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Integrated dating of the construction and restoration of the Modena cathedral vaults (Northern Italy): preliminary results

Tirelli G.[1], Lugli S.[1], Galli A.[2] Hajdas I.[3], Lindroos A.[4], Martini M.[5], Maspero F.[5], Olsen J.[6], Ringbom Å.[7], Sibilia E.[5], Caroselli M.[8], Silvestri E.[9], Panzeri L.[5]

1 Department of Chemical and Geological Sciences, University of Modena and Reggio Emilia, Italy

2 Department of Materials Science, University of Milan –Bicocca, INFN CHNet and CNR-IBFM, Italy

3 Laboratory of Ion Beam Physics, ETH Swiss Federal Institute of Technology Zurich, Switzerland

4 Faculty of Science and Engineering, Åbo Akademi University, Finland

5 Department of Materials Science, University of Milan -Bicocca and INFN CHNet, Italy

6 AMS 14C Dating Laboratory, Department of Physics and Astronomy, University of Aarhus, Denmark

7 Faculty of Arts, Psychology and Theology, Åbo Akademi University, Finland

8 Scuola universitaria professionale della Svizzera italiana, Switzerland

9 Studio Silvestri - Ingegneria e architettura, Modena, Italy

ABSTRACT

After the last damaging earthquake in 2012, an anti-seismic reinforcement project of the cathedral of Modena was formulated giving us the opportunity to investigate and date the building materials.

Radiocarbon, OSL and TL dating techniques were performed on the vaults with the aim to: (i) clarify the construction timing; (ii) define the history of the restorations; (iii) explore the possible correlation of the main restoration works to the earthquake chronology deduced from the historic catalogue.

Preliminary results show that medieval older bricks were re-used for most of the original construction. Only lime and not gypsum mortar was used for the original construction in the 15th century and for later repair of damage caused by earthquakes in the 16th and 17th centuries. Gypsum mortar was used for later repair in the 18th century. The results show much stronger damage due to earthquakes than previously thought.

INTRODUCTION

The seismic event that hit the Emilian plain in 2012 caused an extensive damage to the historical and artistic heritage (Decanini et al. 2012; Dolce et al. 2012; Indirli et al. 2012; Andreini et al. 2014; Graziani et al. 2015). The conditions of the UNESCO World Heritage Site of Modena and in particular the cathedral vaults were alarming (Baraccani et al. 2015; Baraccani et al. 2017; Baraccani et al. 2019). An anti-seismic reinforcement project was planned with the aim to determine the vulnerability of the cathedral analyzing the damage suffered in the past. Since historical documents leave unclear issues regarding the chronology of the construction phases, absolute dating of building materials represents the only way to obtain reliable data. The study and dating of the building materials of the Cathedral have been previously carried out analyzing other parts of the building (Lubritto et al. 2015; Panzeri et al. 2019).

The Modena cathedral construction phases are subject of discussion (Silvestri 2013), but the vaults were progressively added to the main framework during the 15th century (Dondi 1896; Baracchi and Giovannini 1988; Baracchi 1993).

Our survey carried out on the extrados of the vaults revealed unexpected data: the vaults were built using either a gypsum or a lime binder and carry the scars of several earthquakes, repaired using different binders, resulting in a complex patchwork of lime and gypsum mortars (fig. 1).

The main objectives of the project are dating the original vaults construction and the restoration portions built after the main earthquakes and correlating them to the earthquake chronology deduced from the historic catalogue (Rovida et al. 2016). Several damaging, moment magnitude (Mw) and high Peak Ground Acceleration (PGA) value earthquakes occurred after the vaults construction with epicenters located in

various areas in years: 1474 (Modena, Mw 4.30, PGA 0.232), 1501 (Emilian Apennines, Mw 5.82, PGA 0.187), 1505 (Bologna, Mw 5.41, PGA 0.055), 1584 (Tuscan-Emilian Apennines, Mw 5.99, PGA 0.025), 1586 (Spilamberto, Mw 4.53, PGA 0.091), 1660 (Modena, Mw 4.25, PGA 0.172), 1671 (Rubiera, Mw 5.23, PGA 0.110), 1688 (Romagna, Mw 5.85, PGA 0.028), 1828 (Staffora Valley. Mw 5.55, PGA 0.012) and 1850 (Modena, MW 4.53, PGA 0.144).

