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Abstract The study shows how the unique combination of characterization techniques with the single grain OSL dating of mortar allows acquiring relatively complete information about the monument for which we miss the other, written or archaeological, source of information. Important differences in preparation technology of mortar are observed. The mortar appears here as an element of the crucial informative value and reveals the history of the monument and its transformation from the Late Antiquity mausoleum to a sacral building.

1 Introduction

In 1998, UNESCO recognised the historical heritage of the Church Saint Seurin, adding it to the world heritage list as a part of the pilgrim route to Santiago de Compostela. The actual church, built in 12th century in Romanesque style and subjected to numerous reconstructions during its existence, incorporates the crypt being in the centre of our interest in this paper. First mentions witnessing the existence of the crypt appear in manuscripts from 11th and 13th century but none of these documents allow to precise its nature and its architecture. The ancient remains in the actual crypt were discovered in 1840 by Cirot de la Ville [1] repeatedly studied in 1966, 1995-2000 and 2006 and finally identified as a Late Antiquity mausoleum [2, 3, 4] built between the second half of the 4th and 5th century AD according to archaeomagnetic and thermoluminescence dating of bricks used for its construction [5, 6].

In spring 2014, a deeper archaeological study of the complicated stratigraphy, directed also by Anne Michel, lead to the creation of detailed archaeological records proving the existence of nine stratigraphic levels between the Late Antiquity and 19th century [7]. However, apart from the oldest layer corresponding to the mausoleum, the chronology of all subsequent construction phases and their relationship remains to be clarified.

1.1 Objectives of the study

The crypt was subjected to numerous reconstructions resulting today in discontinuities of stratigraphic levels in many zones which makes observations more difficult and complicates archaeological interpretations. Therefore, an archaeometric study of mortars was carried out so as to shed light on the complex history of the crypt in the Early Medieval Period. The undertaken research had dual objective: first to compare the mortars sampled in different zones and archeaological levels of the crypt in order to reveal the extent of different building phases, to study the changes in preparation technology of mortar and possibly to identify the provenance of the sand used, second to date selected mortar samples by optically stimulated luminescence (OSL) applying the methodology by single grain technique developed recently in the laboratory IRAMAT-CRPAA [8, 9, 10].

2 Sampling & Methodology

The crypt consists of three parallel naves ended by chapels. The oldest part of the actual crypt built on the remains of the Late Antiquity mausoleum is situated in the chapel of the central nave. Firstly, several mortar and charcoal samples for the preliminary study were taken in the different levels of the south-east angle of this central chapel (Fig. 1). Few months later, the second sampling campaign comprising 25 mortars and 13 charcoals was integrated into the archaeological research carried out in the entire crypt with the objective to support emerging hypothesis. The archaeometric study focused on the oldest stratigraphic levels.

The samples were studied by standard analytical methods used for mortar characterization [11,12] such as optical polarizing and SEM-EDX microscopy with cathodoluminescence (thin sections), X-ray fluorescence (homogenized mortar powder), X-ray diffraction and laser granulometry (mortar aggregate).

The radiocarbon dating of charcoals was carried out by the external analysis in the radiocarbon dating platform ARTEMIS via the *Centre de datation par le radiocarbone Lyon* (*CNRS Univ Lyon 1*). The dating of mortars was performed using optically stimulated luminescence (OSL). Even if this approach is more largely used in geology, its application to historic mortar in building archaeology is very recent [13-16]. The deeper research on dating mortars by OSL single grain between 2012 and 2015 in laboratory IRAMAT-CRPAA allowed establishing more robust methodology whose first application on an undated structure was undertaken in the crypt of Saint Seurin basilica presented in this paper [8, 9, 10].

