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COMPARISON OF OSL AND ^{14}C DATES ESTIMATED FROM PALEOLITHIC PALEOSOL OF THE SUHEOL-RI SITE IN CHEONAN, KOREA

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ABSTRACT

Optically stimulated luminescence (OSL) dating and radiocarbon (^{14}C) dating were performed to determine absolute ages of Paleolithic paleosol layers from the Suheol-ri site in Cheonan, Korea. The results obtained from OSL dating of very fine sand-size (63-125 μm) and fine sand-size (125-250 μm) quartz showed that the OSL dates agreed well with each other and that all Paleolithic paleosol layers were buried after complete optical bleaching. The results obtained from ^{14}C dating of acid-, neutral-, and base-humic acids (HA, HN, and HB) indicated that ^{14}C dates of paleosol samples are only reliable when the ^{14}C dates for HA, HN, and HB substances correspond with each other. Comparing between the OSL dates and ^{14}C dates based on the typology of Korean Paleolithic stone tools and geological stratigraphy, we concluded that the OSL dating method is more reliable and effective than ^{14}C dating for reconstructing the absolute chronology of Korean Paleolithic paleosol layers.

KEYWORDS: Suheol-ri site, paleosol, OSL dating, ^{14}C dating

1. INTRODUCTION

Since the Gulpo-ri and Seokjang-ri Paleolithic sites were discovered in the early 1960s, more than 110 Paleolithic sites have been excavated on the Korean peninsula. Approximately 80 of these sites are open-air sites. There are around 30 cave sites, which were discovered in limestone-rich regions of North Korea. It is possible to gather a large amount of information on the living conditions and the environment of the Paleolithic Age from the various stone tools, bone tools, and animal and plant fossils that have been excavated from the archaeological sites (Han, 2011). However, it has been difficult to establish an accurate Korean Paleolithic chronology even though the number of excavation sites has increased in recent years. Because most of the sites are open-air sites, they largely reside on paleosol layers that can be used to characterize different Paleolithic cultural levels.

It is well known that paleosols can provide important clues about past environment, chronostratigraphy, plant habitats, and the characteristics of the climate (Ryu, 2000). Many studies have attempted to use paleosols from Korean Paleolithic sites to establish archaeological chronologies (Han, 2003; Seong, 2004). In these studies, various dating methods were applied such as ^{14}C dating, TL/OSL dating, K-Ar dating, FT dating, and tephrachronology, but these methods have not yet yielded satisfactory results. Accurate dating is particularly important for soil wedges (frost cracks) that were formed by a combination of frost action and dessication during the cold and dry climates of the Upper Pleistocene, because this period is closely associated with major changes in Korean Paleolithic cultural assemblages.

The time during which these soil wedges were formed is still a controversial issue. Thus, periodization of the paleosol layer and soil wedge is very important for the establishment of an accurate archaeological chronology that indicates when the sites were formed, occupied, and abandoned (Yi, 2000; Han, 2003).

Absolute age determinations of Korean Paleolithic paleosol layers were recently performed using ^{14}C dating and OSL dating (Han, 2003; Seong, 2004). The ^{14}C dating was carried out on charcoal and seeds that were mixed in with the paleosol layers. However, some problems were encountered with this method; namely, there were disagreements between the age of the samples and the archaeological context, due to differences between the origin of sample and the deposit condition. Because of these problems, several studies have attempted to use humic acid for ^{14}C dating. Humic acid is a natural polymer that exists within the paleosol layer. Unfortunately, these data were insufficient to be used as a tool to reconstruct a chronology of Korean Paleolithic Age (Scharpenseel and Becker-Heidemann, 1992; Kim et al., 2004; Lee and Kim, 2008).

On the other hand, OSL dating was performed using quartz and feldspar that are the primary natural minerals in the paleosol layer. It is advantageous to determine the moment of deposition of paleosol layer where the Paleolithic site is located; if the paleosol layer was fully exposed to sunlight during the deposition, the OSL signal will be optically bleached and reliable OSL date can be evaluated (Kim, 2006; Kim et al., 2010; Kim et al., 2011). However, because most paleosol layers in Korea are built from a combination of wind-blown loess originating from China and bed rock from the Korean Peninsula (Kim et al., 2012), it is essential to examine whether or not the sample collected from the paleosol layer perfectly became absolute zeroing.

