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DELIMITING THE URBAN GROWTH OF SANTIAGO DE COMPOSTELA (NW SPAIN) BY OSL DATING OF MEDIEVAL ANTHROPOGENIC SEDIMENTS

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ABSTRACT

A group of 22 hollows filled with sediment were found excavated in the ground during the construction of an auditorium in the city centre of Santiago de Compostela (NW Spain). This city is part of the UNESCO World Heritage. The hollows were from 3.30 m to 0.8 m depth and from 1.5 to 1 m diameter. Fourteen of them were excavated and some materials recovered (sediment, charcoal and fragments of vessels) to date the infill. The hollows were used to move and store water (as most of them are connected by channels), and their characteristics and arrangement are similar to leather tanning workshops found in the same city. Samples of the sediment infill recovered from two hollows and a pottery fragment taken from another one, were dated by OSL and TL, respectively. Recovered charcoals were also dated by AMS ^{14}C for independent age comparison. OSL signals were suitable for dating and evidences of partial bleaching of quartz grains extracted from the sediment were observed from small multi-grain aliquots. The Minimum Age Model was used to calculate the equivalent dose and OSL results provided ages in agreement with independent ages, providing crucial information on the city growth in medieval times.

KEYWORDS: luminescence dating, hollows, sediment infill, anthropogenic sediments, urban growth

1. INTRODUCTION

Optically Stimulated Luminescence (OSL) dating has been widely applied to Quaternary sediments, in particular using quartz grains, and less to archaeological sediments due to the uncertainty about complete bleaching of the residual OSL signal before burial related to the formation of the sites. Such uncertainty is due to the lack of knowledge on the deposition process of the sediments. From first studies of archaeological sediments by Grogler et al. (1958), different experiences (see Roberts, 1997 and Feathers, 2003, for review) have been carried out and archaeological cave sediments (Jacobs et al., 2006), sediments of rock shelters (Jacobs and Roberts, 2007), surroundings of megalithic stones (Liritzis, 1994), colluvial deposits with archaeological materials (Lang and Warner, 1997), anthropogenic soils (Burbidge et al., 2001; Vafiadou et al., 1997) or sediments from tells in the Near East (Sanjurjo-Sánchez et al., 2008; Sanjurjo-Sánchez and Montero Fenollós, 2012) have been dated. In particular, few reports exist on the reliability of OSL dating of sediments of artificially deposited sediments on pits and hollows (Lang et al., 1999; Kinnaird et al., 2007; Liritzis et al, 2013a). These sediments were presumably deposited by human action in the past, so there is uncertainty about whether the geological signal of the sediment grains has been completely and homogeneously reset prior to burial, as information concerning the sediment deposition is unknown.

1.1. Archaeological site

Santiago de Compostela (NW Spain) has been one of the most important catholic centres of Europe from medieval times to the present time. The old town of the city is part of the UNESCO World Heritage. During works for the construction of an auditorium in the old town several archaeological remains were found. Written documents indicated that the studied site was occupied by a medieval palace called '*Pazo de Ximonde*'. The palace was demolished and successive buildings were built in 1893 and 1970. The present remains indicated the situation and arrangement of the former palace, including foundations of the external façade and some internal walls (with a yard). The façade was built on previous hollows (filled with sediments).

Up to 22 hollows were bored in the schist rock, and filled with clay-rich sandy sediments previously to the construction of the palace. Nineteen of them had circular section (they were cylindrical) and other three had a rectangular section (Fig. 1).

