

# OSL DATING OF TERRACOTTA BRICKS OF THE OLD MOSQUE OF MILA, ALGERIA: A CASE STUDY

Kamel BOUZETINE<sup>1,3,\*</sup>, Messaoud HAMIANE<sup>1</sup>, Pierre GUIBERT<sup>2</sup>, Abla BRAHMI<sup>3</sup>, Mourad BELAIDI<sup>4</sup>

<sup>1</sup> Materials, Process and Environment Research Unit, URMPE, M'hamed Bougara University, Boumerdes, Algeria.
<sup>2</sup> Research Institute on Archaeomaterials, Research Center in Physics Applied to Archaeology, IRAMAT-CRP2A-UMR 5060 CNRS - University Bordeaux Montagne, France.

<sup>3</sup> National School for the Conservation and Restoration of Cultural Property, Arab Center for Archeology, Algeria.
<sup>4</sup> Mouloud Mammeri University, UMMTO, Tizi-Ouzou, Algeria.

#### Abstract

The mosque of Mila is considered as the oldest mosque in Algeria, certain historical sources attribute the construction of the latter to Abou Mouhadjir Dinar in 674 AD by the reuse of materials from construction of earlier eras. The architectural stratigraphic study carried out on the mosque revealed that the current plan of the prayer hall of this mosque is organized in six naves and eight spans and seems to be the result of an extension of a previous plan which included four naves and seven spans. The stratigraphic study always gives a relative chronology in which the stratigraphic units are linked together by a relation of Anteriority and posteriority. In order to have an absolute dating and to confirm the hypotheses of construction and structural modification of this mosque, we proceeded to a dating on two samples of architectural bricks coming from the two parts by the optically stimulated luminescence method OSL in Fine grain technical Quartz. The results obtained made it possible to confirm the two hypotheses, the first relating to the construction of the mosque or the measurements display a date of 616±78AD while the second relating to the extension or the measurements display a date of 1007±80 AD. After a comparison with historical and archaeological data we were able to reduce the interval of the construction of the mosque which is located between [665-694] AD during the reign of the Umayyads and the extension Between [1019-1087] AD, under the reign of the Hammadids.

Keywords: Old mosque; Mila; OSL dating; brick; Fine grain technical Quartz

### Introduction

Numerous studies in recent years have highlighted the importance of interdisciplinarity for the analysis of cultural heritage, and more particularly the measurement of time by optically stimulated luminescence OSL.

This dating method gives access to the last exposure to the light of a brick with the minerals, preferably Quartz crystals, taken from its internal part. If we take the Quartz on the surface, we manage to date the insertion of the brick into the masonry and therefore the construction of the wall, and no longer just the production of the material [1].

In this work we will date the manufacture of the bricks used in the construction of the arches by the OSL method of the BDX 01 sample which is located on the second nave and the fourth span and the BDX 02 which is located on the first nave and the eighth bay inside the

<sup>\*</sup> Corresponding author: <u>k.bouzetine@univ-boumerdes.dz</u>

prayer hall in order to have a chronology of construction and structural modification in this mosque.

#### Materials and method

Over time, materials are continuously irradiated by natural radionuclides, mainly uranium, thorium and their descendants, and the radioactive potassium isotope  $^{40}$ K. Minerals such as quartz act as a dosimeter, recording the amount of radiation to which it has been exposed. Quartz is capable of storing inside its crystal structure a small proportion of the energy supplied by radiation (trapped electrons) due to the disintegration of radioactive elements contained in the mineral itself and in its environment, and cosmic radiation. This energy builds up as exposure to radioactive decay continues over time. At a later stage , it will be released, in the form of light (luminescence) when exposed to light, in this case, the luminescence signal in the grain of the quartz disappears (optical whitening) until it is reduced to zero. Once these quartz grains are hidden in the ceramic paste of bricks or mortars away from daylight, electrons accumulate again under the effect of natural radioactivity (Fig.1).



Fig. 1. Evolution of luminescence over time

To produce the luminescence signal, the electrons trapped and stored in the Quartz can be released in the laboratory by exposing it to light (optically stimulated luminescence, OSL). The light emitted when returning to equilibrium is proportional to the number of electron-hole pairs created, itself proportional to the dose received.