We applied an integrated approach using three different techniques to date the vaults construction repair phases: radiocarbon of mortars by sequential dissolution (Lindroos et al. 2018), Optically Stimulated Luminescence (OSL) of the mortar sand aggregate, and Thermo-luminescence of the bricks binded by the analyzed mortars (TL; Panzeri et al. 2019).

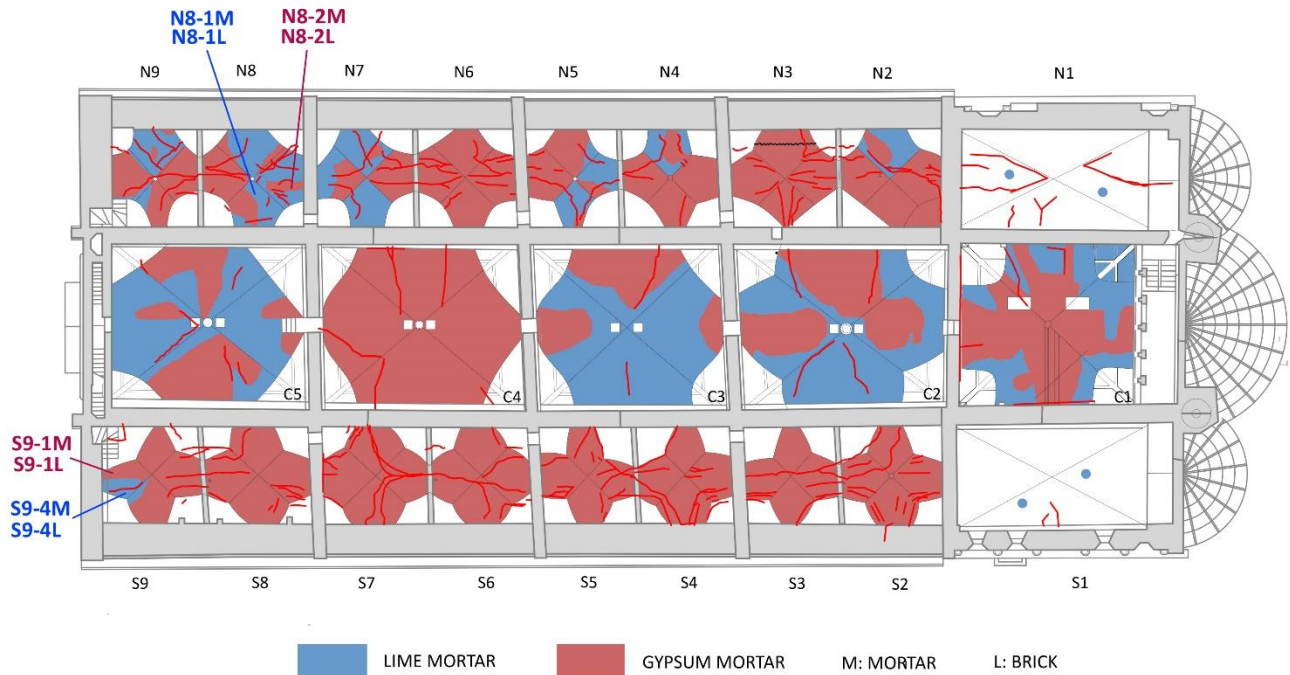This paper illustrates the preliminary results from two vaults (fig. 1).

Figure 1 Modena Cathedral: vaults extrados plan with sampling points of the vaults S9 and N8.

MATERIALS AND METHODS

Petrographic analysis

The mortar samples were consolidated with epoxy resin in order to obtain thin-sections for optical microscope observations. The UNI-Normal 11176 document was used as reference for the petrographic characterization of mortar in order to determine the aggregate composition, the grain size and the texture, the type of binder, the presence of underburned and over-burned limestone fragments and "lime lumps" (Nawrocka et al. 2005; Elsen 2006; Pesce et al. 2012; Michalska and Pawlyta 2019).

Image analysis of thin sections was also carried out to evaluate the binder/aggregate ratio, the aggregate grain size (Carò and Di Giulio 2004: Hayen et al. 2017; Michalska et al. 2017) and for the identification of the supply sources of the aggregate (Lugli et al. 2007).