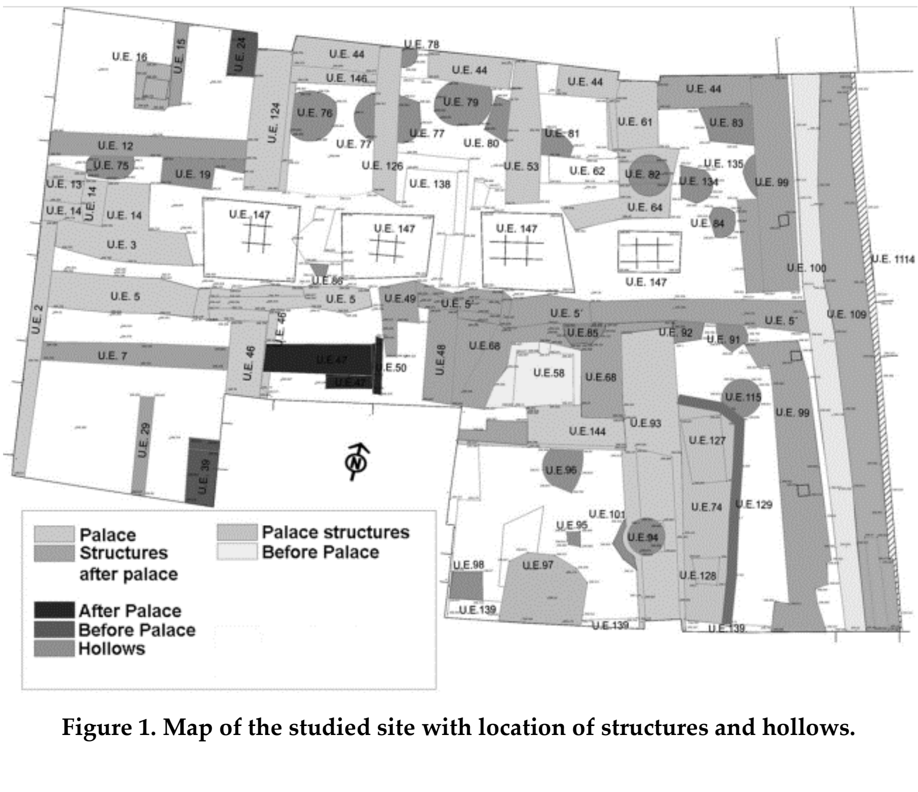

Figure 1. Map of the studied site with location of structures and hollows.

The depth of the hollows was from 3.30 m to 0.8 m and the diameter section from 1.5 to 1 m (Fig. 2).

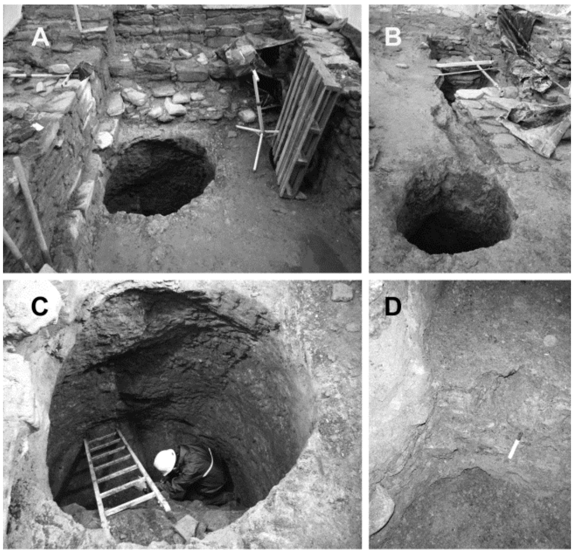

Figure 2. Pictures of the studied hollows: (A) hollow P-76; (B) circular hollow P-115 connected by a channel with rectangular cross-section hollow P-128; (C) sampling within the hollow P-76; (D) sediment profile of the hollow P-76.

They were connected by a channel excavated on the ground rock with a smooth slope for water movement, in the same manner that in other nearby fields of silos. Such structures have been described in other parts of the same city as workshop of leather tanning (Armas Castro, 2003). The studied site is located inwards the second city walls of the city (Fig. 3).

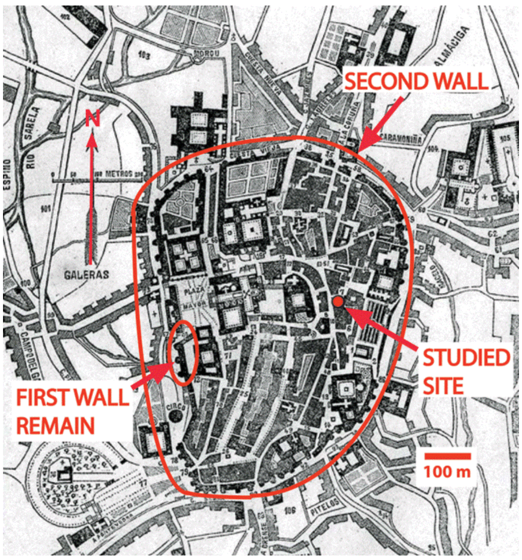

Figure 3. Map of the present centre of Santiago de Compostela. The second wall can be clearly noticed in the map and the studied site is marked with a circle and an arrow.

1.2. Previous chronological data

Santiago de Compostela was founded between 830-880 AC (*Anno Christi*), with the construction of a Sanctuary and an enclosure. The first city walls were built by the Bishop Sisnando II (960-968 AC). The city was destroyed in 997 AC by Muslims and reconstructed with a new size and a second city wall, outwards the first walls, by the Bishop Cresconio (1037-1066).

Some written documents refer these second wall (between 1037 and 1140 AC) and there exist some remains, so there is agreement among archaeologists about their location, although the finishing age of the construction of such walls is unknown (López Alsina, 1989; Armas Castro, 2003; González Méndez and Luaces Anca, 2009). However, there is a lack of knowledge on the arrangement of the first wall and diameter of the first city before destruction.

