IDENTIFICATION, EXTRACTION, AND PREPARATION OF RELIABLE LIME SAMPLES FOR ^{14}C DATING OF PLASTERS AND MORTARS WITH THE "PURE LIME LUMPS" TECHNIQUE

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ABSTRACT. Radiocarbon dating was first applied to historic lime mortars during the 1960s. However, despite the relative simplicity of the technique in principle, a number of subsequent studies have highlighted important aspects that should be considered. One of the most significant of these challenges arises from sample contamination by carbonaceous substances such as incompletely burnt limestone and aggregates of fossil origin containing "dead" ^{14}C. More recent studies have shown that in the majority of old lime-based mixtures the contamination problem can be avoided through selection of pure lime lumps. These particular types of lumps are believed to originate from areas where the lime is incompletely mixed with the aggregate. It has been demonstrated that even a single lime lump can provide sufficient material for a ^{14}C date of the mortar from which the lump was taken (Pesce et al. 2009). This paper describes the practical challenges associated with location, extraction, and preparation of 4 lime lumps extracted from 2 new sites for ^{14}C dating. These include distinguishing the lime lumps from other lumps present in the matrix and the removal of material surrounding the lime lump. The coherence of ^{14}C dating with other archaeological information on the chronology of historic sites is highlighted through case studies.

INTRODUCTION

A number of previous studies describe the application of radiocarbon dating mortars (Folk and Valastro 1976; Pachiaudi et al. 1986; Van Strydonck et al. 1992; Berger 1992; Sonninen and Junger 2001; Hale et al. 2003; Nawrocka et al. 2005; Lindroos et al. 2007; Heinemeier et al. 2010). The basic principle of the method can be described simply as follows: lime is produced from limestone (essentially calcium carbonate) of geological origin ("dead" in terms of ^{14}C concentration) that, in the past, was burnt at about 1023–1173 K to produce CaO (quicklime; Boynton 1980; Goren and Goring-Morris 2008). Quicklime was then slaked with water and mixed with sand (aggregates) to produce products such as plasters and mortars. When in place, the calcium hydroxide within the material hardened by carbonation according to the equations below, forming calcium carbonate:

$$CO_2 + H_2O \rightarrow HCO_3^- + H^+$$

$$HCO_3^- \rightarrow CO_3^{2-} + H^+$$

$$Ca(OH)_2 \rightarrow Ca^{2+} + 2(OH)^-$$

$$Ca^{2+} + CO_3^{2-} \rightarrow CaCO_3$$

The calcium carbonate contained in the material reflects the atmospheric ^{14}C concentration at the time of hardening and, thus, this material can be used for ^{14}C dating.

Despite the fact that the use of lime for ^{14}C dating is very simple in principle, several studies have highlighted various challenges and factors that should be considered (Van Strydonck et al. 1986). These arise mainly from the contamination of samples with carbonaceous substances such as incompletely burnt limestone and aggregates of fossil origin (e.g. limestone sand containing "dead" ^{14}C).

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Recently, studies have shown that accurate sample processing treatments significantly reduce these error sources (Sonninen and Junger 2001). However, adoption of a sampling procedure based on the careful selection of lumps of incompletely mixed lime, which are often embedded in the mortar matrix, provides an interesting alternative that evades the problems of contamination. Earlier studies exploiting this method have been reported (Gallo et al. 1998; Pesce et al. 2009).

LUMPS IN LIME MIXTURES: DESCRIPTIVE BACKGROUND

The internal structure of historic lime mixtures, including renders, plasters, and mortars, contain at least 4 different types of lumps, each of which can be recognized individually. These include under-burned limestone (Leslie and Hughes 2002; Ingham 2005; Elsen 2006); over-burned limestone (Leslie and Hughes 2002; Elsen et al. 2004; Ingham 2005; Elsen 2006); burned limestone containing high concentrations of silicon compounds (these arise when the stone used for the lime production contains impurities of silica and in production of hydraulic lime these lumps are called "grappiers"; Bakolas et al. 1995; Elsen et al. 2004); and pure lime lumps due to the carbonation of lime putty not mixed with aggregate (Bugini and Toniolo 1990; Franzini et al. 1990; Bakolas et al. 1995; Leslie and Hughes 2002; Elsen et al. 2004; Ingham 2005; Elsen 2006).