The basic premise in dating of mortar by optically stimulated luminescence is that quartz in the sand used for making mortar is optically zeroed during the preparation process. The moment to be dated is the last exposure of mortar to light, before being embedded within the masonry and hidden from light. For OSL dating application, the individual archaeological doses are measured by OSL on individual quartz grains extracted from the mortar, and the mean annual dose is determined by on-site dosimetry (environmental component of the radioactivity) and by high resolution low back ground gamma spectrometry of natural radio nuclides of the mortar sample (beta and alpha components). For further details, we refer the reader to the more detailed publications [8, 10, 17].

Fig. 1 The south-eastern angle of the central chapel with marked samples from the first intervention in the crypt: BDX 16496, BDX 16498 and BDX 16500 correspond to the Late Antiquity mausoleum, BDX 16493 and BDX 16492 to two consequent construction phases

3 Results

3.1 Characterisation of mortars

The general character of mortars from the original mausoleum is coarse and heterogeneous, the sample BDX 16500 (Fig. 2a) being more homogeneous in comparison with the samples BDX 16496 and BDX 16498. These mortars contain monocrystalline phases (quartz, microcline, muscovite) as well as metamorphic rock fragments (quartzite, mica schist). The aggregate is rather poorly sorted and sub-angular with the minimum size of 100 µm and the maximum size about 3 mm. The amount of the aggregate is about 30 % for BDX 16500 and about 35 % for BDX 16498 and BDX 16496. The mortar BDX 16500 is more porous and some recrystallization can be observed. Whereas the samples BDX 16498 and BDX 16496 comprise *cocciopesto* (brick) fragments noticeable by macroscopic observation, the sample BDX 16500 contains *cocciopesto* fragments of millimetric scale and a fragment of reused mortar with degraded binder.

The sample BDX 16500 makes part of the concrete mortar in the foundations of the Late Antiquity structure. It is extremely rich in tiny *cocciopesto* fragments. In matrix of the mortar BDX 16500, numerous reaction rims between *cocciopesto* fragments and lime binder are visible (Fig. 3a). The calculation of the hydraulicity index (H.I.) based on the quantification of hydraulic oxides in the binder by SEM-EDX analysis (Fig. 3b) [18] shows that this mortar is highly hydraulic whereas the samples BDX 16496 and BDX 16498 are not. As discussed in the following paragraph, the mortar BDX 16500 was also quite well-bleached. These observations make us think about the careful preparation process employed, which is not the case of the two other samples BDX 16496 and BDX 16498 originating from the same construction phase, but located higher in the perimeter wall and being extremely heterogeneous and poorly bleached. In addition, these two samples contain numerous lime lumps linked to insufficient slaking indicating imperfections in the preparation procedure. An intentional differentiation in the preparation technology within the same construction phase is thus observed.

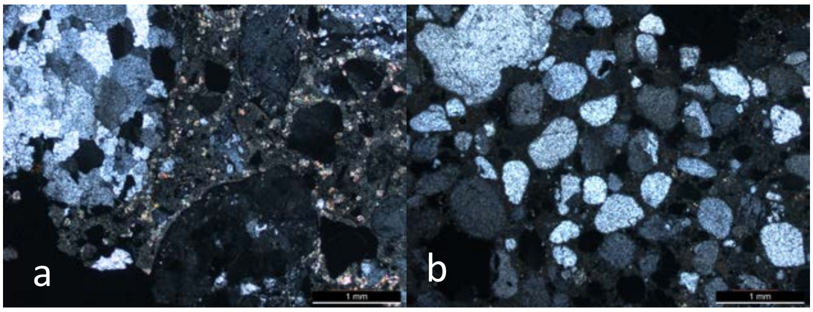

Fig. 2 Thin sections of the samples BDX 16500 (a) and BDX 16493 (b)