In medieval times, some activities were considered as unhealthy or harmful and were moved outwards when cities were enlarged. Such activities included workshops of leather tanning (Armas Castro, 2003). The studied site is located inwards the second walls but it is probably to be located outwards the first walls. Therefore, the hollows were probably filled to move the workshop outwards the new walls due to the urban growth.

1.3. Objectives

The aim of this work is to date the anthropogenic sediment infill of some of the hollows by OSL (Fig. 2).

The sediments were artificially deposited (probably using small tools), so there is uncertainty about the origin of the infill and the bleaching of the OSL signal in the sediment grains.

Charcoal dating provides independent ages, but due to the probable use of nearby soil material, such charcoal could correspond to burned wood of older age and overestimate the age of the sediment infill. If infill of the hollows is dated we can get information on both the age of construction of the second city walls and the location of the first walls in this side of the city, as there is not agreement about the arrangement of such first walls.

Some researchers think that the first walls were located surrounding the area of the ancient sanctuary and present cathedral (inwards the studied site, Fig. 3), but others think that the first city was larger, and the walls were located outwards the studied site (López Alsina, 1989; Armas Castro, 2003; González Méndez and Luaces Anca, 2009). OSL dating could help to the location of the first walls in this side of the city, considering the archaeological information of the studied site.

2. METHODS

2.1. Sample collection

Sample collection was performed during the archaeological survey. After excavation of the first metres of sediment of several hollows the bottom 1.5 m of sediments were partially excavated (a half of the infill) leaving a sediment profile for visual inspection of sediments and sampling. PVC cores 30 cm long and 10 cm diameter were introduced hammering into the sediment infill profile. Two sediment samples and charcoal were collected from one of the circular hollows (P-76) and another one from a hollow of rectangular cross-section (hollow P-128, sample PL-1). The latter had 0.7 m depth and the core was taken from the middle of the sedimentary profile.

To test the reliability of the OSL ages, both a vessel fragment and charcoal were taken from the sediments of a next hollow (P-115) and dated by TL and AMS radiocarbon, respectively, to crosscheck OSL dates (see table 1 for more details). Radiocarbon analysis were performed by Beta Analytic Inc., and resulting ages calibrated by Oxcal 4.2 (Bronk Ramsey et al., 2010) based on IntCal09: Northern Hemisphere curve (Reimer et al., 2009),

Table 1. Results of the radionuclide specific activities and content (P-115C*) of the samples.

Sample	Depth (m)	Water content (%)	Activity Concentration (Bq kg^{-1})						Element Concentration		
			^{232}Th	^{235}U	^{238}U	^{226}Ra	^{210}Pb	^{40}K	Th (ppm)	U (ppm)	K (%)
P-76-1	2.45	28±5	62.2±2.6	2.7±0.9	51±12	66.9±3.9	99±12	788±35	-	-	-
P-76-2	2.05	27±4	50.4±2.2	1.6±0.8	39±10	69.2±4	67±10	735±33			
P-115C*	1.7	16±3	-		-	-		-	15.20±0.76	16.15±0.81	3.05±0.15
P-115C	1.7	26±4	64.6±2.6	2.7±0.9	46±11	80.7±4.6	119±12	719±32			
PL-1	0.35	15±2	73.4±3.2	2.1±0.6	41±10	47.5±2.8	43±8	688±30			

2.2. OSL and TL equivalent doses

For OSL dating the sediment cores were opened in subdued red light. Grains from the central part were dried and sand grains of 90-180 μm were extracted by sieving. After sieving, grains were water-washed and treated with 10% HCl and 10% H_2O_2 to remove carbonates and organic matter, respectively. Concentrated HF was firstly applied to remove feldspars and to remove the surface of quartz grains to eliminate the alpha contribution. 10% HCl was applied again to remove any remaining soluble fluorides and grains were dried. Aliquots of all samples were checked with infrared (IR) stimulation to ensure the absence of feldspar contamination.

A blue-OSL (BL-OSL) single-aliquot regenerative dose (SAR) protocol was used to estimate the equivalent doses of quartz grains (Murray and Wintle, 2000). Measurements were performed at 125°C for 40 seconds. The first 0.8 seconds were used to measure OSL and the last 4 seconds for background. Preheat temperatures were chosen after performing preheat temperature tests for all samples. The test-dose response was measured after heating to 160°C (cut-heat). Moreover, recovery tests (Murray and Wintle, 2003) were carried out in all subsamples. Aliquots were first bleached in the OSL reader (200 s blue light at room temperature). A known beta dose was given approximately equal to the natural dose.

For the pottery sample a 2 mm layer from each fragment surface was removed by sawing with a diamond wheel. The sample was gently crushed in a vice and grains within the diameter range 90-180 μm were extracted by sieving. The same chemical procedure used for sediments was applied to such grains. The quartz extract was used to assess the equivalent doses by TL using the multiple aliquot additive dose protocol (Liritzis et al., 1997). Beta irradiation was performed two weeks before TL measurements. Also, a regeneration glow curve was performed after first glow and normalized by performing TL measurements on the previously irradiated and measured aliquots with a same beta dose. To test for sensitivity changes first and second TL growth curves where compared.

