

Electrom Spin Resonance Dating of Laterite from the Muang Sing and Kamphaeng Phet Historical Parks

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ABSTRACT

Muang Sing and Kamphaeng Phet historical parks are the only two historical parks in Thailand constructed with laterite. Laterite samples from both of these parks were analyzed using electron spin resonance spectroscopy to determine their age. The electron spin resonance age of the laterite from Muang Sing historical park was found to be 5,450 years \pm 580 years. This age falls within the Holocene Epoch. The laterite from Kamphaeng Phet historical park was not datable by electron spin resonance spectroscopy because it was too young.

KEYWORDS: Muang Sing historical park, Kamphaeng Phet historical park, electron spin resonance dating; laterite

INTRODUCTION

Thailand, with its long history of more than 700 years, has been endowed with a unique combination of art and culture that continuously developed from the ancient Sukothai kingdom, Ayuthaya, and Thonburi to the present Rattanakosin period. Ancient monuments and artifacts from the past found in Thailand bear evidence of the continuous prosperity of the Thai nation. Such treasures of Thailand's cultural heritage are highly regarded by Thais, as well as by people from all over the world. Many of these treasures are in the ten historical parks that are managed by the Fine Arts Department of the Ministry of Culture. Of these historical parks, only two of them, Muang Sing and Kamphaeng Phet historical parks, were constructed mainly of laterite.

The electron spin resonance dating technique has been widely used in geology and archaeology as a new dating technique. The pioneering work on electron spin resonance dating was done by Ikeya in which he used electron spin resonance to date a speleothem (Ikeya, 1975). A complete review paper on electron spin resonance dating and electron spin resonance applications in Quaternary science and archaeometry was written by Rink (1997).

The electron spin resonance dating technique is based on the accumulation in laterite, during a period of burial, of a measurable absorbed radiation dose from cosmic rays, uranium, thorium, and potassium and their decay products. The electron spin resonance technique can estimate the accumulated dose to which the sample was exposed up to the time of measurement by administering to

the sample an additive known as radiation exposure (Ikeya, 1975). An electron spin resonance dating experiment involves determination of the accumulated dose, AD, received by the sample under the natural radiation field and determination of the annual dose rate, D, of the radiation field. The electron spin resonance age is obtained by the formula $T = AD/D$, where T equals age (Ikeyda, 1993; Ulusoy, 2002).

HISTORICAL BACKGROUND AND GEOLOGY OF THE STUDY AREA

Muang Sing historical park

Muang Sing historical park is located in Sing sub-district of Sai Yok district in Kanchanaburi Province. It is about 45 kilometers from Kanchanaburi and about 175 kilometers from Bangkok. The altitude of this ancient city Muang Sing is about 340 meters above sea level. Figure 1 shows the location of Muang Sing historical park in Kanchanaburi Province.

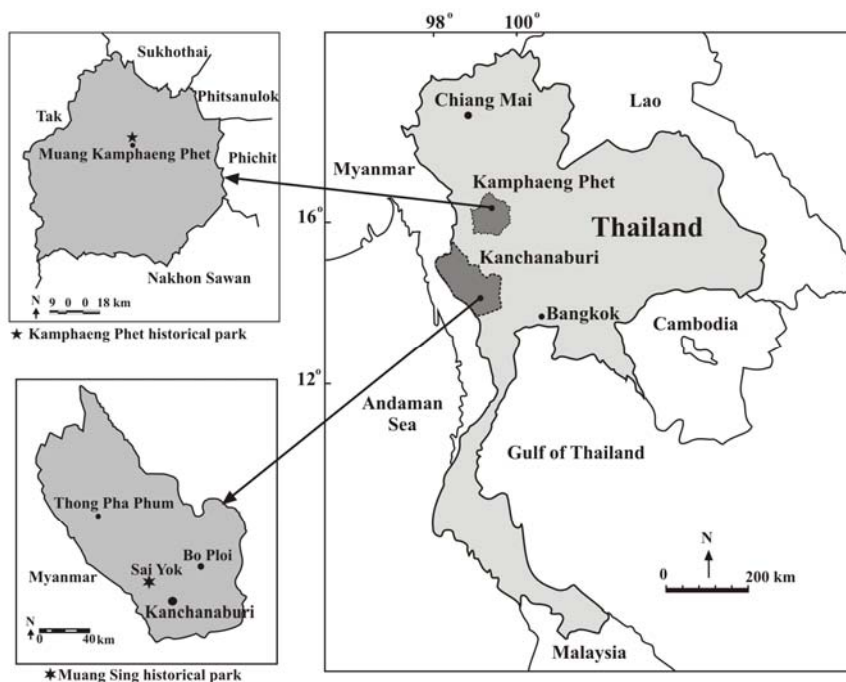


Figure 1. Location of Muang Sing and Kamphaeng Phet historical parks in the map of Kanchanaburi province with respect to locations surrounded by some provincial cities.

