



Research article

Dating the historical old city walls of Songkhla Thailand using thermoluminescence technique

Tidarut Vichaidid^a, Sorasak Danworaphong^{b,*}^a Division of Physics, Faculty of Science and Technology, Prince of Songkla University, Pattani, 94000, Thailand^b Division of Physics, School of Science, Walailak University, Nakhon Si Thammarat, 80160, Thailand

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ABSTRACT

The Old Historical Wall, located in Bo Yang, Songkhla Province, Thailand, is an archaeological icon believed to have been constructed during the reign of Rama III, as indicated in the royal archives, around 1837–1840 CE. However, the recorded age is the result of unofficial documentation. The establishment was based on speculation from circumstantial evidence and local stories. The wall is made of bricks that underwent a heating process before being used for the construction. We therefore propose the use of the thermoluminescence technique for dating the wall. The samples include brick rubble from three excavation sites next to the wall. To determine the age, we estimate the radiation dose rate and the accumulated dose for each sample. The dose rate of the sample is determined using gamma spectroscopy with a high-purity germanium detector. For the accumulated dose, we employ the additive dose method using Co-60 with a dose range of 0–100 Gy. Glow curves are then deconvoluted using the general-order kinetics model. The results yield three superposition glow peaks at three different temperature ranges. Plateau tests are also carried out to find a proper temperature for dating purposes. Comparing the plateau test and the result of deconvolution, we find that the glow curve temperature suitable for dating was within 200–310 °C. The accumulated doses are then evaluated using the area under the curve of the peak temperature. The date is then determined as the ratio between the accumulated dose and the dose rate. The results indicate that the age is approximately 174–192 years, so that the wall was built around 1827–1841 CE, with one standard deviation interval. The duration is in agreement with the recorded age of the wall inscribed in the country's historical archives.

1. Introduction

The Old City Wall of Songkhla is located at 7°11'7.2"N and 100°35'24.8"E, 4 m above sea level, in Bo Yang Muang Songkhla, Songkhla province, southern Thailand. It is among a number of archaeological sites where historical evidence was found as a result of urban development and natural disasters through the years. The historical record indicates that it was built due to city relocation, as King Rama III suggested. It was also the king who demanded the construction of the wall. Without concrete written evidence, the construction was thought to have begun in 1836 and completed in 1842. During a long period of negligence, the town wall disintegrated from multiple adverse events. In 1894, the Thai government issued an order to repair the damaged wall. The renovation process lasted until 1905. As the restoration and other urban developments were carried out, archaeological artifacts were consistently located under the wall. In 2011,

a massive storm caused the wall to collapse, and the governmental agency later restored it. This restoration was reported by the official site of the Tourism Authority of Thailand (2018) and the 13th Fine Arts Department (2010) [1]. Although subjected to a capricious environment, parts of the wall still stand in the city, as shown in Fig. 1. It has become the icon of the city and a trendy location for a weekly night market. This work aims to validate the initial belief of the wall age. Such validation was recently performed using the optically-stimulated luminescence (OSL) technique [2]. However, to confirm the age, OSL often works alongside thermoluminescence (TL) [3, 4]. We therefore propose the use of the thermoluminescence technique for verifying the age of the wall. The technique is a predecessor of OSL and was applied to analyze feldspars in the ceramic industry in 1938 [5] and routinely used by Aitken [6, 7, 8] for archaeological dating purposes. It is widely used for dating potteries, sediments, and soil, which have quartz as an inclusion [9, 10, 11]. It was also used to date brick samples [12, 13,

* Corresponding author.