All measurements were made on an automated Risø TL/OSL-DA-15 reader equipped with an EMI 9635 QA photomultiplier tube, and using an internal $^{90}Sr/^{90}Y$ source that provides 0.130±0.003 Gy s^{-1}. To measure OSL a little amount of sample grains (<100 grains) were mounted on stainless steel discs using silicone oil. TL measurements were made mounting quartz grains on stainless steel cups. Both OSL and TL measurements were carried out with an optical filter Hoya U-340.

2.3. Annual dose rates

The annual dose rates were estimated in the laboratory using High-Resolution γ-Spectrometry (HRGS) for the sediment samples, for measuring the ^{238}U, ^{235}U, ^{232}Th and ^{40}K decay chain activities. Also, HRGS was used to estimate the gamma dose rate of the sediment portion surrounding the pottery sample. To estimate the beta-dose of the pottery the U, Th and K content were measured by Inductive Coupled Plasma-Mass Spectrometry (ICP-MS).

Conversion factors of Adamiec and Aitken (1998) were used to calculate the beta and gamma contribution to the annual dose rate. Comparison with updated factors are within the historical means corresponding to errors except for the Th-232 alpha dose and beta for K-40 (~5%) (Liritzis et al., 2013b). But all fall within final age equation error. The alpha contribution was neglected and the beta attenuation was considered (Mejdahl, 1979), as the HF step eliminated the surface layer of quartz grains by etching. The water content and saturation were assessed in the laboratory by weighing before and after drying, and measuring the maximum water content on saturated samples, respectively. The cosmic dose rates were calculated according to Prescott and Hutton (1994).

Table 2. Calculated dose rates for the studied samples.

Sample	Beta dose (mGy a^{-1})	Gamma dose (mGy a^{-1})	Cosmic dose (mGy a^{-1})	Dose Rate (mGy a^{-1})
P-76-1	2.24±0.4	1.48±0.27	0.16±0.01	3.88±0.48
P-76-2	2.03±0.3	1.39±0.21	0.17±0.01	3.59±0.37
P-115C	4.52±0.26	1.59±0.25	0.18±0.01	6.27±0.18
PL-1	2.19±0.43	1.57±0.32	0.16±0.01	3.93±0.54

3. RESULTS AND DISCUSSION

3.1. Annual dose-rate

The HRGS measurements of the radioactive content of the sediment samples and the U, Th and K content of the pottery sample are shown in the Table 1. Some disequilibrium has been observed in most sediment samples. Samples from the hollow P-76 show similar activities for ^{238}U, ^{226}Ra, ^{235}U and ^{40}K, but differences in the ^{232}Th decay chain and ^{210}Pb activity (enrichment in the upper sample P-76-2). However, the calculated ratios for $^{238}U/^{232}Th$, $^{226}Ra/^{232}Th$ and $^{210}Pb/^{232}Th$ (Degering and Krbetschek, 2007) are not far from 1. Such slight disequilibrium would come (at least partially) from the sediment used for fill in the hollow or could be caused by slight loss of ^{238}U, above all in the sample upper P-76-2. In any case, the differences in the dose rate of P-76-1 and P-76-2 would be of 1.5% and 2.7%, respectively. The difference in age results amount for 14 and 25 years, respectively, within the limits of measuring uncertainties. A higher depletion of ^{238}U (respect to ^{226}Ra) is observed in the sediment surrounding the pottery sample P115-C. Also, important addition of ^{210}Pb is observed in this sediment. Such disequilibrium provides a difference of 4% in the dose rate, if we assume equilibrium when the sediment was deposited. However, as the sediment dose rate accounts for a part of the gamma dose, the difference in the age result is only about 10 yr. The sample PL-1 shows very little loss of ^{238}U and a difference of 0.5% in the resulting dose rate assuming U loss.

The water content results more difficult to assess with precision. The samples P-76-1, P-76-2 and P-115C were taken at less than 1 m of the bottom schist rock, in the vadose zone but next to the present water table. Santiago de Compostela is located in a very humid area with about 2000 mm of precipitation per year. Thus, such samples were probably near saturation most of the burial time, although some summer drought could be expected. Such oscillation of the water table makes difficult to carry out a precise calculation of the mean water content.

The sample PL-1 correspond to a rectangular hollow of 0.6 m depth, so the sample was collected at 0.35 cm from the surface and was not saturated most of the burial time. Our estimates provided mean water contents below 30% for samples P-76-1, P-76-2 and P-115-C sediments (16% for the pottery) and 15% for PL-1. Such values fall into typical densities for bulk sediments (Guerin and Mercier, 2012), but the uncertainty is the cause of an error higher than expected.