The Muang Sing ancient city is surrounded by moats, ramparts, and laterite walls. The laterite wall around the city is about 800 meters by 1,400 meters and has gates on its four sides. The south side of the wall winds along the Kwae Noi River and there is an earth embankment on each of the other inner sides of the wall, of which the outer sides are enclosed with seven moats and ramparts. These moats

and ramparts were probably constructed not only for fortification purposes, but also as a water-control system. There are also six ponds that were possibly used for religious and irrigation purposes.

Muang Sing, or Lion City, was most probably named according to the folk tale about King U-Thong who escaped from King Vechasuwanho and built this city. Folklorists point out that several other place names in this area are the result of newcomers making up a name to explain a locality of which they have no knowledge. However, this name is not mentioned in any official documents prior to the 18th century Rattanakosin period. It was first known in the reign of King Rama I, who founded Muang Sing as a front line city pertaining to Muang Kanchanaburi. King Rama IV later conferred the title of Phra Saming Sing Burin to the ruler of Muang Sing. And since the reformation of provincial administration in the reign of King Rama V, Muang Sing had been reduced to the status of a sub-district. The ancient monuments uncovered in Muang Sing and Prasat Muang Sing can presumably be dated back to the 11th and 12th centuries. The Khmer bayon style of the city layout and the architecture indicate the relationship between Muang Sing and the ancient Khmer Kingdom during the reign of King Jayavarman VII, 1177 to 1237 A.D. This makes the age of Muang Sing historical park about 800 years. The location of the laterite used to construct Muang Sing is about 6 kilometers east of Muang Sing historical park.



Figure 2. The principle monument, monument no. 1. All laterite samples were collected from the area of this monument.

There are four monuments inside the city wall of Muang Sing. The principle monument, monument no.1, is almost in the middle of the city, just a bit diverted to the southeast, and faces the eastern gate. This monument is a building complex comprising wall, gates, gallery, and several buildings. It is built of laterite blocks and was plastered and decorated with stucco reliefs. A main building, the Prasat, is a single tower-like ruin standing at its center (Figure 2). There are four gateways, one on each side of the gallery wall. In the northern gallery, the wall has a low relief carving of a four-handed person believed to be the Bodhisattva Avalokitesavara. In front of the Prasat, there is a small rectangular building on the southeast corner inside the gallery boundary which may be a library. If so, it was built to house Buddhist texts (Pisnupong, 1999).

The laterite samples for electron spin resonance dating were small pieces collected from the floor and wall in the vicinity of monument no.1.

Kamphaeng Phet historical park

Kamphaeng Phet historical park is located in Muang Kamphaeng Phet district in Kamphaeng Phet Province. It is about 360 kilometers north of Bangkok. Figure 1 shows the location of Kamphaeng Phet historical park in Kamphaeng Phet Province.

Ancient Kamphaeng Phet is on the east bank of the Ping River. It was laid out in a trapezoidal plan, parallel to the river and enclosed by a town wall 2,200 meters long on the north, 2,000 meters on the south, 500 meters on the east, and 250 meters on the west. About 475 meters of the southern wall was partially demolished.

The town wall of Kamphaeng Phet was originally composed of earthen ramparts and a moat. A laterite wall was later built and fortified with battlements and parapets, with gates and watchtowers on all four sides. There are still traces of earthen ramparts forming the middle and outer walls on the north. The inner laterite wall probably belongs to the Ayutthaya period. According to the Ayutthaya Chronicle by Van Vliet, Somdet Phra Borommatrai Lokanat, 1448 to 1488 A.D., constructed fortifications to defend important towns, including Kamphaeng Phet. Ten gates allowed access to Kamphaeng Phet. Only nine of the original watchtowers remain.