To calculate an age, two quantities must be determined:

- The equivalent dose (or paleodose), which corresponds to the amount of energy per unit of mass stored by the Quartz since its last exposure to light;

- The annual dose, which represents the amount of energy per unit of mass accumulated in a year by the Quartz.

Age is determined by dividing the equivalent dose by the annual dose:

$$Age = \frac{(\text{Equivalent dose in (Gy)})}{(\text{Annual dose (Gy / an)})}$$
(1)

We have chosen to use the technique of small quartz inclusions (fine grain technical quartz) for dating brick. For this, the fraction less than  $12\mu m$  follows the following treatment protocol:

- Attack with a dilute solution of HCl (1M) to remove the carbonates, followed by several rinses with water;

- Attack with a 30% hydrogen peroxide solution for 48 to 72 hours in order to remove organic matter; followed by several rinses with water;

- Screening under water at 40µm. The article size fraction greater than 40µm is retained;

- Attack with a mixture of hexafluorosilicic acid ( $H_2SiF_6$  31%) and nitric acid ( $HNO_3$  68%) (mixture of concentrated solutions in a respective volume ratio 9:1) for 48 to 72 hours in order to dissolve the feldspars and the aluminosilicates of the material;

- Rinses with dilute nitric acid, then with water and drying;

- The 3-12µm fraction is selected by successive decantations in test tubes containing acetone. It contains almost exclusively quartz crystals. The powder is stored before its OSL study;

- For the OSL study, the powders are suspended in water (4mg of powder for 1mL of water) and small standard volumes are pipetted into equivalent volumes (150µL) using of a micro-pipette then deposited on stainless steel cups of 8mm internal diameter. The cups are dried in an oven and the material thus deposited in a homogeneous thin layer is ready for the OSL study.

OSL measurements on architectural terracotta quartz were carried out using a luminescence reading device of the FREIBERG type SMART type equipped with a beta irradiation source  ${}^{90}$ Sr/ ${}^{90}$ Y with a dose rate 0.148  $\pm$  0.004 Gy. For the stimulation used in the IRSL test, a laser of 300mW of power emitting at a wavelength of 850nm is used. The detection system included in the luminescence reader consists of a Hamamatsu H7360-02 photomultiplier tube and a 2.5mm thick Hoya U340 filter. The luminescence data obtained is processed by the ANALYST 4.31.9 software [2].

Low background gamma spectrometry measurements were performed using a high purity Germanium detector. It is a U-shaped liquid nitrogen well detector of the Eurisys Measurements brand, model EGPC 200 P17. The active volume of the detector is 200 cm<sup>3</sup> and the dimensions of the well are 17mm in diameter and Spectral data is acquired and processed by GENIE2000 software (Canberra/Myrion Technologies).

## History of mosque

Mila (Arabic: ميلة) is a city in the northeast of Algeria and the capital of Mila Province. In antiquity, it was known as Milevum (in Latin; as such still a Latin Catholic titular see) or Miraeon, Μιραίον (in Ancient Greek) and was situated in the Roman province of Numidia.

Between 675 and 682 the city have conquered by the Umayyad Arabs commanded by Abu al-Muhajir Dinar.

In multiple book mentioned precisely City Mila conquered by Abu Muhajer General Umayyad Dinar in 675 AD in it, says in "The Berbers: study on the conquest of Africa by the Arabs, according to the printed Arabic texts", Volume 1, by Henri Fournel [3]. The Mosque Sidi Ghanem of Mila (Fig. 2) was built around 675 by Abu Muhajer Dinar in the tenth century AH, historian and geographer Abu Ubayd-Allah Abd Al-Bakri quoted the mosque of Sidi Ghanem as "the first Mila mosque adjoining Dar El Imara" (House of Command).



Fig. 2. Geographic location of the ancient mosque of Mila

#### Sampling

The BDX 01 sample was taken from the arch of the second nave and the fourth span while the BDX 02 sample was taken from the arch of the second nave and the eighth span (Fig. 3).



