

Article

Luminescence Dating of Rock Surface. The Case of Monoliths from the Megalithic Sanctuary of Ossimo-Pat (Valle Camonica, Italy)

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Featured Application: Dates obtained by optically stimulated luminescence (OSL) of rock artefacts and the underneath soils were shown to be in very good agreement with the archeological evidence. Highly satisfying results contribute to the understanding of a megalithic sanctuary.

Abstract: Ossimo-Pat megalithic sanctuary (Valle Camonica, BS, Italy) is one of the most relevant archaeological findings of the southern alpine region, for the variety of its structures and the quality of its engraved monoliths. Its unique state of preservation gives the opportunity to apply the luminescence dating of the rock surface method. Here, we investigate the use of optically stimulated luminescence (OSL) for dating five cobbles from the site and compare cobble-surface derived ages to quartz OSL ages from sediments and to archaeological evidences. The obtained ages confirm the archaeological studies and open the way to a new hypothesis.

Keywords: optically stimulated luminescence; surface dating; megalithic sanctuary

1. Introduction

There are many examples of rock surfaces, rock art and stone structures of unknown age. In particular, in prehistoric archaeology, long-lasting building traditions can sometimes be difficult to date, if the context does not offer useful chronological tools, such as artifacts or organic remains associated to the structures. Moreover, this particular issue is not a marginal one, if we consider that the study of megaliths, drystone walls and other structures, such as cairns or mounds, is often crucial to our understanding of the society and traditions of very ancient cultures, devoid of other forms of communication.

In this scenario, the Ossimo-Pat (Valle Camonica, BS, Italy, see Figure 1) megalithic sanctuary is an exemplary case.

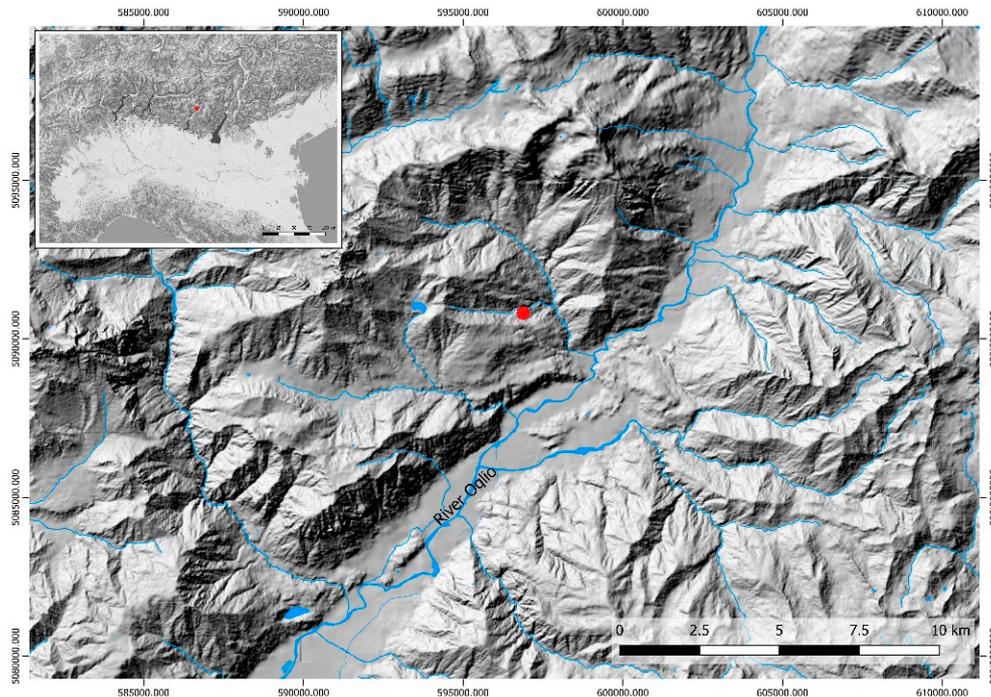


Figure 1. Location of the Ossimo-Pat site in Middle Valle Camonica (GIS elaboration on LIDAR DTM base).