In this study, OSL dating and ^{14}C dating were carried out to evaluate the absolute ages of paleosol layers from the Suheol-ri Paleolithic site in Cheonan, Korea. Comparisons between the resulting OSL dates and ^{14}C dates were then used to determine which method was more effective for obtaining absolute age determination. Finally, the chronostratigraphy of the Suheol-ri site was estimated with the information of the typology of stone tools and geological stratigraphy.

2. SITE AND SAMPLING

The Suheol-ri site (36° 53' 28.54'' N, 127° 8' 30.23'' E), which was excavated by the Chungcheong Research Institute of Cultural Heritage, is located about 80 km south of Seoul, Korea (Fig. 1).

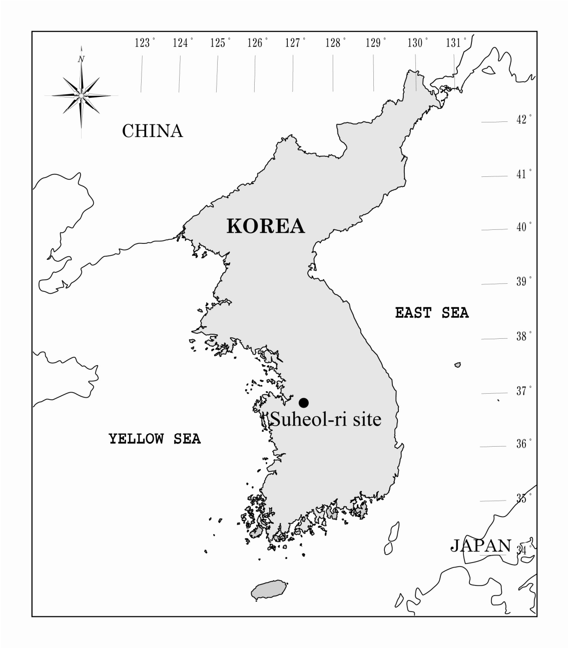

Figure 1. Location of the Suheol-ri Paleolithic site.

This open-air site is situated approximately 35 m above the sea level on a gentle slope and about 10 m higher than the alluvium around Seonghwan creek. As shown in Fig. 2, the Suheol-ri site consists of six geological layers that were formed during the Upper Pleistocene.

Two cold and dry episodes marked in Layer 3a (dark brown soil) and Layer 4 (reddish brown soil) are characterized by the soil-wedges and polygonal structures. There were 454 stone artifacts unearthed from three distinct cultural levels: the first cultural level of Layer 2, the second cultural level of Layer 3a, and the third cultural level of Layer 4.

Using the Korean Paleolithic chronology and geological evidence, it was possible to estimate that the stone artifacts obtained from the Suheol-ri site should be attributed to Middle Paleolithic (third cultural level) and Upper Paleolithic (second and first cultural levels). Raw materials were basically composed of quartz and quartzite.

Hornfels were very rarely found on the first cultural level, but the use of blade or micro-blade techniques was not observed. Pic and biface were only discovered in the third cultural level. The Suheol-ri site has been evaluated as one of central places where Paleolithic culture was transported from the middle to the southern Korean peninsula (Han and Kang, 2009).

