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Research Article
Thermoluminescence Dating of Ancient Pottery Sherds from Ban Pong Manao Archaeological Site, Lopburi Province, Thailand

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Abstract

Background and Objective: During the archaeological excavation in early 2000 at Ban Pong Manao archaeological site in Lopburi province, central Thailand, a collection of ancient pottery sherds were found. Dating these objects in absolute means was important to provide information on many archaeological aspects, including a chronology. The aim of our study, therefore, was to estimate the thermoluminescence (TL) age of those pottery sherds. **Materials and Methods:** To determine the TL age, we performed a multiple-aliquot regenerative-dose procedure to estimate the equivalent dose of quartz grains extracted from the samples of pottery sherds. The annual doses were obtained by measuring the radioactivity concentration of ^{238}U, ^{232}Th and ^{40}K in the pottery sherd samples and their surrounding soil. **Results:** The TL dating results of the samples yielded ages of 2.942±176, 3.152±184, 1.921±122, 1.668±104, 3.014±194 and 3.223±165 years before the present, which were in good agreement with the relative and absolute ages estimated from previous studies. **Conclusion:** We conclude that activities were undertaken on the site in the prehistoric period of Thailand and TL dating of pottery sherds from this site is possible and can complement other dating techniques.

Key words: Multiple-aliquot regenerative-dose procedure, thermoluminescence, pottery sherds, prehistoric period, radioactivity, quartz grains

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Competing Interest: The authors have declared that no competing interest exists.

Data Availability: All relevant data are within the paper and its supporting information files.

INTRODUCTION

Archaeologists have recognized the unique value of pottery as a basis for dating due to its abundance and durability. At the early stage of archaeological studies, different relative dating techniques were used to determine the chronological sequence of pottery. However, the disadvantage of the relative dating techniques is that they cannot provide a numerical age or date range in the number of years for pottery[1]. Therefore, the so-called absolute dating techniques are appealing because they produce precise and accurate numerical ages for pottery. Among the absolute dating techniques, Thermoluminescence (TL) dating is recognized as well-suited for pottery mainly because of the observations of Wang[2], he discovered natural TL emitted from the samples of ancient pottery. This has led to the progressive development of TL as a means of absolute dating techniques for pottery as well as large numbers of archaeological artifacts ever since.

TL is the emission of light from insulating or semiconducting materials (i.e., quartz and feldspars) which have been previously exposed to ionizing radiation under conditions of increasing temperature[3]. Unlike other luminescence processes, heat only acts as a stimulant, whereas ionizing radiation plays the critical role of an exciting agent, which produces free electrons and holes in the electronic system of the materials. Therefore, the signal intensity of natural TL is directly related to the energy absorbed in the materials caused by naturally occurring radioactivity from Uranium-238 (^{238}U), Thorium-232 (^{232}Th), Potassium-40 (^{40}K) with a small contribution from cosmic rays. Ancient pottery is made from clay, which typically contains these radioactive elements together with lesser amounts of quartz and other minerals moulded into desire shapes when plastic. Therefore, part of the energy released from these elements is absorbed by the quartz. On the initial firing for the ancient pottery to become a hardened product, sufficient heat causes all of the absorbed energy to drain out and subsequently sets the TL clock in the crystal to zero. As time goes on, the ionizing radiation around the quartz causes the energy to be restored in the crystal. By comparing the emitted light resulting from the heating of the crystal with the concentration of these three abundant radioactive elements from the soil surrounding the pottery, the TL age can be estimated using the following Eq.[4]:

$$\text{TL age} = \frac{\text{Equivalent dose (ED)}}{\text{Annual dose (AD)}}$$

where, TL age is the TL age in annual (a), the equivalent dose is the absorbed dose in the selected minerals in the sample over its exposure to naturally-occurring ionizing radiation with the unit grey (Gy) and the annual dose is the rate at which the ED is absorbed by the selected minerals in the sample with the unit milli grey per annual (mGy a^{-1}).

Ban Pong Manao archaeological site is one of archaeological significance and is located in Lopburi province, Thailand. During the excavation at the site, several archaeological artifacts were uncovered along with pottery sherds of different sizes and types. Dating in absolute means of these pottery sherds have a high potential for providing excellent information on many aspects of the past, including a chronology. However, the lack of their absolute ages has resulted in insufficiently completing the picture of past human activities occurring at the site during certain numerical ages, this issue is the topic of investigation of this study. The study performed TL measurements on quartz grains extracted from samples of pottery sherds collected from the site to determine the absolute age of the samples. The resulting TL ages can contribute to establishing a chronological framework for the site as well as other archaeological sites and this can also help with further archaeological studies.