Radiocarbon dating of mortars: sequential dissolution

Two lime mortar samples from the vaults N8 and S9 were prepared for radiocarbon dating following the sequential dissolution methodology. This procedure is fundamental in order to identify the contaminants that could occur in historic mortar. Underburned limestone fragments and carbonate aggregate could contaminate the radiocarbon dating results and provide older ages, while a rejuvenation effect can be

induced by the presence of secondary calcite within the calcareous binder, produced by later exchange with the atmospheric CO_2. The dissolution method is described in detail in Lindroos et al. (2007). The bulk mortars were carefully crushed and dry sieved into decreasing grain-size fractions ranging between 75-500 μm. The grain-size fraction <100 μm was subsequently wet sieved and, since the dissolution time decreases with grain size, a narrow grain-size window (46-75 μm) was isolated and dried for ^{14}C dating. After the mechanical separation, cathodoluminescence (CL, see e.g. Marshall 1988) analysis was carried out to identify the possible contaminants from limestone grains added in the aggregate sand and the alkalinity test with phenolphthalein (1%) dissolved in alcohol was performed on the coarser grain size fraction (301-500 μm). An alkaline reaction may indicate the presence of calcium hydroxide, which will react with the modern CO_2 in the atmosphere, leading to a younger age. Therefore, alkaline samples are rejected for AMS dating.

The chemical separation was done in a vacuum system with 85% phosphoric acid in order to release CO_2 for dating. The mortar sample S9-4M was analyzed in two CO_2 fractions, while the radiocarbon dating of the sample N8-1M was performed only on a soft lime lump (Pesce et al. 2012) crushed before hydrolysis and analyzed in two CO_2 fractions.

The vials containing CO_2 gas were submitted to the AMS [14]C Dating Centre at Aarhus University and ETH Zurich. The conventional ^{14}C dates were calibrated using OxCal v 4.3.2 program (Bronk-Ramsey 2017).

All calibrated results are reported at the 95% confidence level (2 sigma).

OSL dating of mortars

OSL dating was performed on the same lime mortar selected for ^{14}C dating: N8-1M and S9-4M. Mortars were sampled using a chisel and a hammer under dim red light and they were put into a black plastic bag. In laboratory, the surface layer was removed and the inner part was used for luminescence analysis. All the laboratory procedures were carried out under dim red light. The measurements were performed using the quartz inclusion technique (180-250 μm; Fleming 1970) with the Single Aliquot Regenerative (SAR) protocol (Murray and Wintle 2000). Both Multi-Grain (MG) and Single Grain (SG) techniques were applied. OSL measurement were performed with TL-DA-20 Risø reader and photons were detected by a bialkali photomultiplier tube (EMI 9235QB) coupled to a 7.5 mm Hoya U-340 filter. More details on OSL application to mortars are discussed elsewhere (Galbraith et al. 1999; Zacharias et al. 2002; Stella et al. 2018; Panzeri et al. 2017; Urbanovà et al. 2018; Panzeri et al. 2019; Panzeri et al, in this volume and other references therein).

TL dating of bricks

We analyzed two brick fragments for each of the two vaults (N8-1L, N8-2L, S9-1L, S9-4L). The outer layer of the brick fragments was removed in the laboratory. The polymineral fine grain technique was used and the Multiple Aliquot Additive Dose (MAAD) protocol was applied (Aitken 1985). TL dating was performed using an in-house system equipped with a photomultiplier tube (EMI 9235QB) coupled to a blue filter (Corning BG12).

Dose rate determination

For dose-rate determination, ^{238}U and ^{232}Th concentrations were derived from alpha counting using ZnS (Ag) scintillator discs and assuming a Th/U concentration ratio equal to 3.16 (Aitken 1985). Contribution due to ^{40}K content was obtained from the total concentration of K measured by flame photometry. The attenuation of the beta particles in coarse grain quartz used for mortar dating was taken into account (Bell 1979). For dose rate calculations, an amount of water corresponding to 20 ± 5% of saturation was assumed. The external dose rate was evaluated on site with an ionization chamber for environmental dosimetry, to avoid the problem of inhomogeneity of the building materials around the samples.

Parameters used for age determination are reported in table 1.

