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LUMINESCENCE AND MINERALOGY OF PROFILING SAMPLES FROM NEGATIVE ARCHAEOLOGICAL FEATURES

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ABSTRACT

Ditch and pit fills from four archaeological sites in the Baixo Alentejo region of Portugal were studied to explore the relationship between Luminescence Profiling (IRSL, OSL, TSL, sensitivity and sensitization) and X-ray diffraction (XRD) analyses. Series of small (ca. 10 g) samples were collected, through the different fill materials. XRD results show that the predominant mineral is calcite (40-90%), and this exhibited the strongest relationships with luminescence behaviour. In the present sites it was often related to high absorbed doses, and so to residual signals rather than low dose rates. Commonly, the calcite content and the absorbed dose value tended to increase from the top to the bottom of a section. Layers with lower calcite content and residual luminescence signals were used to stratigraphically define different phases of accumulation. The best reset luminescence signals for dating at these sites come from the layers with the lowest calcite content. In other cases similar absorbed doses through archaeological stratigraphies, independent of composition, were interpreted to indicate that signals in the different mineral fractions were well reset prior to accumulation in the structure.

KEYWORDS: luminescence, archaeology, profiling, mineralogy

INTRODUCTION

The construction of the irrigation network related to the Alqueva Dam in Alentejo, Portugal, is exposing a wide range of negative archaeological features thought to relate chronologically to the better known Megalithic cultures of SW Iberia (Valera, 2010; Valera et al., 2010a, 2010b).

Ditch and pit fills from four sites in the Baixo Alentejo region of Portugal were studied: Cortes (COR), Monte Carrascal II (CII), Horta dos Quarteirões 1 (HQ1) and Covas (COV). The structures were thought to be Chalcolithic or Neolithic (Valera, 2010; Valera et al., 2010a, 2010b) and since the human abandonment occurred, they suffered stratified accumulation of materials and artefacts.

The negative structures were originally excavated in poorly consolidated calcite-rich geological substrates in the Ossa-Morena Zone (OMZ): CII in Miocene deposits overlying gabbros of Beja, HQ in acid metavolcanic rocks of Moura-Ficalho antiform (Schist and calco-schists), COR and COV included in Miocene deposits of Moura basin formation (Araújo, 2006; Barros e Carvalhosa and Galopim de Carvalho, 1970; Oliveira, 1992; Pais et al., 2012).

Mineralogical analysis by X-ray diffraction (XRD) is based on the detection of maxima in diffraction of a monochromatic beam of X-rays by crystalline structure at different angles, according to the Bragg equation. Data are provided in the form of diffractograms that allow the identification and the semi-quantification of the potentially present minerals (Brindley and Brown, 1980; Schultz, 1964).

To this end, the obtained data are comparing with precisely known reference angle and intensity data and the semi-quantification is made considering the principal peak and weighting factors estimated data for each present mineral phase.

Being a rapid method that allows the rapid identification of mineral assemblage, it is commonly used as a first approach to the study of soils, sediments and archaeological materials as well as in the study of mineralogical profile variations. Previous works showed that the predominant mineralogy in the geological context of the present study is essentially calcite associated with some quartz, feldspars (in very small quantities) in proximity of granitic rocks and clay minerals, mainly palygorskite, smectite and kaolinite (Barros e Carvalhosa and Galopim de Carvalho, 1970; Marques et al., 1990; Abreu et al., 1990; Dias, 1998).

The mineralogical composition of this carbonate is variable in function of geological contexts, in particular in function of the bedrock below these layers (Pimentel, 2002; Durand et al., 2006; Kaplan, 2013).

Mineralogical analysis is commonly conducted on materials from the same unit but exhibiting different degrees of weathering, and on complete weathering profiles. In the region of study, dolomite is sometimes found, depending on weathering state, and hematite is observed in more reddish soils (Pimentel, 2002; Prudêncio et al., 2011).

Prudêncio et al. (2011) describe a mineralogical profile through a similar context (pedogenic calcrete profile at Tunisia).

