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CHRONOLOGY OF THE CRYPT OF *SANT'AGATA LA VETERE* IN CATANIA OBTAINED BY THERMOLUMINESCENCE

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ABSTRACT

During the restoration works of the *Sant'Agata La Vetere* church in Catania, a campaign of archaelogical excavations was conducted in order to study the complex site around the religious building. According to tradition and to historical sources, the site was very interesting because a part of the church, in particular the crypt, was built incorporating an apse of the roman *praetorium* palace. Starting from this assumption, the present study is focused on the resolution of the chronological question about the different construction phases of the crypt.

In agreement with archaeologists and architects, samples of bricks are collected in specific area. The ThermoLuminescence (TL) methodology was applied performing measurements on polymineral fine grain phases extracted from each brick. The Equivalent Dose (*ED*) values were evaluated from TL signals through Added Dose procedure. The annual Dose Rate (*DR*) was calculated using the radioactive content of samples estimated by ICP-MS analysis and environmental and cosmic dose rates obtained by *in situ* gamma measurements.

From the obtained ages, three phases of construction were individuated: 3^{rd}, 7^{th}-8^{th} and 17^{th} centuries. In particular, the first one could be correspond with the end of the roman period, and it seems to confirm the archaeological hypothesis based on the historical sources.

From a methodological point of view, the archaeological issue contributed to study the behaviour vs time of the alpha efficiency coefficient *k*. The results showed that the value obtained after 30 days has to be considered.

KEYWORDS: bricks, architecture, *praetorium* palace, historical building, fine grain.

1. INTRODUCTION

The church of *Sant'Agata la Vetere* is located in a complex area of urban stratification. In fact, it is situated along an imaginary line that connects the three religious buildings: *Sant'Agata alla Fornace*, *Sant'Agata al Carcere*, and the same *Sant'Agata la Vetere*. All these churches are closely linked to the religious tradition and the workship of *Sant'Agata*, Catania patron's Saint.

The S*ant'Agata la Vetere* church is considered the first cathedral of Catania. It is collocated on *Montevergine* hill, occupied from the Greek foundation of the city, in an area of fundamental defensive importance until the sixteenth century (Arcifa, 2002; Barbera et al., 1998; Correnti et al., 1999). According to tradition, this church was built on the site where *Agata* was martyred. For this reason, the bishop *Everio*, in the 3rd century, erected an aedicule and then a church was built by bishop *Severino,* in the period from 380 to 436 AD. In 776-778 AD, Leone II rebuilt and expanded the religious building in a *basilica* (Rasà Napoli, 1984). In the axonometric plan drawing (Fig.1) of Spannocchi (1578) the presence of the *praetorium* palace area and the church are indicated (Spannocchi, 1578).

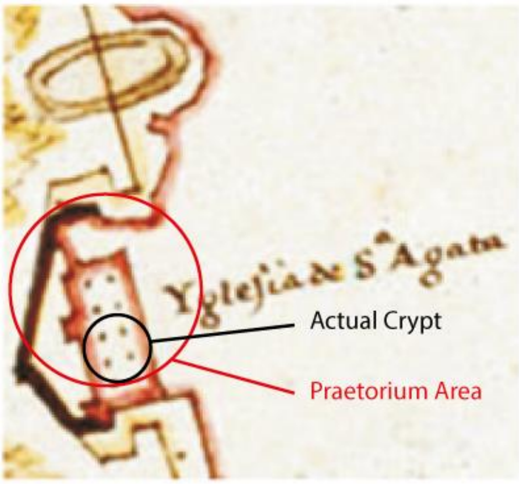


Figure 1. Axonometric plan drawing of the praetorium area indicated by Spannocchi (1578).