MATERIALS AND METHODS

Study area and sample: Ban Pong Manao archaeological site is located in Lopburi Province in central Thailand with 14.92 N latitude and 101.25 E longitude. The site lies on a river terrace at an elevation of approximately 200 meters above mean sea level.

This site was excavated systemically in early 2000 to late 2004 with several subsequent excavations by teams from the Department of Archaeology, Silpakorn University, with the assistance and support of the Tourism Authority of Thailand, the Fine Arts Department of Thailand as well as local villagers[5,6]. Now, the TL study has later conducted from October, 10, 2020-2021. The excavations on the site have revealed several funerary deposits richly equipped with well-preserved human and animal remains, stone axes, ornaments made from stone, glass and bronze, iron tools and weapons and so on, indicating that the site was an ancient burial ground with preliminary results indicating the age of activity from the neolithic (prehistoric) to the dvaravati (historic) period of Southeast Asia[7]. In addition to the great numbers of these archaeological remains, a considerable collection of sherds of earthenware pottery vessels were also uncovered. These objects are of different types and vary in size, however, there are no data about their absolute age.

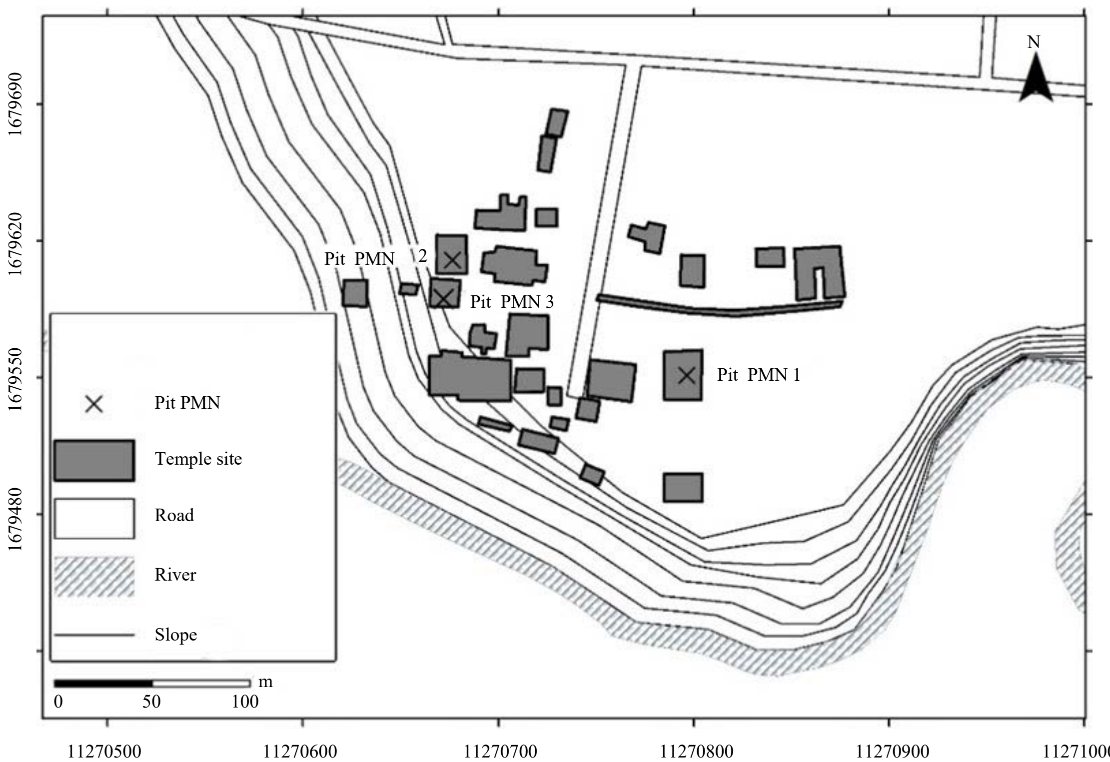

Fig. 1: Plan for the site
Source: Ciarla[5]