In this profile calcite and phyllosilicates dominate, but calcite increases with depth while phyllosilicates decrease. K-feldspars were absent at the surface but increased with depth, while plagioclase occurred only at the surface.

Luminescence profiling is a semi-quantitative method to rapidly determine absorbed dose and evaluate variations in the behaviour of luminescence signals using stratigraphically detailed series of samples taken through archaeological or geomorphological sequences.

This approach was applied in to sand-sized quartz and polymineral fractions by Sanderson et al. (2001; 2003), Kinnaird (this issue), and also to polymineral silt-sized grains by Burbidge et al. (2007).

Different stimulated luminescence signals: thermal (TSL) or optical with blue and infrared light (OSL; IRSL), from different mineralogical and grain-size fractions: quartz-enriched coarse (QZC), and polymineral coarse and fine fractions (PMC and PMFG) are related to differences in light-exposure history, accumulation mechanism, source material or mixing of source materials, and mineralogy.

This can contribute to efficient assessment of a site and/or sediment's suitability for luminescence dating, and help to select samples and methods for more intensive analysis, and improve understanding of sedimentary process at these sites, using its spatial resolution to identify changing environmental conditions.

Similar measurements have been applied in exploratory luminescence studies and initial tests of samples for quantitative dating (Hamel and Huntley, 2003; Spencer et al., 2003; Burbidge et al., 2010; Roberts et al., 2009).

In this case the analyses are used to circumvent lengthy preparation and measurement procedures and obtain a rough approximation of the OSL age, or to evaluate protocols for absolute dating.

The aim of the present work is to relate luminescence behaviour and mineralogy for samples from profiles through four archaeological sequences from fills of negative features cut into poorly consolidated calcitic substrate, by exploring the relationship between luminescence profiling and X-ray diffraction analysis.

From the relationships that can be established, the main goal is focuses on the contribution to the understanding of the processes involved in use / closure of the archaeological structures under study

MATERIALS AND METHODS

Profile sampling was made using small tubes, of around 10 g each, from the bottom of each structure to the modern soil. Identification of the strata of interest for sampling was based on differences in the colour and texture of the accumulated fill materials.

At the archaeological site of COR, a 1 metre deep ditch was sampled. In this ditch, were identified four layers: at the bottom, fine clay with various carbonate nodules; followed by two light red/brown colluvium layers, in which there was a decrease of carbonate nodules towards the surface; these layers were sealed by brown modern soil (Fig. 1, a).

At the CII site, the sampling was made through two distinct archaeological phases through a 2.3 m deep ditch: The upper fills (Fig. 2, a) are composed of poorly differentiated "colluvium" (yellow/brown silty clay with non-oriented stones and white nodules), thought to relate to closure of the ditch.

The deeper strata (Fig. 2, a) include a series of fine layers of light brown clay and white/red nodules. These are interpreted as circulation-floor contexts relating to hypogea encountered in the ditch wall (Valera, 2010a). Two samples were also collected from lateral ditch fills, for comparative purposes (Fig. 2, a)

The HQ1 site presented a sequence at the intersection of two ditches. The fills consisted of a thick upper layer of poorly differentiated colluvium (yellow/brown silty clay with white nodules) sealing a stony layer (schist). Below this is a sequence of red/brown soils interleaved with white, calcite rich layers (Fig. 3, a).

In COV archaeological site two different profiling sequences were obtained down a 1.5 metre deep pit, a main sequence and a lateral sequence (Fig. 4, a). The main sequence consisted of brown/red silty clay with white nodules, thought to represent gradual infill of a recut.

The lateral sequence consisted of cream-coloured clay material more similar to the calcitic substrate. The lower part of this sequence was thought to represent the original infill of the feature, but the uppermost sample was taken from a shallow recut.