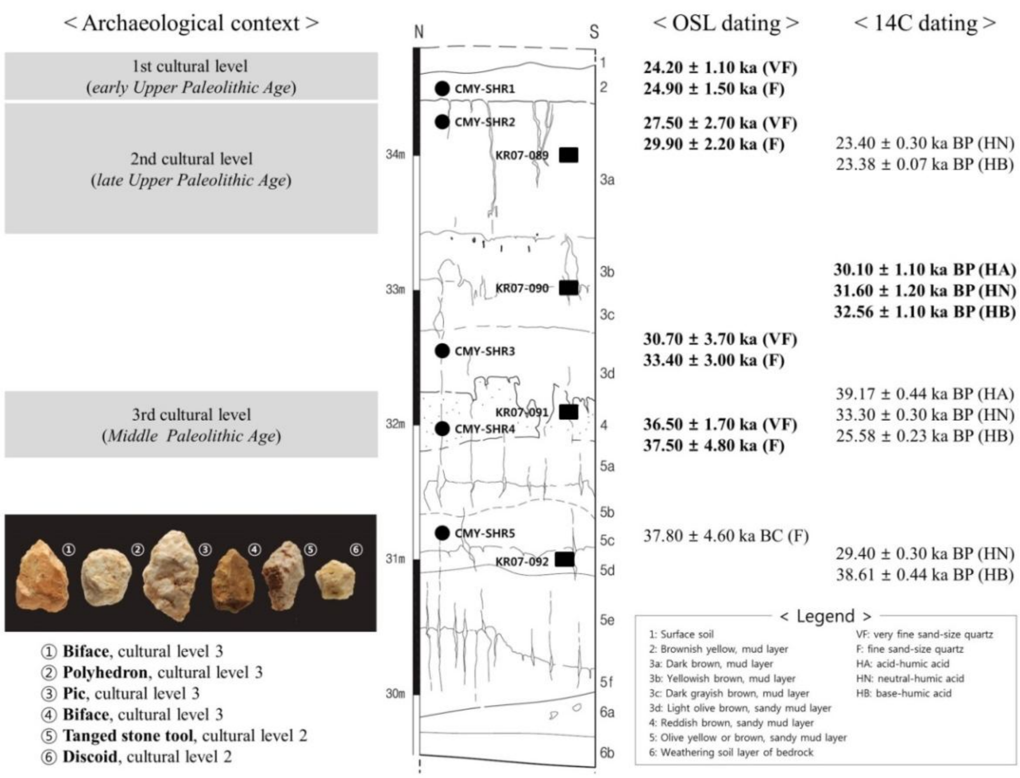

Figure 2. Schematics for sample collection and comparison of the resulting OSL dates with ^{14}C dates with archaeological contexts.

Five paleosol samples were collected for OSL dating from the Suheol-ri site (Fig. 2). The sample CMY-SHR1 (top) was taken from the first cultural level in Layer 2, the sample CMY-SHR2 from the second cultural level in Layer 3a which contained the first soil wedge, and the sample CMY-SHR4 from the third cultural level. The sample CMY-SHR3 and SHR5 were from Layer 3d and 5, respectively. These layers were mainly composed of olive or yellow brown sandy mud. All the OSL samples were obtained by hammering steel cylinders into the face of the layers. After being removed from the section, the steel cylinders were immediately covered at both ends with thick black tape, preventing any light leakage.

Four paleosol bulk samples for ^{14}C dating were also collected for comparison purposes. As shown in Fig. 2, the samples KR07-089 and 091 were obtained from the same layers in which the OSL samples were collected. However, the sample KR07-090 was taken from Layer 3b, a yellowish brown mud layer, and the sample KR07-092 was taken from Layer 5d, an olive yellow sandy mud layer, for selecting the more organic rich soils. Although the collection positions of the samples KR07-090 and 902 did not coincide with the OSL samples, there will be proved the reliability of the resulting OSL and ^{14}C dates from their stratigraphic relationship. After paring away the surface of the soil profile, all of the ^{14}C bulk samples were immediately collected and wrapped in aluminum foil, preventing the contamination caused by modern or dead carbon.

3. OSL DATING

3.1. Sample preparation and equipments

The paleosol samples that were collected by the use of steel cylinders at the Suheol-ri site were divided prior to analyses. The material located at both ends of the cylinder, which may have been exposed to daylight during sampling, was used for the assessment of the annual dose rate. Also, the water content was estimated from measurements of the water content when collected and the saturation water content. The sample remaining in the middle part of the cylinder was processed to chemically extract quartz grains that were used to evaluate paleodose.