Cosmic ray contribution is low but some problems should be considered to calculate them. Although the depth of the dated samples in the hollows has probably remained, a medieval building, and up to other two buildings were constructed on them, and so the cosmic dose have probably oscillated. However, calculated changes in such cosmic ray dose are lower than a 1% of the total dose rate. The resulting dose rates are very similar for the sediment samples (3.5-3.9 mGy yr^{-1}).

3.2. *Equivalent doses*

The OSL signal showed bright and fast signal decay, and high signal to noise ratio, higher than expected for medieval samples with low expected doses (Fig. 4a). Prior to the OSL measurements, preheat tests showed similar equivalent doses for all samples (Fig. 4b) at different preheat temperatures (from 160°C to 280°C, five aliquots for each preheat temperature) with recuperation below 10% and recycling ratios between 0.9 and 1.1 at all temperatures. A 200°C preheat temperature for 10 s and 180ºC cutheat were chosen for SAR measurements.

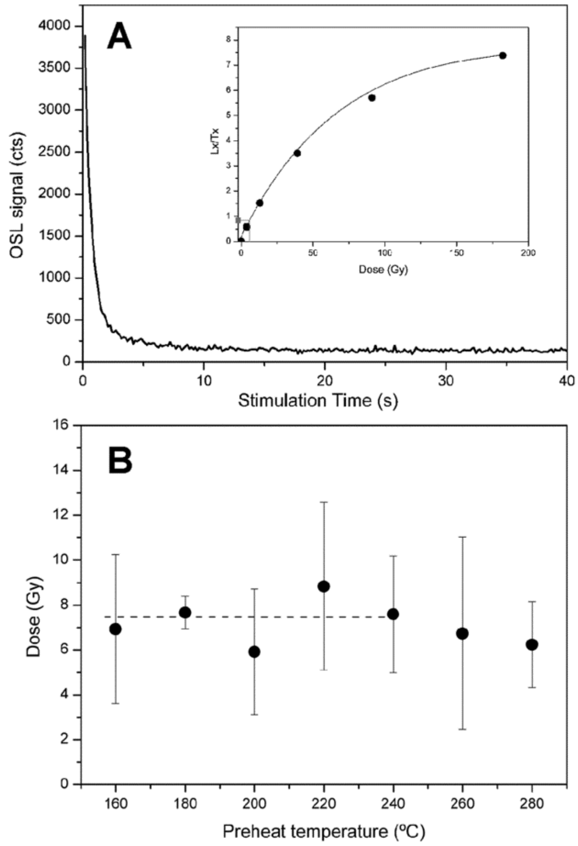

Figure 4. (A) OSL decay signal and OSL growth curve (inset) of an aliquot of the sample P-76-2, (B) Results of the preheat test of the sample PL-1.

Thermal transfer tests (Rhodes, 2000) were also performed and they showed negligible transfer of charge from thermally shallow light-insensitive traps to deeper light insensitive traps when aliquots were preheated between 180°C and 300°C for 10 s (Fig. 5).

The recovery tests (Fig. 6) showed acceptable ratios within errors, in the range between 0.9 and 1.1 although important errors probably due to the OSL sensitivity variability within grains (Murray and Wintle, 2003).

To assess if the OSL signal is dominated by the fast OSL component and it is quickly bleached by light exposure, the natural Linearly Modulated OSL (LM-OSL) has been measured in quartz grains of the three sediment samples. The LM-OSL has been used to study the easy to bleach trapped charge in quartz comparing the LM-OSL curves of quartz following a laboratory beta dose and different bleaching times (Agersnap Larsen et al., 2000).