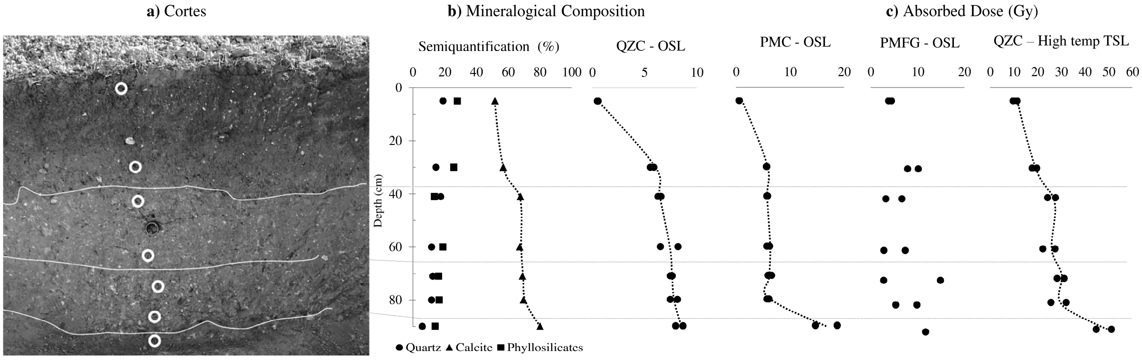

Figure 1. Cortes: Sampled profile, semiquantification and estimated absorbed dose.

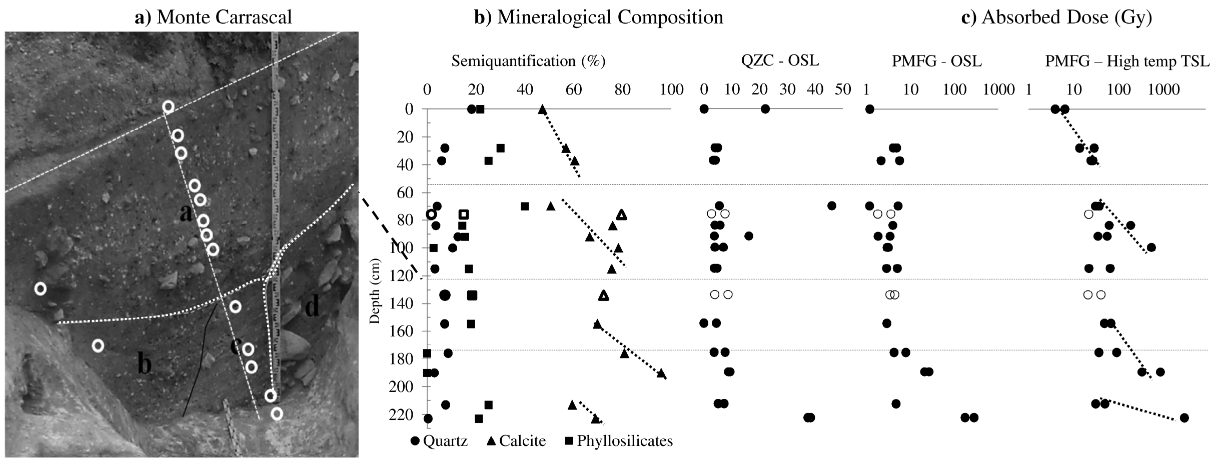

Figure 2. Monte Carrascal: Sampled profile, semiquantification and estimated absorbed dose.

