthonous carbonate (i.e., the carbon fraction produced *in situ* by the uptake of atmospheric CO_2). Mortar ^{14}C preparation methods achieve this discrimination by a combination of mechanical and chemical separation. The mechanical separation is based on binder carbonate being soft and porous compared with hard limestone contaminants (Folk and Valastro 1976; Van Strydonck *et al*. 1986; Heinemeier *et al*. 2010). Mechanical processes that favour crumbling material and small particles therefore enrich binder carbonate in the small grain fraction. Chemical separation employs acid or high temperature decomposition (up to 900°C), where the binder carbonate releases its carbon faster or slower than the contaminant carbonates (Labeyrie and Delibrias 1964; Van Strydonck *et al*. 1986; Heinemeier *et al*. 1997).

The quality of the preparation methods should be checked by extracting multiple fractions of CO_2 from the aliquot for dating. Accelerator mass spectrometry (AMS) can then radiocarbon date such a series of CO_2 fractions (Heinemeier *et al*. 1997). A ^{14}C age profile of multiple fractions serves as a diagnostic tool for evaluating the homogeneity of the ^{14}C signal and conclusiveness of the dating results. Heinemeier *et al*. (2010) state objective criteria for the conclusiveness of mortar dating based on ^{14}C age profiles of fractions. With these criteria, some mortar samples are well suited for the extraction of pure binder material. For example, in a study of 150 lime mortar samples from the interior of medieval churches on the Åland Islands, Finland, Heinemeier *et al*. (2010) found 80% of the samples yielded conclusive results. Furthermore, 50% of the 150 samples had independent age control from dendrochronology or ^{14}C dating of wood. Of these, 95% of the mortar dates agreed with their age control.

When ^{14}C analysis of several sequential CO_2 fractions demonstrates that samples provide reliable and accurate dates, mortar dating can present a key to the chronology of a stone construction.

TYPES OF MORTAR

Mortar belongs to a broader category of binder materials, also called cements in cement chemistry nomenclature (Lea and Desch 1937; Blake 1968; Dodson 1990; Pavia and Bolton 2000). Binder materials have an internal cohesion, which on hardening glues itself and any embedded material together. A mortar is a mixture of a binder material and a fine aggregate, usually with sand-sized grains (Dodson 1990). Concrete is a mixture of a binder material, a fine aggregate and a coarse aggregate (Dodson 1990), and concrete can cast structural units. Given the context of various binder materials, different types of mortar exist at archaeological sites (Lucas 1926; Lea and Desch 1937; Anstetts 1948; Thomsen 2019). Mortar was of concern to ancient builders and architects, and the Roman architect and author Vitruvius devoted a section to ancient mortars in his *De architectura* (first century BCE) (Vitruvius and Morgan 1914a). This section is often used as a point of departure, and it is widely cited in works on ancient mortar. However, one must be aware that while Vitruvius is cited as a standard work and point of reference, it is indeed not known to which degree his work was available across the Roman Empire and later. In fact, the analysis of historical mortars shows mixing ratios varying from Vitruvius' work (Pavia and Bolton 2000; Hobbs and Siddall 2011). This section presents three types of mortar common in archaeological contexts: lime mortar, pozzolana mortar and cocciopesto mortar.

Lime mortar

Production of lime mortar involves mixing slaked lime and an aggregate (often sand) (Fig. 1). Vitruvius advises the right proportions for the mixture (Vitruvius and Morgan 1914a). For pit sand, use three parts sand to one part lime. For river-sand or sea-sand, use two parts sand to one part lime. Lime mortar hardens through the reaction:

$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O \tag{1}$$

Note that lime mortar consumes CO_2 and produces water when hardening, and therefore it cannot harden under water (Hobbs and Siddall 2011); this type of mortar is defined as non-hydraulic. Equation (1) shows how lime mortar stores atmospheric CO_2 as binder $CaCO_3$. This process makes lime mortar the simplest type of mortar, with a clear preservation of the atmospheric ^{14}C signal.

Pozzolana mortar

Roman pozzolana mortar is of a special type, mixed from pozzolana and slaked lime (Blake 1968; Lechtman and Hobbs 1987; Lancaster 2009; Marra *et al*. 2013). Pozzolana is a volcanic ash abundant in the region surrounding the Bay of Naples, and it originates from the Campi Flegrei volcanic field, including Mount Vesuvius (Hobbs and Siddall 2011; Marra *et al*. 2013). Pozzolanas in Rome usually comprise local volcanic rocks (Jackson *et al*. 2010). Vitruvius advises mixing the powder from the country from Cumae to the promontory of Minerva, i.e. the country of the bay of Naples, and slaked lime in the proportions two to one (Vitruvius and Morgan 1914b).

Pozzolana mortar has good compressive strength, and it can set under water as well as in air (Oleson *et al*. 1984; Lechtman and Hobbs 1987; Binda and Baronio 1988; Dodson 1990). The term 'hydraulic mortar' denotes a mortar able to set under water (Anon. 1858; Dodson 1990; Pavia and Bolton 2000; Hobbs and Siddall 2011). This hydraulic ability comes from direct reactions between the slaked lime and the pozzolana, which eliminates the need, and thus also partly the capture, of atmospheric CO_2.