Its construction started in the first half of the fourth millennium BCE and was structured and used during the Copper Age (late 4th–3rd millennium BCE), with ceremonial structures and engraved monoliths articulated in a north-south alignment. The southernmost structure is a sequence of ceremonial mounds, built upon a concentric series of stone circles enclosing an inner rectangular space, which usually contained objects, such as flint weapons and tools or ornaments. The central part of the sanctuary was an alignment of monoliths of various lithologies, mainly sandstones of diverse granulometry, carved with figures and symbols and probably depicting mythical ancestors or gods. The alignment led to a monumental tomb and then to a series of at least five votive stone circles, which contained, similarly to the southern mounds, a number of flint, stone, metal and ceramic artefacts, probably the result of unknown cult activity [1,2]. The site was frequented until its sudden, partial, dismantling and further abandonment, at the very beginning of Bronze Age, at the end of the 3rd millennium BCE. Later frequentation during the final Bronze Age and the whole Iron Age is attested by a series of fireplaces that were lit in front and at the backside of the monoliths still vertically standing. Ossimo-Pat is one of the most relevant archaeological findings of the southern alpine region, for the variety of its structures and the quality of its engraved monoliths, as well as for its unique state of preservation, which gives the opportunity for a documentation of the exact moment of its disuse. As part of the UNESCO World Heritage Site n.94 “Rock Drawings in Valle Camonica,” the sanctuary occupies an area of more than 4000 square meters, and it is located on the right hydrographic flank of the medium Valle Camonica, at an altitude of 810 m asl. The site is arranged on the edge of a terrace overlooking a steep drop into the Valle dell’Inferno (the strikingly named “Valley of Hell”), placed on a wide elevated plateau comprised by two dominant peaks: the Pizzo Camino (2491 m asl) to the southwest and the Concarena (2549 m asl) to the north-east. The rocky substrate of the area is mostly made of Triassic (from the Scitic to the Carnic) limestones, while the surfacing lithology is often covered by morainic deposits, left by the glaciers, which can be encountered up at an altitude of 1650 m asl. (source: F. Franzoni, Piano di Governo del Territorio, Comune di Ossimo, 2012 (http://www.comune.ossimo.bs.it/Allegati/all_50012_GEO_Relazione%20Geologica.pdf)). The monoliths chosen to be engraved are very likely coming from these deposits, but the geological survey and study is still ongoing. Excavations and analyses are being carried on since 1994 and are still ongoing, under the direction of Raffaella Poggiani Keller (Figure 2).

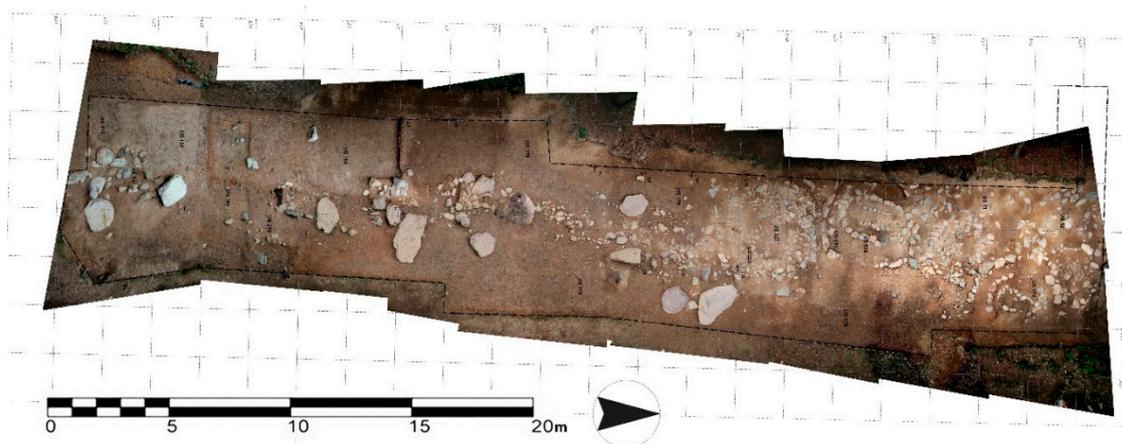


Figure 2. Ortophotograph of the Ossimo-Pat site.

There are two potential methods for dating the surfaces of such artefacts: the measure of cosmogenic radionuclides concentrations [3] and the application of luminescence dating (in particular Optically Stimulated Luminescence, OSL) [4,5]. The first approach depends on cosmogenic nuclides (e.g., ^{10}Be , ^{26}Al , ^{36}Cl) build-up with time in minerals exposed to cosmic rays. Therefore, measuring their concentrations allows determination of how long rocks have been exposed at or near the surface of the Earth. In this paper, we focus on the alternative approach based on OSL. This method was originally developed to date the deposition of sediments [6] and it depends on the ability of some minerals, such as feldspar and quartz, to absorb and store energy from environmental ionizing radiation. When the light stimulates these minerals, they release the stored energy in the form of light, from which the term luminescence is derived. Measuring the amount of energy released in conjunction with a determination of the rate at which the energy was accumulated allows to calculate an age, indicating the time that has elapsed since the storage of energy began.