Quartz extraction was undertaken under subdued red light to prevent affecting the dating signal. The paleosol samples, grain size 63-250 μm, were first treated with HCl and H_2O_2 to remove carbonates and organics and then treated with concentrated HF for 1 hour to reduce feldspars and etch the quartz. Because of the size reduction during HF etching, HF-etched quartz grains were re-sieved to remove grains that were smaller than 63-250 μm. Feldspar contamination was detected using infrared stimulated luminescence (IRSL), which was measured on a few aliquots of each paleosol quartz sample (Spooner et al., 1990). Finally, the paleosol quartz was classified into two sub-groups, very fine sand-size (63-125 μm) and fine sand-size (125-250 μm) quartz (Kim, 2006). However, in case of the sample CMY-SHR5, a sufficient amount of very fine sand-size quartz could not be extracted.

All OSL measurements were performed by using an automated Risø TL/OSL reader (Risø TL/OSL-DA-20). The reader is equipped with a heater unit to heat a sample at linear heating rate ranging from 0.1 to 10°C s^{-1} from room temperature to 700°C and with an optical stimulation units based on blue LED (470±20 nm) arrays delivering about 50 mW cm^2 to the sample through GG-420 filters. The emitted light signal was measured by a bialkali PM tube after passing through a 7.5 mm thick U340 filter in front of the PM tube. Laboratory irradiation was carried out using a $^{90}Sr/^{90}Y$ beta source that delivered an equivalent dose rate of 0.09 mGy s^{-1} at the sample position, which was obtained from the 5.1 Gy Riso calibration quartz.

For assessing annual dose rates, the concentrations of the natural radionuclides ^{238}U, ^{232}Th, and ^{40}K were determined by using HPGe detector (Canberra Ltd., p-type, 40%) installed at the central laboratory of Kangwon National University.

3.2. Paleodose estimation

The single aliquot regenerative-dose (SAR) protocol introduced by Murray and Wintle (2000) was used for paleodose determination. Details of the procedure used in this study are shown in Table 1.

Table 1. Sequences of the SAR method.

Step	Sequence	OSL intensity[a]
1	Regenerative dose, D_x	
2	Preheating at T°C [b] for 10 s	
3	Blue LED stimulation for 40 s at 125°C	L_x
4	Test dose irradiation, D_T	
5	Cut-heat to 220°C	
6	Blue LED stimulation for 40 s at 125°C	T_x
7	Repetition from step 1-7	

[a] OSL intensity was derived from the initial 0.5 s integration, which was subtracted using the background signal estimated from the last 2 s of the OSL decay curve.

[b] For the plateau test of paleodose, the four preheating temperatures, T °C, varied from 220°C to 280°C with 20°C intervals were used.

First, the natural OSL signal was measured from an aliquot (8 mm masking size) of the sample. Then, successively larger known laboratory regenerative doses were administered and the resulting OSL signals were measured. During this procedure, the sensitivity changes of the sample aliquot due to successive measurements were corrected for by monitoring the luminescent response to a subsequent small radiation dose (so called "test dose"), which was kept constant throughout the experiment.

Samples received 40 s of blue light exposure, and the first 0.5 s of the OSL signal measured at 125°C was integrated to construct growth curves. Fig. 3 shows examples of results that were derived by the SAR protocol for very fine sand-size (Fig. 3a) and fine sand-size (Fig. 3b) quartz from the sample CMY-SHR1 (see Liritzis et al., 1997).

To examine the dependence of paleodose values on preheating temperatures, four different preheating temperatures (ranging from 220 to 280°C with 20°C intervals) were employed (Kim, 2006). Five aliquots were analyzed for each preheating temperature. Fig. 4 shows the paleodose plotted as a function of the different preheating conditions for the sample CMY-SHR1.