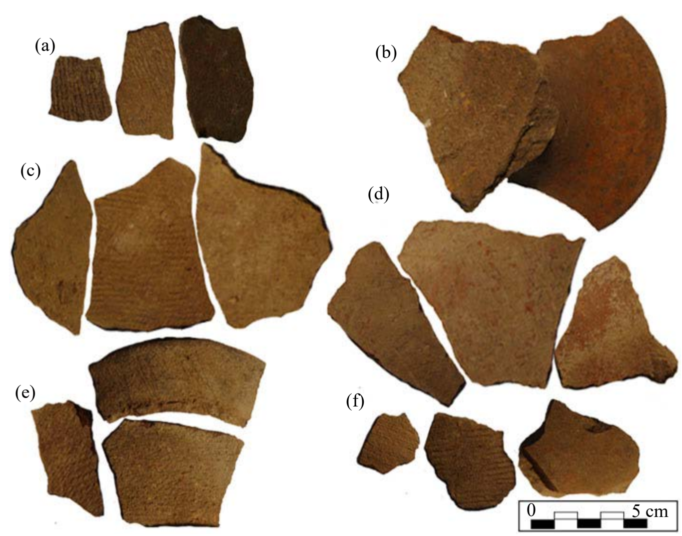

Fig. 2(a-f): Photograph of samples, (a) PMN_1-35 ED, (b) PMN_1-65 ED, (c) PMN_2-50 ED, (d) PMN_2-95 ED, (e) PMN_3-63 ED and (f) PMN_3-70 ED

For this study, several pottery sherds as well as their surrounding soil, were collected from pit PMN_1, PMN_2 and PMN_3 (Fig. 1) and were placed in opaque bags to prevent TL signal loss. At pit PMN_1, two groups of samples were taken for ED and AD measurements at 35 cm (PMN_1-35 ED, PMN_1-35 AD) and 65 cm (PMN_1-65 ED, PMN1-65 AD) below the present surface (Fig. 2a-b). At pit PMN_2, samples PMN_2-50 ED and PMN_2-50 AD and samples PMN_2-95 ED and PMN_2-95 AD, were taken from 50 cm and 95 cm below the present surface, respectively (Fig. 2c-d). At pit PMN_3, two groups of samples were taken at 63 cm (PMN_3-63 ED, PMN_3-63 AD) and 70 cm (PMN_3-70 ED, PMN_3-70 AD) below the present surface (Fig. 2e-f).

Experimental methods

Determination of annual dose: In this study, internal doses due to the contribution of alpha and beta particles were determined from the concentrations of ^{238}U, ^{232}Th and ^{40}K present in the pottery sherd samples, whereas the contribution of gamma radiation taken from those three radioactive nuclides of the surrounding soil samples and cosmic radiation were evaluated for external doses. The measurements were performed for 290 g of bulk samples, which were kept at room temperature for 1 month to allow secular equilibrium between ^{226}Ra and its progenies to be achieved by using gamma-ray spectrometry with a 76×76 mm NaI scintillator crystal detector. Uncertainties of the measurements were estimated to be 5% according to the previous study[8]. The contribution from the cosmic rays was set to be 0.1 mGy a^{-1}, based on the calculation in Takashima *et al.*[8]. Using the dose-rate conversion factor proposed by Tsakalos *et al.*[9] the contribution of gamma rays and alpha and beta particles were evaluated, for this calculation, the quartz grain size was set to 0.15 mm for the beta ray correction based on petrographic observations.

Determination of equivalent dose: The outer 2-3 mm surface of each sample was removed, pulverized and sieved to obtain fractions in the size range of 63-250 µm. Next, magnetic minerals were removed from the fractions using a hand-magnet and a Frantz isodynamic magnetic separator. The fractions were washed with 35% hydrochloric acid (HCl) for 1 hr to eliminate carbonate minerals and then etched with 24% Hydrofluoric acid (HF) for 1 hr to remove the feldspar grains and alpha-irradiated region of quartz grains. Finally, the fractions were washed with 35% HCl for 1 hr to eliminate fluorides that may have formed during the previous step.