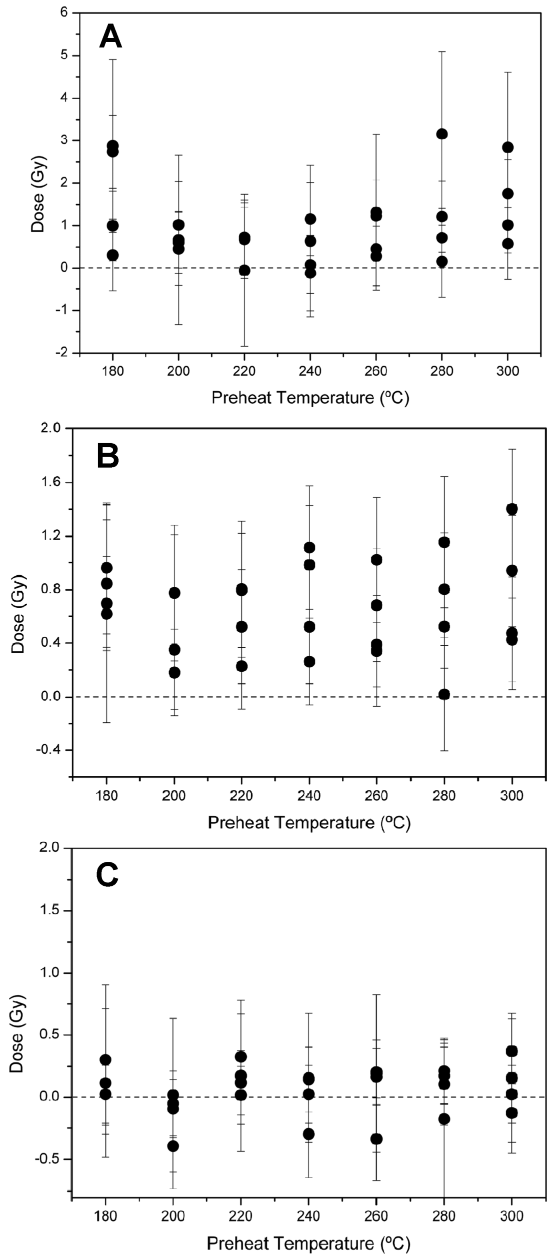

Figure 5. Thermal transfer test of the samples (A) P-76-1, (B) P-76-2, (C) PL-1.

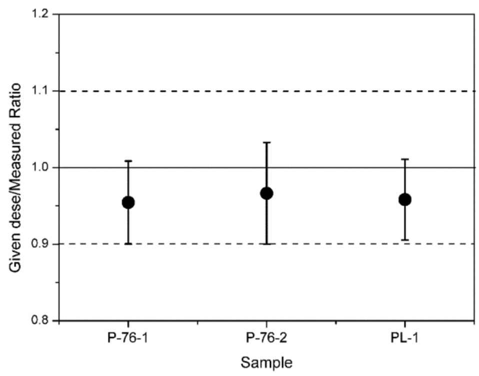

Figure 6. Recovery dose tests of the samples P-76-1, P-76-2 and PL-1.

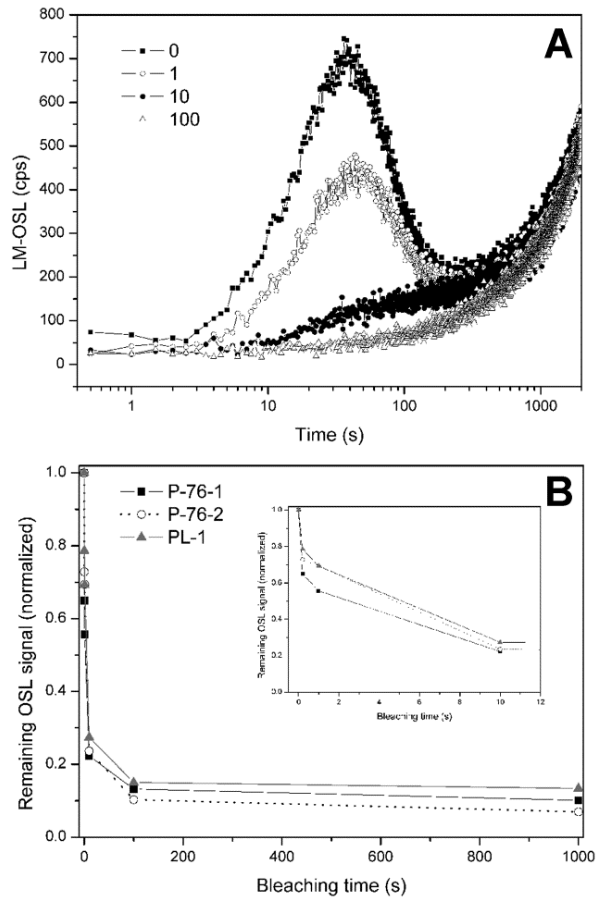

Figure 7. (A) LM-OSL curve of the sample P-76-2 after bleaching with blue diodes for 0, 1, 10 and 100 seconds; (B) bleaching curves of integrated fast LM-OSL component after different bleaching times with blue diodes for the sediment samples (inset with detail of the section after bleaching 0.5, 1 and 10 seconds).

Such observations indicated that the OSL of our samples was dominated by a fast component (Fig. 7a), so the doses measured by the SAR protocol will correspond to a dominant fast OSL signal. Moreover, exposure of the irradiated samples to light exposures (with blue diodes in the Risø equipment) of 0.2, 1, 10 and 100 s, showed that most of the fast signal (~75%) was bleached in all the samples after 10 s of exposure (Fig. 7b).

Radial-plots and histograms were used to assess possible partial bleaching (Fig. 8).

Samples from the hollow P-76 showed aliquots with both high equivalent doses (higher than expected) and inter-aliquot dispersion of doses.