Several historical sources are agree in indicating *Sant'Agata la Vetere* as site in which a roman *praetorium* palace were. Carrera (Carrera, 1639) and Sciuto Patti (Sciuto Patti, 1892) reported the notice of the church next to the *Quinziano*'s palace ruins. Furthermore, in a Bernardino Negro painting representing the *Sant'Agata* martyrdom, the *praetorium* palace in ruins is visible (Negro, 1588). According to tradition, the *praetorium* probably remained until a natural event destroyed it. The event could be dated with the earthquake of 251-252 AD or one of the 361-365 AD reported by ancient and medieval literary sources and documented by INGV (*Istituto Nazionale di Geofisica e Vulcanologia*) in the Parametric Catalogue of Italian Earthquakes (www.ingv.it). After archaeological excavations, a study was conducted in order to date the historic building, particularly the crypt. The crypt (Fig.2) is collocated in the *Sant'Agata la Vetere* church, in correspondence of presbytery at an altitude of 6 meters lower relative to the first floor.

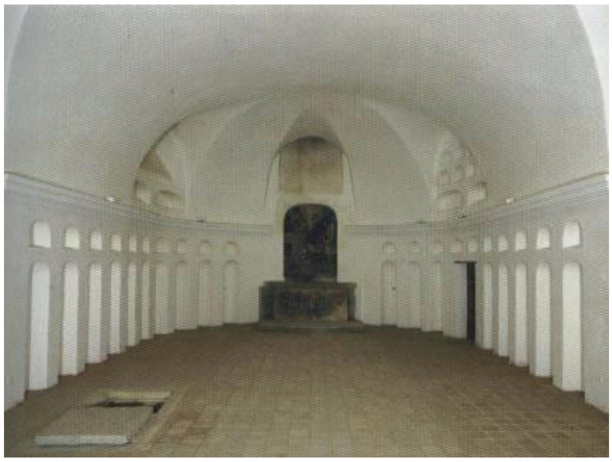


Figure 2. The Sant'Agata la Vetere crypt.

The discovery in 1767 of the stone lava block studied and analyzed by the philologist Finnish Kalle Korhonen (Korhonen, 2008) probably confirmed the archaeological hypothesis that the crypt incorporated a part of a roman *praetorium* palace. The stone lava building block reported the inscription "*L(ucio) Rubrio Proculo, Hvir(o) quin(quennali), auguri*". The inscription is referred to the praetor and could testify the possibility that the actual crypt was a local of *praetorium* palace. In the 2nd and in the 4th centuries two earthquakes destroyed the old crypt roof and the stone block with inscription was hidden until the discovery in 1767 (Korhonen, 2008).

In order to solve archaeological issues and to date the different construction phases of the crypt, the ThermoLuminescence (TL) technique was applied after sampling of bricks in agreement with archaeologists and architects.

The TL for historical building dating is considered a routine technique (Martini et al., 2006; Bailiff, 2008; Gueli et al., 2009, 2010, 2015; Blanco Rotea et al., 2010; Blain, 2010; Liritzis et al., 2013a; Stella et al., 2013, 2014).

The principles and general aspects of this methodology are described in detail by Aitken (Aitken, 1985).

Referring to detailed literature regarding the TL dating technique (Aitken, 1985; Troja et al., 2000; Martini et al., 2001; Zacharias et al., 2002; 2006; Blain, 2010) the general equation used to determine the age, time from the last firing of the artefact until today, is the ratio between the Equivalent Dose (*ED*) and the annual Dose Rate (*DR*).

The *ED* is the total dose accumulated from the last firing of *terracotta* samples, measured using luminescence signals, while the *DR* is the quantity of the dose absorbed in one year and is measured by independent techniques.

About *ED* evaluation, different methodologies are routinely performed (Zimmermann, 1971; Fleming et al., 1979; Mejdahl et al., 1979; Goedicke et al., 1981; Wintle, 1997). In this study the Age TL was evaluated by the *ED* determination based on the TL emissions obtained with the additive dose procedure of multiple aliquots (Aitken, 1985; Gueli et al., 2009, 2010, 2015; Stella et al., 2013, 2014) on fine-grain fractions extracted from the sample (Zimmermann, 1971). The *DR* contributions from the sample were calculated using the radioactive contents of natural decay series of ^{235}U, ^{238}U, ^{232}Th and of the ^{40}K radioactive isotope while the environmental and cosmic dose rates were obtained by *in situ* measurements.