To investigate factors that might be a primary source of significant uncertainties in the estimation of the age, mineralogical characterization of the fractions in terms of their mineral phase and elemental composition was also analyzed. Identification of mineral phase in the fractions was carried out by element mapping using Scanning Electron Microscopy combined with Energy-dispersive element analysis (SEM-EDS), which could be used as an alternative to X-ray Diffractometry (XRD) if tiny amounts of some phases and mineral samples being present[10]. The operating conditions were 40 kV and 12.5 mA current with 60 sec acquisition time. Then, quantitative point analysis for Li, Na, Mg, Al, P, K, Ti, Mn, Fe and Ge in quartz grains of each sample was analyzed by Agilent 7500 Series Laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS). The grains were ablated by using a laser wavelength of 240 nm and a pulse width of 265 fs with a spot beam diameter of 60-70 µm, a laser energy density of 6 J cm^{-3} and a frequency of 500 Hz. Helium gas was used as a carrier gas at 1,500 mL min^{-1} flow. The analyzed data were calibrated using BHVO-2G, BIR-1G and NIST SRM 610 reference glasses.

The purified quartz fractions were divided into two main portions, one for obtaining the Natural TL (NTL) signal and the other for obtaining the Artificial TL (ATL) signals. The latter portion was heated at 320°C for 5 hrs and precisely irradiated by a ^{60}Co gamma irradiator at the Office of Atoms for Peace, Thailand to obtain doses of 3.5, 10, 18.6 and 39.3 Gy. The irradiated fractions were subsequently annealed at 130°C for 24 hrs to eliminate unstable TL signals generated during the gamma irradiation. The TL signals were measured using Shimaden Temperature Controller series FP 21, Hamamatsu Counting Unit C8855 and a high sensitivity photomultiplier tube with a spectral response from 400-700 nm, using a linear heating rate of 1°C sec^{-1} from 100-400°C in a nitrogen gas atmosphere. Three aliquots of each dose were used to measure the NTL and ATL signals.

RESULTS AND DISCUSSION

Annual dose: The data in Table 1 and 2 represent the radioactivity, the internal and external doses and annual doses of the pottery sherd samples and their surrounding soil. Although the samples yielded extremely low *in situ* water content, ranging from 0.16-5.05%, the influence of water content on the annual doses was not used for the calculation since it may have fluctuated seasonally over the entire burial period of the samples as described in Chuenpee *et al.*[11].

At pit 1, PMN_1-35 ED and PMN_1-35 AD yielded the annual dose of 1.33±0.07 mGy a^{-1}, whereas that of 1.69±0.08 mGy a^{-1} was obtained from PMN_1-65 ED and PMN_1-65 AD. PMN2-50 ED and PMN_2-50 AD and PMN_2-95 ED and PMN_2-95 AD collected from pit 2 yielded the annual doses of 2.22±0.11 and 1.82±0.09 mGy a^{-1}, respectively. At pit 3, the annual doses of 1.62±0.08 and 1.83±0.09 mGy a^{-1} have been observed in PMN_3-63 ED and PMN_3-63 AD and PMN_3-70 ED and PMN_3-70 AD, respectively.

Equivalent dose and TL age: A multiple-aliquot regenerative-dose procedure was performed for estimating the ED, which was determined through glow and growth curves using NTL and ATL glow peaks. The typical glow curve patterns of NTL and ATL glow peaks for all of the samples were characterized by an intense glow peak centred at 290°C with