Two important ancient monuments, Wat Phra Kaeo and Wat Phra That, were in the heart of ancient Kamphaeng Phet. Wat Phra Kaeo, the biggest temple in town, had no monastic precincts. Buildings within the temple were constructed at different periods of time. North of Wat Phra Kaeo is an ancient palace known as Sa Mon. Another significant ancient monument is the Siva shrine, the only Hindu sanctuary in Kamphaeng Phet. Within the town are the remains of about 10 small temples, along with ditches and ponds.

About 500 meters north of the ancient Kamphaeng Phet wall is the Aranyik area, comprising 40 temples clustered on a laterite mound. This group of ancient monuments is regarded as the focal point

of Kamphaeng Phet. All of the ancient monuments were made of laterite. The use of huge blocks of laterite for pillars and bases of buildings indicates the high level of sophistication of construction in the past. Temples in this group include Wat Phra Non, Wat Nak Chet Sien, Wat Phra Si Iriyabot, Wat Khong Chai, Wat Singha, Wat Kamphaeng Ngam, Wat Tao Mo, Wat Chang Rop, and Wat Avasa Yai. Some ancient monuments are scattered to the east and northeast of the town wall. A large number of ancient monuments has been destroyed because of the land being used for agriculture and housing. Remains of important temples include these at Wat Kalothai, Wat Tabaek Lai, Wat Pho Sam Kha, Wat Chang and Wat Dong Wai.

Kamphaeng Phet historical park is on the east bank of the Ping River. It covers an area of 3.4 square kilometers and is divided into two zones. One zone is within the town wall and covers an area of 503 rais. It includes Wat Phra Kaeo, Wat Phra That, the Sa Mon ancient palace, the Siva shrine, the town wall, moat, and fortifications. The other zone is outside the town wall and is known as the Aranyik area. This second zone covers an area of 1,611 rais on a mound. The ancient monuments in this zone consist of 40 large and small temples. Significant temples include Wat Phra Non, Wat Phra Si Iriyabot, Wat Singha, Wat Khong Chai, Wat Nak Chet Siem, Wat Kamphaeng Ngam, Wat Chang Rop, and Wat Avasa Yai.

The group of ancient monuments in the Aranyik area is a unique feature of Kamphaeng Phet. Densely located on a single piece of land on a mound, these ancient monuments were artistically constructed and are examples of indigenous architecture which reflects excellent craftsmanship and the beliefs of the inhabitants of Kamphaeng Phet of that period. Being amidst a well-preserved forest, this group of ancient monuments retains the atmosphere of forest temples of olden days.

Most of the ancient monuments in Kamphaeng Phet are temples dedicated to Theravada Buddhism. They date back to the 14th century in the reign of Phra Maha Dharmaraja Lithai. These Buddhist sanctuaries were built in the Sukhothai style, with influences from Ayutthaya and Lanna. This was a natural result of Kamphaeng Phet's location, connecting Sukhothai and Lanna in the north with Ayutthaya in the south. The perfect combination of these art styles characterizes the typical style of Kamphaeng Phet, known as the Kamphaeng Phet school of craftsmen.

The laterite samples for electron spin resonance dating were small pieces collected from the floor and a wall in the region of Wat Phra Si Iriyabot in the Aranyik area (Figure 3).