Thus, if the OSL signal from mineral grains is normally used to date how long the sediment grains were buried, recent works have shown that luminescence signals can also be used to determine the duration of daylight exposure for rock surfaces [4,7–9].

The OSL surface dating technique exploits the depth-dependence of the resetting (bleaching) of the geological luminescence signal when exposed to daylight. Sohbaty et al. [4,10] proposed a model that reproduces the dependence of the OSL signal on depth and exposure-time and predicts that the longer the exposure duration, the deeper the resetting of the luminescence signal into the rock surface. Analogously, this dependence has been evaluated for renaissance bricks [11].

Thus, there exists chronological information in the luminescence-depth profile, but despite some applications of the OSL surface technique [8,9,12], the bleaching-depth model is yet to be validated experimentally; this is critical for further development of the technique. The emerging OSL surface dating technique has the potential to answer many new questions in the fields of geo- and archaeological sciences.

Dating the sanctuary was mostly done through the combination of stratigraphy observations, typo-chronological study of the artefacts and with the use of a certain number of radiometric measurements (^{14}C) on charred coals from different parts of the site [1], mostly from the southern mounds and the upper layers of the monoliths alignment. Therefore, to test the capability of the surface dating technique, five pebbles were collected from three different areas. Three samples consisted of the pebble itself and the sediment collected directly underneath the pebble. Dating was attempted on both the underside of the pebble and on the sediment under the pebble, on the assumption that neither has been exposed to light since the placement of the rock. In this way, we checked the predictability of the reconstructed luminescence profiles into the buried rock surface.

The measured ages obtained from buried surfaces are in good agreement with those obtained from the sediments and with those obtained through archaeological methodology. Moreover, in some cases the ages obtained with luminescence dating are consistent with available ^{14}C ages.

2. Materials and Methods

All experiments in this study were performed using rock samples and sediments collected from the Ossimo-Pat sanctuary (Figure 3). The Sample “3/2016” is a pebble taken from the stone accumulation behind the big monolith known as “Pat 2,” presumably relative to the first stage of the abandonment of the site. “1/2016” and “US 178” are samples from the northern votive stone circles, which are generally placed in the central phase of the site-life, around the first half of the 3rd millennium BCE, while the samples “Pat12” and “Pat15” were collected from the housing pits of the fallen monoliths “Pat12” and “Pat15.” All the cobbles have a diameter in the range of 100–150 mm.