Fig. 3. Diagram showing the place of collection of the two samples BDX 01 and BDX 02

#### **Results and discussion**

### The equivalent dose

For the measurement of the equivalent dose, we used the technique of small quartz inclusions (fine grain technical quartz), since the brick fragments presented a very fine ceramic [4]. The technique consists in using quartz of dimensions such that the alpha particles coming from the ceramic pass through the crystals. The particle size chosen was  $3-12\mu$ m.

The protocol that was used to determine the beta equivalent dose of bricks is based on the papers of Ian Bailiff [5, 6]. It is a protocol derived from SAR (Single Aliquot Regenerative), a procedure very universally used for dating sedimentary deposits, as described by Murray and Wintle [7]; Wintle and Murray [8]). It is important to specify that on the same aliquot the measurements of the natural OSL are directly compared to those of the OSL of the regeneration doses, which makes it possible to deduce an equivalent dose from a single test sample. However, it is necessary to specify that this technique supposes a multiplication of test intakes in order to evaluate experimentally the statistical uncertainty of the archaeological dose measurement. Unlike the traditional SAR protocol, the protocol implemented for bricks takes into account the fact that the materials have been strongly heated in the past and that inevitably there is a progressive variation in the sensitivity to irradiation during measurements, linked to the progressive filling of very deep traps with electrons from "optical" or photo-sensitive traps [9].

Table 1 describes the protocol used. The measurement of the residual signal at all stages of the protocol makes it possible to highlight a possible evolution of the zero level of the OSL during the measurement. Similarly, the evolution of the test signal, Tn or Ti, makes it possible to correct the changes in sensitivity of the material. Since the regeneration doses in stages 1 and 5 for the study of preheating or in stages 2 and 5 for the measurement protocol for equivalent doses are the same, it is possible to check the adequacy of the sensitivity corrections by measurement course (refresher test). The OSL resulting from the dose test in step 0, was used as a reference for a so-called "recovery" test intended to verify that the dose measurement is indeed consistent with that which was administered (recovery test). Finally, several preheating temperatures are possible, generally ranging between 180 and 240°C. The purpose of

preheating is to minimize the influence of thermally unstable components on natural, regenerated and test signals.

 Table 1. Protocol for measuring the equivalent dose of fine grains extracted from architectural brick for a test sample (disc containing a thin layer deposit of fine quartz grains)

Step	Operation	Measure
		height
	Natural signal measurement	
	Preheatig 10s@T <sub>p</sub> (10 seconds at preheating temperature Tp)	
	Reading signal OSL Nat at 125°C during 40s	L <sub>n</sub>
	Preheating 10s@T <sub>p</sub>	
	Reading signal OSL residual at 125°C during 40s	$RL_n$
Step 0	Irradiation by dose test $D_0(2.98 \text{ Gy})$	
	Preheating $10s@T_p$ (10 seconds at $T_p$ preheating temperature)	
	Lecture signal OSL test à 125°C durant 40s	$T_n$
	Preheating 10s@T <sub>p</sub>	
	Reading signal OSL residual at 125°C during 40s	RT <sub>n</sub>
	Measurements of regenerated OSL signals (steps i = 1 to 5)	
	Irradiation with a regeneration dose	
	Di = {2.98; 5.96; 8.94; 0; 2.98, 11.92} Gy for the preheating tests	
	Di = {1.49; 2.98; 4.47; 0; 2.98, 5.96} Gy for the measurement of equivalent doses	
	Preheating $10s@T_p$ (10 seconds at $T_p$ preheating temperature)	
	Lecture signal OSL Nat à 125°C durant 40s	$L_i$
	Preheating 10s@T <sub>p</sub>	
	Reading signal OSL residual à 125°C during 40s	$RL_i$
	Irradiation by dose test D <sub>0</sub>	
Step 1	Preheating $10s@T_p$ (10 seconds at $T_p$ preheating temperature)	Ti
	Reading signal OSL residual at 125°C during 40s	
	Preheating 10s@T <sub>p</sub>	
	Reading signal OSL residual at 125°C during 40s	RT <sub>i</sub>
	If i <6 Go to the next step, otherwise go to the next aliquot	