Among these types, only the lumps belonging to the latter group, pure lime lumps, are suitable for ^{14}C dating because they originate from the carbonation process of calcium hydroxide contained in lime putty (Elsen et al. 2004; Elsen 2006). Consequently, carbon contained within these lumps represents that of atmospheric carbon dioxide at the time of carbonation. In comparison, over-burned pieces of lime originally containing sintered calcium oxide are less reactive with water (Elsen 2006) and, consequently, if carbon is contained within these samples, it must not be considered representative of atmospheric carbon dioxide at the time of mixing. Unburned pieces of limestone contain the carbon dioxide of geological origin, while calcium contained inside the "grappiers" is mainly bonded to the silicon dioxide (Bakolas et al. 1995). A consequence of this is that little atmospheric carbon dioxide originating from the mixing time is expected to be contained within the samples.

To date, relatively few studies have involved lumps of pure lime; however, according to these researchers, their composition is similar to that of the original binding material (Franzini et al. 1990; Bakolas et al. 1995; Bruni et al. 1997). From a micromorphological point of view, these lumps are composed of very small, well-packed crystals in a compact structure and differ completely from that of the surrounding matrix. This observation led some authors to hypothesize that the crystals present in lumps developed over a shorter time period in comparison to the crystals of the surrounding binder (Bruni et al. 1997).

Recarbonation of calcium carbonate is a process reported to occur in almost all old mortars and would be expected to affect the results of ^{14}C dating (Karkanas 2007). However, few studies have been carried out to address this phenomenon in lime lumps, which is probably due to the associated difficulties in identification of primary and secondary calcite. Primary calcite is formed by carbonation of the binder, while secondary calcite is formed by dissolution and reprecipitation of calcium carbonate in lime mortars (Leslie and Hughes 2002).

Results of dating Medieval mortars (Pesce et al. 2009 and this paper) suggest that any "rejuvenation" process involving recarbonation of calcium carbonate within the lumps could affect the ^{14}C date. Almost all the dated samples were obtained from ruined walls of archaeological sites where the penetration of rising dampness or rain was expected. These cases are assumed to represent the most common situations and, consequently, we can temporarily hypothesize that lime lumps would be affected by the recarbonation process.

SAMPLING METHOD

The procedure used to sample lime lumps for ^{14}C dating involves on-site extraction of a small amount of mortar containing the lump and then removal of suitable pure lime lumps for laboratory analysis. The method used on-site to sample lime lumps must be tailored to accommodate the individual requirements of the structure from which the samples are taken. Buildings above ground, underground walls, fresco layers, mosaic substrate, all require slight variations in technique. In the field of building archaeology, it can be difficult to reach the inner part of the walls, especially if the thickness of mortar joints is not large enough to allow selection of suitable samples. This problem is particularly prevalent in most parts of Roman and Medieval constructions containing squared-off blocks laid upon very thin mortar joints. In these cases, it is only possible to proceed if a cross-section of the wall is accessible. When the inner part of the masonry is accessible, it is important to consider the possible recarbonation of lime lumps and the depth from which samples within the walls should be taken. Care should also be taken to avoid unusual situations such as water pockets.

In the event of an unusual situation being recognized, care must be taken to avoid mixtures not representative of the original structure (such as pieces of plaster applied on the wall after its construction and mortar deep inside the wall) where incompletely or delayed carbonated lime may be present. The carbonation process, in fact, initiates from the external surfaces of the walls and progresses towards the inner region at decreasing speed due to constricting pore section reducing carbon dioxide diffusion. As a consequence, it is possible that lime situated in the inner regions of the walls carbonate many years (decades or even centuries) after the mortar was laid. In this case, ^{14}C dating of these lumps would not represent the correct time of construction, and incorrect data would be introduced into the archaeological framework.

When a suitable depth of sampling is reached (the sampling point should be close to the surface of the wall in order to avoid delay in carbonation but deep enough to avoid repair mortars), a lump of suitable mass containing sufficient material for the ^{14}C dating must be identified. If an accelerator mass spectrometer is used, at least 20 mg of calcium carbonate is required. In the case of a single lump containing insufficient material, multiple samples from the same region of the masonry can be used (in archaeological terms this is from the same stratigraphic unit).