The chemistry of setting of pozzolana binder is more complex than that of slaked lime. Pozzolana is a very fine and highly porous powder of weathered volcanic glass and silicate and hydroxide minerals rich in Al, Na, K, Mg, Ca and Fe (Massazza 2003; Hobbs and Siddall 2011). When mixing slaked lime and pozzolana, the slaked lime provides a highly alkaline environment for pozzolana. The alkaline environment and large surface area of the powder enhances reactivity, and pozzolanic reactions occur. For simplicity, equation (2) only considers the pozzolanic reaction of silica, SiO_2 (Dodson 1990):

$$Ca(OH)_2 + \text{pozzolana} + H_2O \rightarrow \text{CSH (binder)} \qquad (2)$$

Chemistry considering further pozzolanic reactions of aluminosilicates and other oxides can be found in the literature (Lechtman and Hobbs 1987; Dodson 1990; Hobbs and Siddall 2011). Cement chemists use CSH as a shorthand notation where C = CaO, S = SiO_2 and H = H_2O. The reaction product CSH, or calcium silicate hydrate, is the binder in pozzolana mortar and pozzolana concrete. The shorthand notation is useful because it avoids specifying calcium silicate hydrate stoichiometry, which is not universal (Dodson 1990). Note in equation (2) how pozzolana sets through hydration, consuming water. Indeed, pozzolana mortar can harden under water, and in the presence of air it must be kept wet during hardening (Hobbs and Siddall 2011).

From the perspective of mortar dating, the absence of atmospheric CO_2 in equation (2) is important. Consequently, pozzolana that hardened underwater has no immediate interaction with the atmosphere, and we do not recommend sampling it for radiocarbon dating. For pozzolana that hardened in the presence of air, excess $Ca(OH)_2$ can react with atmospheric CO_2 to produce $CaCO_3$, as described in equation (1) (Ringbom *et al*. 2014). Lime lumps composed mainly of calcite are commonly found in pozzolana mortars, and pozzolana hardened in the presence of air has the potential for carbon dating (Stuiver and Smith 1965; Lindroos *et al*. 2018; Nonni *et al*. 2018).

Cocciopesto mortar

Cocciopesto is a mortar mainly made from lime and crushed fired ceramics and pottery (Lancaster 2009; Hobbs and Siddall 2011; Ringbom *et al*. 2014). There are pre-Roman cocciopesto floors in Sicily, so the technique may originate from Greece or North Africa (Harden 1962). The modern name cocciopesto originates from Renaissance Italy, while the Romans called it *opus signinum* (Hobbs and Siddall 2011). Vitruvius describes one recipe with pounded tile mixed with lime, in the proportions three to one (Vitruvius and Morgan 1914c). In another recipe he advises mixing river or sea-sand, lime and burnt brick pounded up and sifted in the proportions two to one to one (Vitruvius and Morgan 1914a).

Cocciopesto is hydraulic, waterproof and has greater compressive strength than lime mortar (Pavia and Bolton 2000; Ringbom *et al*. 2014; Mota-Lopez *et al*. 2018). Smaller domestic buildings rarely used volcanic pozzolana mortar but used cocciopesto instead (Harden 1962).

Europe has widespread use of artificial pozzolanas, such as cocciopesto, due to the absence of local sources of natural pozzolana (Pavia and Bolton 2000). Clay minerals are hydrous aluminosilicates, and examples of clay minerals are kaolin, mica, talc, etc. (Barton and Karathanasis 2002). Aluminosilicates are abundant in both clays and volcanic pozzolana ash, and the two materials have chemical similarities (Massazza 2003; Hobbs and Siddall 2011). Firing clay, at a not too high temperature (450–900°C), followed by crushing to increase fineness, can activate pozzolanic properties in clay (Dodson 1990; Mota-Lopez *et al*. 2018). Similarly to pozzolana, cocciopesto hardened in the presence of air has the potential for radiocarbon dating on account of the excess $Ca(OH)_2$ reacting with atmospheric CO_2. However, the results obtained in the international mortar dating intercomparison study (MODIS) show that radiocarbon ages of cocciopesto are affected by certain complications that make this potential problematic to realize (Hajdas *et al*. 2017). The sections Sampling hydraulic mortar and Discussion below elaborate further on complications for cocciopesto.

GENERAL SAMPLING STRATEGY

This section presents a general mortar-sampling strategy which applies regardless of specific mortar dating complications or the types of mortar involved.

It is highly recommended to use a hammer and a chisel when extracting a mortar sample from masonry. The sample size must be about a handful of mortar (50–100 g) and have enough material for sample characterization, preparation and AMS radiocarbon dating. Avoid drilling with drill-bits, as this will alter the grain distribution of the mortar. This obstructs the desired mechanical separation of the sample by gentle crushing, which is intended to separate the mortar binder from the aggregate without significant alteration of the grain size distribution of the sample (Heinemeier *et al*. 1997; Nonni *et al*. 2018; Ponce-Anton *et al*. 2018).

When sampling from a building unit, consider which structural components are likely to be original and which are likely later repairs or refurbishments (Heinemeier *et al*. 2010; Ringbom *et al*. 2014). The use of a site's archaeological excavation's documentation system can help ensure congruence. Furthermore, it is important to have clearly defined chronological research questions formulated before sampling. For example, if the aim is to date the age of construction, then repairs or refurbishments should be avoided, whereas if the usage period of the building is the aim of the investigation, then repairs or refurbishments will be of greater importance. For original construction, rework is rare for masonry near inner wall corners or out-of-the-way locations such as rough walls in attics or basements. Mortar protruding between the stones is ideal for sampling because it secures the original mortar unaffected by surfacing, later repair or repointing. Conversely, a surface plaster covering a wall is a poor location as it may be a renovation.

When dating a building, extract *in situ* mortar samples from a unit of the building. This is more likely to guarantee a secure context between the age of the building unit and the dating of samples (Boaretto 2009). Avoid sampling scattered mortar on the ground as it may have been transported from different units, or organic acids may have weathered it. This is especially important for collapsed ruins and rubble. A secure context sample is a fundamental first step for a successful ^{14}C mortar result, no matter what research the chronology aims for.

COMPLICATIONS IN MORTAR DATING

This section discusses carbon, not reflecting atmospheric CO_2 at the time of hardening, associated with mortar samples and mortar dating. Samples affected by such complications may produce inconclusive results as discussed in the section Mortar dating studies. The issues are multiple, and they can come from the mortar itself (i.e., the type of mortar) or from interaction with the environment up to the present day. This section also presents strategies to address the complications, and it encourages the reader to use them in the field. The section Sampling mortar on-site presents a more compact mortar-sampling guide. Communication and understanding between field workers and radiocarbon workers are paramount for successful mortar dating. Ideally, a person from the dating team should participate in the sampling.