#### Determination of the preheating temperature

For each sample, the prior determination of the best preheating conditions is necessary. Tests were carried out for separate preheating temperatures (180, 200, 220 and 240°C), on 12 discs. We used 3 discs for the same preheating temperature to test the reproducibility of the observations. Table 2 presents the results obtained. In this work we have retained the preheating temperature which produces the slightest changes in the recycling ratio (determined between step 1 and step 5 of the SAR protocol), otherwise expressed, we use the conditions of preheating of the material which induces the slightest disturbances in the sensitivity of the material while allowing the erasure of the thermally unstable components. We finally opted for the following conditions:

- 200°C for BDX 01 - 210°C for BDX 02

 
 Table 2. Determination of the conditions for measuring the equivalent dose of brick on the basis of OSL recycling reports

Sample	Preheating temperature	180°C	200°C	220°C	240°C
BDX 01	Recycling ratio	$0.96 \pm 0.06$	0.95±0.09	$1.04\pm0.11$	1.24±0.15
BDX 02	Recycling ratio	1.21±0.14	0.88±014	1.13±0.13	1.32±0.10

#### Alpha efficiency factor

The efficiency factor alpha was not measured here but set equal to  $0.03\pm0.01$ , which corresponds to the usual values of this parameter for the OSL of quartz.

## Determination of equivalent doses

The same protocol as that presented in Table 1 was used, but with defined temperature preheats (210°C for BDX 02 and 200°C for BDX 01). However, we have modified the regeneration doses so as to adapt them to the value of the equivalent dose in Table 3 which we were able to estimate more precisely with the measurements made during the preliminary study of the preheating conditions.

Sample	Number of discs for measuring the equivalent dose Average	Quantity of material deposited in the discs	Equivalent dose (Gy)
BDX 01	19	0.3mg	$3.65 \pm 0.08$
BDX 02	16	0.1mg	2.52±0.14

**Table 3.** Determination of equivalent doses of brick

The recycling report was reassessed based on SAR measurements. The table presents the mean and standard deviation on the mean of this ratio, data which are therefore evaluated from a larger number of aliquots than when studying the preheating conditions. The measured values, close to 1, confirm a posteriori the correctness of the preheating choices.

## Annual radiation dose

The annual radiation dose corresponds to the dose absorbed by the luminescent minerals from which the archaeological doses are determined. These are therefore small inclusions of quartz in the case of terracotta, and individual grains of quartz in the case of lime mortars.

Natural radiation comes from alpha particles, beta particles, gamma photons and cosmic rays which give up all or part of their energy to the material to be dated. Due to the very different path of these various particles and radiations in matter ( $20\mu$ m for alphas, 2mm for betas, 30cm for gammas, several meters for cosmics) we distinguish several components:

- The alpha component originates from the decay of uranium and thorium: alpha irradiation comes from the immediate environment of luminescent grains but also quartz grains when these contain significant traces of radioelements;

- The beta component, resulting from the decays of K, U and Th, comes essentially from the environment of quartz grains, over a radius of about 2mm. If the grains are far from an irradiation discontinuity (more than 2 mm from the interfaces between two building materials for example), the beta dose comes exclusively from the sample to be dated, the brick. If the luminescent grains themselves contain radioactive elements, a small part of the irradiation can also come from this source;

- The gamma component comes from the radioelements K, U and Th, present in the sample to be dated and in its environment;

- The very penetrating cosmic component is attenuated by the architectural elements or the burial materials which screen their path towards the sample;

The annual dose is therefore the sum of these various contributions, according to,

$$\dot{d} = \mathbf{k}. \ \dot{d}_a + \dot{d}_\beta + \dot{d}_\gamma + \dot{d}_{cosm}$$
 (2)

The k factor is the efficiency factor of alpha particles compared to beta and gamma. Generally, this factor is very small, which means that at equal dose, the alpha particles provide an OSL signal which is a fraction k (of the order of a few%) of the signal supplied by beta or gamma. The origin of this difference is related to the nature of alpha particles, compared to beta and high energy secondary electrons generated by the interactions of matter with gamma rays or cosmic rays, and their ionization capacity of the irradiated material. It is determined experimentally by comparative OSL experiments between calibrated alpha and beta irradiations [10].