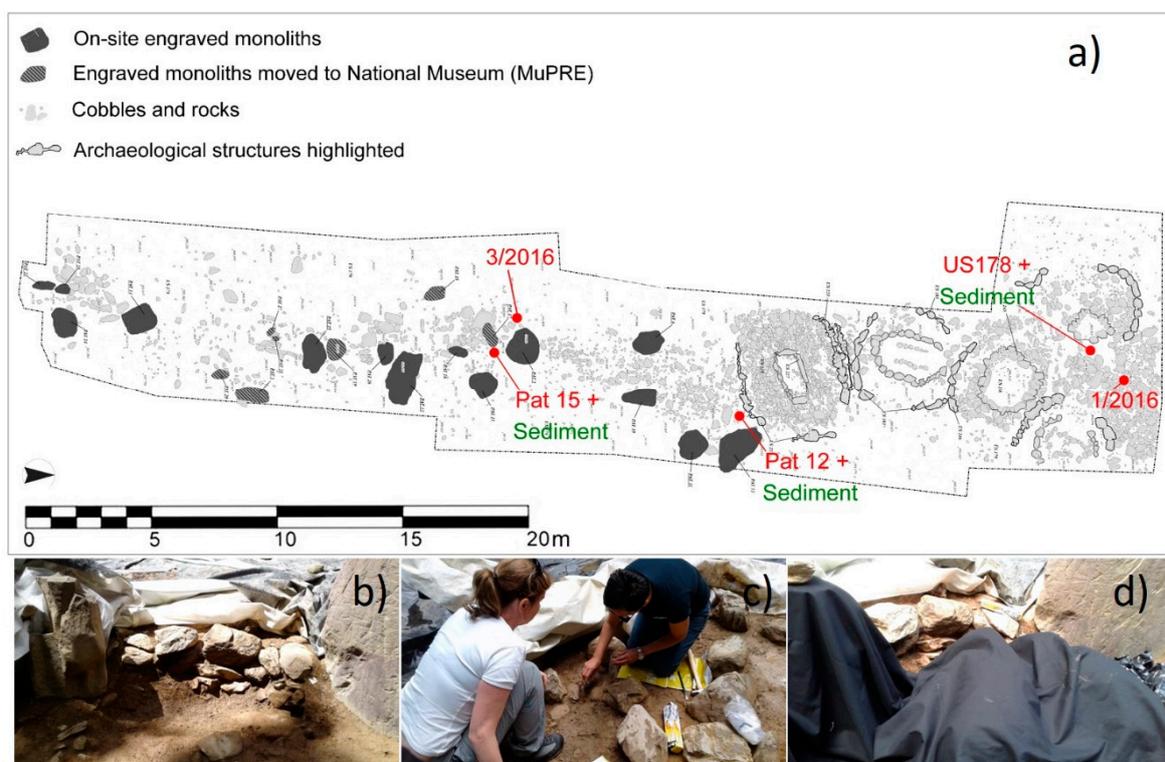


Figure 3. (a) Site map with sampling points; (b) detail of sample US178; (c) during the sampling operation under the sunlight and under the dark (d).

To measure the OSL depth profiles and the absorbed doses, the cobbles have been drilled under dim red light in laboratory. The reached depth was checked with a digital precision caliber. The polymineral fine grain fraction with grain sized between 4 and 10 micrometers was used.

For the sediments, the quartz grains have been extracted using the conventional procedure [13–16] under dim red light and the luminescence measurements were conducted on coarse quartz grains (180–250 μm).

All samples, both cobbles and sediments, were mounted on stainless steel discs. The OSL measurements were performed with a Risø TL/OSL DA20 reader. The samples were stimulated by blue LEDs ($470 \pm 30 \text{ nm}$, 54 mW/cm^2) or by IR LEDs ($\lambda = 830 \text{ nm}$, 360 mW/cm^2) and the emitted photons were detected with an EMI 9235QB photomultiplier coupled with a UV filter (7.5 mm Hoya U-340) in case of blue stimulation or with a blue filter (Schott BG39/Corning 7–59 filter combination) in case of IR stimulation. Blue stimulation was used for the samples in which the main dosimeter was the quartz and IR stimulation [13] for the samples rich in feldspars.

To evaluate the absorbed dose the Single Aliquot Regenerative dose protocol (SAR) was applied for all samples. It is a well-known standard procedure for the optically stimulated luminescence dating in which the same aliquot is irradiated, heated and illuminated, during experimental cycles that are repeated (Table 1). The obtained signal is integrated, normalized and plotted in function of dose and the absorbed dose is calculated from interpolation of the natural signal on the constructed curve.

Table 1. Single Aliquot Regenerative dose protocol applied (L_i and T_i were calculated starting from the optically stimulated luminescence (OSL) curve, integrating the first 0.8 s minus a background estimated from the integral of the last 8 s).

Step	Rock Samples SAR Cycle	Sediments SAR Cycle
1	Give dose, D_i ^a	Give dose, D_i ^a
2	Preheat at T_0 for 10 s ^b	Preheat at T_0 for 10 s
3	Stimulated for 100 s at 125 °C (OSL) or 50 °C (IRSL), get L_i	Stimulated for 40 s at 125 °C (OSL)
4	Give test dose, D_t	Give test dose, D_t
5	Cut heat to 220 °C for 0 s	Cut heat to 180 °C for 0 s
6	Stimulated for 100 s at 125 °C (OSL) or at 50 °C (IRSL)	Stimulated for 40 s at 125 °C (OSL)
7	OSL or IRSL at 290 °C for 40 s	OSL at 290 °C for 40 s
8	Return to 1	Return to 1

^a For the natural sample $i = 0$ and $D_0 = 0$. Four regenerative doses plus a zero-dose measurement and a repeat dose were used; ^b T_0 depends on the sample.

The pre-heat value was experimentally derived on the basis of the results of a dose recovery pre-heat plateau test [17] (see Table 2).