Recrystallization

Complication Recrystallization, also called diagenesis, occurs in circumstances where binder $CaCO_3$ is not entirely stable, but may grow new crystals (Boaretto 2009; Heinemeier *et al*. 2010; Nawrocka *et al*. 2009; Lindroos *et al*. 2020). In the presence of ambient water, mortar binder $CaCO_3$ may dissolve, react with fresh atmospheric CO_2 and redeposit (MacLeod *et al*. 1991). Recrystallized $CaCO_3$ then has a ^{14}C age younger than the time of construction (Fig. 2). Ambient water, affecting mortar, can come from various weathering sources: rainfall, surface water and groundwater.

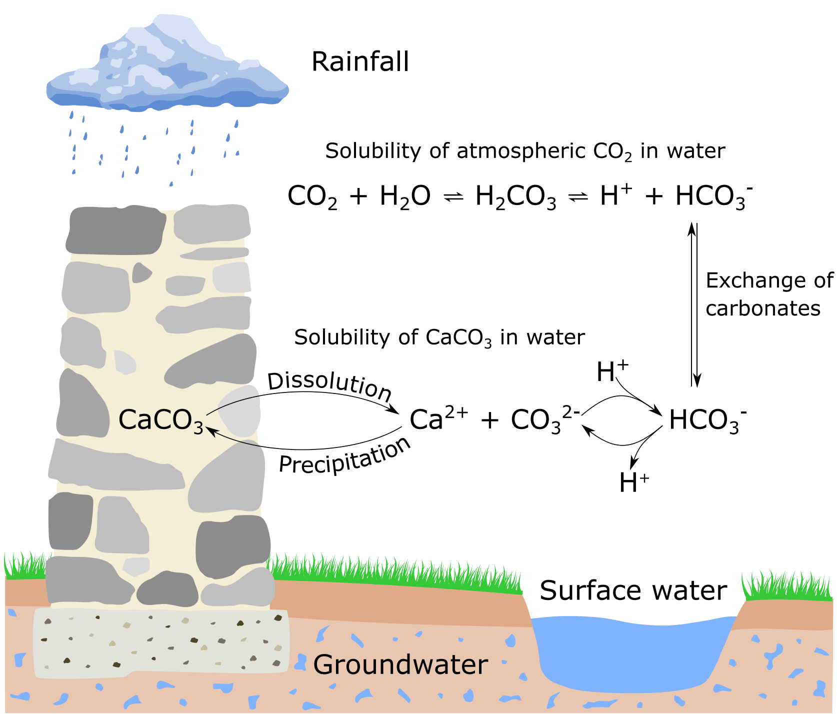

FIGURE 2 *Mortar exposed to wet conditions is at risk of having its $CaCO_3$ binder rejuvenated with younger atmospheric CO_2. If this happens, it compromises the original ^{14}C signal.*

Strategy To avoid recrystallization, the best place for sampling is from a sheltered and dry place in the building, and preferably above ground (Heinemeier *et al*. 1997; Ringbom *et al*. 2014). In churches, Heinemeier *et al*. (2010) carefully sampled from the sheltered space under the roof and above the masonry vaults, and achieved high rates of conclusive results and accurate dates (see the Introduction above). Lindroos *et al*. (2018) found recrystallization in the mortar from the ancient bridge in Parma, Italy, and this structure was exposed to weathering and in contact with water. The ancient bridge is located where the ancient road Via Emilia crossed the Parma stream in the city of Parma. In the laboratory, petrography can produce further information on the binder matrix and identify secondary calcite depositions (Hobbs and Siddall 2011; Nonni *et al*. 2018).

Delayed hardening

Complication Delayed hardening constitutes a complication due to the possibility of mortar hardening significantly later than the time of construction (Zouridakis *et al*. 1987; Sonninen and Jungner 1989; Van Strydonck *et al*. 1989; Heinemeier *et al*. 2010; Lindroos *et al*. 2020). Mortar hardens by uptake of atmospheric CO_2, and hardening starts at the surface and progresses inward by diffusion of CO_2 from the surface. The hardening slows down as it progresses because the innermost parts are reachable only by diffusion through partly hardened mortar. Consequently, the inner parts of a thick wall are prone to a delay in the order of decades, or centuries, compared with the time of construction, and such samples produce an age that is too young (Pesce *et al*. 2012). Furthermore, it is common that such samples are alkaline and can absorb modern CO_2 in the field or laboratory when exposed to ambient air. Figure 3 illustrates delayed hardening in a wall. Delayed hardening also relates to mortar chemistry. If dolomitic limestone has been used as a raw material, the Mg component is carbonated very slowly (Michalska *et al*. 2017).

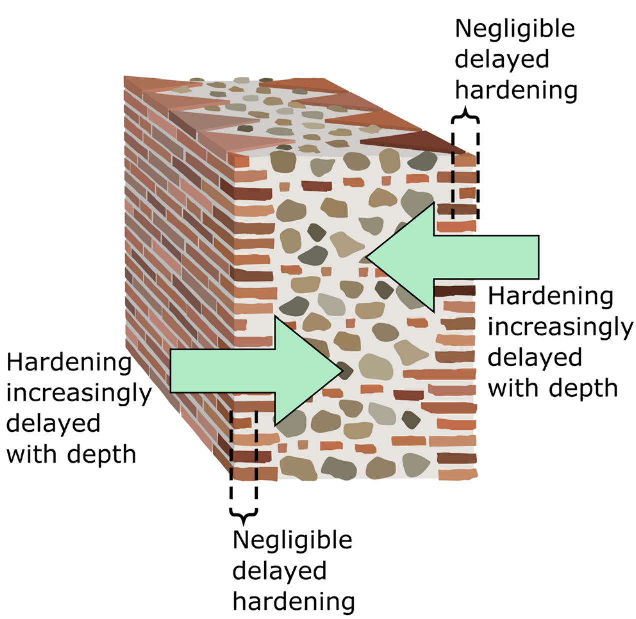

FIGURE 3 *Hardening of mortar starts at the surface of a wall, and deeper parts harden with an increasing delay (Sonninen and Jungner 1989). It can even contain uncarbonated portlandite ($Ca(OH)_2$) that absorbs modern CO_2 when sampled. Near the surface of a wall, delayed hardening is negligible, and the ^{14}C age relates to the time of construction.*