E-mail address: dsorasak@mail.wu.ac.th (S. Danworaphong).<https://doi.org/10.1016/j.heliyon.2021.e06166>

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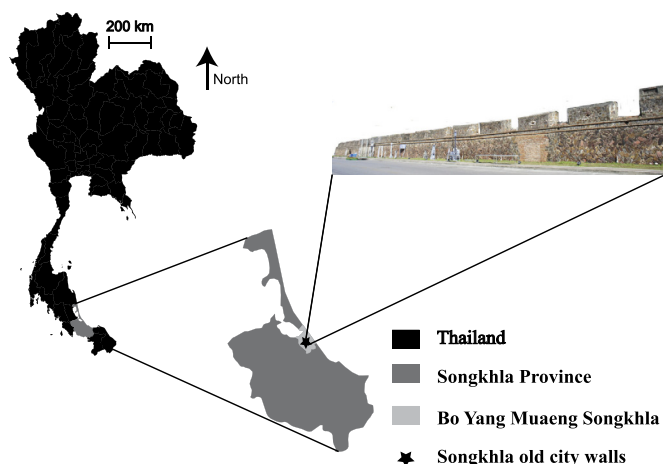


Fig. 1. Location of the excavation site “Songkhla old city walls”, Bo Yang, Mueang, Songkhla province.

Table 1. Sample codes, their descriptions, and areas of excavation sites.

Excavation sites	Sample codes	Sample features	Area (m ²)
TP1	SKTP1	Sediment collected from the center of the remaining wall	3 × 6
TP2	SKTP2	Sediment collected from the area near the Payaknamruangrit town gate of the walls	2 × 4
TP3	SK012, SK013, SK016	Sediment collected from the area near the west side of the second excavation	3 × 3

14]. The technique requires heat as a stimulus for freeing trapped electrons or holes within crystalline defects. The energized electrons then recombine with their counterparts setting off electromagnetic wave radiation [6]. This radiation is the thermoluminescence signal. Quartz behaves like a dosimeter temporally registering radiation doses from its surroundings and is considered a TL material. Therefore, we use quartz as the luminescence material. Its glow peaks are then deconvoluted for the determination of accumulated dose by general-order kinetics [15]. Our results show that the thermoluminescence method provided a comparable age to that of OSL and the historical record, thus consolidating the age of the wall.

2. Materials and methods

2.1. Sample collection

Samples consisted of the sediment containing the brick debris collected from the excavation site of Songkhla Old City Wall by the 13th Regional Office of Fine Arts Department Songkhla. The rubble was acquired from the unprotected area of the site. The historic wall extended about 1,200 m from east to west and 1,000 m from north to south. Its average thickness was 2 m. Initially, there were a total of eight fortresses and ten city gates. Today, the remaining wall is located only in the north, as shown in Fig. 2(a), starting from the edge of the Payaknamruangrit town gate to Taewas turret, according to the data published on the official site of the Tourism Authority of Thailand (2018). Three excavation sites—TP1, TP2, and TP3—were near the remains of the wall with a digging depth of 1 to 2 meters, as seen in Fig. 2(a) and (b). We collected samples SKTP1 and SKTP2 from TP1 and TP2, respectively, and three samples, SK012, SK013, and SK016, from TP3. Table 1 shows their designations and extraction sites. Each sample weighed roughly two kilograms with an average diameter of 3–5 cm.

2.2. Preparation of samples

Brick rubble was extracted from the collected sediment for further processing. Under red light, the rubble was gently scraped to remove approximately 3 mm off its surface layer. It was washed and cleaned in an ultrasonic bath and later rinsed with distilled water and dried at 40 °C. The dried samples were carefully ground with a mortar, and the grains were sieved to obtain a size of 90–150 μm. At this stage, the samples were divided into two different grain sizes, 1–90 μm, and 90–150 μm, which were significantly smaller than initial brick sizes. We then canned the samples separately in a temperature-controlled room at 25 °C, under red light. Each cup weighed one hundred grams. The canning helped seal the samples away from the surrounding air, allowing them to reach secular equilibrium [6, 16, 17]. All specimens were left in the room for a month. The fine-grain samples (1–90 μm) underwent gamma-ray measurement for determining radionuclide concentrations. In contrast, the coarse-grain ones were further etched with 15% HCl for 40 minutes and then again etched in 48% HF for 40 minutes to remove the sample skin and other contaminants [18, 19]. The samples went through an additional etching process, 15% HCl for 15 minutes to remove fluoride ions. Finally, the samples were cleaned again in distilled water and allowed to dry at 40 °C.