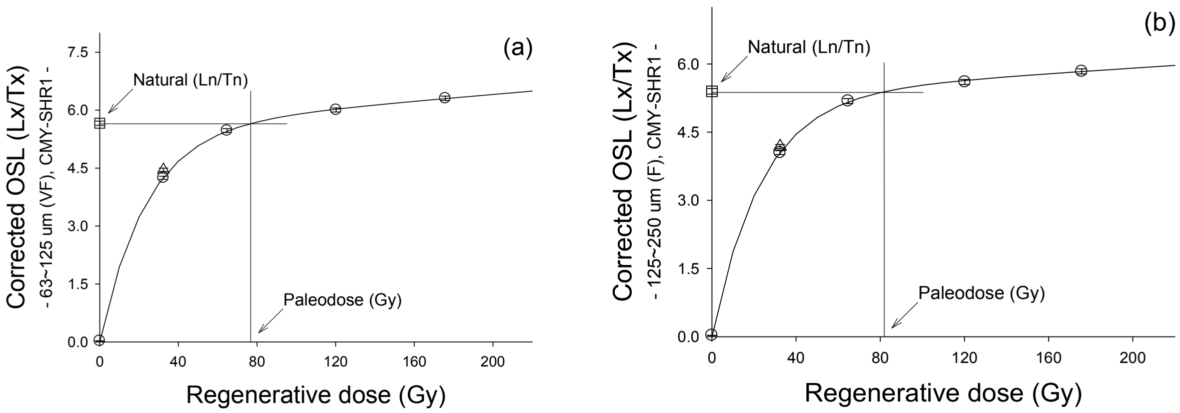

Figure 3. Examples of paleodose determinations based on the SAR protocol for very fine sand-size (Fig. 3a) and fine sand-size (Fig. 3b) quartz of the sample CMY-SHR1. Each growth curve was obtained from curve-fitting by a single saturation exponential function to the regenerated data points (Lx/Tx). The paleodose was determined by the interpolation of natural OSL intensity (Ln/Tn) into the curve.

The paleodose of both very fine sand-size and fine sand-size quartz of all samples used in this study had the plateau region in the range from 220 to 260°C, with the recycling ratios between 0.9 to 1.1 and the recuperation below 5% at all temperatures.

Thus, the averaged paleodose values from preheating temperatures ranging from 220 to 260°C were used for OSL age calculations (i.e., 15 aliquots for each quartz sample). The uncertainties quoted in Table 2 refer to the standard error for the number of samples used.

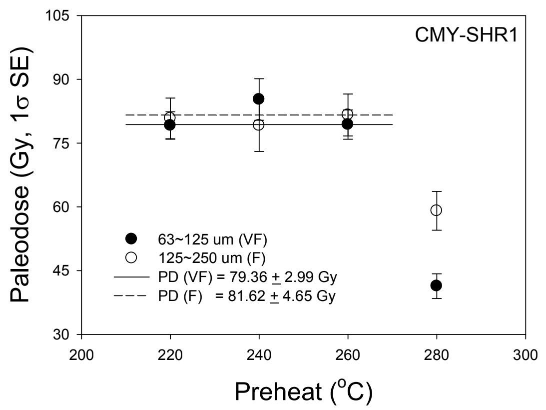

Figure 4. Plot of paleodose values as a function of preheating temperature for very fine sand-size (VF) and fine sand-size (F) quartz of the sample CMY-SHR1. Each line indicates an average of the paleodose (PD) in the range from 220°C to 260°C.

3.3. *Annual dose rate calculation*

For the annual dose rate assessment, approximately 100 mg of the paleosol was used for laboratory gamma-ray spectrometry with the HPGe detector. The dried sample was packed into a plastic holder that had been stored for 40 days in order to achieve secular equilibrium between radon and its daughter nuclides. The concentrations of the natural radionuclides ^{238}U, ^{232}Th, and ^{40}K were calculated by comparing the ratio of each full-energy peak area to that of RGSet (RGU-1, RGTh-1and RGK-1) certified by IAEA (Kim and Burnett, 1983).

Beta and gamma dose rates were evaluated using the conversion factors for the concentrations of natural radionuclides. These values were corrected for water content using the formula of Zimmerman (1971) and beta attenuation through the grains by a factor of 0.9. The contribution from cosmic rays was calculated by using the formula of Prescott and Hutton (1994) in which the density of soil was assumed to be 1.85 g cm^{-3}.