Strategy The ideal sample is from a depth close enough to the surface of the wall to avoid delayed hardening, yet deep enough to avoid near-surface recrystallization, due to weathering, or possible later surface repairs or repointing. The exact sampling depth depends on factors such as the type of mortar and the mortar's permeability. This paper advises a sampling depth of a few centimetres or less. Clean the outermost layer gently with a chisel and sample the mortar from the cleaned surface with a clean chisel (Heinemeier *et al*. 2010). Sometimes there is original mortar still protruding between the stones of rough, unsurfaced walls, especially in inner corners that are difficult to access. Such mortar is ideal for sampling because it hardened quickly and is clearly unaffected by later activity. Heinemeier *et al*. (2010) used this strategy and reported results with high rates of conclusiveness and accuracy (see the Introduction above). Lindroos *et al*. (2020) have radiocarbon dated mortar from various wall depths and demonstrated that delayed hardening increases with depth.

A solution of 2% phenolphthalein dissolved in alcohol can test mortar samples for alkalinity. Spray the solution on a lump of mortar. If the sample turns pink, the mortar is alkaline. An alkaline mortar sample is likely to have absorbed modern CO_2, and it should be rejected for AMS dating (Lindroos *et al*. 2020). Samples for radiocarbon mortar dating must be unstained by phenolphthalein.

Groundwater and soil moisture

Complication Groundwater and soil moisture may contain solute geological carbonates (i.e., an infinite ^{14}C age) and organic carbon (Al-Bashaireh and Hodgins 2012; Nonni *et al*. 2018). When mortar is in contact with groundwater and soil moisture, the solute geological carbon can interact with the mortar binder's $CaCO_3$. As moisture evaporates from the soil, geological carbonates can also precipitate directly onto the mortar. This disturbs the stored ^{14}C signal, and the mortar's ^{14}C age is shifted towards the higher age of the groundwater's carbon (Lubritto *et al*. 2018).

Strategy To avoid the complication of groundwater and soil moisture interaction, sample mortar above ground level in a dry and sheltered location (Heinemeier *et al*. 1997). In addition, petrography and analysis of ^{14}C profiles of multiple CO_2 fractions can help identify depositions of secondary calcite and assess the degree of carbon exchange (Nonni *et al*. 2018). Using petrography, Nonni *et al*. (2018) identified secondary calcite attributed to groundwater in buried mortar from the Temple of Minerva Medica, Rome, and found contamination of old material in the radiocarbon results. Meanwhile, some studies with above ground sampling do not report groundwater issues (Heinemeier *et al*. 2010; Pesce *et al*. 2012; Barrett *et al*. 2020a).

Mortar components containing dead carbon

Complication Mortar dating has further complications, best addressed by the preparation method rather than sampling strategy. Dead carbon (i.e., infinite ^{14}C age) is a complication associated with mortar containing grains of geological carbonate minerals (Labeyrie and Delibrias 1964; Baxter and Walton 1970). The mortar is then a mix of the binder's historical carbonates and the geological carbonates, with the ^{14}C signal of the latter containing effectively no ^{14}C atoms. The sand used as mortar aggregate can be a source of geological carbonate grains. Incomplete burning of limestone when producing quick lime also constitutes a problem of contamination (Stuiver and Smith 1965; Heinemeier *et al*. 1997). Fragments of unburned limestone then end up in the mortar as a source of dead carbon. The situation with dead carbon can be more complicated if a sample contains grains of partially, burned limestone. If binder material is not separated effectively, dead carbon can interfere with mortar dating and produce erroneously high ages.

Strategy Dead carbon and incomplete burning are complications distributed throughout a batch of produced mortar, and sampling strategy can do little to avoid it. In the laboratory, petrography, cathodoluminescence microscopy and scanning electron microscopy (SEM) may identify grains of aggregate carbonate minerals and incompletely burned limestone (Lindroos *et al*. 2014; Michalska *et al*. 2017; Michalska and Pawlyta 2019). An efficient preparation method for extracting binder material may be able to discriminate against the dead carbon contaminants. Geological limestone grains are relatively large and can be sieved, so fine grain size fractions are enriched in binder $CaCO_3$ (Heinemeier *et al*. 2010; Ortega *et al*. 2012). Thus, it is important to sample carefully so the production of aggregate splinters is minimized. Avoiding the use of power tools (e.g., drilling) is particularly recommended in this regard.

Reliability of the results

Complication In some locations, complications can make radiocarbon dating of mortar challenging. In this case and other situations, mortar dating can benefit from having multiple independent age determinations and supplementary materials for dating.

Strategy Take multiple samples from each structural unit, so multiple age determinations are possible. The guide advises at least three samples are analysed per building unit. This will enable the evaluation of the reliability of the result. Heinemeier *et al*. (2010) state objective criteria for conclusiveness by comparing multiple samples from the same structural unit. For example, studies of the Åland churches and water-management installations in Jerash, Jordan, benefitted from a comparison between samples (Heinemeier *et al*. 2010; Lichtenberger *et al*. 2015). When taking samples, keep journals with good photographic documentation of extraction locations. It must be possible to return for repeat sampling or verification of the context.