Quartz was then separated based on density using intermediate specific density chemicals with heavy liquids. A heavy liquid with 2.62 g/cm³ density was prepared using tetrabromoethane and dipropylene glycol [20]. The clean dried sample was put into the heavy liquid and centrifuged at 2,000 rpm for 1 hour. Quartz with a mineral density of 2.65 g/cm³ settled at the bottom. The sample with quartz only was cleaned with distilled water and acetone and kept in small light protection bags for determining the accumulated dose.

2.3. Gamma-ray measurement and dose rate calculation

A gamma spectrometer was employed to determine the concentration of U, Th, and K. The measurement system comprised a P-type HPGe (high-purity germanium) detector with a closed-end coaxial geometry mounted in a vacuum-tight cryostat having 48 mm in diameter. The operating voltage was +4.5 kV. The spectrum analytical system connected to a computer-based MCA multi-port II, and the software in use was Gamma Vision-32 version 3.2. Two isotopes (¹³⁷Cs and ⁶⁰Co) were utilized for energy and efficiency calibration. A standard reference soil (IAEA soil6) was used to determine the concentrations of U, Th, and K. The dose rate (*D*) was then determined from U, Th, and K contents within the sample. The dose of cosmic rays (*D_c*) was also included in the dose rate. The geographical characteristics—latitude, longitude, and elevation—are necessary for calculating the cosmic ray dose. The sample depth lessened the influence of the cosmic dose [21, 22]. The dose rate can be estimated from the equation,

$$D = kD_{\alpha} + D_{\beta} + D_{\gamma} + D_{c}. \quad (1)$$

D_α, *D_β*, *D_γ*, and *D_c* are dose rates resulting from the α-, β-, γ-decay of U, Th, K, and cosmic ray, respectively. Each dose rate must be adjusted by the correction factor because of the effect of moisture or water content in the sample, 0.81%w [23, 24, 25]. The alpha dose is negligible due to the coarse grain size of the sample, *k* ≈ 0 [7].

2.4. Accumulated dose calculation

The quartz samples were irradiated with γ-rays from a Co-60 source at the Office of Atoms for Peace in Bangkok, Thailand. The dose ranged from 0 to 100 Gy with a 20-Gy step, with extra 10-Gy irradiation for each sample. Within 36 hours, the samples were divided into five sets. Each set consisted of seven aliquots. The weight of each sample was about 150 – 250 mg, three of which were irradiated with the same dose for averaging purposes. Each irradiated sample underwent measurement with Harshaw 3500L for the TL signal. The heating rate was