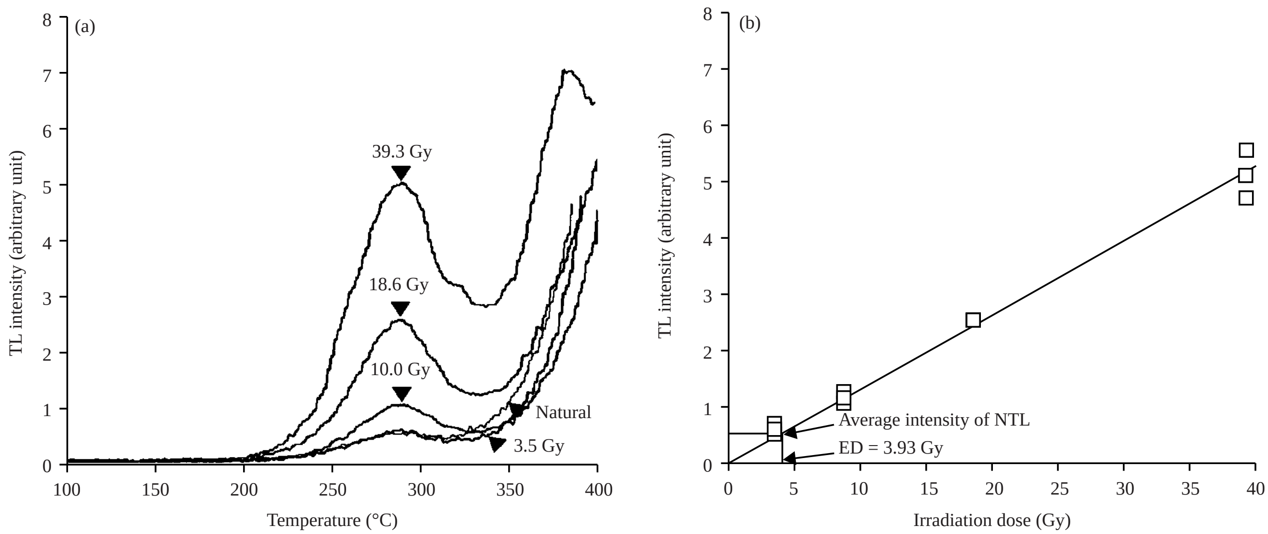

Fig. 3(a-b): (a) Typical glow curve and (b) Growth curve of quartz extracted from the pottery sherd samples

Table 1: Internal and external doses with concentrations of the three radioactive elements and water content of pottery sherd samples and their surrounding soil

Sample type	Sample name	^{238}U (ppm)	^{232}Th (ppm)	K_2O (%)	Water content (%)	Doses (mGy a^{-1})
Pottery sherds	PMN_1-35 ED	6.86±0.34	11.36±0.57	2.78±0.14	2.48	0.63±0.03
	PMN_1-65 ED	6.63±0.33	10.73±0.54	2.52±0.13	1.31	0.58±0.03
	PMN_2-50 ED	4.70±0.23	12.06±0.60	3.51±0.18	1.02	0.68±0.03
	PMN_2-95 ED	5.75±0.29	9.761±0.49	2.32±0.12	3.08	0.53±0.03
	PMN_3-63 ED	5.75±0.29	17.62±0.88	2.18±0.11	0.16	0.77±0.04
	PMN_3-70 ED	6.37±4.31	19.27±17.24	1.98±2.03	4.68	0.77±0.04
Soil	PMN_1-35 AD	1.60±0.08	5.36±0.27	0.71±0.04	2.60	0.71±0.04
	PMN_1-65 AD	2.33±0.12	7.64±0.38	1.67±0.08	1.47	1.11±0.06
	PMN_2-50 AD	3.26±0.16	9.15±0.46	2.92±0.15	1.24	1.55±0.08
	PMN_2-95 AD	2.45±0.12	8.07±0.40	2.39±0.12	3.13	1.29±0.06
	PMN_3-63 AD	2.12±6.42	6.24±0.31	0.89±0.04	0.22	0.85±0.04
	PMN_3-70 AD	2.16±6.42	6.42±0.32	1.82±0.09	5.05	1.06±0.05

^{238}U, ^{232}Th, K_2O, ppm, %, mGy a^{-1} stand for uranium-238, thorium-232, dipotassium oxide (potassium-40), weight in part per million, weight (%) and milligray per annual, respectively

Table 2: Annual doses, equivalent doses and the TL ages were calculated

Sample name	AD (mGy a^{-1})	ED (Gy)	TL age (a)
PMN_1-35 ED	1.33±0.07	3.93±0.07	2.942±176
PMN_1-65 ED	1.69±0.08	5.32±0.09	3.152±184
PMN_2-50 ED	2.22±0.11	4.27±0.08	1.921±122
PMN_2-95 ED	1.82±0.09	3.03±0.06	1.668±104
PMN_3-63 ED	1.62±0.08	4.89±0.10	3.014±194
PMN_3-70 ED	1.83±0.09	5.88±0.06	3.223±165

A stands for annual

subordinate shoulder peaks located at approximately 245 and 320°C. Therefore, the 290°C glow peaks of NTL and ATL signals of the samples were used to construct the growth curve based on four regenerative gamma doses and hence to calculate the ED. Figure 3(a-b) provides typical glow and growth curves of all the samples and Table 2 shows the TL dating results for the six samples.