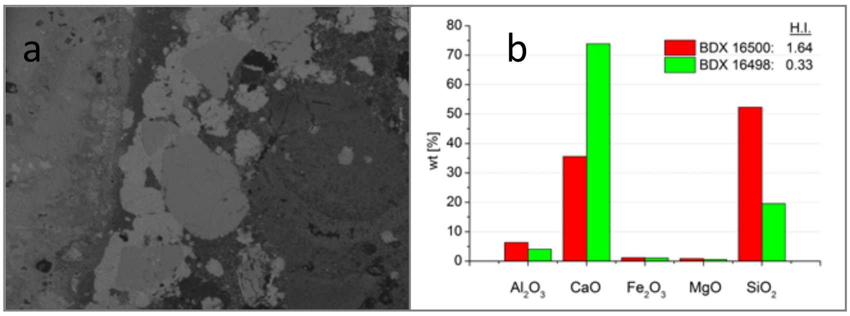

Fig. 3 SEM-EDX image of a reaction rim between a *cocciopesto* fragment and binder in the sample BDX 16500 (a) and the content of selected oxides quantified by SEM-EDX analysis

The samples from the subsequent construction phases show homogeneous character, containing exclusively monocrystalline phases (mainly quartz, sporadically feldspars). The aggregate is well-sorted and rounded with the main size about 400 µm which suggests a sieving stage in the preparation process. The proportion of the aggregate predominates over the binder (more than 50 %) which is cracked. The sample BDX 16493 (Fig. 2b) corresponds to the moment when all the sarcophagi in the crypt where covered by the new level of ground. The high proportion of the aggregate seems to play this filling role. These mortars contain also lime lumps, charcoal pieces and pieces of partially burned lime. All these observations seem to indicate less elaborated preparation procedure.

The provenance of the aggregate in the mortars from the first construction phase (mausoleum) is obviously different from the aggregate of subsequent stratigraphic layers. We also observe the change in the technology; the intentional addition of *cocciopesto* fragments to the mortars from the first phase (mausoleum) is not used in subsequent construction phases.

The microscopic observation does not allow distinguishing between different subsequent construction phases. Therefore, the study continues by the analysis of the aggregate by XRD and laser granulometry. At the same time, the study attempting to identify the geological sources exploited by ancient builders is running.

3.2 Dating of mortar by optically stimulated luminescence (OSL)

The exposure of sand grains in mortar to light is not uniform and only some of them are well-bleached and can thus be used for OSL dating. In other to identify these well-bleached grains, each single grain has to be analysed separately. The sensibility of the studied samples to optical stimulation is sufficiently high and the signal of individual grains shows measurable, which is the first prerequisite to date mortars by OSL. About 4000 grains per sample were analysed. Whereas for the mortars sampled in the mausoleum between 2-3 % of grains gave a signal, for samples from the subsequent construction phases this proportion represents about 5-6 % of all analysed grains.

The measured distributions of archaeological doses for individual grains are positively skewed for all the samples studied, but a very clear frequency maximum is emerging at the beginning of the distributions for the samples BDX 16500, BDX 16493 and BDX 16492. This indicates the presence of well-bleached grains containing the chronological information (Fig. 4a). The distributions of the samples BDX 16496 and BDX 16498 do not show a clear frequency maximum (Fig. 4b). We distinguish clearly the difference in luminescence properties between the BDX 16498 with BDX 16496 and BDX 16500, the mortars originating from the same construction phase.

Probably because of the presence of brick fragments, the mortars from the first construction phase (mausoleum) are more radioactive than the others. Also, these mortars being coarse and heterogeneous reveal complex microdosimetry [17] as shown by beta-radioagraphy and SEM-EDX cartography. This information must be taken into account when determining the age. On the other hand, the smallgrained and homogeneous mortars BDX 16493 and BDX 16492 are not affected by heterogeneous microdosimetric effects which simplifies the dating process.