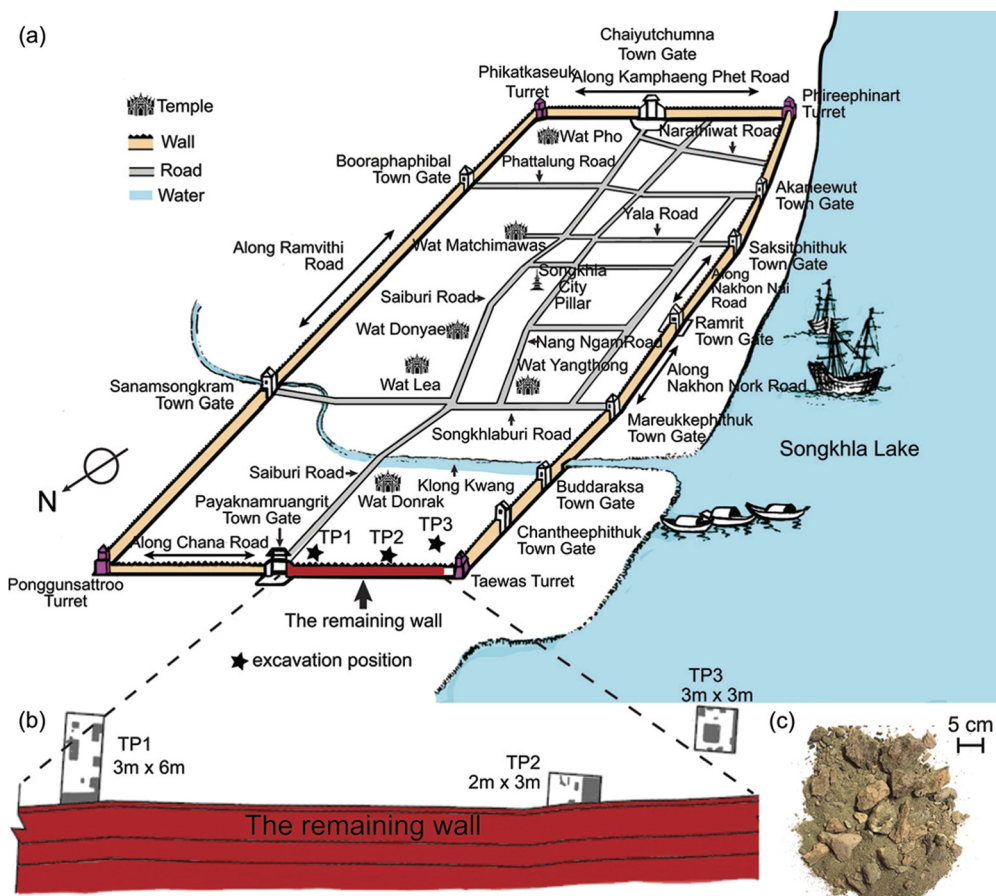


Fig. 2. (a) Schematic representation of the Old City Wall of Songkhla. Enhanced and modified from an old city map. (b) The excavation sites, TP1, TP2, and TP3, in this study and (c) debris sample.

kept at 5 °C/s, and the temperature range was 50–550 °C. The measurement was conducted at room temperature, 25 °C, under a high-purity nitrogen atmosphere. Before entering the detector, the photons were filtered by a neutral density filter with an optical density of 0.5–1.5. The resulting TL signals in terms of temperature, glow curves, for each dose can be seen in Fig. 3(A1)–(C5) where A, B, and C respectively indicate the excavation sites TP1, TP2, and TP3. Using the general-order kinetics to deconvolute glow curves discloses the peak composition of the glow curves [15]. Three superposition peaks are found at different centered temperatures. The plateau test was also conducted to find the appropriate temperature for dating and to verify the deconvolution results from the kinetics model. This confirmed the validity of our measurements. The areas under the curves from the chosen temperature are plotted with the artificial doses, and their x -intercepts yield the accumulated dose, AD .

3. Results and discussion

The concentrations of Th, U, and K, obtained for the five sample sites, are adjusted using a standard reference soil (IAEA6). In TP1 and TP2, the abundance of Th is 8.20 and 10.38 ppm, respectively. The values are slightly off the average, 10 ppm, of that of the Earth's crust but within the typical range, 6–20 ppm. However, in the excavation site TP3, all three samples contain twice the concentration of Th, 19–24 ppm, a factor of two exceeding the global average for the Earth's crust. The reason may be due to the location of the excavation site. It is closer to the beach than the other two, and sand is a good source of Th [26]. Moreover, the bricks were made of fired clay, which is also a good source of Th [27]. The abundance of U is, on the contrary, precisely equivalent to that of the average composition of the Earth's crust. The

typical range of U is 1.5–6.0 ppm, within which our uranium results reside [28]. Lastly, the determined K concentrations, 2.28–4.46%, are higher than the global mean for the crust of 1.18% [28]. This excess potassium is rather arbitrary. It may be inherently related to the raw material for producing the bricks. High activity of K-40 has also been reported previously, as has low activity [29, 30].

The concentration of these three radionuclides is employed to calculate the dose rate (D) imposed on the samples using Eq. (1). The calculation must also take into account the dose of cosmic radiation, which is location-dependent. At the excavation site, the cosmic dose, D_c , is 0.193 ± 0.01 mGy/y [22]. Since the wall is 2 m thick, it is possible to assume that the radioactive elements in the wall are uniformly distributed, and they equally emit and absorb their neighbors' radiative energy. This assumption is called the infinite method [23], and we use this condition to estimate the dose rate. They are found to be ranging from 2.55–4.06 mGy/y with 5–10% deviation, as displayed in Table 2. For age determination, we need to find the value of the accumulated dose (AD) of each sample. The additive dose technique helps to facilitate this task. In Fig. 3 (A1)–(C5), the TL signals from all irradiated samples show peak maximums at around 190 °C regardless of the excavation site. However, when performing the plateau test, samples from each excavation site behave differently. Regardless of artificial doses, as displayed in Fig. 3(A)–(C), samples from TP1 show the plateaus around 200–250 °C, whereas those of TP2 depict the behavior in 260–290 °C. Lastly, samples from TP3 display plateaus in the temperature region of 270–320 °C. Therefore the temperature used for dating purposes depends on the location. These outcomes turn out to agree with the deconvolution using the general order kinetics model. The glow curve data are fitted to the model using nonlinear regression with the Levenberg-Marquardt algorithm. The fitting results are the solid lines embedded