## Radiochemical measurements

The alpha and beta component is determined from radiochemical measurements of the samples to be dated by gamma spectrometry with low background noise [11]. Possible additional measurements by ICP MS are carried out on the luminescent grains which allow the measurement of archaeological dose, when it is estimated that these grains contain radioelements in significant quantity [12-15]. By working on the small inclusions of quartz as we did here on the two brick samples, BDX 01 and 02, it is however not necessary to measure their contents in radioelements because we suppose in this case verified the hypothesis of radiochemical continuity and alpha irradiation between the fine grains and their local environment.

In order to determine the environmental component of irradiation, we generally carry out measurements on site, at precise sampling points, by gamma-ray measurement (using a probe sensitive to gamma and cosmic radiation) and by dosimetry by luminescence using alumina pellets sintered under reducing conditions (Al2O3:C) which have a very high sensitivity to irradiation. In this work, no such measurements were made and we simply proposed a plausible estimate of this component taking into account the position of the samples in the architectural structure and the surrounding materials which are known to have rather low radioactivity (marble and limestone). The value chosen is:  $0.60\pm0.20$ mGy/year reflects this level of radioactivity. It also contains a cosmic term which we estimate to be around 0.2mGy/year.

## The role of humidity

We must emphasize that laboratory measurements are carried out on dry samples, and it must take into account the humidity of materials in an "archaeological" situation, that is to say estimate the average water content since the placement of materials to determine the annual radiation dose. The terracotta samples were taken from elevated structures and were dry when taken in the laboratory. We considered that this dry state was also the archaeological state.

### Results of the annual dose assessments

The measurements of radioelements of the bricks by gamma spectrometry at low background noise are presented in table 4.

Sample	H <sub>2</sub> O (%)	U( <sup>238</sup> U) (ppm)	U( <sup>226</sup> Ra) (ppm)	U( <sup>210</sup> Pb) (ppm)	Th (ppm)	K (%)
BDX 01	0.0	2.16±0.19	2.34±0.04	2.74±0.43	8.41±0.12	1.29±0.03
BDX 02	0.0	$2.01\pm0.17$	2.07±0.03	$1.84\pm0.35$	7.93±0.11	$1.35\pm0.02$

Table 4. Humidity and radioactive elements content of the brick samples. Uncertainty is a standard deviation

For the uranium series, there are three measurement systems depending on the nature of the gamma emitters:

-U (<sup>238</sup>U) is the uranium content determined from <sup>234</sup>Th and <sup>235</sup>U isotopes respectively in equilibrium and in constant relationship with <sup>238</sup>U;

- -U (<sup>226</sup>Ra) is the equivalent uranium content determined from <sup>214</sup>Pb and <sup>214</sup>Bi, under the equilibrium conditions between <sup>226</sup>Ra and <sup>222</sup>Rn (sealed container, waiting for equilibrium for measurement);
- -U (<sup>210</sup>Pb) is the equivalent uranium content determined from <sup>210</sup>Pb.

These three measurements of uranium make it possible to assess the state of equilibrium of the uranium chain, which gives some clues to possible changes in the radioactivity of the material due to the contribution or the impoverishment of certain soluble or mobile elements.

For example, U (<sup>210</sup>Pb) is an indicator of the average activity of radon-222 in the sample for 30 years (radon is an element belonging to the series of chemically inert gases: it is relatively little linked to the material that generates it). A lower value of U (<sup>210</sup>Pb) than U (<sup>226</sup>Ra) is interpreted as a continuous radon flow in the masonry element concerned, conversely a higher value, means in the balance sheet a contribution radon from neigh boring elements.

Here, <sup>210</sup>Pb and <sup>226</sup>Ra are quite close, with near measurement uncertainties. These are nevertheless high for <sup>210</sup>Pb with the well detector reserved for small quantities of material. They do not show a very pronounced imbalance and we can consider that the radon formed remains confined in the material.

In addition, there is a good agreement between U ( $^{238}$ U) and U ( $^{226}$ Ra), an indication of the absence of alteration inherited from the raw material and absence of alteration of the material which occurred during burial or exposure of masonry to rather aggressive environmental conditions.