Name	Sample	Technique	E.D. (Gy)	^{238}U (ppm)	^{232}Th (ppm)	^{40}K (ppm)	Saturation water content (%)	Dose rate
S9 1La	Brick	TL	4.3 ± 0.5	2,29	7,23	1,213875	21	5.52 ± 0.19
S9 1Lb	Brick	TL	3.0 ± 0.4	2,05	6,49	1,13	27	3.91 ± 0.14
S9-4L	Brick	TL	3.6 ± 0.3	2,68	8,46	1,34	19	4.67 ± 0.16
S9-4M	Mortar	OSL MG	4.2 ± 0.9	1,94	6,12	0,31	10	1.32 ± 0.04
		OSL SG	1.4 ± 0.3					
N8-1L	Brick	TL	5.3 ± 0.5	2,30	7,26	1,47	22	5.47 ± 0.19
N8-2Lc	Brick	TL	1.5 ± .1	2,29	7,23	1,26	20	5.44 ± 0.19
N8-2La	Brick	TL	1.4 ± 0.18	2,11	6,68	1,23	20	5.13 ± 0.17
N8-2Lb	Brick	TL	1.2 ± 0.3	2,38	7,52	1,34	22	5.72 ± 0.20
N8-1M	Mortar	OSL MG	2.3 ± 0.2	1,22	3,85	0,51	5	0.97 ± 0.03

Table 1 Parameters used for age determination of OSL and TL dating.

RESULTS AND DISCUSSION

Petrographic analysis of lime mortars

The sample S9-4M is a non-hydraulic lime-based mortar with underburned particles up to 3 mm in size. Lime lumps are scarce and small (< 1 mm). The binder/aggregate ratio is 2.2. The aggregate grain size is very fine to fine sand and moderately well sorted. The sand composition is: micritic limestone and sparry calcite, quartz, feldspar, siltstone, rare fossil fragments, serpentinite fragments, biotite, iron oxide and/or hydroxide. The provenance is probably from the Panaro River (Lugli et al. 2007; fig. 2a).

The sample N8-1M is a non-hydraulic lime-based mortar with underburned particles up to 4 mm in size. Lime lumps are frequent and up to 2 mm across. They are soft and white and of the type that Pesce et al. (2012) and Lindroos et al. (2018) recommend for dating. The binder/aggregate ratio is 1.3. The aggregate grain size is very fine to medium sand and moderately well sorted. The sand composition is: micritic limestone and sparry calcite, quartz, feldspar, siltstone, fossil fragments, serpentinite fragments, biotite, iron oxide and/or hydroxide, glauconite. The provenance is probably from the Panaro River (Lugli et al. 2007; fig. 2b).
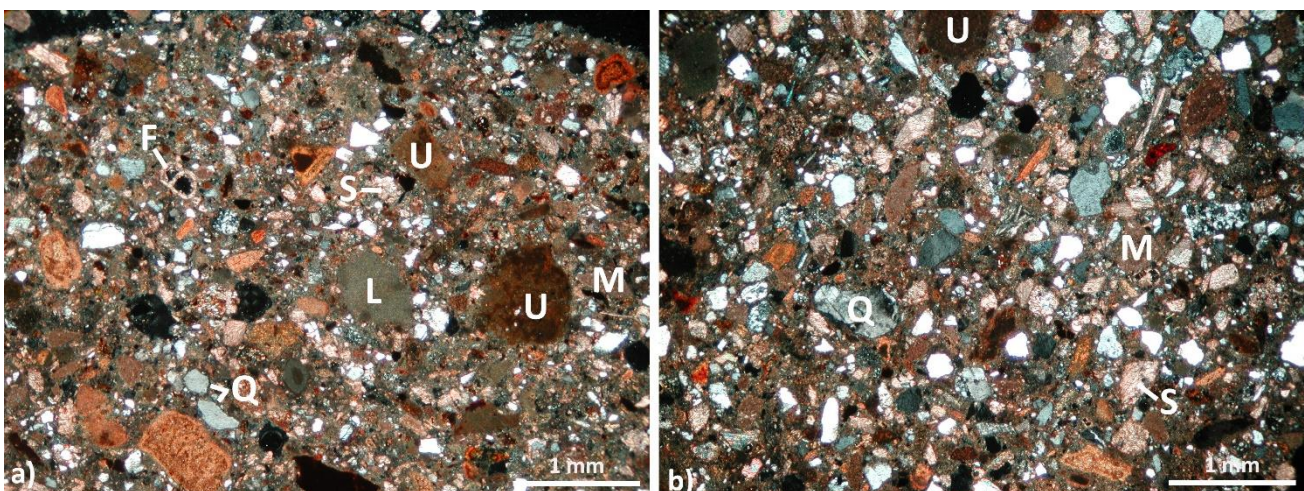

Figure 2 (a) photomicrograph in crossed polars of the mortar sample S9-4M. F: fossil fragments, L: lime lump, M: micritic limestone, S: sparry calcite, U: underburned fragment; (b) photomicrograph in crossed polars of the mortar sample N8-1M. M: micritic limestone, Q: quartz, S: sparry calcite, U: underburned fragment.