Mortar may contain small organic inclusions identifiable by eye, magnifying glass, microscope or X-ray imaging. Organic inclusions are typically charcoal, but could also be wood fragments, hairs, straws, grains or seeds. Only short-lived inclusions are useful for a secure radiocarbon dating in this context. In the Åland churches, archaeologists found wood fragments from surface/bark of the original scaffolding embedded in the mortar, which yielded consistent dates (Heinemeier *et al*. 2010). Identification and extraction of such inclusions enable dating with standard ^{14}C dating methods.

Sampling hydraulic mortars

Complication Hydraulic mortars (i.e., pozzolana or cocciopesto) that hardened under water should be avoided due to the missing interaction and absorption of atmospheric CO_2 (see Types of mortar section). Hydraulic mortars hardened in the presence of air may be useful for ^{14}C dating. However, the complications discussed above for non-hydraulic mortars will apply for hydraulic mortars too. Nevertheless, pozzolana and cocciopesto mortars are found in eventful periods of classical archaeology and can therefore not be completely avoided.

Pozzolana and cocciopesto mortars have several characteristics that make radiocarbon dating challenging. Both types of mortars have a low permeability for air, so deep parts of the masonry have poor contact with atmospheric CO_2, and delayed hardening is often encountered (Ringbom *et al*. 2014; Nonni *et al*. 2018). For example, the pozzolana sample Rome 025 from Trajan's Market showed increasingly delayed hardening with a sampling depth > 3 cm (Lindroos *et al*. 2020). Further, a pozzolana mortar stays chemically active after the time of construction and recrystallizes new carbonates (Michalska 2019). If the pozzolana ash's parent volcanic system interacts with deposits of carbonate minerals, the pozzolana itself may contain dead carbon (Jackson *et al*. 2010). Furthermore, volcanic activity can contaminate the atmosphere with dead CO_2 and the pozzolana with CO_2-rich bubbles in the volcanic glass. If both the absorption of modern CO_2 and dead carbon strongly affect a pozzolana sample, it can be unsuited for conclusive mortar dating (Lindroos *et al*. 2018).

Strategy If a site features different types of mortar, keep in mind that non-hydraulic lime mortar is the most suitable for conclusive mortar dating (Ringbom *et al*. 2014; Van Strydonck 2016). Circumstances in the field, or the research question at hand, may make it necessary to attempt dating a hydraulic mortar (Nonni *et al*. 2018; Lindroos *et al*. 2020). Whenever possible, well-preserved lime lumps should be considered for dating in hydraulic mortars (for examples, see Lindroos *et al*. 2018). However, if the site has sufficient degrees of freedom, choosing non-hydraulic lime mortar samples will increase the proportion of samples yielding conclusive mortar dating results.

In the field, it is possible to make certain observations to assess if a mortar is hydraulic or non-hydraulic. If the mortar is from the waterproofing part of a cistern or other water installation, it is probably hydraulic by necessity (Ringbom *et al*. 2014). Hydraulic mortars typically have a greyish colour, while non-hydraulic lime mortar is whiter. Weathering can change the colour of mortar, so be sure to assess colours on a fresh surface. Hydraulic mortar is harder than lime mortar, and scratching or peeling with a pick or knife can assess hardness. These are relative comparisons, and it is a great help to have reference samples of known non-hydraulic lime mortar and hydraulic mortar. Laboratories have further not-in-field methods to distinguish hydraulic and non-hydraulic mortar, for example, different hydraulic indices (Van Strydonck *et al*. 1986; Bakolas *et al*. 1998; Moropoulou *et al*. 2000).

In pozzolana mortar, dark fragments of volcanic rock are sometimes identifiable by eye. Some volcanic fragments have a reddish colour, notably pozzolana rossa (Jackson *et al*. 2010). Be careful not to mistake them for ceramic fragments. An alkalinity test is especially useful for pozzolana and cocciopesto mortars, which are likely to react with contemporary ambient CO_2, leading to less chance of suitability for dating as noted above.

The hydraulic mortar cocciopesto is recognizable by its abundant ceramic content. Many non-cocciopesto mortars contain a few ceramic fragments, but in cocciopesto, ceramics are very abundant. Ceramic dust gives it a reddish colour, and reddish ceramic fragments from millimetres to centimetres are identifiable by eye (Ringbom *et al*. 2014; Michalska *et al*. 2017).

SAMPLING MORTAR ON SITE

The previous section presented the complications and strategies for mortar sampling. This section presents Table 1, which has the information in compact form as a useful reference in the field.

Table 1 *Compact mortar-sampling guide for fieldwork reference: summary of the complications and strategies considered in this study*

Complication	*Strategy*	*Benefit*
Sample context	Extract samples *in situ* from intact building units	Settles the potential debate about the relation between a mortar sample and a building unit
	Assess if samples come from the original structure or later repairs	Clarifies if a mortar sample is suitable to answer a specific chronological question, for example the date of first construction
Recrystallization	Sample mortar from standing, sheltered building units. Sample above ground level	Avoids weathering, rainfall and groundwater, which can cause recrystallization and rejuvenate the sample age
Groundwater and soil moisture interaction	Sample mortar above ground level	Avoids groundwater and soil moisture, whose geological carbonates can shift the ^{14}C age to a higher age
Delayed hardening	Clean the outermost layer of mortar with a chisel and use a clean chisel to sample from the cleaned surface. Avoid sampling deep into the mortar	Avoids mortar from deep inside structures where delayed hardening is an issue
	Separate a small piece from the sample and test for alkalinity with 2% phenolphthalein in alcohol. Samples sent to radiocarbon dating must be unstained by phenolphthalein	Alkalinity is an indicator of chemically active mortars that can be reactivated when broken. Screen for problematic samples in the field
Reliability of the result	Collect multiple samples from each structural unit	Multiple same-age samples, from the same structural unit, enable evaluating reliability by comparing results
	Look for organic inclusions in the mortar	Wood fragments, bark and other organic inclusions may enable more routine ^{14}C methods. Charcoal in the mortar can yield results too old
Hydraulic mortar	Assess the type of mortar. Prioritize sampling non-hydraulic lime mortar whenever possible	Non-hydraulic lime mortar has the highest success rate in mortar dating. Other types are more difficult or inadvisable to date

Circumstances in the field may very well prevent the fulfilment of all points in the sampling strategy. In this case, it is an option not to follow all the points. The consequence of relaxing the sampling strategy is to expect a lower proportion of samples with conclusive dating, which could be acceptable in an archaeological project. In such a situation, this guide advises that one follows as many of the points in the strategy as reasonably possible.