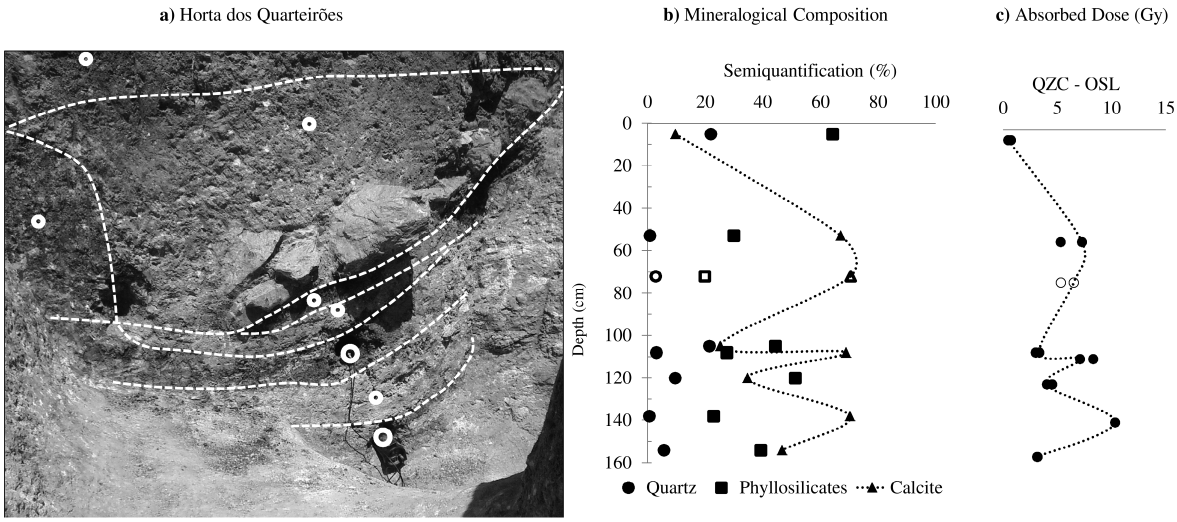

Figure 3. Horta dos Quarteirões: Sampled profile, semiquantification and estimated absorbed dose.

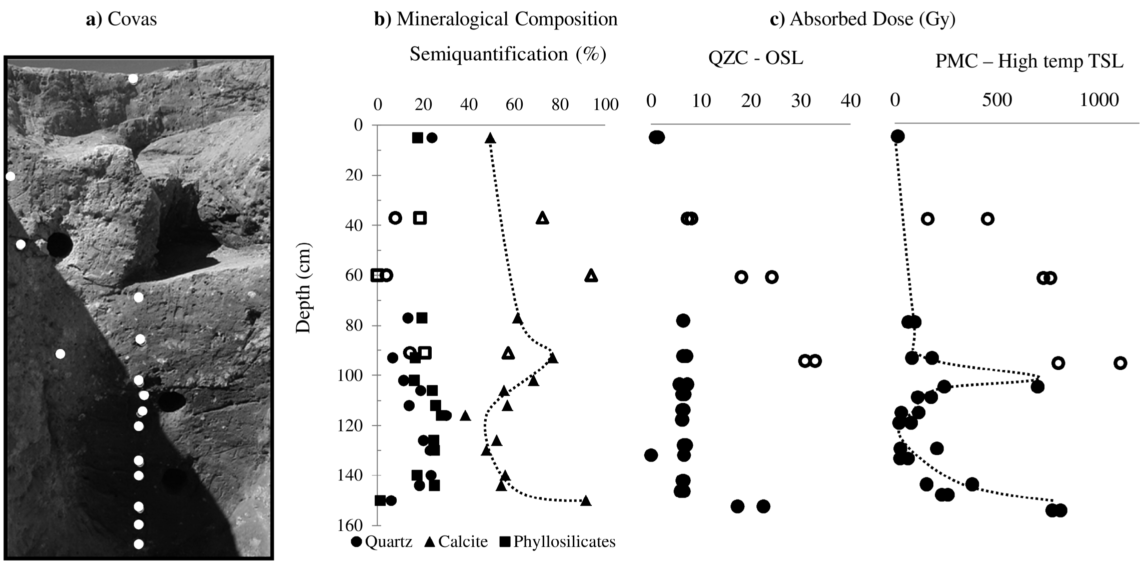

Figure 4. Covas: Sampled profile, semiquantification and estimated absorbed dose.

Sample preparation followed the procedures illustrated in Fig. 5. The material from the ends of each tube was milled to powder for XRD analysis, followed by semi-quantification of the crystalline mineral phases (Brindley and Brown. 1980; Schultz, 1964). XRD measurements were made using a Philips X'Pert Pro diffractometer, with CuKα radiation and operating at 45 kV and 40 mA, on non-oriented powder in the 4-70°2θ range, a 1° divergence slit, and a scan rate of 1° min^{-1}. Diffractograms from samples are compared with reference angle, intensityfor their identification (Brindley and Brown, 1980) estimated data for potentially present minerals. Semi-quantitative analysis of mineral assemblage was undertaken measuring the principal peak areas with intensities correction, using the recommended weighting factors (Schultz, 1964).