Radiocarbon dating

The radiocarbon dating results are listed in table 2.

The alkalinity test reveals that the samples result with a low alkalinity and therefore suitable to be dated.

CL inspection has been performed on the selected grain size fraction (46-75 µm) after sieving and on the same powder after sequential dissolution of the sample S9-4M. The powder before dissolution contains carbonate contaminants and part of silicic aggregates, while after the dissolution, it is only visible the aggregate since the binder and the carbonate contaminants were dissolved.

The ^{14}C profile of the sample S9-4M is shown in figure 3a. It displays an increasing slope, characterized by rapidly dissolving mortar binder and slowly dissolving contaminants. The radiocarbon age of the first CO_2 fraction (523 ± 23 BP) of the sample S9-4M gave a calibrated calendar age 1328-1341 AD (5.2%) and 1396-1439 AD (90.2%), matching with the age provided by historical documents (1404-1433 AD, Baracchi and Giovannini 1988, fig.3b). It follows that despite the presence of carbonate grains in the aggregate and unburned limestone fragments, the sequential dissolution procedure has succeeded in eliminating the possible contaminants effect which would have given an older age than that provided by documents.

As show in figure 3c, the ^{14}C profile of the sample N8-1MLi reveals that the dates seem to be inverted i.e. fraction 1 produced an age slightly older than fraction 2. However, CO_2 fractions 1 and 2 have overlapping ages and according to the Criterion I (Heinemeier et al. 2010; Ringbom et al. 2011; Lindroos et al. 2018) if the first two CO_2 fractions agree within the given error margins (and so the ages are overlapping), it demonstrates absence of contamination and it is considered a reliable result. In this case the combined calibration is assumed to give the true age of the mortar. The combined age of the CO_2 fractions 1 and 2 yields the ^{14}C age 263 ± 43 BP corresponding with a calibrated calendar age 1487-1681 AD (79.2%), 1739-1743 AD (0.4%), 1763-1802 AD (12.4%), >1938 AD (3.5%), younger than the documented construction age (1433-1454 AD, Baracchi and Giovannini 1988, fig. 3d), indicating a later re-construction after significant damage. The catalogue of earthquakes reports several seismic events that occurred after the vaults construction. Four damaging earthquakes in 1474, 1501, 1660, 1671 overlap with the radiocarbon dating and they may be the direct responsible for the reparation works, which are not documented in the historical records.

In table 3 we report the calibrated ages and their interpretation in comparison with the TL and OSL dating results.

Site	Sample subsample / CO_2-fraction	Material	Grain size (µm)	Reaction time (s) from t_0	Carbon yield (tot) (%)	Fraction size (relative 1)	C-14 age (BP)	±	δ13C ‰ VPDB
Modena Cathedral	N8-1MLi.1.1	Lime lump	Crushed	10	3.05 % C	0-0.27	291	56	n.r.
Vault N8	N8-1MLi.1.2			24		0.27-0.52	223	66	n.r.
Modena Cathedral	S9-4M.3.1	Bulk mortar	46-75	13	2.62 % C	0-0.17	523	23	-17,4
Vault S9	S9-4M.3.2			35		0.17-0.36	1207	24	-6,4

carbon yield (tot) (%) = total carbon yield after about one hour of dissolution

n.r. = not reported

Table 2 Hydrolysis data, ^{14}C results and $\delta^{13}C$ values for the dated samples.

OSL dating

The OSL dating results are listed in table 3. Mortar OSL dating gives results not compatible with the construction phases. The OSL ages obtained are much older than the expected ones, regardless of the techniques used, indicating a poor and uncomplete bleaching (Sawakuchi et al. 2011).

TL dating

The TL dating results are listed in table 3. They provide information about the construction timing and restoration of the vaults, especially for those cases where gypsum binder was used. In these cases TL of bricks is the only possible dating technique. The results of the vault S9 suggest that bricks were manufactured in the 13th century (1245 ± 65 and 1235 ± 80, 1250 ± 80) and were re-used later in both lime and gypsum portions of the vault.