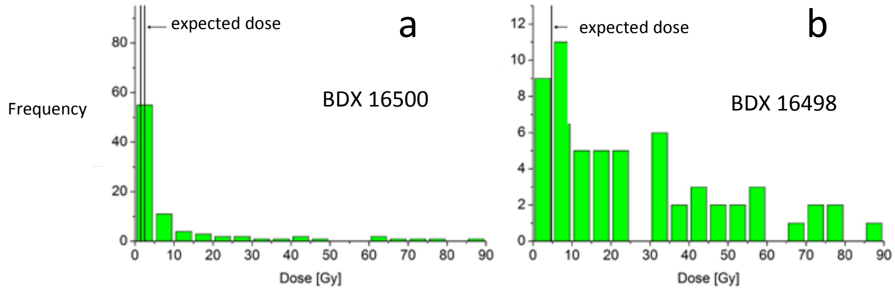

Fig. 4 Distributions of archaeological doses for individual quartz grains measured by OSL

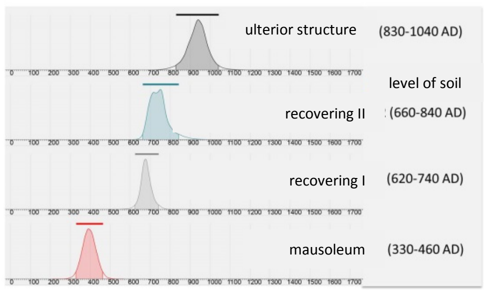

Fig. 5 The combination of the OSL dating of mortars with radiocarbon dating of charcoals, thermoluminescence and archaeomagnetic dating of bricks [5,6] and archaeological observations (stratigraphic relationships between archaeological units). The chronological data are statistically evaluated by the CHRONOMODEL procedure [19] within 95 % level of confidence.

The OSL analysis reveals chronological intervals that are consistent with the stratigraphic layers in which the mortars were sampled. They also agree with the radiocarbon dates of charcoals taken in the same spots. As a result of this interdisciplinary approach, four construction phases are distinguished (Fig. 5):

- the construction of the mausoleum (4th-5th century, mortar BDX 16500)
- the recovering of the sarcophagi by the new level of ground in two stages (7th-8th century, mortar BDX 16493)
- the construction of an ulterior structure (10th-11th century, mortar BDX 16492)

Three interesting observations emerge from the OSL analyses. Firstly, the sample BDX 16500 was much better bleached than the samples BDX 16496 and BDX 16498 which indicates probably the difference in preparation technology within the same construction phase as observed by characterization methods. Moreover, the transformation of the monument by recovering the sarcophagi included probably two levels visible on the stratigraphy but hardly distinguishable by OSL dating and petrography. Either the precision of OSL dating is not sufficient to distinguish this difference or there is only one construction phase realized in two stages. The future dating might help to answer this question. Finally, the sample BDX 16492 seems to be much older than expected following the actual archaeological interpretations and this result stimulates new reflexions concerning the interpretation of this particular unit.

4 Conclusions and perspectives

The mortars from the first sampling stage have been characterized and dated. The combination of archaeological observations, mortar characterization and mortar dating allowed confirming the differentiation of at least four important construction phases which attests the continuity in the occupation of the monument in the Early Medieval Period.

In the second part of the research that is running now, the objective is to realize a comparative study of the mortars sampled during the second intervention on the site and relate them to the identified construction phases. This is being carried out by petrographic and CL examination, XRF analysis, laser diffraction and X-ray diffraction analysis of the mortar aggregate, SEM-EDX analysis of binder and OSL dating of selected samples. The running analyses should allow:

- to distinguish the construction phases on the bases of chemical, petrographic and mineralogical comparison of mortars, to relate different zones in the crypt and thus to clarify how the monument was progressively extended
- to confirm and consolidate the actual dating results
- to attempt identifying the provenance of the aggregate

The paper represents an interdisciplinary approach where physicists, specialists in archaeometry and archaeologists worked in a narrow collaboration contributing to the better understanding of this unique monument. The mortar appears here as an element of the crucial informative value revealing the history of the crypt. The final synthesis including the results of the first and the second part of the research will be presented on the meeting.

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