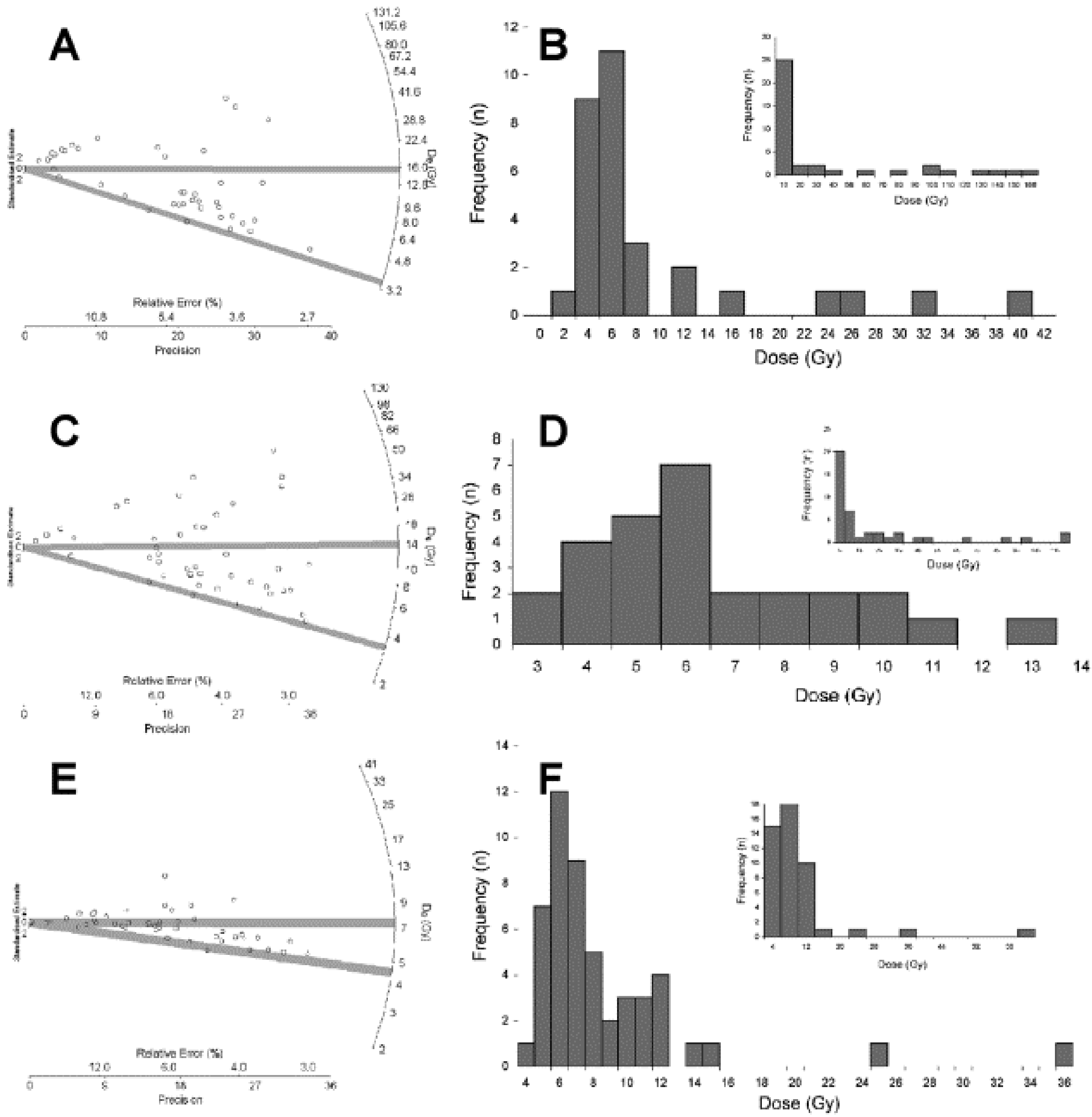

Figure 8. Radial-plots and histograms of the equivalent doses measured on multi-grain aliquots for the samples (A, B) P-76-1, (C, D) P-76-2, (E, F) PL-1.

The equivalent doses calculated by the Central Age Model (CAM, Galbraith et al., 1999) showed high doses and very high overdispersion values (142% and 120% for P-76-1 and P-76-2, respectively).

This can be indicative of incomplete bleaching of the OSL signal. We have used the Minimum Age Model (MAM, Galbraith et al., 1999) to calculate the equivalent doses of such samples. Such age model provided very similar equivalent doses (~3.5 Gy) for both samples. The sample taken from the rectangular hollow P-128 (PL-1) provided less inter-aliquot dispersion and lower equivalent doses.

However, the CAM provided a too high equivalent dose (~7.4 Gy) and an important overdispersion (34%). The MAM provided a more confident equivalent dose (~4.5 Gy).