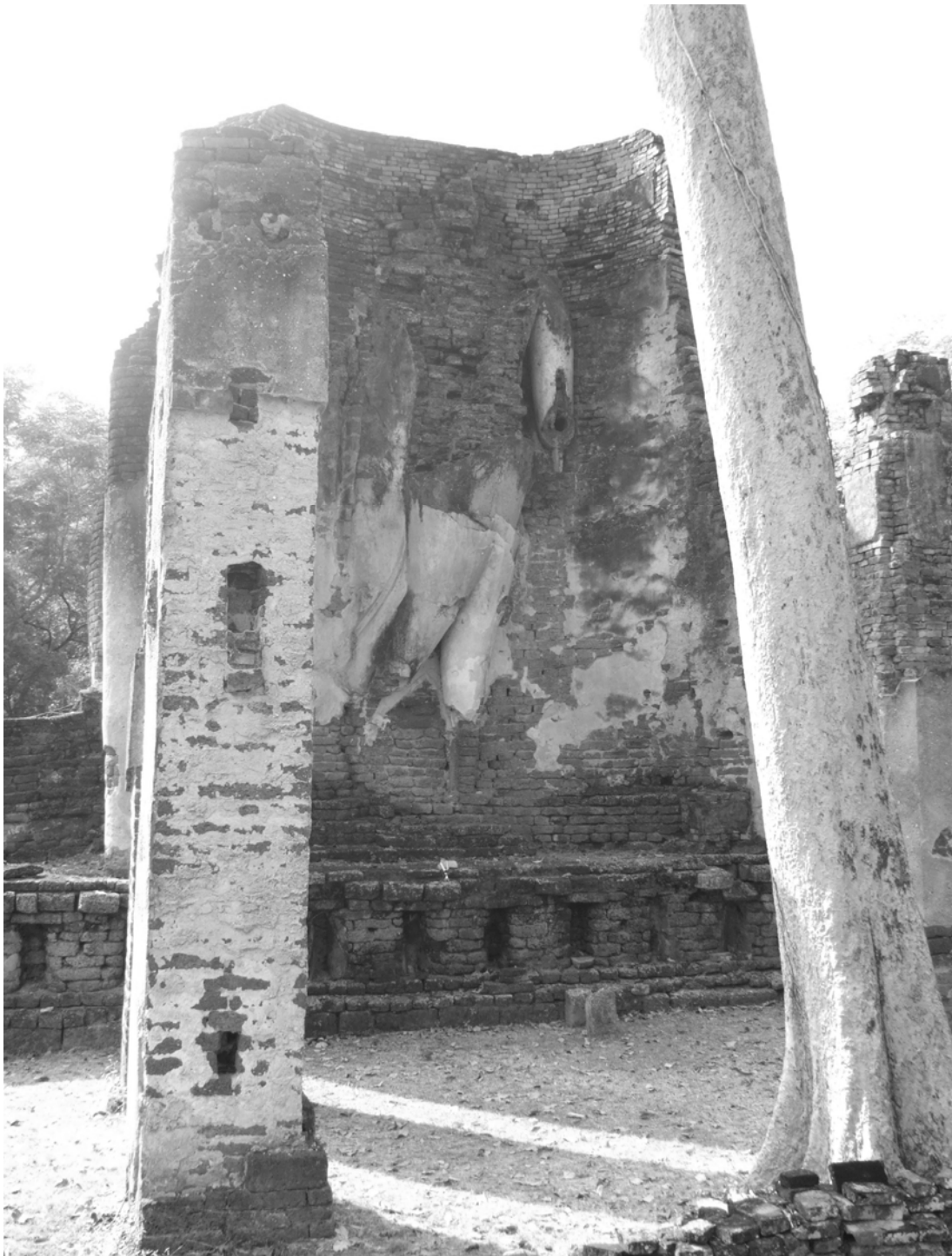


Figure 3. Wat Phra Si Iriyabot .All laterite samples were collected from the area of this wat.

EXPERIMENTAL

Sample preparation

The laterite samples were washed and cleaned by distilled water and annealed to dry at a temperature of 40°C for 2 hours. Following this, the samples were gently ground with a mortar and these grains were sieved in order to separate the size fraction of 75 to 250 micrometers. The separated grains were etched by 10 percent hydrofluoric acid at 40°C for about 1 hour to remove feldspar and quartz grains (Bartoll and Ikeya, 1981; Olley and others, 2004). Finally, the samples were washed again repeatedly in distilled water and annealed to dry at 40°C for 2 hours. All sample preparation procedures were performed in a dim red light to avoid samples being exposed to light. The samples were divided into three sets, each set consisting of 11 aliquots, approximately 500 to 800 milligrams each. All samples were wrapped in aluminum foil to avoid exposure to excessive sunlight. The ^{238}U , ^{232}Th , and ^{40}K content in one laterite aliquot of each set was measured by neutron activation analysis to determine the annual dose rate, D . Another aliquot was un-irradiated and was used for chemical analysis. The remaining nine aliquots were irradiated with different doses for electron spin resonance measurements to determine the accumulated dose, AD .