Finally, overall, the two samples have a very similar radiochemical composition, which could indicate one or more fairly homogeneous sources of clay for the manufacture of these bricks. This is only an index and other elements of characterization are obviously necessary to specify the existence or not of several sources of raw material.

The annual content/dose conversion is carried out using the conversion coefficients calculated and published by Guérin *et al.*[16]. Compared to the original presentation of the authors, we have modified them to take into account the scenarios of potential imbalance in the uranium series of the samples. Thus, we grouped radioactive elements in radioactive filiation according to the nature of the parent isotope and alteration hypotheses. Thus, we assumed that the difference in activity between radium-226 and uranium-238 could be linked to an alteration in the uranium concentration. This leads to grouping the isotopes ranging from <sup>238</sup>U to <sup>234</sup>U activity is associated with <sup>226</sup>Ra for the <sup>238</sup>U series and with the <sup>231</sup>Pa ... <sup>207</sup>Pb fragment for the <sup>235</sup>U series. This table also differentiates the activity of <sup>226</sup>Ra and that of <sup>210</sup>Pb. If we assume the mobility of radon-222, its average activity is represented by that of <sup>210</sup>Pb. From the point of view of the kinetics of the imbalance we have assumed that it has been constant throughout the "archaeological" duration that interests us table 5.

Element	Concentration unit	Series of concerned isotopes	Alpha	Beta	Gamma
U( <sup>238</sup> U)	ppm	<sup>238</sup> U <sup>234</sup> U et <sup>235</sup> U- <sup>231</sup> Th	0.5706	0.056	0.0017
U( <sup>226</sup> Ra)	ppm	<sup>230</sup> Th- <sup>226</sup> Ra et <sup>231</sup> Pa <sup>207</sup> Pb	0.6961	0.0041	0.002
U( <sup>210</sup> Pb)	ppm	<sup>222</sup> Rn <sup>206</sup> Pb	1.5283	0.0855	0.1079
Th	ppm	<sup>232</sup> Th <sup>208</sup> Pb	0.7375	0.0277	0.0479
K	%	<sup>40</sup> K- <sup>40</sup> Ar, <sup>40</sup> Ca	-	0.7982	0.2491

**Table 5.** Specific annual doses from Guérin *et al.* [16] (in mGy/year). We compare different fragments of the U series to take into account the uranium imbalance scenario of the samples (constant imbalance over time originating from the mobility of the U element and that of radon-222)

The annual dose values and their components are presented in Table 6. The most significant loss of precision is linked to the lack of field measurements of the annual

environmental dose. What we do know is that if the environment consisted only of bricks, the maximum annual gamma dose would be that provided by an infinite medium composed of bricks of the same composition, is approximately 1.0mGy/year to which would be added the cosmic contribution, of the order of 0.2mGy/year taking into account the screen effects of masonry in elevation. A priori bricks are minority masonry elements, too, even if we do not know either the proportion of other materials in the environment of the samples, their own radioactivity, or the dimensions of bricks, by comparison with other constructed sites comprising brick levelings or isolated bricks in a limestone environment, we estimate that the value of 0.6±0.2mGy/year a is a realistic approximate value.

Sample	d <sub>a</sub> (mGy/an)	ḋ <sub>β</sub> (mGy/an)	d <sub>env</sub> (mGy/an)	Annual total dose (d) (mGy/an)
BDX 01	$0.40\pm0.14$	$1.60\pm0.05$	0.60±0.20	2.60±0.25
BDX 02	0.34±0.12	$1.55 \pm 0.04$	$0.60\pm0.20$	$2.49 \pm 0.24$

Table 6. Components of the annual dose and total annual dose

Table 7 presents for each sample of architectural terracotta, the archaeological dose, the annual dose and the age in years compared to 2019, as well as the details of the systematic and statistical uncertainties.