Following the on-site sampling and before treatment at the AMS laboratory, it is necessary to check the samples under an optical stereomicroscope to confirm the color, texture, and impurities of the lump and remove mechanically aggregate particles that may still be attached to the sample surface. One of the main problems in ^{14}C dating of old lime-based mixtures is contamination with carbonaceous substances, and among these substances there are commonly grains of limestone sand and pieces of incompletely burnt limestone.

During the on-site work, it is often possible to mistake incompletely burnt limestone resembling small white and rounded lumps, similar to the pure lime lump. But under a magnifying glass, even at low magnification, it is possible to distinguish between these 2 types of lumps as the surface of lime lumps have a floury appearance, while the surface of under-fired lumps exhibit a denser stone-like appearance. Evaluation of hardness can also be used to distinguish between these different types of lumps. Even performing a crude test by hand using a needle point allows these different types of lumps to be distinguished through their surface hardness.

SAMPLE PREPARATION

After identification, all pieces of sand still attached to the surface of lumps must be removed using tools such as a scalpel or needle. In order to remove as many pieces of sand as possible, this work

should be carried out under a stereomicroscope. Great care is essential during this phase of preparation as the sample is very delicate and prone to material loss. The AMS sample processing procedures are described in Pesce et al. (2009).

RESULTS AND DISCUSSION

Pesce et al. (2009) presented an example of this sampling and dating technique. Two samples of lime lumps were collected in the apse of the church of San Nicolò of Campodimonte (Camogli, Genoa, Italy) and dated with the ^{14}C method. Results obtained were evaluated and compared with the ^{14}C dating of organic material. All the results were finally compared with results of other dating methods such as mensiochronology of squared-off blocks (Pesce et al. 2009).

Prior to this case, few researchers carried out ^{14}C dating of lime lumps in archaeological sites: Gallo (2001) on the alto-Medieval castle of Aghinolfi (Massa Carrara, Italy) and Fieni (2002) on the basilica of San Lorenzo Maggiore in Milan. However, since 2008, additional tests have been made by the research group of the Institute for the History of Material Culture of Genoa (Italy). Among them, 4 new samples taken from 2 different archaeological sites were removed and dated following the procedures discussed above. Results were always consistent with the respective archaeological frameworks, and the uncertainty of ^{14}C dating was often reduced by comparison with other archaeological information. In particular, 1 sample was removed from the Medieval castle of Zuccarello (Italy) and 3 samples from the Medieval crypt of the Reggio Emilia cathedral (Reggio Emilia, Italy).

The Medieval castle of Zuccarello (Savona, Italy; Figure 1) is now a ruin located at the top of a hill in the western part of the Liguria region (northwest Italy). Over the past 3 yr, the castle has been subjected to some restoration work and archaeological studies. In order to obtain an archaeological dating of the main walls of this building, several techniques were used including mensiochronology of bricks, chronotypology dating of door and window frames, and historical/artistic dating.

Figure 1 Medieval castle of Zuccarello (Savona, Italy)

Mensiochronology of bricks is a well-established archaeological dating method (Martini and Sibilia 2006; Boato 2008) based on the trend of brick size over time. Its accuracy (a few tens of years on average) is not constant but varies over time depending on various factors such as the amount of data

available for a specific production and the precision of the producer. Results of mensiochronological dating method applied to 5 groups of bricks from the Zuccarello castle are shown in Table 1.

Table 1 Results of mensiochronology dating method applied to some groups of bricks from the Zuccarello castle.

Sample	Mensiochronology age (AD)
M1	1250–1350
M2	1370–1480
M3	1370–1450
M4	1370–1450
M5	1540–1560

Chronotypology of door and window frames is a dating method based on the shape of these details. Like the mensiochronology of bricks, it is a well-established archaeological method that works only on a local scale with an accuracy of a few hundred years (Casarino and Pittaluga 2001; Boato 2008). In addition to these techniques, the ^{14}C dating of a single lime lump from one of the oldest parts of the surrounding wall of the castle was carried out (Figure 2; sample LTL4756A).