Table 2. Pre-heat temperature for rock and sediment samples.

Sample Code	Rock Samples Pre-Heat Temperature (°C)	Sediment Samples Pre-Heat Temperature (°C)
1/2016	260	
US178	220	220
3/2016	200	
Pat12	240	260
Pat15	200	220

Laboratory irradiations were given using a calibrated $^{90}\text{Sr}/^{90}\text{Y}$ beta source (5.50 GBq) mounted on the Risø TL/OSL reader (dose rate: 0.23 Gy/s for fine grain; 0.12 Gy/s for coarse grain).

The absorbed dose is calculated from the weighted mean of values obtained from the analyzed aliquots (at least 10 for sample).

The annual dose-rate absorbed by the samples is due to the contribution of the internal radioactivity of the material and the radioactivity of the surrounding environment. The radioactive families ^{238}U , ^{232}Th and ^{40}K are, in both cases, responsible for the natural imparted dose. There are also minor contributions from ^{87}Rb and from cosmic rays. To take into account any non-homogeneity in the external contribution, the radioactivity of a sphere of the surrounding material (30 cm diameter centered at the sampling point) has to be measured [9]. The heterogeneity effect was considered by measuring the radioactivity concentrations of the surroundings materials, i.e., sediments and cobbles. The contribution of each material to the annual dose-rate was evaluated applying the infinite matrix approximation, with updated conversion factors [18]. After radioactivity measurements, it is possible to assess the contribution of each layer calculating the relative volume of every material within the interaction sphere and normalizing the annual dose fractions in the age equation [19]. ^{40}K concentrations were measured through atomic absorption spectrometry; ^{238}U and ^{232}Th concentrations were obtained from total alpha counting technique. Although the water content of the cobbles is negligible, the surrounding sediment material does contain water, and thus, a water content correction is needed. This was based on the one calculated using the sediment material, specifically a mean water content equal to 75% of saturation value ($20 \pm 10\%$) was used. Attenuation of the beta

dose was taken into account in case of sediments for which the coarse grains fraction was used to determine the absorbed doses [20], while alpha contribution was eliminated by an HF etching.

3. Results

3.1. Luminescence—Depth Profiles

To test the application of luminescence rock surface dating to the surface of buried cobbles, we first assessed the degree of bleaching of the surface itself. To investigate this, we measured the absorbed dose as a function of depth into all the five cobbles. As an example, the Figure 4 shows the luminescence-depth profiles from two different samples. Each point in the profile is obtained by the mean of the absorbed doses measured on 10 aliquots.

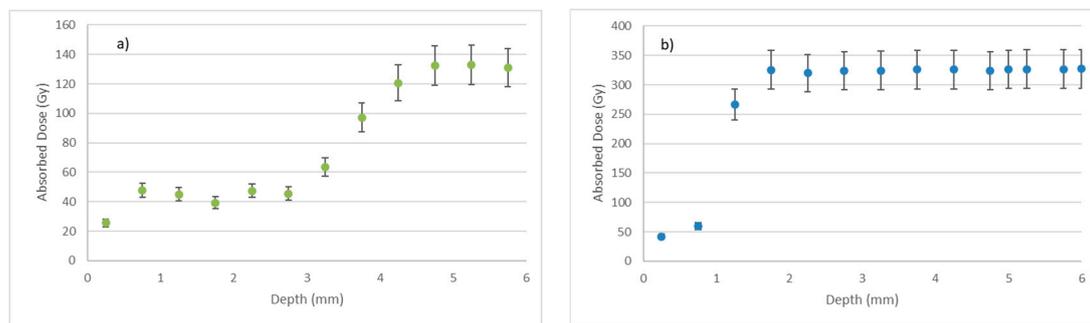


Figure 4. Luminescence depth-profiles (a) 1/2016 sample; (b) 3/2016 sample.

In the left panel, the absorbed dose increases with the depth and reaches a plateau (the so-called field saturation level) in the middle of the cobble. In the right panel, this plateau is reached in the first layers below the surface. It seems to have limited light exposure, insufficient to fully reset the OSL signal in the layers below 1mm from the surface. Presumably, light exposure has not been sufficiently long to totally bleach the luminescent signal throughout the cobble or this sample has a high attenuation coefficient. For both samples, the absorbed dose at the surface is proportional to the time elapsed since the deposition. The calculated ages are reported in Table 3.

Table 3. Cobbles and sediments calculated ages.