2. MATERIALS AND METHODS

2.1 Samples collection and preparation

Four areas (A, B, C and D) were individuated in the crypt and different bricks were collected for TL dating.

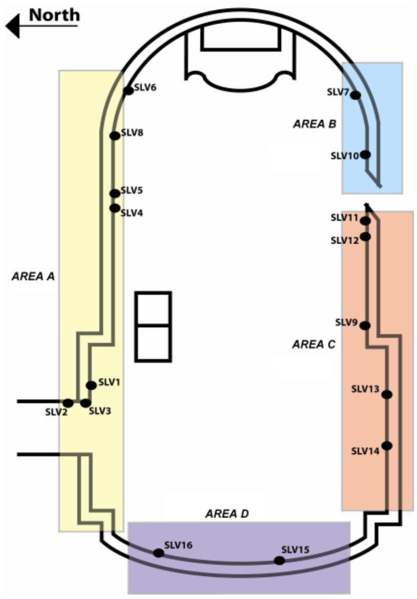


Figure 3. Planimetry of the crypt, with the ID codes of collected samples.

The Fig.3 shows the crypt plan, the sampling points with the relative area and IDentification (ID) codes of each brick.

In agreement with archaeologists and architects sixteen samples were collected. From area A three samples were taken in the entrance wall (SLV1, SLV2 and SLV3) and four samples (SLV4, SLV5, SLV8 and SLV6) in the north wall. From area B, in the south wall, SLV7 and SLV10 samples were collected while SLV9, SLV11, SLV12, SLV13 and SLV14 samples, from area C. In area D, from the west wall, SLV15 and SLV16 samples were taken.

About the sample preparation, the protocol Fine Grain (FG) was applied (Guibert et al., 2009). The first step is the removal of the outer 2 mm from all faces of the sample that influences the DR calculation (Liritzis, 1986). The inner parts of the bricks were then crushed and sieved. The finest fractions (<40 μm) were etched by hydrochloric acid (1 M) for 100 min at Room Temperature (RT) to dissolve carbonates and then treated with hydrogen peroxide to remove organic material. Remaining clay materials that coated luminescence crystals were dissolved by diluted HF (45min@1%) in combination with HCl (30min@10%). The 4–11 μm grains were then selected by sedimentation in acetone and finally deposited onto 9.8 mm diameter discs, approximately 2 mg per aliquots (homogeneous portions of the same sample). At the end of this procedure, entirely performed in dim red light, a polymineral fraction is obtained.

2.2 Equation Age

In the case of Fine Grain polymineral fraction, the age is calculated as shown below (Aitken, 1985):

$$Age = ED/(k \cdot D_{\alpha} + D_{\beta} + D_{env})$$

where *ED* is the Equivalent Dose and *k* is the alpha efficiency, calculated through luminescence measurements. D_{α} and D_{β} are respectively, the Dose Rate contributions derived from alpha and beta decay of the radioactive contents present in the sample. The D_{env} is the the contribution to the Dose Rate resulting from gamma and cosmic emissions of the environment. The evaluation of this last contribution is important because it is linked to meteorological and seasonal factors (Liritzis and Galloway, 1981; Liritzis, 1985).

2.3 ThermoLuminescence measurements

The Added Dose procedure is used for the calculation of *ED*. It is applied on the acquisitions, on different portions of the same sample, of natural TL intensity and that obtained by adding to the sample increasing values of artificial dose. In this case a β calibrated source ($^{90}Sr/^{90}Y$) was used. With Added Dose procedure it is possible to determine the value of the beta dose equivalent to the total natural dose received by the sample from the evaluation of *Q* and of a corrective term associated with nonlinear behaviour at low doses (*q*). The final

ED value is the sum of the experimental *Q* and *q* data (Guibert et al., 2009).

TL glow curves were recorded by heating the aliquots up to 500°C at a uniform heating rate of 5°C/s in an ultrapure nitrogen environment. The inter-aliquot variations in luminescence were corrected by second glow normalisation (Aitken, 1985). Temperature plateau region comprising 250 and 350°C TL peaks of the glow curves was used for calculation of *ED* (*Q*).