The ED values of PMN_1-35 ED and PMN_1-65 ED from pit PMN_1 were 3.93±0.07 and 5.32±0.09 Gy, yielding TL ages of 2.942±176 and 3.152±184 years before the present, respectively. At pit 2, PMN_2-50 ED with an ED value of 4.27±0.08 Gy and PMN_2-95 ED with an ED value of 3.03±0.06 Gy showed younger TL ages of 1.921±122 and 1.668±104 years before the present, respectively. PMN_3-63 ED and PMN_3-70 ED from pit 3 showed ED values of 4.89±0.10 and 5.88±0.06 Gy, resulting in TL ages of 3.014±194 and 3.223±165 years before the present, respectively, these TL ages resulted in ranges similar to those from pit 1. It is worth noting that the TL age results for the samples from pit 2 were in reverse stratigraphic ordering that might be caused by the presence of some mineral phases or some quenching impurities. However as shown in Fig. 4(a-l), the phase of

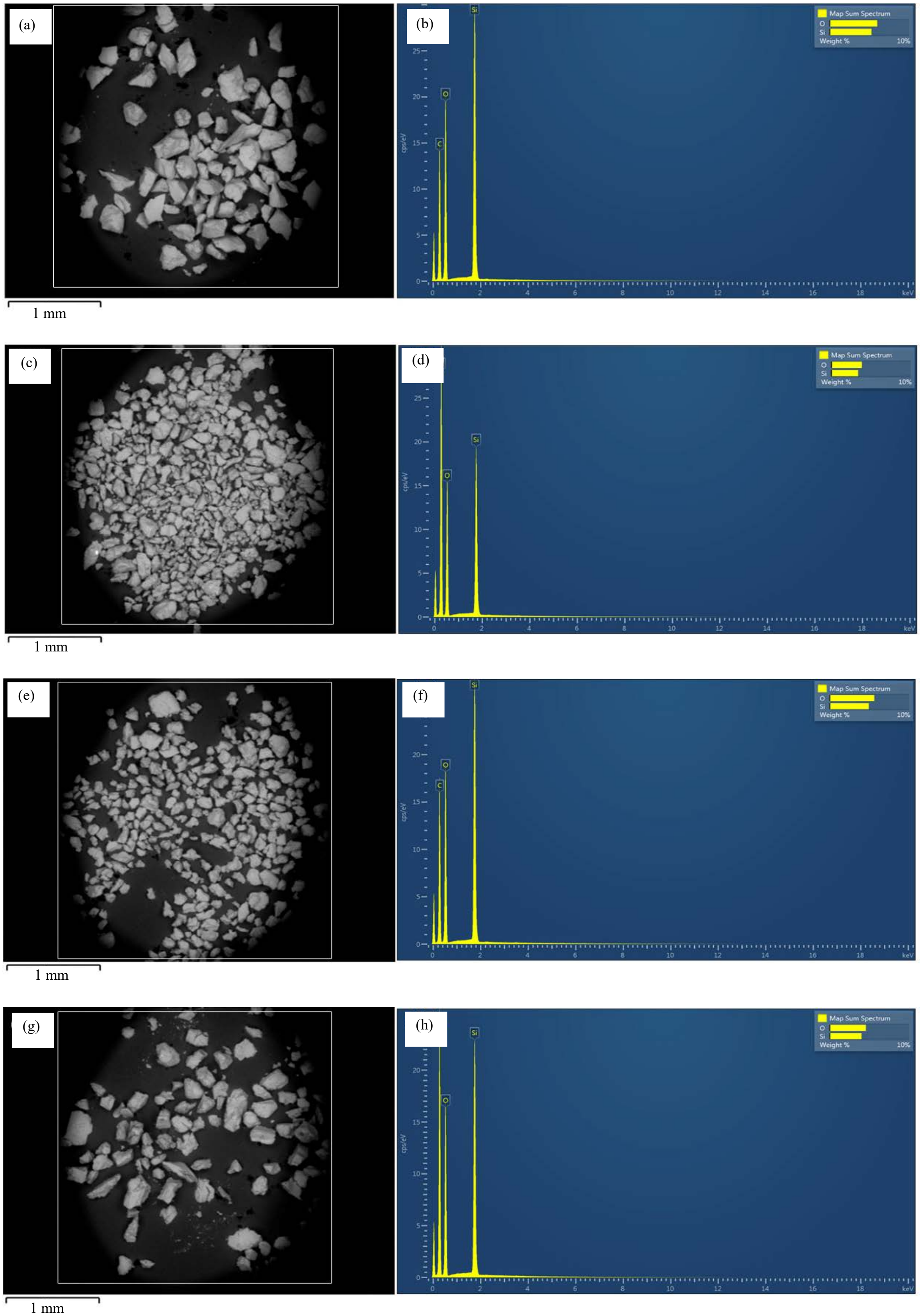

Fig. 4(a-l): Continue

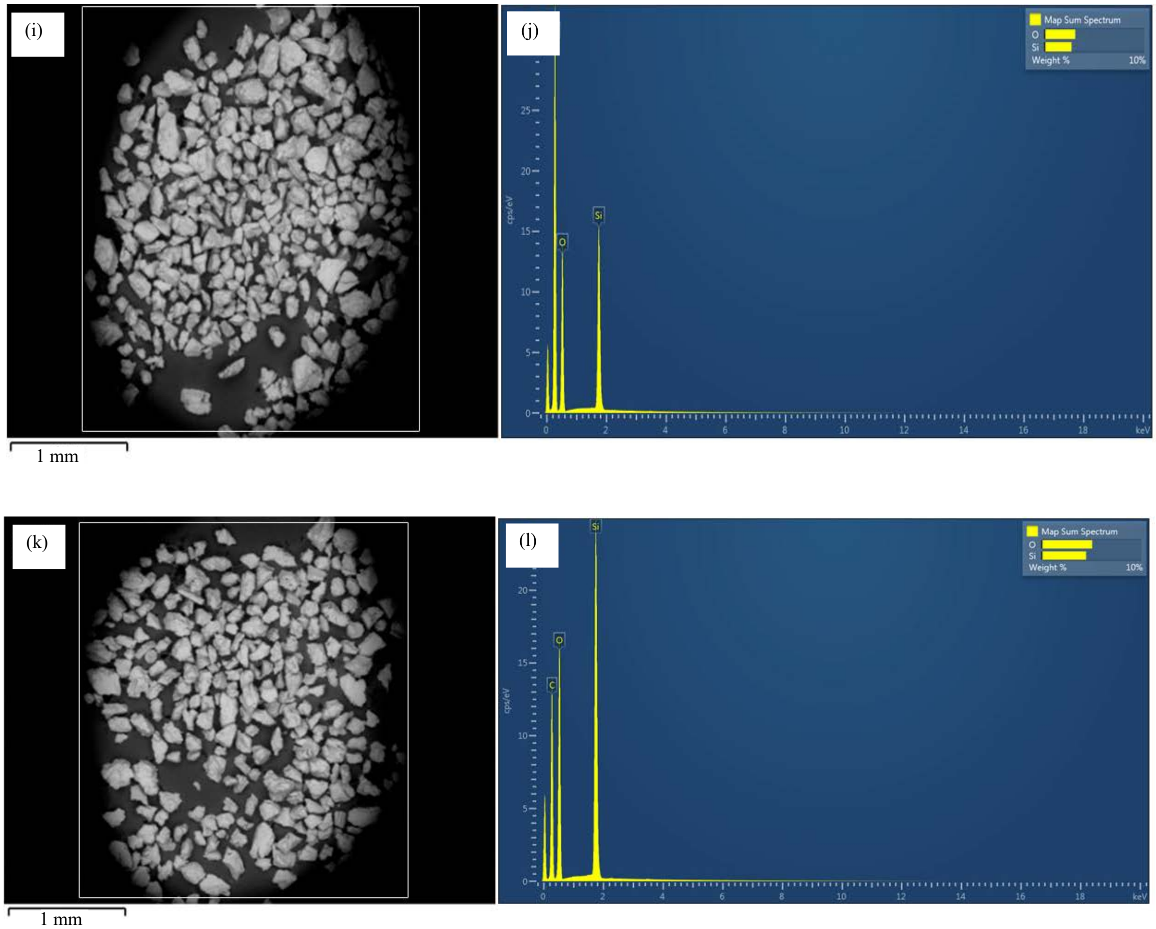

Fig. 4(a-l): SEM images and EDS results of quartz grains extracted from the samples of ancient pottery sherds, (a, b) PMN$_1$-35 ED, (c, d) PMN$_1$-65 ED, (e, f) PMN$_2$-50 ED, (g, h) PMN2-95 ED, (i, j) PMN$_3$-63 ED and (k, l) PMN$_3$-70 ED