CHARACTERIZATION AND PREPARATION

As stated in the Introduction, sampling strategies alone do not guarantee a successful mortar dating study. Characterization methods and preparation methods are also important, and they are briefly discussed below. Readers should consult the primary literature for further details.

Characterization methods

Mortar samples, or powders mechanically separated from mortar samples, are usually subjected to one or more preliminary characterization methods. These give valuable information on the contents of a mortar sample and possible complications or contaminants as described in this paper. A survey of 56 papers, covering the period between 1964 and 2020, yielded 40 mortar dating studies with characterization methods. Figure 4 displays the occurrences of the characterization methods, and the following section discusses the selected methods.

Petrography produces thin sections of samples for polarized light microscopy and that identifies the mineralogy of binder and aggregate grains (Folk and Valastro 1976; Nawrocka *et al*. 2009). Petrography is the most used characterization for mortar dating studies (Fig. 4), and it enables identification of recrystallization and limestone and dolomite aggregate with dead carbon.

Cathodoluminescence microscopy places a powder, or a slice, from a sample in a vacuum chamber, and a beam of electrons excites the sample's luminescent properties (Lindroos *et al*. 2007; Al-Bashaireh and Hodgins 2012). The colour of the luminescence is mineral dependent, and different minerals are identifiable. Cathodoluminescence can identify recrystallization, grains of limestone and dolomite with dead carbon, but not delayed hardening. Mortar dating studies widely use cathodoluminescence (Fig. 4).

SEM places a sample in a vacuum and a focused beam of electrons interacts with the sample, which then emits secondary electrons. The detection of secondary electrons while the beam scans the sample enables the reconstruction of a sample's topology with circa nanometre resolution. Some SEM setups enable energy-dispersive X-ray spectroscopy (EDS or EDX) where the electron beam excites the sample's atoms, which then emit element-characteristic X-rays, and the

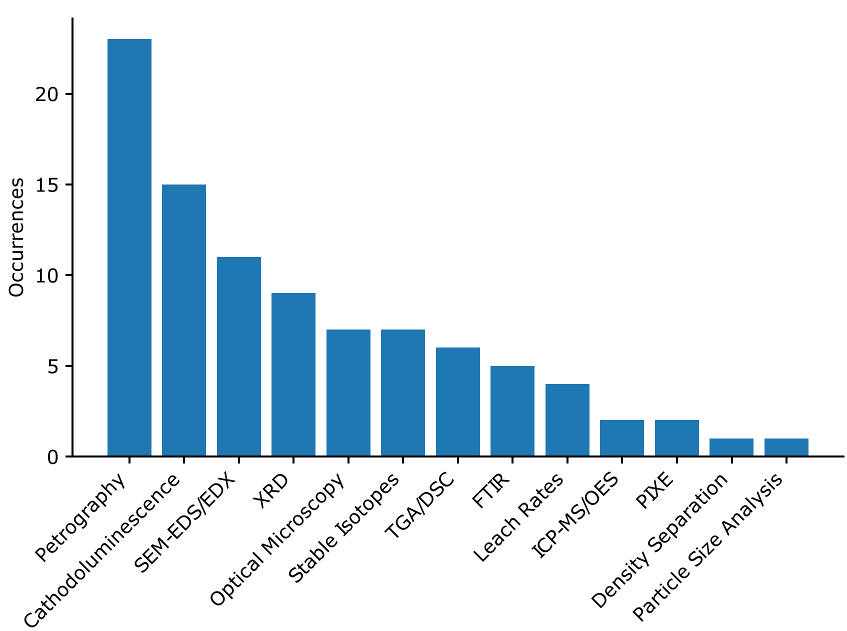

FIGURE 4 *Occurrences of characterization methods in 40 mortar dating studies between 1964 and 2020.*

sample's spatial distribution of elements can be mapped. SEM can identify recrystallization and aggregate grains, for example, limestone and dolomite with dead carbon (Michalska *et al*. 2017; Mota-Lopez *et al*. 2018; Ponce-Anton *et al*. 2018).

Further characterization methods in mortar dating studies have also proven useful for the identification of possible contaminants (Fig. 4). Examples are X-ray diffraction (XRD) (Ponce-Anton *et al*. 2018), optical microscopy (Pesce *et al*. 2009), stable isotopes (Pachiaudi *et al*. 1986; Van Strydonck *et al*. 1986; Ambers 1987), thermogravimetric analysis or differential scanning calorimetry (TGA/DSC) (Ponce-Anton *et al*. 2018; Michalska 2019), Fourier-transform infrared spectrometer (FTIR) (Chu *et al*. 2008; Poduska *et al*. 2012), leach rates (Lindroos *et al*. 2007; Michalska and Czernik 2015), inductively coupled plasma mass spectrometry or inductively coupled plasma optical emission spectrometry (ICP-MS/OES) (Al-Bashaireh 2016;