Figure 2 Medieval castle of Zuccarello (Savona, Italy) surrounding wall (at left) and the sampling point of the lime lump (at right). Note that this was the only lime lump gathered at this archaeological site.

Mortar containing the lump was made of air lime as binder and grains of quartz, sandstone, and limestone as aggregate (mortar analysis have been made by the geologist Roberto Ricci). Uncalibrated ^{14}C and calibrated ages of samples are reported in Figure 3 and Table 2, obtained using OxCal v 3.10 (Bronk Ramsey 1995, 2001) and the IntCal09 atmospheric calibration curve (Reimer et al. 2009). Inspection of the graph shows that, even if the ^{14}C determination shows a normal distribution (red line at left-hand side of figure), the curve of calibrated data is divided into 2 parts with very similar probabilities (49.9% and 45.5%) because of the shape of the calibration curve in this time range.

The reliability and usefulness of this data was evaluated by comparison with results from other dating methods. Figure 4 shows all the data collected, represented on a timeline shown at the bottom of the graph. Each line in the figure corresponds to a specific dating obtained from different methods such as mensiochronology of bricks, chronotypology of doors and windows, and artistic dating.

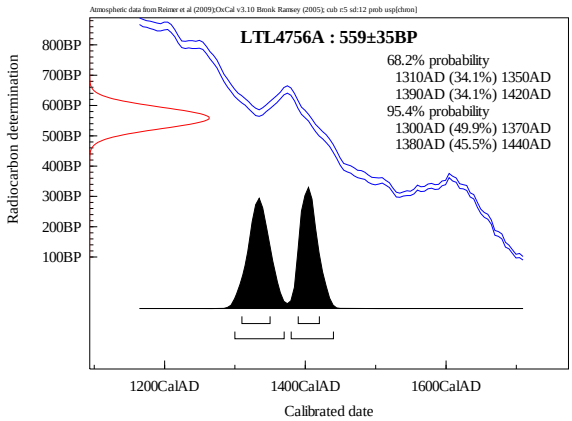

Figure 3 Uncalibrated and calibrated age of the lime lump found in the lower part of the surrounding wall of the Zuccarello castle.

Table 2 Uncalibrated age of lime lumps from both the Reggio Emilia cathedral and the surrounding wall of the Zuccarello castle. Calculation of the uncalibrated ^{14}C age is given with the Libby half-life (5568 yr), compared to the correct value of 5730 yr. AD 1950 was used as the reference year; the reference material was oxalic acid.

Sample	Radiocarbon age (BP)
LTL4752A	1295 ± 45
LTL4753A	1009 ± 40
LTL4754A	933 ± 45
LTL4756A	559 ± 35

The lines corresponding to the dating obtained with the mensiochronology method were obtained from 5 groups of bricks collected in different parts of the castle. Their length represents the chronological range of production. Above these lines, the ^{14}C dating of the lime lump is reported and, above this, the results of the chronotypology dating. Dating from the artistic evaluations of frescos, still visible inside the castle, are given at the top of Figure 4.

This graph highlights the agreement between ^{14}C dating and other dating methods applied on structures of the first and third stage (top of the figure). The first peak of the ^{14}C dating curve between AD 1300 and 1370 matches, in fact, historical records indicating that between AD 1326 and 1335 the historically important family of Del Carretto acquired the castle. At this time, significant expansion of the main building was carried out.

The second part of the ^{14}C date between AD 1380 and 1440, even if in the range of the third stage, is not in agreement with archaeological records describing building techniques of this period and, in particular, with the size and shape of masonry unit mortar joints and the laying technique. Therefore, after comparing ^{14}C data with other archaeological evidence, the surrounding wall of the castle is believed to date between AD 1300 and 1370.

The accuracy with which this date has been made is not typical of the previous studies where much larger variations in data were found. An example is dating of the crypt in the Reggio Emilia cathedral (Figure 5). As with the Zucarello castle, between 2008 and 2010 the crypt of the Reggio Emilia cathedral (northern Italy) was subjected to restoration works. An archaeological analysis of walls was made using a number of techniques including the lime lumps dating method.