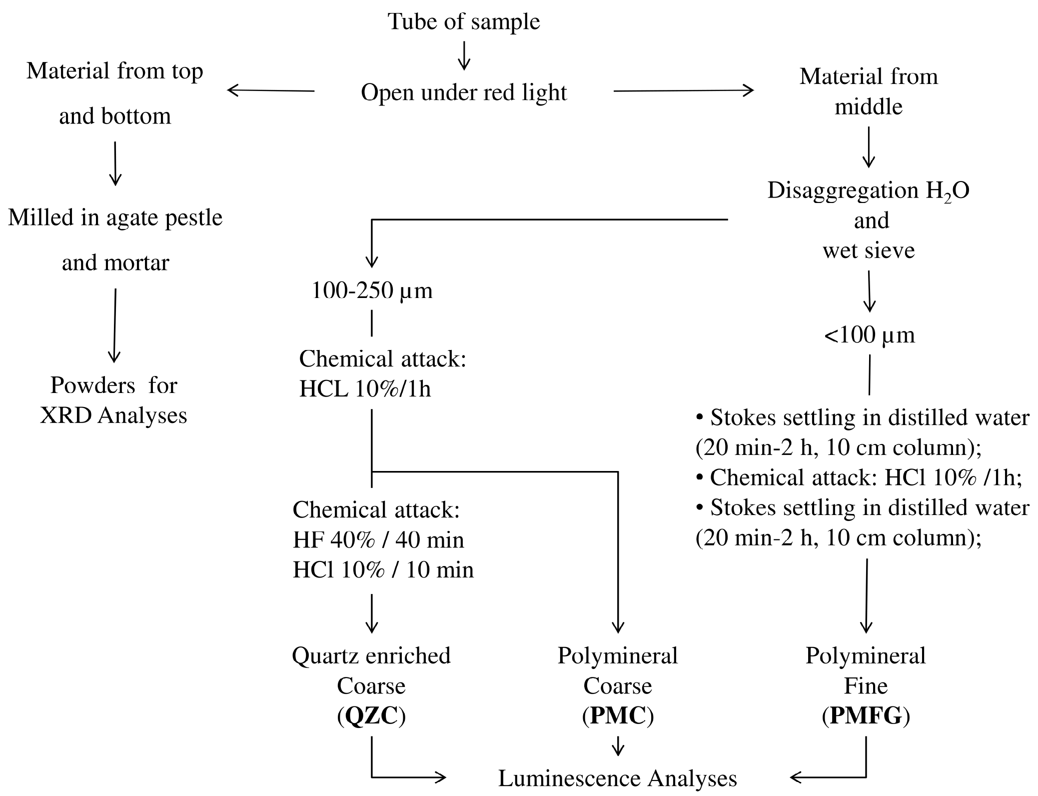

Figure 5. Sample preparation procedure.

For luminescence, the material from middle of the tubes was disaggregated, wet sieved, treated with acid and settled, to produce a coarse polymineral silicate fraction (PMC), a coarse fraction enriched in quartz (QZC), and a fine polymineral fraction (PMFG).

IRSL, OSL and TSL sensitivity, capacity for sensitization, and absorbed dose, measurements were made using a regenerative sequence with two cycles: one natural and another after irradiation with 5 Gy (Table 1).

Measurements were made using a Risø automatic reader with calibrated β source ($^{90}Sr/^{90}Y$) and U340 detection filter.

Table 1. Regenerative sequence with two cycles: one natural and another after irradiation with 5 Gy.

Sequence	2 cycles	
	Natural	5 Gy
Preheat (TSL)	240°C @ 5°C s^{-1}	
IRSL	125s @ 50°C	
OSL	125s @125°C	
Test Dose	1Gy	
Preheat (TSL)	160°C @ 5°C s^{-1}	
IRSL	125s @ 50°C	
OSL	125s @125°C	
TSL	500°C @ 5°C s^{-1}	

RESULTS

The results obtained for the mineralogical composition of the samples from COR (Table 2), showed that there was a gradual increase in quartz and phyllosilicates contents occurs upwards, as well as an decrease of calcite content.

