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OSL dating of mortars from constructive phases of the old chapel San Breixo de Ouvigo (NW Spain)

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Abstract. The use of optically stimulated luminescence (OSL) dating on ancient mortars have provided increasing knowledge of the history of buildings in the last years. In this work, we apply OSL dating on mortars of a key building for the history of NW Spain: the old chapel of San Breixo de Ouvigo. After archaeological excavations, more than 40 years ago, it was generally accepted that this building preserves, at least, a Late Roman (4th and 5th centuries) constructive phase with later modifications introduced in the Early and Late Middle Ages. However, neither stratigraphical nor chronological evidence confirmed this interpretation. Five samples from three different constructive phases have been taken for OSL dating. Small quartz multi-grain aliquots were used for dating. Results provide ages in agreement with expectations for the mortars of the Late Roman phase but, they show the need of a new interpretation of the chronological model assigned to the building. Such model could be corroborated in a next phase of the project that intends to characterize the mortars and using radiocarbon dating.

1. Introduction

In the last years, the use of optically stimulated luminescence (OSL) and radiocarbon for dating ancient mortars have provided increasing knowledge of the history of architecture. The development of OSL dating of mortars is still in progress aimed towards improving results [1-3]. OSL dating provides the age of manufacture of mortars, as it indicates the moment when quartz aggregates remain shielded from daylight. Although problems, such as age overestimation, or age imprecision can arise when dating [1,3], the use of OSL for dating mortars have proven to be successful in NW Spain, partially due to the high radiation dose of the stone materials used in ancient buildings [4-6]. In this work we apply OSL dating on mortar samples to date a key building for the history of NW Spain: the old chapel of San Breixo de Ouvigo.

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1.1. The old chapel of San Breixo de Ouvigo

The old chapel of San Breixo de Ouvigo (Os Blancos, Ourense, NW Spain) is one of the key buildings that could help us to understand the Late Roman architecture in the NW of Spain. After the archaeological excavations of A. Rodríguez Colmenero more than 40 years ago [7], it was generally accepted that this building preserves, at least, a Late Roman constructive phase, with later modifications introduced in the following centuries. However, neither stratigraphical nor chronological proofs were given in order to confirm this interpretation. The site is also distinctive in NW Spain by the (quite exceptional) use of lime mortar, the building plan (figure 1) and its orientation.

The chapel was probably abandoned between the Middle Ages and the beginning of the modern ages [7]. The present remains consists of the south wall (reused as the wall of a stone building) and a part of the north, east and west walls. It is interpreted as a one nave church, rectangular in plan of 9×6.5 m, with a partition at its eastern side to delimiting apse.

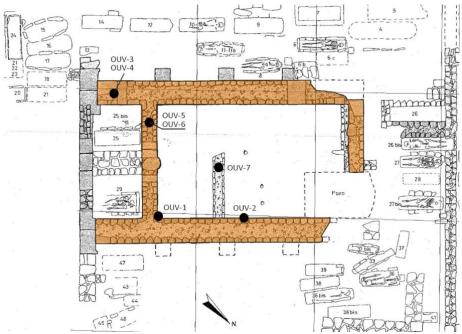


Figure 1. Plan of the building with location of samples.

As mentioned, the site was excavated between 1972 and 1981 [7]. Five constructive phases were then identified:

- (i) an early roman building, without built remains, but the roman materials being reused;
- (ii) the present building with a stucco render in the inner walls;
- (iii) the reinforcement of the walls, adding 0.4 m and a new stucco render;
- (iv) the opening of a well as storage silo in the NE corner;
- (v) abandonment of the building.

The study of the surrounding necropolis provided roman and late roman graves, anthropomorphic graves and a tombstone with an inscription dated to the year 909 AD. Roman, late roman and medieval pottery was also recovered during excavations.

Based on the excavation findings, the main question is the chronology of the second phase. Although Rodríguez Colmenero [7] admits that it could be medieval, he thinks that the materials, constructive technique and stuccos correspond to a late roman building (4th and 5th centuries). This author proposes that such building would have a nartex and a nave with an altar in the opposite side, with walls covered by stucco and paintings and mosaics on the ground. At the beginning of the 6th century, the building could have been destroyed, and rebuilt shortly after. In such period, the chapel should be surrounded by a necropolis, with graves made of *tegula* first, bathtub graves later, and "lauda" covers in medieval

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times. From the Early Mediaeval phase (8th-9th centuries) only a few archaeological objects and the necropolis with graves excavated on the rock would remain. In the 12th century a neighboring Romanesque church was built (Covelas) this building being converted into a chapel, probably in ruins in the 16th century.

The constructive technique of this chapel of Ouvigo is different to other early medieval buildings recently studied in Galicia [5]. Moreover, the use of lime mortar is also different, as other buildings of the period in the neighboring areas have earth mortars, at least between the 8th and 11th centuries. In Ouvigo, they constructed two-faced walls with an infill of stones and lime mortars. The walls have a mixed rigging, alternating three rows of ashlars with rows that combine ashlars and large stone blocks. Between both means of rigging, small regularization rows are used in order to equilibrate the different heights. Although this rigging could have similarities with others such as San Martiño de Pazó (Allariz, NW Spain) (10th century), the truth is that the construction technique of Ouvigo is quite unique.