Sample Code	Sample Type	Technique	Absorbed Dose (Gy)	Dose Rate (mGy/a)	Date
1/2016	ROCK	OSL	24.5 ± 3.5	5.15 ± 0.15	2760 BCE ± 500
US178	ROCK	OSL	20.8 ± 3.5	2.95 ± 0.20	2675 BCE ± 500
US178	SEDIMENT	OSL	15.4 ± 0.5	2.95 ± 0.07	3220 BCE ± 330
3/2016	ROCK	IRSL	42.3 ± 7.5	10.75 ± 0.30	1920 BCE ± 250
Pat12	ROCK	IRSL	30.5 ± 5.0	4.20 ± 0.12	5240 BCE ± 600
Pat12	SEDIMENT	OSL	17.2 ± 0.4	2.40 ± 0.08	5090 BCE ± 360
Pat15	ROCK	IRSL	22.9 ± 4.0	6.50 ± 0.25	2470 BCE ± 500 *
Pat15	SEDIMENT	OSL	12.8 ± 0.4	3.00 ± 0.05	2270 BCE ± 260

* Fading-corrected date.

3.2. Fading Correction and Cobbles Ages

By applying the well-known equation age [6], the cobble burial ages were simply derived by dividing the cobbles surface absorbed dose values by the dose rate at the surface of the cobbles. However, the luminescence signal from feldspars could be unstable: this phenomenon is known as ‘anomalous fading’ and can be taken into account by the conventional g value measurement [21]. For the samples Pat12 and Pat15, we have quantified the fading rate as a percentage of IR signal loss

with storage time after irradiation. Three aliquots from each sample were bleached in the reader using an IR stimulation (for 500 s at 50 °C repeated after 60 s); a known dose has been imparted, and then the absorbed dose were measured, both immediately after irradiation and after a delay time of 6 h, 1 day, 19 days and 33 days. For the sample Pat12, the ratio between the imparted dose and the dose measured after the time delay are indistinguishable from unity, (Figure 5), thus, it possible to deduce that the phenomenon of anomalous fading is negligible. The cobble Pat15 is in a different condition: its signal fades by 10% in about a month. Therefore, we evaluate the fading rate with a g value of $2.4 \pm 0.5\%$, and in Table 3, the fading-corrected date is reported.

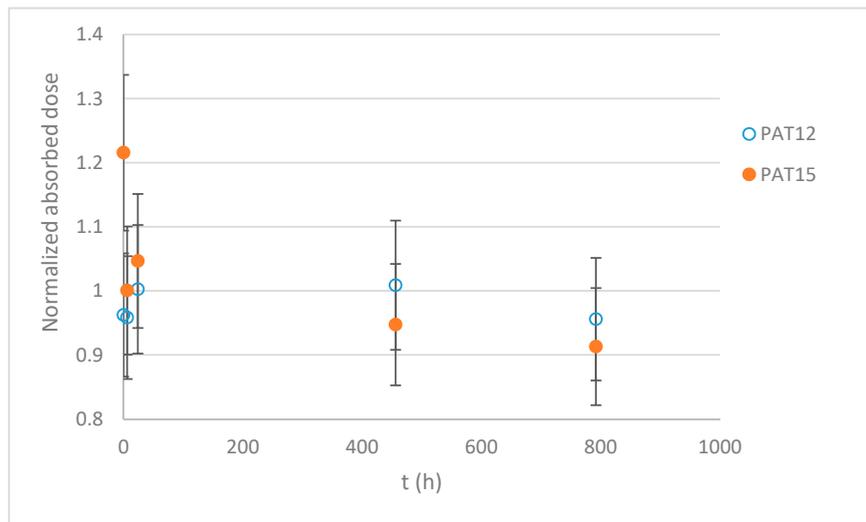


Figure 5. Normalized absorbed dose versus time.

3.3. Sediments' Ages

The absorbed dose histogram, relative to more than 50 aliquots for each sample, as reported in Figure 6 for sample Pat15, shows a symmetric distribution with rather low dispersion of the data, evidencing a similar dose absorbed by all the aliquots. This is an indication that the surface sediments were fully bleached before the cobbles' deposition. This allows to use the weighted mean to calculate the absorbed doses by the samples that are reported in Table 3, together with the annual dose rates and the OSL dates.