Absorbed doses obtained by OSL on coarse quartz grains (Fig. 1, b) show a tendency for a gradual increase with depth, except for samples corresponding to modern soil which have a value near zero. This can be interpreted as a gradual ditch infill.

Rather than the gradual increase indicated by the quartz enriched fraction, OSL from the polymineral coarse fraction gave similar results for all archaeological samples, but with higher doses at the base of the section. This can be interpreted as rapid infill. Results from the fine grains were more variable, but again gave higher doses at the base of the section.

Higher doses were obtained through the section using TSL on the quartz enriched fraction, but they were less than 50 Gy and the tendency was similar to the OSL (Fig. 1, c).

Along the profile with depth, at CII, it is possible to distinguish four different patterns of behaviour as regards the mineralogy (Table 2; Fig. 2, b): an upper layer with modern soil, where calcite content tends to be lower, and the content of phyllosilicates tends to be higher; a second layer where the mineralogical composition suffers little variation with depth, but calcite content is higher; below this is visible an increase of calcite content with depth, to values around 90%; in the bottom layer the mineralogical composition is similar to the upper layer.

In the archaeological levels a relationship is visible between the mineralogical composition and the absorbed dose determined by OSL on QZC (Fig. 2, c). That is: samples with higher calcite content tend to show higher values of absorbed dose.

The sample from the bottom also shows a higher absorbed dose, but the calcite content of this material was not particularly high. Looking at results obtained by TSL and OSL of polymineral fine grains, the same relationship can be seen but the distinction between the different phases is stronger (logged axes for the polymineral results in Fig. 2, c).

For the case of HQ1 site there is a clear correlation between the content of calcite and absorbed dose determined by OSL on quartz coarse grains (Table 2; Fig. 3, b and c): layers with high absorbed doses had high calcite content.

This switching behaviour is also evident in the graphs of OSL for polyminerals (fine and coarse grains) and permits define different phases of accumulation with different fill materials, which relates the observations in field relatively to colour and texture of them. As opposed to what was observed for the other sites, in the main sequence at COV the absorbed dose values for quartz OSL are consistent with depth (ca. 6 Gy with only minor variations evident) and independent of the content of calcite, in archaeological levels (Fig. 4, b). Infill of this (recut) ditch appears to have occurred rapidly.

At COV the lateral sequence (empty markers in Fig. 4 and italic lines in Table 2) shows more geological contribution to absorbed dose and higher content of calcite. This pattern is similar to the deepest samples from main sequence. However, the pattern of mineralogy found for this site has some relation with the results of absorbed dose obtained by high temperature TSL in the coarse polyminerals: for archaeological levels the calcite content and the absorbed dose tends to decrease with depth. For this profile, quartz luminescence behaviour is more independent of the calcite content than PMC. This behaviour suggests that quartz signals are more rapidly reset because they are less protected by calcite, or the quartz grains are from an external source and therefore have different origin and "history".

DISCUSSION

The mineralogical profiles of the studied sites are consistent with examples from sites with similar lithology in the literature (e.g. Marques et al., 1990; Abreu et al., 1990; Prudêncio et al., 2011): calcite associated with phyllosilicates, where calcite content commonly decreased upwards, plus some quartz and feldspars and occasional minerals like dolomite and hematite. Only minor indications of stratigraphically consistent variation in sensitivity or sensitization were observed.

Table 2. Mineralogical composition and semiquantification of samples from sites COR, CII, HQ1 and COV, along the archaeological ditch. Lateral sequence of sampling for values in italic.