1.2. Aim of the work

Given the importance of obtaining a good chronological sequence for this site, in 2016, two mortar samples from this building were sampled and dated by ¹⁴C, using their bulk lime [8]. Unfortunately, such samples did not provide accurate ages, as explained later. Thus, the purpose of this work is to date lime mortars of several constructive phases (figure 1) by optically stimulated luminescence dating (OSL), on the basis of the sequence proposed by Colmenero.

2. Methods

The samples were taken by cutting a block of mortar in all cases, and the block was transferred to the Luminescence Laboratory of the University of A Coruña (Spain). Under subdued red light, the outer part of the samples was removed (0.3-0.5 cm), extracting the inner part to obtain quartz from the 250-180 µm grain size of the aggregate by procedures specified elsewhere [9]. For the luminescence measurements, small multigrain aliquots (1 mm in diameter) were prepared, mounted on stainless steel discs. The measurements were taken in a Riso DA-15 automated TL/OSL reader equipped with blue light emitting diodes (LEDs) (470 \pm 30 nm) for stimulation and a 9235QA photomultiplier. A Hoya U-340 filter was placed between the photomultiplier and the samples. To irradiate the samples, beta doses were used, utilizing a 90 Sr/ 90 Y source which provided a dose rate of 0.110 \pm 0.003 Gy s-1. To estimate the equivalent doses (D_e), the single-aliquot regenerative dose (SAR) protocol was used and recovery tests were carried out [10-11].

Bulk samples were analyzed by X-Ray Fluorescence Spectroscopy and Mass Spectrometry with Inductive Coupling Plasma (ICP-MS) to assess the concentration of K, U and Th. To estimate the annual dose (D_r), the alpha dose was ignored and the beta dose corrected due to an HF etching step in the quart purification procedure, using dose attenuation coefficients [12]. Conversion factors of Guerin et al. [13] were employed. The gamma and cosmic doses were estimated by means of a GF Instruments Gamma-Ray spectrometer model Gamma Surveyor Vario, measuring with and without the use of a lead collimator. A geometrical model was considered as proposed by Feathers et al. [14] to check results (including an estimate of the cosmic dose following Prescott and Hutton [15]).

3. Results

The concentration of U, Th and K radioisotopes is high in both lime mortars and the surrounding rock, the obtained D_r s ranging between 4.44 and 5.17 mGy a⁻¹. The distribution of aliquots is different in the first two samples (OUV-1 and OUV-2) and the others (table 1). In such samples, non-skewed distributions are observed with a low overdispersion of the Central Age Model (CAM) [16]. For these two samples this is the model used to assess the age. For the other samples, a lightly skewed distribution is observed, the overdispersion values being higher in samples OUV-5 and OUV-7. In such cases, we have used the Minimum Age Model (MAM) [16] for assessing the age.

Results provide a similar age for samples OUV-1 and OUV-2 that are very similar, corresponding the periods 302-406 AD (1663±52) and 241-409 AD (1692±84), respectively (table 1). These ages

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correspond to the second phase of the building following Rodríguez Colmenero's chronological proposal [7] and fall in the expected historical period considering archaeological data. Samples OUV-3 and OUV-7 provide similar ages, 993-1129 AD (956±68) and 1103-1270 AD (830±84), respectively. These ages could correspond to the same phase, indicating that the identified phase should be younger than expected [7]. The sample OUV-5 correspond to an intermediate phase, dated between 667 and 764 AD (1302±48). A sample taken in 2016 in the same building, and dated by radiocarbon of carbonate contained in the mortar was incorrectly calibrated as marine sediment in the laboratory and published as 9th-13th centuries [8]. However, it actually provided an age of 545-642 AD, close to the OSL age. A second sample was intended to date organic matter from the mortar but the obtained age was 14th century AD, probably because of later plant contamination. Thus, OSL dating results seems to provide consistent period that fit some of the archaeological expectations but requiring the need for a new interpretation of the chronological model assigned to the building. Such model could be corroborated in a next phase of the project that intends to characterize the mortars and using radiocarbon dating. In the same way, it will be necessary to carry out a stratigraphic reading that will allow the results to be framed within the phases preserved in the building.

Table 1. Mortar types, corresponding constructive phase and expected age of the samples. Estimated dose rates (D_r) , equivalent doses (D_e) and obtained OSL ages.

Sample	Mortar type	Phase	Expected age	D_r	D _e (Gy)	Age (a)
				$(mGy y^{-1})$		
OUV-1	Lining mortar	2	4 th -5 th century	4.55±0.10	7.34 ± 0.36	1663±52
OUV-2	Joint mortar	2	4 th -5 th century	4.44±010	7.30 ± 0.28	1692±84
OUV-3	Wall infill mortar	3?	4 th -5 th century	4.74±0.11	4.62±0.31	956±68
OUV-5	Wall infill mortar	3?	7 th -10 th century?	4.66±0.11	6.07 ± 0.18	1302±48
OUV-7	Wall infill mortar	4	12th-15hth century	5.17±0.11	4.18±0.41	830±84

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