Table 2. The estimates of dose rate, accumulated dose, and age from TL analysis.

Sample Code	Concentration			D (mGy/y)	AD (Gy)	Age (y)
	Bulk Th (ppm)	Bulk U (ppm)	Bulk K(%)			
SKTP1	8.20 ± 0.31	3.99 ± 0.07	2.28 ± 0.03	2.55 ± 0.14	0.48 ± 0.05	187 ± 22
SKTP2	10.38 ± 0.33	2.32 ± 0.05	4.36 ± 0.09	3.78 ± 0.21	0.66 ± 0.10	174 ± 28
SK012	19.24 ± 1.44	2.07 ± 0.03	4.46 ± 0.03	4.06 ± 0.42	0.75 ± 0.08	184 ± 27
SK013	24.74 ± 2.06	3.48 ± 0.05	3.91 ± 0.10	4.04 ± 0.47	0.75 ± 0.08	185 ± 29
SK016	19.89 ± 1.47	2.14 ± 0.04	3.75 ± 0.04	3.60 ± 0.37	0.69 ± 0.08	192 ± 28

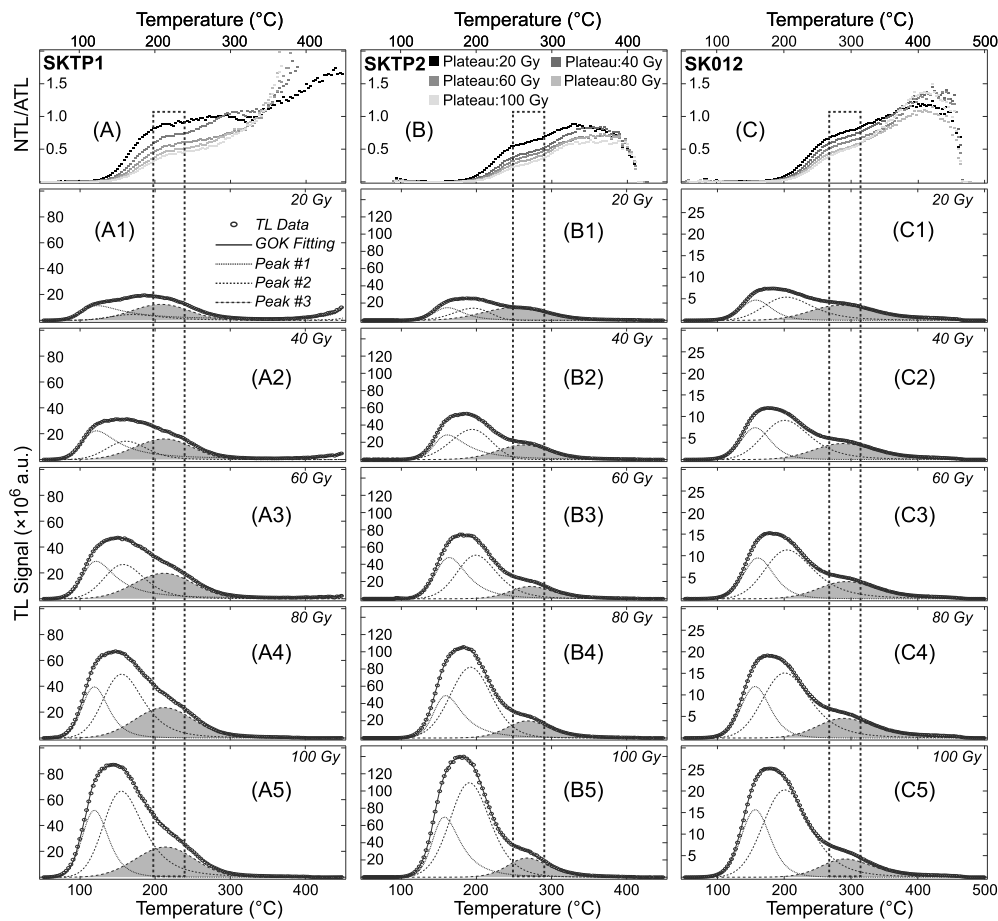


Fig. 3. (A)–(C) show the plateau, Natural TL/Artificial TL (NTL/ATL), for samples, SKTP1, SKTP2, and SK012, from three sites—TP1, TP2, and TP3. In (A1)–(C5), TL signals for different additive doses are shown as oval markers, whereas the solid lines illustrate the fitting result of the general-order kinetics (GOK) model. The fitting results reveal the superposition of three peaks displayed by different dashed lines—Peak #1, #2, and #3. Relating the temperatures where the plateaus reside to the deconvoluted peaks, the crossing dashed rectangles, it is apparent that peak #3’s are suitable for dating. The temperature regions are slightly different from sample to sample but stay within 200°–320 °C. The integration of the area under curves, shaded regions, is used to determine the accumulated dose, y-axis in Fig. 4.

in the oval markers in Fig. 3 (A1)–(C5). The model and the TL data are in agreement. The model also reveals that the glow curve data consist of three superposition curves, and fitting parameters such as the order of kinetics and trapping energies are available online [31]. The first and second peaks are located around 150 °C and 200 °C, respectively. Though the first and second peaks show strong TL signals, the plateau test indicates the instability of the trapped electrons. Thus, these