The dates obtained from the vault N8 reveals that the lime portion of the vault was built with re-used bricks from the 11th century, while bricks from the gypsum portion are younger (1740 ± 20, 1740 ± 30, 1805 ± 20) than the original construction phase of the vault. These data suggest that significant restoration works were carried out using gypsum mortar in the 18th and 19th centuries after the damaging earthquakes of 1671, 1688, 1828, 1850.
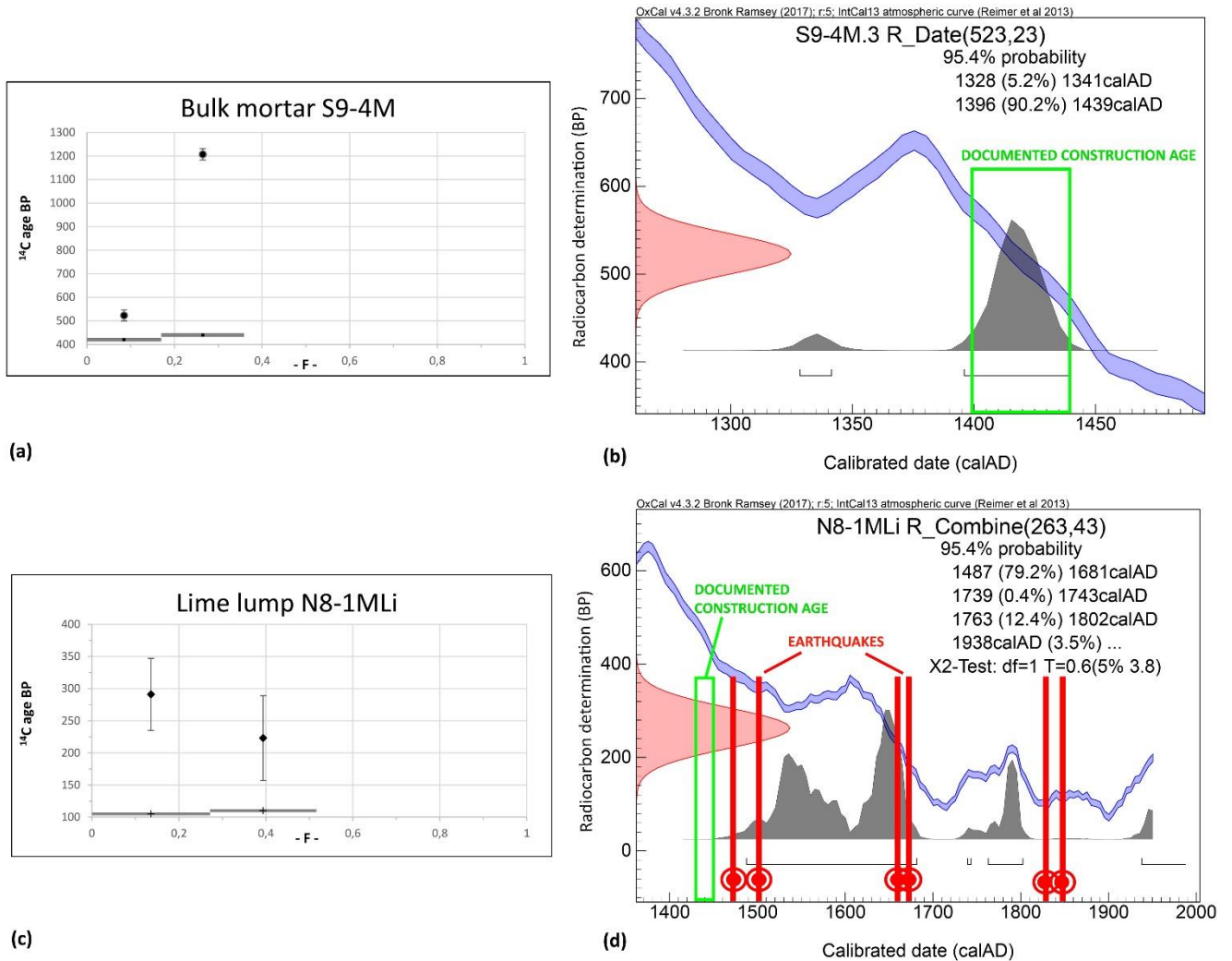