Chemical analysis

Chemical analysis of laterite samples was carried out by laser ablation inductively coupled plasma mass spectroscopy. In this work, laser ablation inductively coupled plasma mass spectroscopy was performed on a Q-switch Nd:YAG laser operating at 266 nm (Cetac, LSX-500) coupled to a quadrupole mass spectrometer (Perkin-Elmer, Elan 6000). A homogenized laser beam with a flat-top beam profile was imaged onto the sample surface using mirror optics, homogenizer arrays, and a spectrographic microscope. This system allowed focusing the laser beam onto the sample while the operator observed the sample during ablation. The focused laser light caused very rapid heating on the sample surface and volatilized, or ablated, the matrix. This ablated material was carried to the inductively coupled plasma mass spectrometer in an argon gas carrier stream for analysis. Thus, the quantities of major oxides present in laterite samples, such as Fe_2O_3 , SiO_2 , and Al_2O_3 , were determined. For each sample, the laser ablated five positions on the sample surface and the measurements were repeated three times. These measurements were done on three sets of laterite samples. The average results are shown in Table 1.

Table 1 The content of major oxides (in wt %) in laterite samples from Muang Sing and Kamphaeng Phet historical parks.

Laterite Sample	Fe_2O_3	SiO_2	Al_2O_3	Others
Muang Sing	51.32	29.20	16.87	2.61
Kamphaeng Phet	46.43	34.20	17.86	1.51

Accumulated dose determination: Artificial irradiation and electron spin resonance measurements

All except one of the nine aliquots laterite samples were packed in a cylindrical container to under go artificial irradiation. These eight aliquots were irradiated by using a ^{60}Co source (GC-220E) which delivered the gamma rays with 3.404 gray per second at the gamma ray irradiation laboratory of the Office of Atoms for Peace in Bangkok. The artificial doses imparted were 20, 50, 100, 150, 200, 250, 300, and 350 gray. After irradiation, to eliminate unstable paramagnetic centers, the irradiated samples were kept at the recommended 100°C for 2 hours (Engin and others, 2006; Lyons and Brennan, 1991; Prescott and Stephan, 1982). The samples were allowed to cool to room temperature before the electron spin resonance measurements were made. Following this cooling, the eight irradiated samples and the one un-irradiated sample were transferred to tubes for electron spin resonance measurements. All electron spin resonance measurements were done at room temperature. The electron spin resonance signal intensities were obtained using a Bruker spectrometer, model E500 operating in the X band frequency, $\nu = 9.80$ gigahertz. The spectrometer operating conditions adopted during the experiment were 350 millitesla central magnetic field, 12 millitesla scan range, 0.1 mT field modulation amplitude, 100 kilohertz modulation frequency, 1 milliwatt microwave power, 5.12-millisecond conversion time, and 81.92-millisecond time constant. DPPH with a g-factor of 2.0028 was used as an internal standard for g-factor calculations (Nambi and Sankaran, 1985; Blackwell, 1998). Signal growth curves of nine aliquots were obtained by irradiation from 0 gray up to 350 gray. In order to obtain accumulated dose, AD, value for the dating signal, the additive dose method was used (Ikeya, 1993). Signal growth was fitted to an exponential saturation function of the following form (Jonas, 1997; Kinoshita and others, 2002):

$$I = I_s [1 - e^{-(D_{ab}+AD)/D_s}] \quad (1)$$

where I is measured electron spin resonance intensity at a g value of 2.0028, I_s is saturation, or maximum, intensity, D_{ab} is absorbed dose of artificial irradiation, AD is accumulated dose and D_s is dose at 63 percent of the saturation intensity value.

The accumulated dose AD value was obtained by extrapolating the growth curve to the zero ordinate. All data points are the average of three different sample aliquots. The electron spin resonance signal intensities were normalized to the sample weights and reported in arbitrary units.

Annual dose rate determination

The annual dose rate has two parts, the internal dose rate, D_{in} , and the external dose rate, D_{ex} . The internal dose rate is due to radiation that originated from the ^{238}U , ^{232}Th and ^{40}K content in a laterite sample. The external dose rate is due to radiation from the same three elements that are present in the

surrounding environment. Since the samples of laterite from the region of Muang Sing monument no.1 were surrounded by the same laterite and if radioactive elements exist uniformly in all Muang Sing laterite, then the matrix radiation energy emitted by the samples would be equal to the dose that the laterite samples had absorbed. Given this, the laterite sample size can be considered to be infinite. In this case, the external dose rate, D_{ex} , can be ignored (Ikeya, 1993).