The TL measurements were performed using automated Risø reader TL-DA-10 (Bøtter-Jensen, 1997) with EMI 9235QA photomultipliers. TL glow curves were recorded using Corning 7-59 and Schott BG-12 optical filters in the detection phase.

2.3.1. Alpha efficiency measurements

The use of polymineral fine grained fraction for thermoluminescence dating requires the measurement of alpha efficiency for the estimation of this effective contribution to the dose rate. The luminescence signal induced by alpha particles is related to its range (with an average of 20 μm in quartz), and it is a linear function of the integrated alpha flux (Aitken, 1985). In order to evaluate the alpha efficiency during the initial phase of TL dating of bricks, Zimmerman (Zimmerman, 1971) introduced a *k* value to describe the efficiency of alpha irradiation relative to beta irradiation, which was defined as the ratio of the TL per unit absorbed alpha dose to the TL per unit absorbed beta dose.

From artificial luminescence signals induced by calibrated beta and alpha irradiations, it is possible to determine the luminescence efficiency coefficient *k* necessary to correct the alpha contribution to the annual dose rate (Gueli et al., 2009; Guibert et al., 2009; Blain et al., 2010; Polymeris et al., 2011).

This coefficient is determined experimentally according to the following formula:

$$k=(L_\alpha/D_\alpha)/(L_\beta/D_\beta)$$

where L_α is the luminescence obtained from alpha artificial irradiation (D_α) and L_β is the luminescence obtained from artificial beta radiation (D_β). Artificial alpha irradiations were performed with an external ^{241}Am calibrated alpha source delivering 2.1 Gy/min. In order to assess the effects of alpha radiation in the determination of *k* coefficient, the measurements sequence shown in Table I was used.

For each sample, preheat temperature was determined by tests that consist in the comparison between the natural and the artificial signals of TL induced by beta radiation using different preheat temperatures.

Table I. Steps of measurements for the evaluation of the alpha radiation effects in the k determination.

Steps	Treatment	Observed
1	*Bleaching@ 650°C for 1 h*	
2	*Given β-dose (5 Gy)*	
3	*TL@500°C, preheat@x°C for 120 sec*	L_β
4	*Given α-dose (50 Gy)*	
5	*TL@500°C, preheat@x°C for 120 sec*	L_α

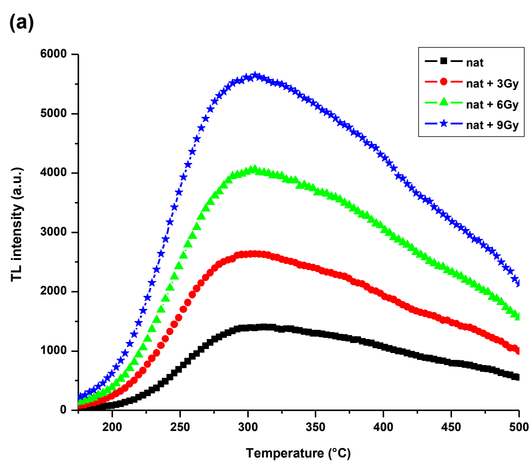
For applying the procedure shown in Table I, several aliquots of each collected sample were measured. The *k* measurements were performed at different time intervals from the irradiation phase. The signals were detected from a starting time, just after the irradiation of the aliquots, the relative coefficient is called k_0, after 15 days, 30 days and 45 days, to obtain the corresponding coefficients called, respectively, k_{15}, k_{30} and k_{45}.

2.4 ICP MS measurements

Internal Dose Rate of bricks were determined using radioelement concentrations of U, Th and K measured from Inductively Coupled Plasma Mass Spectrometry (ICP-MS). The concentration values were then converted into the corresponding dose rate contributions using the factors re-evaluated by Liritzis et al. (2013b). The influence of moisture was evaluated considering experimental parameters porosity, W, and saturation fraction, F, useful to correct the dose rate contribution (Zimmermann, 1971; Aitken, 1985; Gueli et al., 2017).