two curves are unsuitable for dating. On the other hand, the last peaks of all samples are located within the plateau regions, 270–320 °C, illustrated in vertical dashed rectangles in Fig. 3. Consequently, we use the area-under-curve (total photon count) of these peaks to determine the AD, the shaded areas in Fig. 3 (A1)–(C5). The total count of all samples is then plotted against the irradiated doses. The x-intercept represents the AD, the value in the box beneath the x-axis in Fig. 4(a)–(e). The absolute values are written in Table 2. Two of the TP3 samples, SK012 and SK013, yield an identical value of AD, whereas SK016 is slightly different. However, if we consider the deviation, all three values are

alike. In fact, only that of TP1 shows a different AD since the AD value of the sample from TP2 is 0.66 ± 0.10. Finally, we calculate the age of all five samples using the simple division of the accumulated dose and the dose rate, AD/D. The resulting ages are shown in Table 2. The estimates are in the range of 174 ± 28 years to 192 ± 28 years. The results are also in agreement with the online dose rate and age calculator [32] for quartz using the conversion factors and beta-grain size attenuation from Guerin et al. [33, 34]. The beta-etch attenuation factor is derived from Bell [35]. Input data for DRAC and its results are available online at [31]. Besides, since the original location of the brick rubble was unknown, the contribution of the surroundings, either from air or soil, may cause variations in age prediction. We explore this effect by adding extra doses, about 5% of the internal dose (α, β, and γ), to the calculated dose rate. The predicted age is changed by approximately 3% from its original predicted value. To visually compare the results with those recorded in the national archive, we display the ages with their deviation superimposed on the recorded construction period,