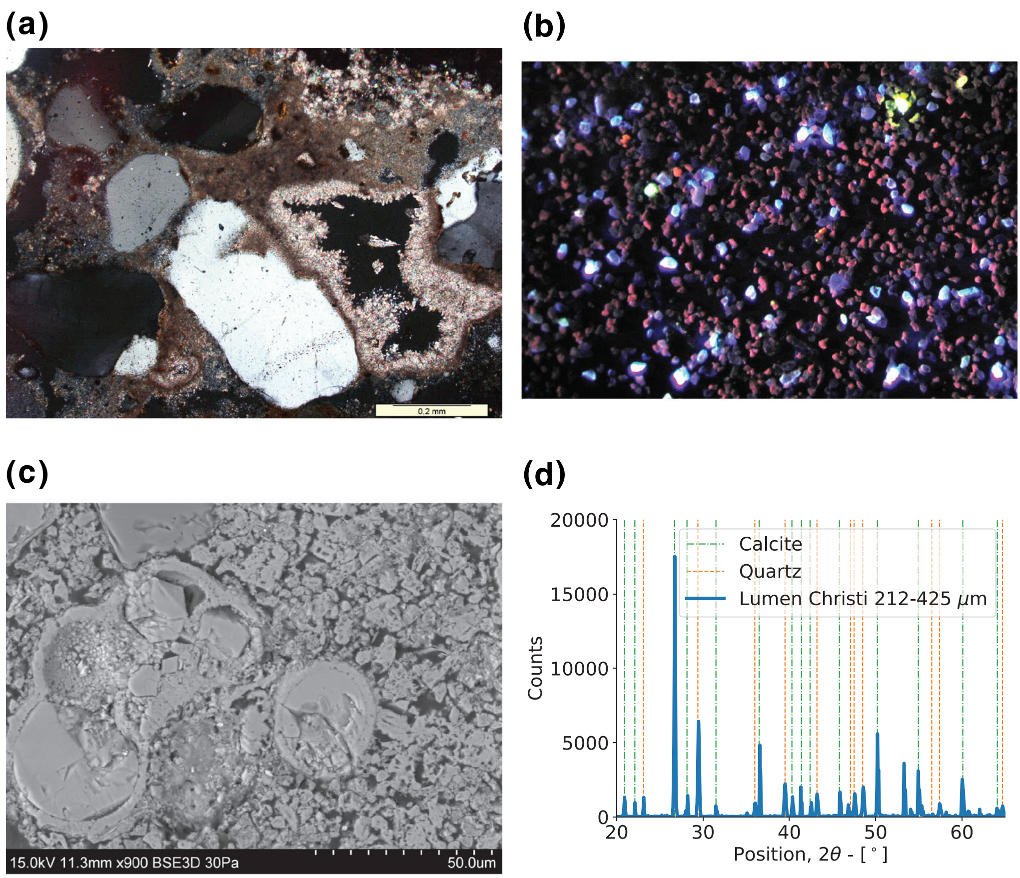

FIGURE 5 *(a) Petrograph showing recrystallization within the binder and in the pores. Sample A from Góra Przemysła, Royal Castle, Poznan, Poland. (b) Cathodoluminescence of powder with a grainsize of 75–125 μm. Blue grains are quartz; green grains are feldspar; bright yellow grains are zircon; bright red grains are limestone or dolomite; and dark red-brown grains are binder calcite. Sample 2, Hammarland church, Hammarland, Finland. (c) Scanning electron microscopy (SEM) image with foraminifera within the mortar binder. Sample from Jericho, Palestine. (d) XRD spectrum showing peaks from quartz and calcite. Powder with a grainsize of 212–425 μm. Spectrum originally published in Barrett et al. (2020a). Sample from Lumen Christi, Northern Ireland, UK.*

Michalska 2019), proton-induced X-ray emission technique (PIXE) (Lindroos *et al*. 2014), density separation (Toffolo *et al*. 2020) and particle size analysis (Ortega *et al*. 2012). Figure 5 presents examples of selected characterization results.

Preparation methods

Identifying the most appropriate preparation method is difficult as this will often depend on the type of contaminants present in the sample. For complicated mortar samples, it may be necessary to test and refine the preparation in order to radiocarbon date samples conclusively.

The cryo-breaking method aims to improve the mechanical separation of binder material compared with conventional crushing of samples (Marzaioli *et al*. 2011; Michalska *et al*. 2017). Some studies use cryo-breaking and report accurate results (Marzaioli *et al*. 2011; Michalska *et al*. 2017; Barrett *et al*. 2020a).

The cryo2sonic method is a mechanical separation (Marzaioli *et al*. 2011; Ortega *et al*. 2012; Nonni *et al*. 2018). It differs from cryo-breaking by having steps involving suspension and ultrasound. The method is an option when dealing with dead carbon contamination. There are studies where cryo2sonic successfully suppresses dead carbon (Ortega *et al*. 2012; Nonni *et al*. 2018), and others where dead carbon persists (Nonni *et al*. 2018; Ponce-Anton *et al*. 2018).

Second portion of forced suspensions is a mechanical separation method (Michniewicz *et al*. 2007; Michalska 2019). Michalska (2019) reports second portion of forced suspension reducing contamination from unburned limestone, and mortar radiocarbon dates in agreement with existing chronologies.

Sequential dissolution is a form of chemical separation that performs well for mortars with moderate complications from dead carbon, recrystallization or alkalinity (Folk and Valastro 1976; Van Strydonck *et al*. 1986; Lindroos *et al*. 2007; Michalska and Czernik 2015). A severe dead carbon contamination can challenge sequential dissolution (e.g., Lichtenberger *et al*. 2015).

Thermal decomposition is a chemical separation method (Labeyrie and Delibrias 1964; Daugbjerg *et al*. 2020; Toffolo *et al*. 2020). The literature reports thermal decomposition conclusively and accurately dating complication-free mortar samples and mortar samples complicated by recrystallization or alkalinity. Samples with considerable dead carbon can challenge the method (Barrett *et al*. 2020b; Daugbjerg *et al*. 2020).