3. RESULTS AND DISCUSSION

Fig.4 shows, as example representative of all samples, the glow curves of SLV5 and the relative growth straight line from which the values of *Q*, Fig.4(a) and (b), and the correction q, Fig.4(c) and (d), were obtained.



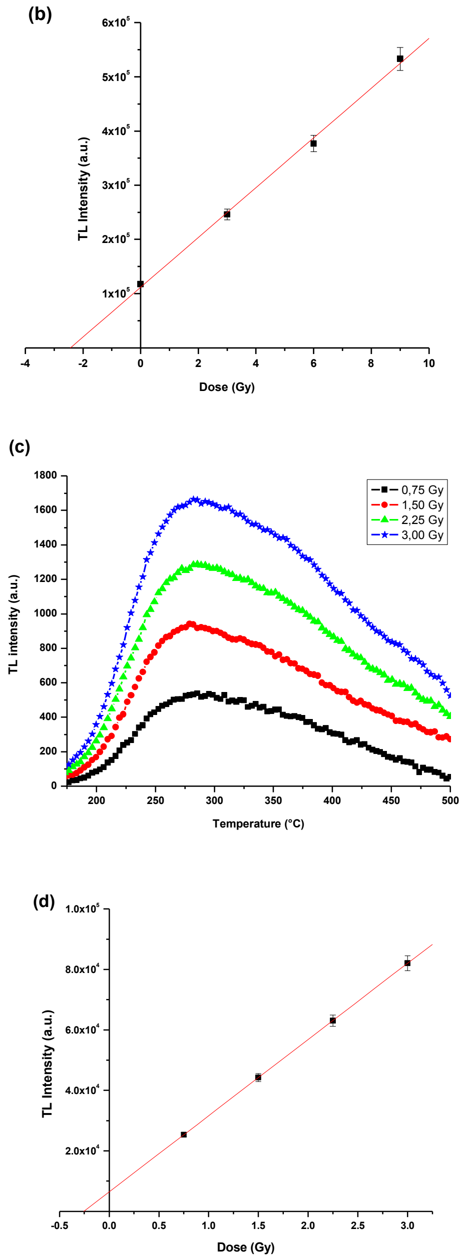


Figure 4. SLV5 sample: (a) TL signals obtained without (nat) and with 3, 6 and 9 Gy added beta dose irradiation; (b) growth curve useful to obtain equivalent dose Q; (c) glow curves related to artificial irradiation at low doses (0,75; 1,50; 2,25; and 3,00 Gy); (d) growth curve related to low doses for q calculation.

Fig.5 shows, as an example for the SLV11 sample, the behaviour of *k* values vs the time intervals between alpha irradiation and detection of the related luminescence signal. The data show that *k* coefficient reaches a constant value after 30 days.

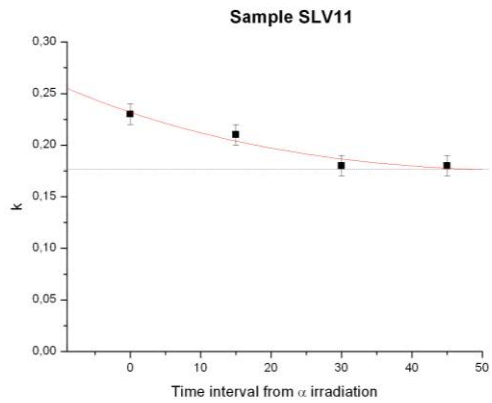


Figure 5. k value obtained for SLV11 sample.

Table II shows the values of *k* related to different temporal intervals from alpha irradiation: k_0 obtained from measurements just after the irradiation, k_{15}, k_{30} and k_{45} that correspond to a time interval from irradiation to reading phases equal to, respectively, 15, 30 and 45 days. Luminescence loss was evaluated in terms of percentage difference between the k_{45} and the k_0 values. The results highlight a loss causing a difference from a minimum of 15% to a maximum of about 30%. This evidence suggests the use of the *k* value related to a time interval of at least 30 days.