Archaeological site	Depth (cm)	Semiquantification (%)						
		Quartz	Phyllosilicates	Calcite	Feldspars	Hematite	Micas	Dolomite
COR	5	19	28	52	2	-	-	-
	30	15	26	57	3	trace	trace	-
	41	18	14	68	trace	-	-	-
	60	12	19	67	2	-	-	-
	71	13	16	69	2	-	-	-
	80	12	17	70	2	trace	-	-
	90	6	14	80	-	-	-	-
CII	0	18	22	47	13	-	-	-
	28	7	30	57	5	-	-	-
	37	6	25	61	8	-	-	-
	70	4	40	51	5	-	-	-
	76	*2*	*15*	*80*	*4*	-	-	-
	84	4	14	76	6	-	-	-
	92	13	16	67	6	-	-	trace
	100	11	3	78	8	-	-	-
	115	3	17	76	4	-	-	-
	134	*7*	*19*	*72*	*2*	-	-	-
	155	7	18	70	5	-	-	-
	176	9	0	81	11	-	-	-
	190	3	0	96	1	-	-	-
	213	8	25	60	6	trace	-	-
	223	0	21	69	10	-	-	-
HQ1	5	22	64	10	3	1	-	-
	53	1	30	67	3	trace	-	-
	72	*3*	*20*	*71*	*5*	*2*	-	-
	105	21	44	25	7	2	-	-
	108	3	28	69	trace	-	-	-
	120	10	51	35	3	1	-	trace
	138	1	23	70	6	-	trace	-
	154	6	39	47	7	1	-	1
COV	5	24	18	50	9		trace	-
	37	*8*	*19*	*72*	*1*	-	-	-
	60	*4*	-	*94*	*2*	-	*trace*	-
	77	13	19	62	6	-	trace	-
	91	*14*	*21*	*57*	*7*	-	-	-
	93	7	17	80	-	-	trace	-
	102	11	16	68	5	-	-	-
	106	19	24	55	2	-	trace	-
	112	14	25	57	4	-	-	-
	116	30	28	39	4	-	-	-
	126	20	25	52	3	-	trace	-
	130	23	25	48	4	-	-	-
	140	24	17	56	3	-	trace	-
	144	18	25	54	2	-	trace	-
	150	6	1	91	1	-	-	-

The presence of calcite and associated low concentrations of radionuclides tends to produce low dose rates and should therefore result in lower absorbed doses, but in the present sites it was often related to high absorbed doses. Very high dose values were almost always associated with geological samples or those from levels closest to substrate (generally rich in calcite), but in modern soils absorbed dose was always low, regardless of calcite content. This indicates that the substrate material retains large residual luminescence signals, but that can be well reset during reworking and re-deposition.

Commonly, from the bottom to the top of a section the calcite content and the absorbed dose value tended to decrease. The gradual increase of absorbed dose with depth can be interpreted as a result of a gradual ditch fill. This was observed for archaeological layers at COV and CII.

Where residual doses were interpreted to be present in archaeological layers, these were generally higher for polymineral fractions, and IRSL and TSL signals: IRSL signals may be less easily reset than OSL in this environment or the polymineral fractions may be better protected from light (in calcite agglomerates) than the quartz. In these cases the calibration dose was similar to the absorbed doses so that the difference is not expected to relate to saturation effects and hence quartz content (*cf.* Burbidge et al., 2007). Layers with lower calcite content and residuals were identified at CII and HQ1, and these were used to define different phases of accumulation. The best reset luminescence signals for dating at these sites come from the layers with the lowest calcite content, as has also been observed in karst caves (Burbidge et al., 2007). At COV, OSL absorbed doses from QZC were independent of calcite content in the archaeological layers, but those from less light sensitive TSL signals showed a positive relationship. However, at COR, OSL absorbed doses from QZC increased with depth and calcite content, while OSL from PMC was independent of calcite content through the archaeological layers. Similar absorbed doses through archaeological stratigraphies, independent of composition, may indicate that signals in the different mineral fractions were well reset prior to accumulation in the structure.