The annual dose rate for laterite samples was calculated from the relative contributions of alpha, beta, and gamma from ^{238}U , ^{232}Th and ^{40}K using standard tables and assuming radioactive equilibrium (Ikeya, 1993; Engin and others, 2006; Nambi and Aitken, 1986). The water content in the laterite samples was measured and the water effect for each dose rate was taken into account for each sample (Ikeya, 1993; Aitken, 1985). The defect production efficiency, the k-value, by α -particle in the laterite is 0.18 and the g-factor equals 2.0028 (Nambi and Sankaran, 1985; Blackwell, 1998). These values were used for the internal dose rate calculation. In addition, cosmic rays have to be considered as dose corrections. Cosmic ray dose rate, D_{cosm} , depends on the geographic latitude, longitude, and altitude of the samples, the attenuation of the rays being small at high altitudes. The dominant portion of the hard, or high energy, component of cosmic rays is muons, which compose 75 percent of all particles at sea level. Thus, cosmic rays dose rate should be added to obtain the accurate age estimation (Ikeya, 1993; Sankaran and others, 1985). The annual dose rate, D , can be calculated from internal dose rates of alpha, beta, and gamma, $D_{in,\alpha}$, $D_{in,\beta}$, and $D_{in,\gamma}$, and the cosmic ray dose rate, D_{cosm} .

RESULTS AND DISCUSSION

The electron spin resonance spectra of Muang Sing's laterite samples corresponding to un-irradiated and irradiated laterite with radiation doses of 20, 50, 100, 150, 200, 250, 300, and 350 gray are shown in Figure 4. Figure 5 is a plot between electron spin resonance signal intensity and radiation dose. The signal used for the dating was at $g = 2.0028$. The electron spin resonance intensity increased with increased radiation dose. For laterite from the Kamphaeng Phet historical park, the electron spin resonance spectra of un-irradiated and irradiated samples were the same. A typical electron spin resonance spectrum result is shown in Figure 6. This figure has no electron spin resonance signals for dating, this because no accumulated dose, AD, was obtained. Consequently, the age of the laterite samples from the Kamphaeng Phet historical park cannot be determined. This result is due to the age of the Kamphaeng Phet historical park laterite being too young and, thus, not suitable for electron spin resonance dating.