In Table III the U, Th and K of all samples with the corresponding D_α and D_β contributions, the total contribution (gamma and cosmic) of the environment (D_{env}) to the *DR* are reported. The *ED* values are reported in Table IV. This last table shows also *DR* total values obtained from contribution corrected considering the effective *in situ* water content. This correction was calculated by the porosity *W* values and the saturation *F* factors experimentally estimated for each sample. The individual dating (TL age in *anni*) results and the corresponding calendar dates (in *Annus Domini, AD*) calculated by the Age equation are also listed. Taking into account the statistical uncertainties, the results obtained for all samples individuate three different temporal phases (Fig.6).

The SLV7 and SLV10 samples, taken in the southeast area near the altar of the crypt (area B), belong to the first older phase centred to 3rd century. This area probably corresponds to the period from the edification of a crypt by bishop *Everio* (256 AD) to construction of the first church dedicated to Saint'*Agata* (380 - 436 AD).

Table II. k values obtained after irradiation, k_0, and after, respectively, 15 (k_{15}), 30 (k_{30}) and 45 (k_{45}) days from irradiation. The last column shows the percentage of luminescence loss from irradiation to reading after 45 days.

Sample	k_0	k_{15}	k_{30}	k_{45}	% loss
SLV1	0,18 ± 0,01	0,16 ± 0,01	0,14 ± 0,01	0,14 ± 0,01	22 ± 2
SLV2	0,30 ± 0,01	0,26 ± 0,01	0,24 ± 0,01	0,24 ± 0,01	20 ± 1
SLV3	0,28 ± 0,01	0,26 ± 0,01	0,24 ± 0,01	0,23 ± 0,01	18 ± 1
SLV4	0,24 ± 0,01	0,20 ± 0,01	0,17 ± 0,01	0,17 ± 0,01	29 ± 2
SLV5	0,22 ± 0,01	0,20 ± 0,01	0,18 ± 0,01	0,17 ± 0,01	23 ± 2
SLV6	0,23 ± 0,01	0,20 ± 0,01	0,19 ± 0,01	0,19 ± 0,01	17 ± 1
SLV7	0,29 ± 0,01	0,27 ± 0,01	0,25 ± 0,01	0,24 ± 0,01	17 ± 1
SLV8	0,21 ± 0,01	0,18 ± 0,01	0,16 ± 0,01	0,16 ± 0,01	24 ± 2
SLV9	0,20 ± 0,01	0,18 ± 0,01	0,16 ± 0,01	0,16 ± 0,01	20 ± 2
SLV10	0,26 ± 0,01	0,24 ± 0,01	0,23 ± 0,01	0,22 ± 0,01	15 ± 1
SLV11	0,23 ± 0,01	0,21 ± 0,01	0,18 ± 0,01	0,18 ± 0,01	22 ± 2
SLV12	0,27 ± 0,01	0,25 ± 0,01	0,22 ± 0,01	0,22 ± 0,01	19 ± 1
SLV13	0,25 ± 0,01	0,23 ± 0,01	0,21 ± 0,01	0,21 ± 0,01	16 ± 1
SLV14	0,22 ± 0,01	0,19 ± 0,01	0,18 ± 0,01	0,18 ± 0,01	18 ± 1
SLV15	0,17 ± 0,01	0,15 ± 0,01	0,13 ± 0,01	0,12 ± 0,01	29 ± 3
SLV16	0,22 ± 0,01	0,19 ± 0,01	0,17 ± 0,01	0,17 ± 0,01	23 ± 2

Table III. Radiochemical composition and dose rate contributions for the examined samples and the related sampling areas (see text for symbols explanation).