Table 3: Average trace element compositions of quartz grains were extracted from the samples of ancient pottery sherds

	Content (ppm)									
Sample name	Li	Na	Mg	Al	P	K	Ti	Mn	Fe	Ge
PMN$_1$-35 ED	3	8	1	20	29	<0.000	8	<0.000	<0.000	<0.000
PMN$_1$-65 ED	5	6	3	28	31	<0.000	7	<0.000	<0.000	<0.000
PMN$_2$-50 ED	4	9	3	26	28	<0.000	8	<0.000	<0.000	<0.000
PMN$_2$-95 ED	3	6	4	23	25	<0.000	8	<0.000	<0.000	<0.000
PMN$_3$-63 ED	3	5	1	23	28	<0.000	8	<0.000	<0.000	<0.000
PMN$_3$-70 ED	3	1	1	23	28	<0.000	8	<0.000	<0.000	<0.000

minerals identified in the fractions of each sample was quartz, due to the significant amount of Silicon (Si) and Oxygen (O). This indicates that the fractions can be separated properly after employing usual chemical treatments and cannot be contaminated with other luminescent minerals, such as feldspar and zircon. Trace element concentrations in the six quartz samples are quite similar and have a low level or are under the detection limit, except for Al and P concentrations as listed in Table 3. In addition, Fe impurity, which is a common luminescence quencher in quartz crystal and plays a vital role in luminescent properties[12,13] is below the detection limit in all quartz samples. Therefore, it is reasonable to consider that the reverse stratigraphic ordering in the TL age results has a close association with burial activities at different times and different depths, PMN$_2$-95 ED taken at 95 cm. below the present surface was buried later in a deeper pit compared with that at a shallower depth.

The TL ages of the six pottery sherd samples from Ban Pong Manao archaeological site yielded ages ranging from 3.223±165 to 1.668±104 years before the present, which coincides with the prehistoric period of Thailand. Details concerning the models for the timing of archaeological ages in Southeast Asia are presented in Higham *et al.*[14]. The dating results of the samples are in good agreement with previous studies, both in relative and absolute means. According to the relative means, the TL ages obtained coincided with ages of approximately 2.340-2.310 and 2.500-1.000 years before the present, topologically estimated from a comparison of the archaeological finds found on the site with those with known ages found at the other archaeological sites in Thailand[15]. Through absolute dating, the resulting ages were seen to well correspond with the conventional radiocarbon date of 2,900-2,800 years before the present calculated from charcoal aside from the human remains found at the site[16]. Thus, our study suggests that activities on the site were conducted in the prehistoric period of Thailand.

CONCLUSION

TL dating was performed on quartz grains extracted from ancient pottery sherds at the Ban Pong Manao archaeological site in Lopburi province in Thailand. The dose rate is estimated from three naturally occurring radionuclides, i.e., ^{238}U, ^{232}Th and ^{40}K. The typical glow curve patterns for all of the samples were characterized by an intense glow peak centred at 290°C. We, therefore, use this intense glow peak for evaluating the ED. The six samples of pottery sherds yielded TL ages of 2.942±176, 3.152±184, 1.921±122, 1.668±104, 3.014±194, 3.223±165 years before the present, which is in good agreement with the relative and absolute ages estimated from previous studies. Therefore, the TL ages obtained suggest that activities were undertaken on the site in prehistoric Thailand. In addition, our resulting TL ages can contribute to establishing a chronological framework for the site as well as other archaeological sites.

SIGNIFICANCE STATEMENT

Ban Pong Manao archaeological site is one of archaeological significance in Thailand. During the excavation at the site, several archaeological artifacts were uncovered along with pottery sherds. Dating these objects in absolute means was important to provide information on many archaeological aspects, including a chronology, this issue is the topic of investigation of this study. This study, therefore, performed TL measurements on quartz grains extracted from samples of pottery sherds collected from the site to determine the absolute age of the samples. The resulting TL ages can potentially contribute to establishing a chronological framework for the site as well as other archaeological sites and this can also help with further archaeological studies.

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