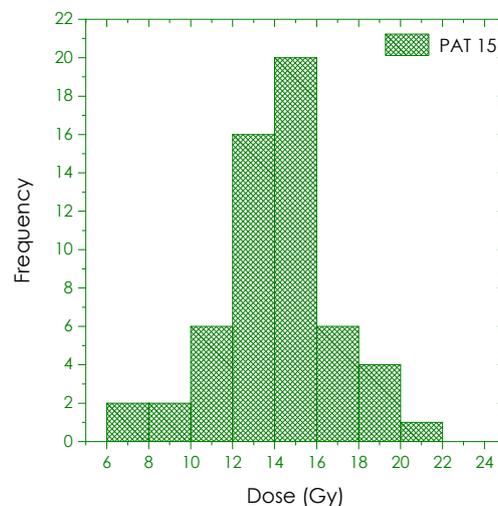


Figure 6. Sample "Pat15." Distribution of absorbed doses.

4. Discussion and Conclusions

The doses absorbed by the cobbles were directly derived by the aliquots taken from the first layer. Although the uncertainty in the dose is derived from the standard error of 10 aliquots, the relative error spans between 15% and 30%, it is quite high, especially if compared with that obtained with sediments (5–6%). Due the scarcity of material for each considered layer, it has been impossible to extract quartz or feldspar grains from the samples. This may have led to luminescence signals with inadequate characteristics (slow decay rate, high background) that introduced a high error in the fitting to calculate the absorbed dose.

For US178, Pat12 and Pat15, the age from cobble is in agreement with the sediment age at a level of 1 standard deviation, thus, they are consistent with each other.

From a methodological point of view, these preliminary results show that it is possible to apply luminescence dating to the surfaces of the artefacts present in the Ossimo-Pat site. Despite the high error obtained on rock surfaces, the data obtained on sediments under the rock were more precise. This suggests that for future application of surface dating of megalithic structure, it is recommended to sample both rocks and the underlying sediments in order to reduce the age errors.

Moreover, the results of the dating offer, from an archaeological point of view, an interesting feedback to the existing timeframe of the site. Even considering the range error of each date, we can compare the results with what was already established about the site, chronology-wise. The first sample (the rock “1/2016,” 2760 BCE \pm 500) was taken from the northernmost of the votive stone circles. Its date is compatible with the other two samples from the nearby, similar, stone structures (samples “US 178,” rock, 2675 BCE \pm 500, and sediments, 3200 BCE \pm 330). There are no 14C dates available from these circles that have been dated to the first half of the 3rd millennium BCE based on the objects found inside the circles, namely a specific type of arrowheads, usually related this period. The three OSL dates from the area would seem to confirm the current chronological framework.

The IRSL date from the rock sample “3/2016” (1920 BCE \pm 250) seems equally aligned with the current chronology of the site. This rock was collected from the accumulation layer located behind the big monolith called “Pat 2,” one of the few still vertically standing. The small accumulation of soil and rocks is probably related to the first stage of abandonment of the site, right after its partial destruction and dismissal, and is usually dated around the 23rd–20th centuries BCE thanks to a charred coal 14C dating [1].

The samples taken from the monolith pits are the most intriguing ones, as we still do not have a full comprehension of the precise life cycles of these monuments. The samples taken from the housing pit of the monolith “Pat15” are related to its filling, which happened in time, after the dismissal of the site and the overthrowing of the monolith, which was pushed to the ground, out of its proper housing. The dating of rock and relative sediment (2470 BCE \pm 500 and 2265 BCE \pm 115, respectively) are perfectly coherent with the said action, which probably happened, as said, between the end of the 24th and the 21st centuries BCE.

The dating of the samples from the housing pit “Pat12” (Rock: 5240 BCE \pm 600; sediment: 5090 BCE \pm 360), on the other hand, offer a very interesting result, related back to the end of the sixth millennium BC, before the foundation of the sanctuary. Nevertheless, these dates are perfectly coherent with another dating, obtained by 14C on a charcoal sample from the layer 181 [1], which was documented in this very sector of the site thanks to a deep stratigraphic trench. Layer 181, rich in charred coal and burnt wood, is believed to be the outcome of the very first action carried out in this place, namely a slash/burn activity, usually performed during the late Neolithic to open new pasture areas in medium and high-altitude locations. Evidently, the digging activity performed during the Copper Age to prepare the housing pit for the huge monolith “Pat12,” which is longer than 2 m and very heavy, led to reach the deeper and more ancient level of the site, to which our samples appear to belong.

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