The corrected total annual dose rates for age calculations were comprised of the beta dose rate, gamma dose rate and cosmic ray component, as shown in the Table 2.

Table 2. OSL dates of very fine sand-size (63-125 μm) and fine sand-size (125-250 μm) quartz extracted from the paleosol.

Sample[a]	Layer (depth, cm)	Grain size (μm)	Paleodose[b] (Gy)	Annual dose rate (Gy ka^{-1})			Total annual dose rate [c] (Gy ka^{-1})	OSL age (ka, 1σ SE)
				Gamma	Beta	Cosmic ray		
CMY-SHR1	2 (50)	63-125	79.36 ± 2.99	1.52 ± 0.04	2.40 ± 0.08	0.19 ± 0.01	3.28 ± 0.07	24.20 ± 1.10
		125-250	81.62 ± 4.65					24.90 ± 1.50
CMY-SHR2	3a (70)	63-125	92.84 ± 8.82	1.53 ± 0.04	2.54 ± 0.09	0.18 ± 0.01	3.38 ± 0.08	27.50 ± 2.70
		125-250	101.16 ± 7.14					29.90 ± 2.20
CMY-SHR3	3 (240)	63-125	91.29 ± 10.73	1.24 ± 0.04	2.37 ± 0.09	0.15 ± 0.01	2.98 ± 0.08	30.70 ± 3.70
		125-250	99.51 ± 8.52					33.40 ± 3.00
CMY-SHR4	4 (305)	63-125	121.30 ± 5.02	1.43 ± 0.04	2.64 ± 0.10	0.14 ± 0.01	3.33 ± 0.08	36.50 ± 1.70
		125-250	123.06 ± 15.67					37.50 ± 4.80
CMY-SHR5	5c (380)	63-125	N/ A	1.65 ± 0.04	2.70 ± 0.10	0.12 ± 0.01	3.54 ± 0.08	N/A
		125-250	133.93 ± 16.02					37.80 ± 4.60

[a] All samples were collected in 2007 AD.
[b] Each paleodose was evaluated by using the SAR method.
[c] For calculating the total annual dose rates, a mean water content of 20 ± 5% was applied to all the paleosol samples.

3.4. OSL age determination

The OSL age was calculated from the ratio of the paleodose to the annual dose rate by following equation:

$$\text{Age (ka)} = \frac{\text{Paleodose (Gy)}}{\text{Annual dose rate (Gy ka}^{-1}\text{)}}$$

All resultant OSL dates were summarized in Table 2. Except for the sample CMY-SHR5, the OSL dates from both the very fine sand-size and fine sand-size quartz agreed well with each other within error ranges. These results suggest that the Paleolithic paleosol layers at the Suheol-ri site were buried after complete optical bleaching and that each OSL date was identical to the time that the paleosol layer was deposited (Buylaert et al., 2008; Timar-Gabor et al., 2011).

Additionally, we believed that the OSL date of the fine sand-size quartz from the sample CMY-SHR5 is also reliable, considering the typology of Korean Paleolithic stone tools and geological stratigraphy (Fig. 2).

4. ^{14}C DATING

4.1. Sample preparation and equipments

Humus existed in paleosol is a natural polymer that is created through the decomposition of soil organic matter and combined with clay. So, it is impossible to separate the humus from sample by physical treatment. For ^{14}C dating of the paleosol samples at the Suheol-ri site, plant roots were first eliminated using props. After this step, humin, fulvic acid and humic acid were sequentially extracted through chemical pretreatment based on A-A-A (acid-alkali-acid) method (Kim et al., 2004).

Normally, ^{14}C dating of paleosol samples has been performed on the entire fraction of humic acid, and it is known that the measured ^{14}C age of humic acid material is generally younger than the true age of paleosol because of continuous input of organic material into the paleosols (Wang et al., 1996).