At the COR site, four stratigraphic phases were evident in the field, and variations in mineralogical composition support this. OSL on PMC and High temperature TSL on QZC results indicate three fill phases: a layer where residual signals prevail, archaeological fill, and then cover by modern soil. However, absorbed doses obtained by OSL on QZC (Fig. 1) indicate that these different materials accumulated gradually in one phase, the top of which was then covered by or reworked to form the modern soil. In this case it is possible to say that there was a gradual colluvial re-deposition with resetting of geological OSL signals in the QZC fraction, but responses of other luminescence signals from other fractions related closely to changes in source material.

At the CII site both the luminescence and mineralogical results indicate four stratigraphic phases rather than the two identified in the field (Fig. 2). Higher calcite content and residual luminescence signals were observed in the lower halves of the circulation-floor context and the colluvial fills above it. Absorbed doses were similar in samples with lower calcite contents from the upper halves and for a sample with low calcite content near the base of the sequence (4-8 Gy, QZC-OSL). Both these observations indicate that infill of the ditch occurred during a relatively brief period. At the base of the sequence geological material appears to have fallen onto the floor context and been trodden in, resulting in even higher signals, while the infill sealing this context appears initially to have been a high energy process, perhaps rapid collapse.

For HQ1 the ditch infill may have occurred in two distinct phases, one before collapse / placement of the layer of stones (with thin layers of materials of different composition that alternate with each other) and other after this event. OSL results from the QZC fraction indicate that the first accumulation (under the layer of stones) occurred during a relatively short period of time: for the levels with low calcite content, the absorbed dose varies around 3 Gy (Fig. 3). The samples collected from the colluvial fills above the stony layer show similar composition and absorbed dose values (around 6 Gy), indicating that they are in fact part of the same archaeological context. These colluvial fills accumulated after the collapse / placement of the layer of stones, but contain residual signals associated with high calcite content and rapid re-deposition: these were largely reset in the superficial layer (modern soil), with absorbed dose around 0,4 Gy.

At COV three distinct archaeological phases were encountered during excavation between the modern soil and the geological substrate. These phases are evident in the luminescence results (Fig. 4): an older phase, with more residual signals (18-31 Gy) (see empty marks in absorbed dose plots); above this and below the modern soil it was possible observe another layer (empty marks in Fig. 4, at 37 cm depth) with less absorbed dose at OSL on QZC (8 Gy); the main sequence appears to represent a rapid infill of the recut with constant absorbed dose (around 6 Gy from OSL on QZC) with slight mineralogical variation of fill materials. However, the absorbed doses for high temperature TSL on PMC show a relation with calcite content. In this archaeological structure, the QZC signal on OSL was better reset than PMC, which were more insensitive to sun light may have been protected by calcite agglomerations.

CONCLUSIONS

In this study, four negative anthropogenic structures were analysed with the objective of relating luminescence behaviour with mineralogical composition. The mineralogy of the fill materials is mainly calcite (40-90%). Variations in mineralogical composition allowed different phases of infill to be distinguished (more marked for CII and HQ1 samples).

Calcite is the mineral that most influences the luminescence behaviour of the analysed materials, mainly the absorbed dose. Calcite rich contexts may contribute with mineral grains to residual luminescence signals and/or change dose rates by precipitation/dissolution and alteration of water retention properties. Calcite may promote the protection of quartz grains from the sunlight by agglomerates formation or by the inclusion of quartz grains in geological calcrete, from which residual signals may not be completely removed. In most of the studied cases residual signals appear to be the factor that most strongly influenced the estimated absorbed doses.

For three sites high calcite content was related with residual absorbed doses, particularly in TSL and IRSL signals, and less so for OSL. Fluctuations in such residual signal levels, and comparison between the results from different signals through the stratigraphies, made possible the identification of different phases of accumulation and their relative chronology.

Luminescence profiling analyses made it possible to define different phases of accumulation, best reset luminescence signals for dating, influence of mineralogy on different measured luminescence signals and an estimative of absorbed dose. This study provide stratigraphic resolution of the luminescence behaviour and mineralogical composition, using small amount of sample and rapid and simple measurement techniques.

Through the relationship established between mineralogical composition and luminescence behaviour it was possible to obtain relevant information about the processes of fills accumulation in these negative structures, particularly about the different accumulation phases and their relative duration, as well as the materials involved.

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