Table 7. OSL dating results for two bricks from the mosque								
Sample	Equivalent dose (Gy)	Annual dose (mGy/an)	Age (years)	incertainty. (years)	Date (years)			
BDX 01	3.65±0.08	2.60±0.25	1403	78	616±78 AD			
BDX 02	2.52±0.14	2.49±0.24	1012	80	1007±80AD			

Гał	ole	7.	OSL	dating	results	for two	bricks	from	the	mosque
-----	-----	----	-----	--------	---------	---------	--------	------	-----	--------

We also give the calendar age of architectural brick according to the common calendar (CEA: common era or AD according to the Christian calendar [17].

The results obtained by the OSL datig in single grain on the brick of the sidi Ghanem mosque shows that these in the medieval period with certain uncertainties for the first sample BDX 01 is dated in 616AD more at least 78 years, which corresponds to an interval of manufacture of this brick located between 538 - 694AD, if we project these dates on the Hijri calendar we obtain the interval of -84 - 72H (Fig. 3), the dates before the Installation of the banū umayya dynasty or the Umayyads 661 to 750AD [18] because under the reign of the latter which begins the conquests towards the southern shore of the Mediterranean and the Middle East and in particular under the command of Oqba ibn Nāfi (665-689) that had the second invasion of the Maghreb by Oqba ibn Nāfi which arrived at the shore of the Atlantic Ocean [19].

So we can admit that the interval between 42 to 72H, 665 - 694AD as logical interval is acceptable from the manufacture of terracotta BDX 01 and the construction of the primitive part of the Mosque if you consider the time between the manufacture of this brick and its insertion into the masonry is very negligible (Fig. 4).



Fig. 4. Chronological of the construction of the mosque

For the second terracotta BDX 02, it is manufactured between 927-1087AD which corresponds to 305-465H between the Ziride dynasty or banū Zīrī (972-1014) and the dynasty of Banū Hammād or Hammadides 1019 - 1152AD [20] the most probable hypothesis is that the mosque was enlarged by the Hammadids since elements of decoration of Kufic inscription resembling that found by Golvin at the Qala'a of Banū Hammād where the city of Mila knew a considerable development for that we can admit that the interval 1019 - 1087 as interval of modification of this mosque (extension), figure 4.



Fig. 5. Chronological modification of the mosque

#### Conclusions

The OSL dating results, a unique grain on the fine fraction produced on the architectural terracotta tiles of the Sidi Ghanem Mosque, confirm the hypotheses of construction and modification (enlargement) of the latter. It is a medieval mosque built in a very advanced date of the Muslim conquests, the date by OSL displayed a date  $616\pm78AD$  c-ad in 538 - 694AD which corresponds to -84 to 72 of the Hégirien calendar although the interval of uncertainty is rather important we were able to reduce this interval using a cross analysis with the historical events which marked this period, can admit as the interval between 665 - 694AD which corresponds to 42 to72H, as possible interval of the construction of this mosque. For the extension of this mosque, the OSL analysis shows a date of  $1007 \pm 80AD$  cad 927-1087AD which corresponds to 305-465H, a relatively high interval this time covering two dynasties that of Banū Zīrī and that of Banū Hammād. For this we made a cross analysis with the objects found during the excavations inside the prayer hall of the mosque which confirmed the strong presence of the Banū Hammād and the absence of the remains relating to banū Zīrī for this we have admitted that the interval 1019 -1087AD as possible interval for the extension of this mosque.

#### Acknowledgments

The Authors Would Like to Thank the Financial Support From Boumerdes University. Moreover, the Authors are Grateful to Research Institute on Archaeomaterials, Research Center in Physics Applied to Archaeology, Iramat-Crp2a-Umr 5060-Cnrs – University Bordeaux Montagne, for the Collaboration in Laboratory Testing.

#### References

[1] E. Vieillevigne, *The potential of optically stimulated luminescence for medieval building; A case study at Termez, Uzbekistan,* Radiation Measurements, 41(7), 2006, pp. 991-994.