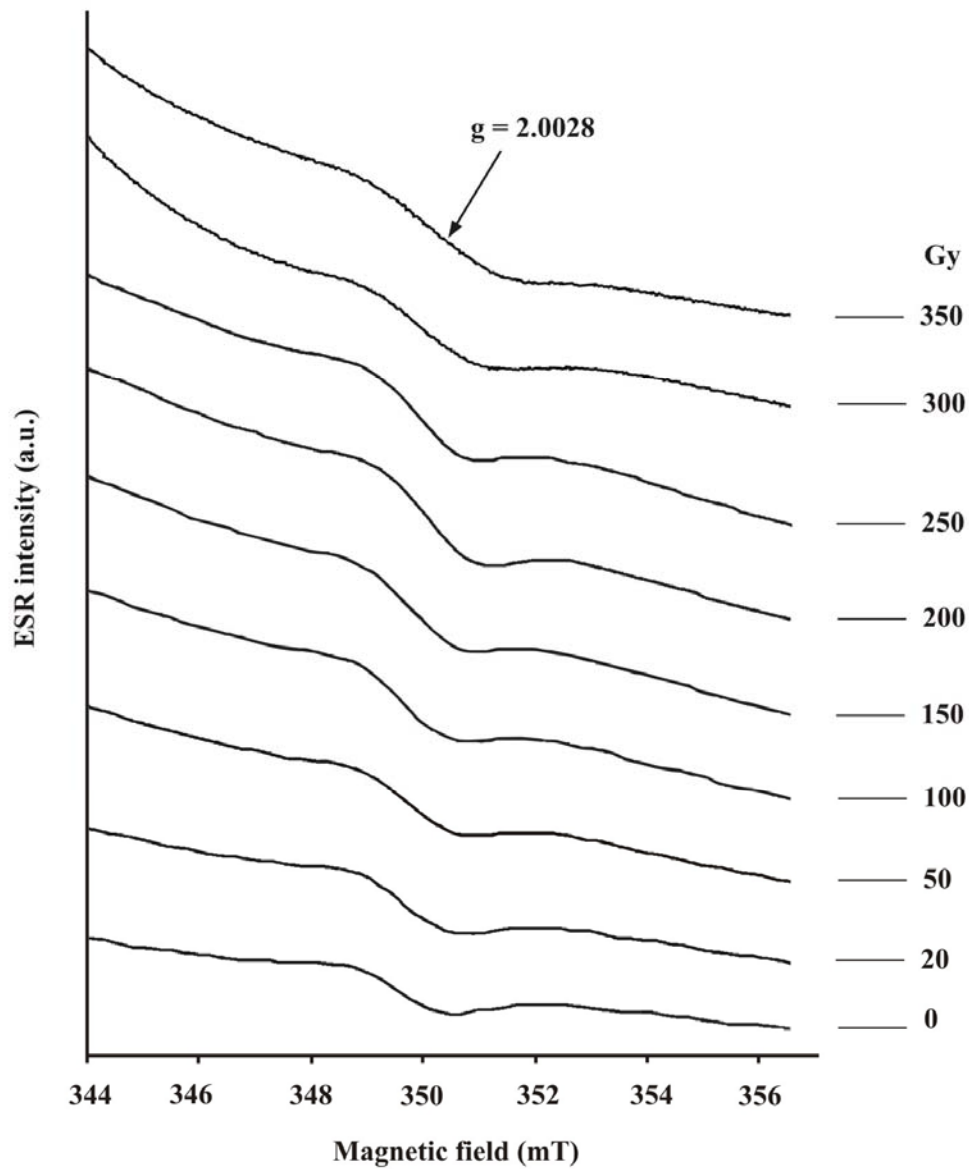


Figure 4. Electron spin resonance spectra of the laterite samples from Muang Sing historical park with radiation doses of 0, 20, 50, 100, 150, 200, 250, 300 and 350 gray.

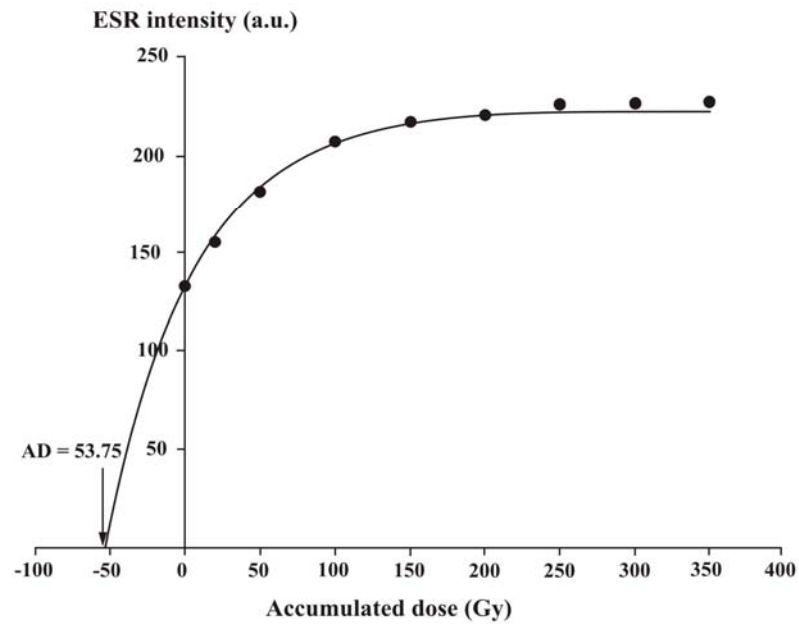


Figure 5. Electron spin resonance growth curve of the $g = 2.0028$ center in laterite samples. The intensity increased exponentially by gamma doses. Symbol (experimental), solid line is the best fitting line.