Figure 3 (a) ^{14}C age profile of the samples S9-4M. F is the fraction of reactive material transformed from carbonate to carbon dioxide. The horizontal gray bars along the x-axis denote the size of the extracted CO_2 fractions relative to the total carbon yield (=1); (b) calibrated result of the bulk mortar S9-4M, 1328–1341 (5.2%) and 1396–1439 (90.2%). The date coincides with the historical documents age (1404–1433) marked as a green rectangle; (c) ^{14}C age profile of sample N8-1M. F is the fraction of reactive material transformed from carbonate to carbon dioxide. The horizontal gray bars along the x-axis denote the size of the extracted CO2 fractions relative to the total carbon yield (=1); (d) calibrated result of the lime lump N8-1MLi, 1487–1681 (79.2%), 1739–1743 (0.4%), 1763–1802 (12.4%), >1938 (3.5%). The dates are younger than the historical documents age (1433–1454) marked as a green rectangle. Several earthquakes, marked as red vertical lines, overlap with the ^{14}C age, indicating later repair after significant seismic damage.

Name	Sample	Expected age	Absolute dating techniques	Date	Notes	Interpretation
S9-1La	brick (gypsum mortar)	1404-1433	TL	1235 AD ± 80	RE-USED brick	Lime mortar for original construction (1404-1433) with re-used bricks; gypsum mortar for later repair with re-used bricks
S9-1Lb	brick (gypsum mortar)		TL	1250 AD ± 80	RE-USED brick	
S9-4M	lime mortar		^{14}C Sequential dissolution ETH Zurigo	Bulk: [1328-1341 AD] 5.2% [1396-1439 AD] 90.2%	the date is in AGREEMENT with expected age	
S9-4M	lime mortar		OSL multi grain	1140 BC ± 720	the date is NOT in agreement with expected age	
S9-4M	lime mortar		OSL single grain (MAM)	960 AD ± 200	the date is NOT in agreement with expected age	
S9-4L	brick (lime mortar)		TL	1245 AD ± 65	RE-USED brick	
N8-1L	brick (gypsum mortar)	1433-1454	TL	1050 AD ± 90	RE-USED brick	Lime mortar for later repair with re-used bricks after earthquakes (1474, 1501, 1660, 1671); gypsum mortar for interventions of 18th century
N8-1M	lime mortar		^{14}C Sequential dissolution Aarhus AMS Centre	Lime lump: [1487-1681 AD] 79.2% [1739-1743 AD] 0.4% [1763-1802 AD] 12.4% [>1938 AD] 3.5%	the date is YOUNGER than expected age. Later repair after earthquakes: 1474, 1501, 1660, 1671	
N8-1M	lime mortar		OSL multi grain	390 BC ± 220	the date is NOT in agreement with expected age	
N8-2Lc	brick (gypsum mortar)		TL	1740 AD ± 20	Later repair after earthquakes: 1671, 1688, 1828, 1850	
N8-2La	brick (gypsum mortar)		TL	1740 AD ± 30		
N8-2Lb	brick (gypsum mortar)		TL	1805 AD ± 20		

Table 3 Results of absolute dating of the vaults S9 and N8.

CONCLUSION

A detailed survey of the 15th century vaults of the Modena medieval cathedral revealed a complex and unexpected structure consisting of different mortars used for multiple modifications.

The dating of mortars and bricks clarifies the chronology of the construction phases and the history of complex restorations, which are probably related to damage suffered after earthquakes.

Absolute dating results suggests that the vault S9 was originally built in 1404-1433 using lime mortar and older bricks, and was subsequently partially reconstructed with gypsum mortar and re-used bricks. As gypsum mortar can not be directly dated, the re-use of older bricks does not allow to pinpoint the timing of the reconstruction and the possible triggering earthquake.

Two different restoration phases were recognized on the vault N8. A first repair by lime mortar, and re-used bricks, was carried out immediately after the original construction, possibly as a result of the 1474, 1501, 1660 and 1671 earthquakes. A second intervention was then necessary to repair damage probably caused by the 1671, 1688, 1828 and 1850 earthquakes. As building materials for this second repair new bricks were used, kept together by a gypsum mortar.

The preliminary results of this study demonstrate that the vaults suffered much larger damage induced by earthquakes than previously assessed by mapping fracture and crack networks alone.

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