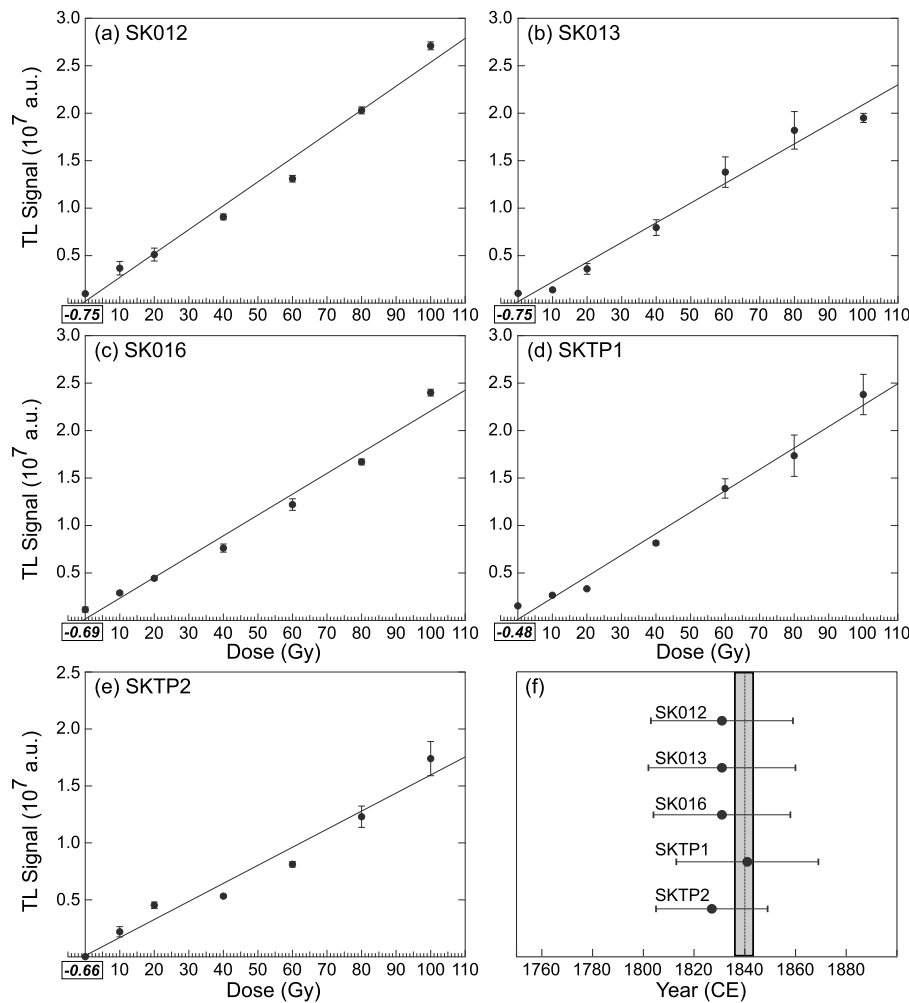


Fig. 4. (a)–(c) display the TL signals as a function of a given dose incident on the samples from the excavation site TP3, whereas (d) and (e) are those from TP1 and TP2. The x -intercepts of the linear fitting lines represent the accumulated doses (AD), written in the dedicated box under the x -axis. Dividing the resulting AD with the dose rate gives the age of the brick sample. (f) illustrates the determined ages of the samples in terms of the current epoch (CE). The shaded bar shows the period when the construction of the wall began and finished.

1836–1842 (the shaded bar). It can be seen that our results are in agreement with the age reported by the government agency and are also in accordance with those resulting from the optically-stimulated luminescence technique [2]. All predicted ages are older than the written years of the construction period. We speculate that the bricks were possibly prepared for years before the construction. Since the samples are debris from bricks, basically baked clay, the TL signal was reset at the time of their manufacturing process. Bleaching or zeroing is, therefore, not a concern for this type of sample. As a result, TL is effectively sufficient.

4. Conclusion

The debris from fallen bricks of the Old Historical Wall is investigated for its age by the thermoluminescence (TL) dating technique. The dose rate is estimated from three radionuclides, i.e., thorium, uranium, and potassium. The general kinetic order function deconvolutes the glow curves. The deconvolution gives three peaks at three different temperatures, 154–158 °C, 195–199 °C, and 277–288 °C. The plateau test shows thermal stability at about 200 °C. We therefore use the corresponding curve for evaluating the accumulated dose, AD . The resulting age of the debris ranges from 174 ± 28 to 192 ± 28 years old, which agrees with the wall's recorded history, i.e., 169–175 years old or built roughly between 1827–1841 CE. The TL technique is still well suited for estimating the age of the heated samples, in this case, brick rubble.

Declarations

Author contribution statement

T. Vichaidid: Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper. S. Danworaphong: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

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Data availability statement

Data associated with this study has been deposited on the OSF (maintained and developed by the Center of Open Science) at <https://doi.org/10.17605/OSF.IO/4NTQ9>.

Declaration of interests statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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