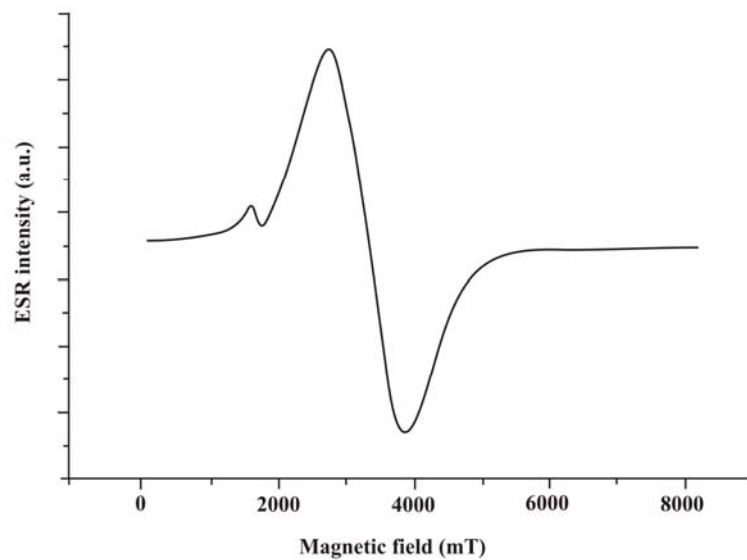


Figure 6. The typical electron spin resonance spectrum of un-irradiated and irradiated laterite samples from Kamphaeng Phet historical park.

The ^{238}U , ^{232}Th , ^{40}K and water content in laterite samples are shown in Table 2. The calculated annual dose rate, D , is shown in Table 3. The accumulated dose, AD , for the laterite from the Muang Sing historical park, obtained by fitting the electron spin resonance signal growth curve in Figure 5 with an exponential saturation function, such as 1, is 53.75 ± 1.87 gray. The electron spin resonance age of the laterite was estimated by dividing the accumulated dose by the annual dose rate. The result was an age of 5,450 years, \pm 580 years, indicating that the laterite in Muang Sing formed during the Holocene Epoch.

Table 2. U, Th, K and water contents in laterite samples from Muang Sing and Kamphaeng Phet historical parks.

Laterite Sample	^{238}U (ppm)	^{232}Th (ppm)	^{40}K (%)	Water (%)
Muang Sing	3.37	34.29	0.30	0.62
Kamphaeng Phet	4.53	22.57	0.71	0.60

Table 3. Age and annual dose rate evaluations for laterite samples from Muang Sing and Kamphaeng Phet historical parks.

Laterite Sample	Internal dose (mGy/a)			D_{cosm} (mGy/a)	Annual dose rate (mGy/a)	Accumulated dose (Gy)	ESR age (ka)
	$D_{\text{in},\alpha}$	$D_{\text{in},\beta}$	$D_{\text{in},\gamma}$	D_{cosm}	D	AD	T
Muang Sing	34.04	1.42	2.16	0.15	9.86 ± 1.00	53.75 ± 1.87	5.45 ± 0.58
Kamphaeng Phet	28.53	1.26	1.78	0.15	8.32 ± 0.84	-	-

Much research work on electron spin resonance dating has been done in past years. However, little of this concerned the dating of laterite (Nambi and Sankaran, 1985; Sankaran and others, 1985). Nambi and Sankaran (1985) reported the electron spin resonance dating of laterite of basaltic origin. The laterite sample of their study was from Goa in western India. This laterite was derived from basalt of the Deccan lava flows and was rich in pyroxene and feldspar minerals. The electron spin resonance spectra of basaltic laterite should also be comparable to that of feldspar and clay minerals. The electron spin resonance age obtained by Nambi and Sankaran was about 1 million years.

The chemical analyses of laterite samples from Muang Sing historical park show that the laterite consists of Fe_2O_3 , SiO_2 , and Al_2O_3 in the amount of 51, 29, and 17 percent, respectively. The Fe_2O_3 ,

SiO₂ and Al₂O₃ content of the Kamphaeng Phet laterite is 46, 34 and 18 percent, respectively (Table 1). These results are not comparable with those of Nambi and Sankaran.

The laterite in Thailand was first studied by Pendleton and Sharasuvana (Pendleton and Sharasuvana, 1946). They reported the chemical composition of the laterite in northeast of Thailand. The results from some of their locations are shown in Table 4. The chemical composition of the Muang Sing laterite is quite similar to that of Table 4.

Table 4. Chemical composition of laterites from some provinces in the northeastern of Thailand.

Location (Province)	Fe ₂ O ₃	SiO ₂	Al ₂ O ₃	Others
Srisaket	48.74	21.73	17.74	11.79
Maha sarakam	41.20	39.78	10.80	8.22
Roi-et	49.84	32.60	8.30	9.26
Nakon Ratchasima	31.36	46.58	18.27	3.79

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