Sampling area	Sample	U (ppm)	Th (ppm)	K (%)	D_α (mGy/a)	D_β (mGy/a)	D_{env} (mGy/a)
Area A	SLV1	2,4 ± 0,1	10,0 ± 0,1	2,20 ± 0,01	13,19 ± 0,11	2,26 ± 0,01	0,96 ± 0,04
	SLV2	2,7 ± 0,1	10,4 ± 0,1	1,78 ± 0,01	14,80 ± 0,10	2,06 ± 0,01	0,89 ± 0,03
	SLV3	2,2 ± 0,1	9,7 ± 0,1	1,78 ± 0,01	12,76 ± 0,12	1,95 ± 0,01	0,90 ± 0,03
	SLV4	2,1 ± 0,1	8,3 ± 0,1	3,25 ± 0,01	10,96 ± 0,12	2,92 ± 0,01	0,82 ± 0,03
	SLV5	2,4 ± 0,1	11,2 ± 0,1	2,10 ± 0,01	13,64 ± 0,10	2,17 ± 0,01	0,83 ± 0,03
	SLV6	2,6 ± 0,1	10,4 ± 0,1	2,02 ± 0,01	12,97 ± 0,09	2,04 ± 0,01	0,92 ± 0,04
	SLV8	2,7 ± 0,1	11,3 ± 0,1	1,98 ± 0,01	14,27 ± 0,09	2,10 ± 0,01	0,90 ± 0,03
Area B	SLV7	2,0 ± 0,1	7,9 ± 0,1	1,77 ± 0,01	10,10 ± 0,13	1,74 ± 0,01	0,89 ± 0,03
	SLV10	2,7 ± 0,1	11,5 ± 0,1	2,02 ± 0,01	13,94 ± 0,09	2,08 ± 0,01	0,91 ± 0,03
Area C	SLV9	2,3 ± 0,1	10,2 ± 0,1	2,03 ± 0,01	11,69 ± 0,10	1,95 ± 0,01	0,94 ± 0,04
	SLV11	2,1 ± 0,1	8,8 ± 0,1	1,96 ± 0,01	11,58 ± 0,13	2,01 ± 0,01	0,96 ± 0,04
	SLV12	2,4 ± 0,1	9,9 ± 0,1	1,97 ± 0,01	13,38 ± 0,11	2,11 ± 0,01	0,96 ± 0,04
	SLV13	2,2 ± 0,1	9,8 ± 0,1	1,70 ± 0,01	12,71 ± 0,12	1,87 ± 0,01	0,92 ± 0,04
Area D	SLV14	3,0 ± 0,1	13,2 ± 0,1	2,47 ± 0,01	16,93 ± 0,09	2,63 ± 0,01	0,96 ± 0,03
	SLV15	2,0 ± 0,1	9,2 ± 0,1	1,80 ± 0,01	10,93 ± 0,13	1,80 ± 0,01	1,10 ± 0,04
	SLV16	2,2 ± 0,1	9,7 ± 0,1	2,58 ± 0,01	11,61 ± 0,11	2,38 ± 0,01	1,03 ± 0,04

Table IV. Ages and dates calculated for the samples by TL dating (see text for symbols explanation).

Sampling area	Sample	*ED* (Gy)	*W*	*F*	*DR* (mGy/a)	TL Age (*a*)	Date (AD)
Area A	SLV1	2,24 ± 0,10	0,148±0,001	0,201±0,002	5,07 ± 0,14	443 ± 23	1570 ± 20
	SLV2	2,76 ± 0,17	0,169±0,001	0,017±0,002	6,50 ± 0,15	424 ± 29	1590 ± 30
	SLV3	7,87 ± 0,39	0,133±0,001	0,099±0,003	5,78 ± 0,26	1361 ± 91	650 ± 90
	SLV4	7,50 ± 0,41	0,120±0,001	0,379±0,003	5,60 ± 0,22	1338 ± 91	670 ± 90
	SLV5	2,55 ± 0,15	0,189±0,001	0,256±0,002	5,31 ± 0,68	481 ± 68	1530 ± 70
	SLV6	7,12 ± 0,15	0.173±0,001	0.049±0,002	5,43 ± 0,39	1312 ± 99	700 ± 100
	SLV8	6,94 ± 0,34	0,148±0,001	0,401±0,003	5,28 ± 0,15	1315 ± 75	700 ± 80
Area B	SLV7	8,92 ± 0,33	0.169±0,001	0.739±0,003	5,06 ± 0,31	1763 ± 125	250 ± 130
	SLV10	10,50 ± 0,53	0,170±0,001	0,486±0,001	6,06 ± 0,42	1734 ± 148	280 ± 150
Area C	SLV9	2,00 ± 0,10	0,195±0,001	0,570±0,001	4,76 ± 0,24	420 ± 29	1590 ± 30
	SLV11	2,35 ± 0,12	0,165±0,001	0,175±0,002	5,05 ± 0,12	464 ± 26	1550 ± 30
	SLV12	8,17 ± 0,49	0,186±0,001	0,081±0,001	6,03 ± 0,14	1357 ± 87	650 ± 90
	SLV13	2,51 ± 0,17	0.190±0,001	0.355±0,003	5,46 ± 0,26	459 ± 39	1550 ± 40
Area D	SLV14	2,84 ± 0,18	0.232±0,001	0.690±0,002	6,64 ± 0,34	428 ± 35	1580 ± 40
	SLV15	1,89 ± 0,08	0,138±0,001	0,520±0,001	4,21 ± 0,33	449 ± 41	1560 ± 40
	SLV16	2,20 ± 0,12	0,758±0,001	1,515±0,001	5,38 ± 0,24	409 ± 29	1600 ± 30