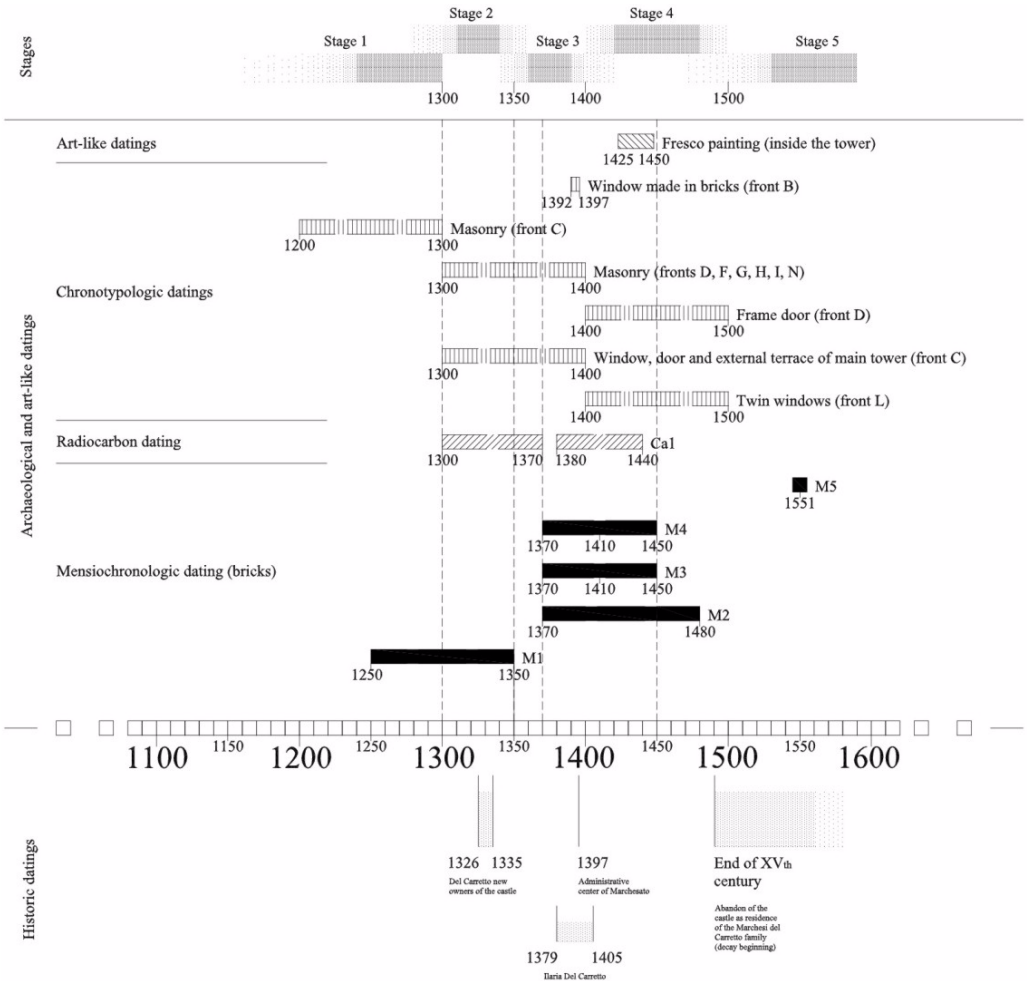

Figure 4 Comparison of results obtained from the different dating methods applied at the Zuccarello castle

Sample LTL4752A was obtained from the remains of an old apse discovered during archaeological excavation and believed to be representative of the oldest part of all structures within the perimeter of the cathedral. Sample LTL4754A was collected from a wall built with lime and pebbles (US 103), and sample LTL4753A was collected from a pillar built in bricks at the top of the wall. All the mortars containing the lumps were made of feebly hydraulic lime as binder and grains of quartz and schist as aggregate.

Locations were chosen to represent the stratigraphic sequence of construction. The structure dictates that the apse was built first. However, it is unclear whether the pillar was built after or at the same time of the wall. This was investigated by comparison to the ^{14}C dates, shown in Figure 9.