Isotope fractionation corrections of bulk mortar dating is a method that aims to determine a sample's content of dead carbon from aggregate and correct the dating result accordingly (Pachiaudi *et al*. 1986; Ambers 1987; Van Strydonck *et al*. 1989). Michalska and Pawlyta (2019) present a study where dating results are accurately corrected.

The pure lime lumps technique uses local inhomogeneity in the mortar where binder material is concentrated and aggregate material absent (Van Strydonck *et al*. 1992; Pesce *et al*. 2009; Pesce *et al*. 2012; Lindroos *et al*. 2014). Lime lumps probably form during lime storage, mixing or slaking. Other lump types that are undesirable for dating also exist, and Pesce *et al*. (2012) list under-burned limestone, over-burned limestone and burned limestone containing high concentrations of Si compounds. The pure lime lump technique identifies and extracts material from pure lime lumps, which is inherently free of contamination from external sources of $CaCO_3$, for example dead carbon. When sampling pure lime lumps on-site, select mortar with small, round and white lumps. In the laboratory, optical stereomicroscope confirms pure lime lumps as white lumps having a floury appearance, being soft and delicate, while other types of lumps are denser and look like stone (Pesce *et al*. 2012). After identification, and still under the stereomicroscope, a scalpel or a needle can clean and extract the pure lime lump material, which is then available for chemical separation.

DISCUSSION

The ^{14}C mortar preparation methods were recently tested in an international mortar dating intercomparison study (MODIS) involving five radiocarbon laboratories (Hajdas *et al*. 2017; Michalska *et al*. 2017). The MODIS comparison revealed good agreement between ^{14}C mortar analysis and independent age control for lime mortars, whereas more complicated mortars (e.g., cocciopesto) were unable to provide accurate ^{14}C results.

Numerous attempts of ^{14}C dating of cocciopesto and pozzolana mortars are found in the literature, and almost all describes serious difficulties (Jackson *et al*. 2010; Ringbom *et al*. 2014; Michalska *et al*. 2017). Without exception, Roman cocciopesto samples have given result far too young, which serves as a serious warning against using cocciopesto mortars for ^{14}C analysis (Ringbom *et al*. 2014). There are, however, examples of successful pozzolana ^{14}C dating even with conclusiveness rates as high as 50% (Ringbom *et al*. 2014; Nonni *et al*. 2018). In contrast, lime mortars are reported to be very successful, in particular if the above guidelines are followed (Lindroos *et al*. 2007; Nawrocka *et al*. 2009; Heinemeier *et al*. 2010; Ortega *et al*. 2012; Ringbom *et al*. 2014; Van Strydonck 2016; Michalska 2019).

A fundamental assumption for the radiocarbon method is that a sample's ^{14}C content, after cessation of biological or chemical CO_2 uptake from the environment (e.g., a plant's death), is subject only to radioactive decay and not artificially elevated or reduced in concentration due to natural or anthropogenic causes, that is, the sample is a closed system. As has been described here, this is often not the case for mortars in building structures, which may interact chemically with the surrounding environment through various processes, that is, an open system. Therefore, it is important to stress an open mind towards alternative materials such as wood or bark inclusions. Tubbs and Kinder (1990) have studied wood/charcoal inclusions in an early attempt to take advantage of the small sample size in AMS to date mortar. However, they found that inclusions of wood and charcoal generally yielded far too old dates, by several thousand years, compared with historical records due to the old wood effect (Tubbs and Kinder 1990). Here, the origin of the wood is the reason for this discrepancy. For example, if old wood/charcoal is used as fuel for limestone burning or is included in the aggregate, the wood/charcoal already has an age at the time of construction (Folk and Valastro 1976; Van Strydonck *et al*. 1986; Heinemeier *et al*. 1997). Embedded charcoal can be especially troublesome for dating because it may originate from sediments used as aggregate. On the other hand, Al-Bashaireh and Hodgins (2011) dated charcoal in the mortar in Petra, Jordan, with the assumption that only young bushes were used in lime burning. However, if alternative absolute dating materials or methods cannot be found, then it is essential to ensure that samples are taken from mortars with the least chance of being exposed to conditions that may cause chemical interactions.

Mortar ^{14}C preparation techniques and analysis are designed to handle the risk of open system condition by preserving only those fractions or lime binder components that are assumed to represent closed-system conditions. Research into new and more sophisticated ^{14}C mortar preparation techniques is an active research field; for example, experimental preparation methods based on thermal decomposition or preheating before sequential dissolution show good results in reducing problems with recrystallization (Barrett *et al*. 2020b; Daugbjerg *et al*. 2020).

CONCLUSIONS

A comprehensive discussion of mortar dating has been presented, including a description of the types of mortar involved, an overview of possible complications and a mortar-sampling guide. With recommended strategies to avoid a range of complications, this guide advises sampling schemes to improve the proportion of samples with conclusive mortar dating.

When lime mortar hardens, a reaction between slaked lime and atmospheric CO_2 produces $CaCO_3$, which stores the atmospheric ^{14}C signal. A proper preparation method can extract the signal allowing the mortar to be radiocarbon dated. However, certain complications may interfere with mortar dating, for example: delayed hardening and reactivation, recrystallization, and a mismatch between sample provenance and chronological investigation. For each complication, this guide presents sampling strategies to mitigate the issues. It also provides a description and advice in relation to three commonly encountered types of mortar: lime mortar, pozzolana mortar and cocciopesto mortar. Particularly, it addresses these mortars' constituents, hardening reactions and emphasizes the difference in behaviour between hydraulic and non-hydraulic mortars. Lime mortar is non-hydraulic and has a high success rate for mortar dating. Hydraulic pozzolana mortar is difficult to date; and hydraulic cocciopesto mortar is even more difficult and best avoided.

Finally, this article provides a compact summary table of strategies for easy reference. Field workers are encouraged to consult this guide when sampling mortar in the field.

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