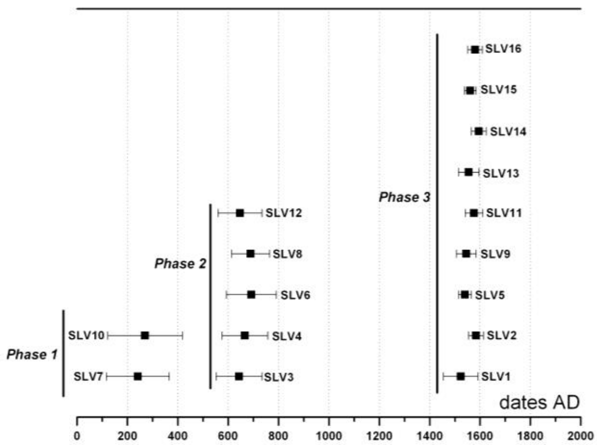


Figure 6. Chronological phases obtained for the construction of Sant'Agata la Vetere crypt.

The obtained ages show that the northern entrance area, the north and the south walls (SLV3, SLV4, SLV6, SLV8 and SLV12 samples) were built during a reconstruction and/or expansion step between the 7th – 8th centuries. This period overlaps the data of the rebuilding of the church by Leone II in the 776 AD.

A more recent phase (17th century) corresponds to a general intervention on the fabric as indicated by the age of the samples SLV1, SLV2 and SLV5 from area A, SLV9, SLV11, SLV13 and SLV14 from area C, SLV15 and SLV16 collected in the area D.

4. CONCLUSIONS

The Sant'Agata la Vetere church is linked to the religious tradition and the workship of Sant'Agata, Catania patron's Saint.

A campaign of archaelogical excavations was conducted in order to study the complex site around the religious building. According to tradition and to historical sources, the site was very interesting because a part of the church, in particular the crypt was built incorporating an apse of the roman *praetorium* palace.

For the reconstruction of the chronology of the fabric, in agreement with archaeologists and architects, some samples are collected in specific areas of the crypt for TL measurements.

The samples collected allow us to perform a methodological study aimed to evaluate any loss of luminescence after alpha irradiation. The results obtained for the k coefficient showed that the evaluation of effective alpha contribution to the dose rate requires a correction. The losses, considering different time intervals from irradiation and reading steps, were in fact evaluated about 15-30%.

Considering the data related to the constant k values after 30 days, several construction phases were obtained.

The oldest phase resulted the area B (SLV7 and SLV10 samples) probably built during the construction of the first church dedicated to Saint'*Agata* (380 - 436 AD).

A second phase belongs to 7th and 8th centuries (SLV3, SLV4, SLV6, SLV8 and SLV12 samples) when the church, from historical sources, was rebuilt in the 776 AD.

In addition, the results obtained by samples collected in different points of area A, C and D, suggest that the crypt was interested by a last intervention in the 17th century.

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