- [2] G.A.T. Duller, *The Analyst software package for luminescence data: overview and recent improvements*, Ancient TL, 33(1), 2015, pp. 35-42.
- [3] H. Fournel, Les Berbers : étude sur la conquête de l'Afrique par les Arabes, d'après les textes arabes imprimés. Tome 1, Impr. Nationale, Paris, (1799-1876) (1875–1881).
- [4] S. Blain, P. Guibert, A. Bouvier, E. Vieillevigne, F. Bechtel, C. Sapin, M. Baylé, 2007. TLdating applied to building archaeology: The case of the medieval church Notre-Damesous-Terre (Mont-Saint-Michel, France), Radiation Measurements, 42, 2007, pp. 1483-1491.
- [5] I.K. Bailiff, N. Holland, *Dating bricks of the two last millennia from Newcastle upon Tyne: a preliminary study*, **Radiation Measurements**, **32**, 2000, pp. 615- 619.
- [6] I.K. Bailiff, Methodological developments in the luminescence dating of brick from English late-medieval and post-medieval buildings, Archaeometry 49, 2007, Article Number: 827e851. <u>http://dx.doi.org/10.1111/j.1475-754.2007.00338.x</u>.
- [7] A.S. Murray, A.G. Wintle, *Luminescence dating of quartz using an improved single-aliquot regenerative dose protocol*, **Radiation Measurements**, **32**, 2000, pp. 523–538.
- [8] A.G. Wintle, A.S. Murray, A review of quartz optically stimulated luminescence characteristics and their relevance in single-aliquot regeneration dating protocols, Radiation Measurements 41(4), 2006, pp 369–391
- [9] S.W.S. McKeever, R. Chen, *Luminescence models*, Radiation Measurements, 27(5-6), 1997, pp. 625-661.
- [10] M.J. Aitken, Thermoluminescence Dating, Academic Press, London, 1985, p. 359.
- [11] P. Guibert, C. Lahaye, F. Bechtel, *The importance of U-series disequilibrium of sediments in luminescence dating: a case study at the Roc de Marsal cave (Dordogne, France),* Radiation Measurements, 44, 2009, pp. 223-231.
- [12] F. De Corte, D. Vandenberghe, J.-P. Buylaert, P. Van den Haute, J. Kučera, *Relative and k<sub>0</sub>-standardized INAA to assess the internal (Th, U) radiation dose rate in the 'quartz coarse-grain protocol' for OSL dating of sediments: Unexpected observations*, Nuclear Instruments and Methods in Physics Research A 564, 2006, pp. 743–751.
- [13] D. Vandenberghe, F. De Corte, J.-P. Buylaert, J. Kučera, P. Van den Haute, *On the internal radioactivity in quartz*, Radiation Measurements, 43, 2008, pp. 771 775.
- [14] P. Guibert, I.K. Bailiff, S. Blain, A.M. Gueli, M. Martini, E. Sibilia, G. Stella, S.O. Troja, Luminescence dating of architectural ceramics from an early medieval abbey: The St-Philbert intercomparison (Loire-Atlantique, France), Radiation Measurements, 44(5-6), 2009, pp. 488-493.
- [15] P. Urbanová, A. Michel, N. Cantin, P. Guibert, P. Lanos, P. Dufresne, L. Garnier, A novel interdisciplinary approach for building archaeology: The integration of mortar "single grain" luminescence dating into archaeological research, the example of Saint Seurin Basilica, Bordeaux, Journal of Archaeological Science: Reports, 20, 2018, pp. 307-323.
- [16] G. Guérin, N. Mercier, G. Adamiec, *Dose-rate conversion factors: Update*, Ancient TL, 29, 2011, pp. 5-8.
- [17] \* \* \*, CEA (collectif anonyme), Statistique appliquée à l'exploitation des mesures, Tom.1, Ed. Masson, Paris, 1978, 148p.
- [18] M. Andrew, Rituals of Islamic Monarchy: Accession and Succession in the First Muslim Empire, Edinburgh University Press, 15 avril 2009, p. 99.
- [19] E. Gibbon, F. Guizot (éditeur et réviseur), Histoire de la décadence et de la chute de l'Empire romain, Tom. 10, Paris, Lefèvre, 1819, p. 283.

[20] Ibn Khaldoun, Les prolégomènes d'Ibn Khaldoun, Vol. 2, (French Edition), Hardcover, 2018, 506 p.

*Received: June 30, 2020 Accepted: May 28, 2021*