The plateau test of the pottery sample (P-115) provided a plateau between 280°C and 340°C (Fig. 9). The first and second TL growth curves provided an equivalent dose of (~6 Gy).

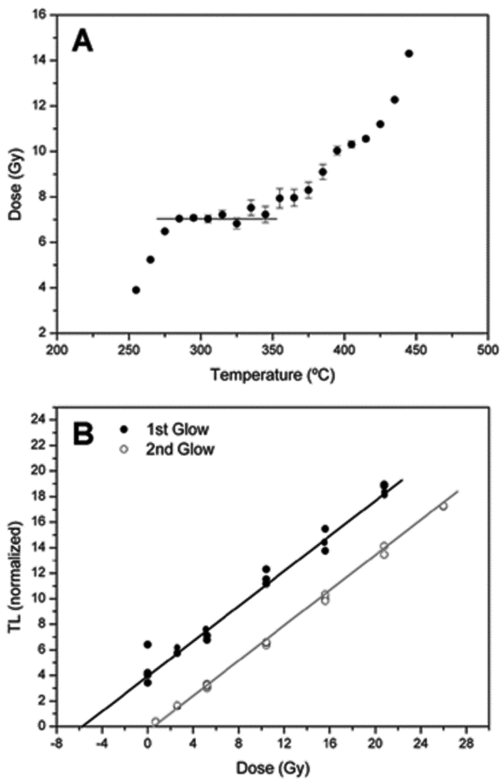

Figure 9. Plateau test and TL growth curve (first and second glow) of the sample P-115C.

3.3. Ages

The OSL age of the sediments taken from the hollow P-76 fit very well and they are in agreement with the dated charcoal. The OSL ages indicated that this hollow it was filled between the end of the 10th and the beginning (destruction of the city) of the 13th Centuries although the calibrated radiocarbon age error provides a shorter time span corresponding to 1020-1120 AC (Table 3).

The OSL age of the infill of the hollow P-128 (sample PL-1) also agrees with these OSL within errors, but we cannot discard the hypothesis that it was filled before (653-1050 AC). This possibility will be consistent with our hypothesis as this hollow was completely different than the silos. The TL age of the pottery recovered from the hollow P-115 is in agreement with the charcoal calibrated age and it also matches the P-76 ages.

Considering all ages (sediment, charcoal and pottery) and assuming that they correspond to the same period (this can be considered a straightforward regarding the history of the city, our archaeological hypothesis and the resulting ages), the ages are coincident in an age interval between 950 and 1020 AC. The weighted mean of the ages give a time span of 990-1052 AC. This time intervals fit the age of destruction of the city (997 AC).

Thus, the hollows were probably filled and sealed immediately after the destruction of the city. This is the moment when the city is reconstructed and the new walls built. This is indicative that the studied site was located outwards the first walls (second half of 9th Century to the end of the 10th Century) and inwards the second walls (from the first half of the 11th Century) of the city.

Table 3. Equivalent doses and final OSL and radiocarbon ages of the studied samples. * Anno Christi: luminescence or calibrated ^{14}C.

Sample	Depth (m)	Lab code (AMS)	Grain size (μm)	Aliquots (n)	Dose Rate (mGy a^{-1})	Equivalent dose (Gy)	Luminescence Age (yr)	^{14}C Age (yr BP)	Calendar Date (AC)*
P-76-1	2.45		90-180	43	3.88±0.48	3.52±0.39	908±151		1103±151
P-76-2	2.05		90-180	45	3.59±0.37	3.44±0.61	959±196		1052±196
P-76-S-RC	2.05	Beta-2 73038	-	-	-	-	-	950±40	1070±50
P-115C	1.7		90-180	50	6.27±0.18	6.01±0.86	958±140		1053±140
P-115-RC	1.7	Beta-273039	-	-	-	-	-	1080±40	955±50
PL-1	0.35		90-180	50	3.93±0.54	4.55±0.47	1157±198		854±198

4. CONCLUSIONS

We have dated artificially deposited sediments that fill medieval hollows by OSL on small multi-aliquots of quartz grains. Despite the apparent incomplete bleaching, OSL seems to be reliable if the equivalent doses are calculated with the Minimum Age Model (MAM). The calculated ages are in agreement with independent radiocarbon ages of charcoal found in the sediments and expected archaeological ages. The TL of a pottery age also has provided an age that fit the OSL ages.

The use of OSL dating in similar archaeological sites can be useful to reconstruct and locate urban structures such as ancient city walls (e.g. ditches, pits). In this case, luminescence allows locating the maximum area reached by the first medieval wall in a city side of Santiago de Compostela (Spain).

Moreover, similar studies in other similar sites (some of them recently found in the city) could allow reconstruct a more complete location of the first walls.

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