The uncalibrated ^{14}C ages for these samples are listed in Table 2 and the calibrated ages are shown in Figures 6, 7, and 8. The sample collected in the apse (LTL4752A; Figure 6) is clearly the oldest. However, some overlap is present between the pillar (LTL4753A; Figure 7) and in the wall below (LTL4754A; Figure 8). This suggests that they were built at the same time considering that the slight shift observed is within the variability of the technique.

Figure 5 Façade of Reggio Emila cathedral

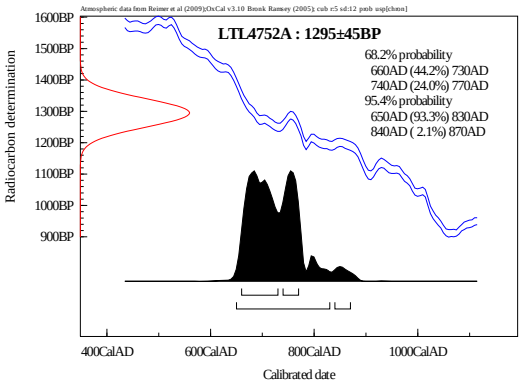

Figure 6 ^{14}C dating of lime lump from the old apse of the Reggio Emilia cathedral

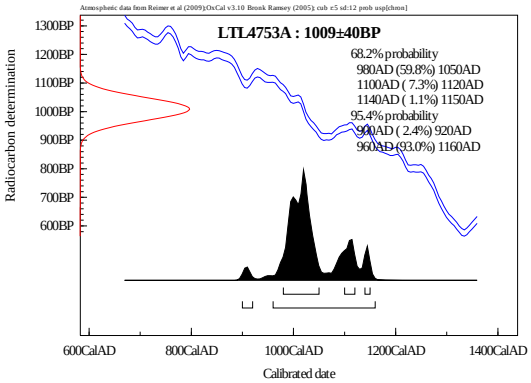

Figure 7 ^{14}C dating of lime lump from the pillar inside the crypt of the Reggio Emilia cathedral

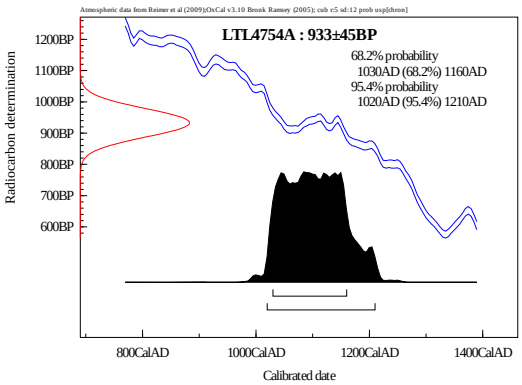

Figure 8 ^{14}C dating of lime lump taken from US 103 (wall made of pebbles) inside the crypt of the Reggio Emilia cathedral

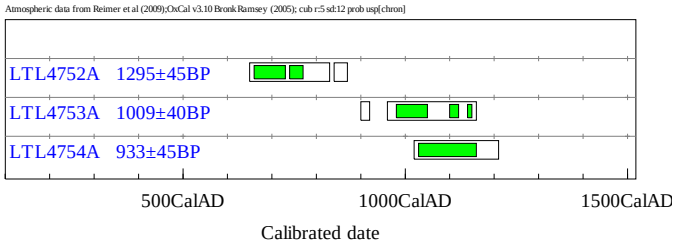

Figure 9 Comparison of the calibrated ages of lime lumps from Reggio Emilia cathedral.

CONCLUSIONS

Results obtained in recent years demonstrate that the ^{14}C dating of pure lime lumps (lumps originated by the carbonation of unmixed lime putty) is a viable method for the absolute dating of old structures built with mixtures of air lime. Application of this sampling and dating technique is simple and does not need specific equipment or processes. However, the reliability is incumbent on suitable lime lumps being obtained. Among the different types of lumps usually found embedded within old lime-based mixtures, only a "pure lime" lump of total mass at least 20 mg is suitable for the ^{14}C dating. The results of studies carried out over recent years have not highlighted drawbacks on the ^{14}C dating of lime lumps, and this will be an important aspect of future studies. Further studies will address issues concerning the accuracy and precision of dating obtainable with this technique, the recarbonation of lime lumps, and the differences in the chemical make-up of hydraulic lime.

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