In this study, to confirm the reliability of ^{14}C dates for the paleosol samples, we extracted the humic acid further into acid, neutral, and base components by the process shown in Fig. 5 (Lim et al., 2008).

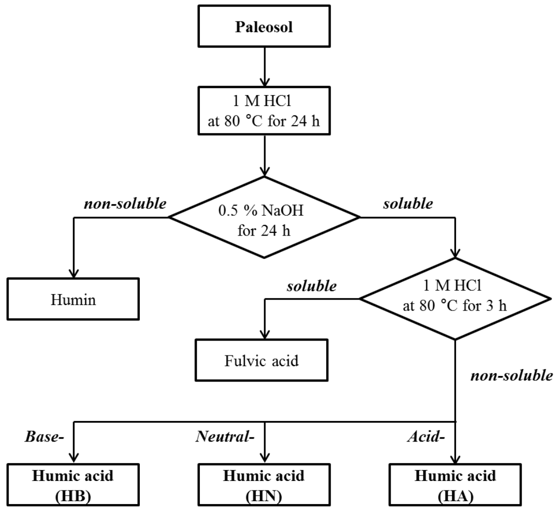

Figure 5. The chemical pretreatment process used for extracting three types of humic acid from the Paleolithic paleosol sample.

Hereafter, we refer to these specific components as acid-humic acid (HA), neutral-humic acid (HN), and base-humic acid (HB). However, a sufficient amount of HA material could not be extracted from the sample KR07-092. An automatic elemental analyzer (Flash EA 1112 series, Thermo Fisher Scientific, Inc.) that installed at the central laboratory of Chungnam National University was used to determine the carbon content of the chemically extracted humic acid components. The results showed that the HA material had higher carbon contents than the other materials, and that the organic content of the paleosol layers decreased with depth (Table 3).

Sample graphitization for ^{14}C dating by using accelerator mass spectrometer (AMS) was carried out by our self-developed system (Kim et al., 2010). Initially, a quartz tube that contained the resulting humic acid components was placed into a muffle furnace and combusted at 850°C for 2 hours to substitute for CO_2 gas. For the purpose of collecting the sample CO_2 gas, the quartz tube was broken in a flexible bellows, and the released CO_2 gas was separated from the entrained H_2O vapor using a liquid nitrogen/alcohol trap (–40°C). The gas was then cryogenically transported to a vessel (a volume of 11 cm^3), where the sample CO_2 was stored at about 400 torr for latter graphitization. The catalytic reduction method was then employed at a graphitization temperature of 625°C and a vacuum of 10^{-6} torr.

Table 3. ^{14}C dates of the chemical residuum obtained from the paleosol.

Layer (Depth, cm)	Lab No.	Material	C (%)	$\delta^{13}C$ (‰)	Conventional ^{14}C yr BP.
3a (100)	KR07-089	HA	8.97		N/A
		HN	0.76	-36.77	23,400 ± 300
		HB	0.30	-38.08	23,380 ± 70
3b (190)	KR07-090	HA	5.23	-27.02	30,100 ± 1,100
		HN	1.16	-26.30	31,600 ± 1,200
		HB	0.40	-24.63	32,560 ± 1,100
4 (295)	KR07-091	HA	2.61	-21.91	39,170 ± 440
		HN	1.08	-23.59	33,300 ± 300
		HB	0.31	-22.66	25,580 ± 230
5d (400)	KR07-092	HA	N/A		N/A
		HN	0.62	-27.63	29,400 ± 300
		HB	0.41	-24.65	38,610 ± 440

4.2. ^{14}C age determination

^{14}C dating was conducted with AMS measurement at the National Center for Inter-university Research Facilities in the Seoul National University. All conventional ^{14}C dates obtained from the three types of humic acid were summarized in the Table 3. The results represent an average of three different measurements. In this study, the ^{14}C date of the HA material from the sample KR07-089 was not calculated despite its high carbon content.

The only ^{14}C dates that agreed with each other were those from the HA, HN and HB materials derived from the sample KR07-090. Moreover, there was no chronostratigraphic correlation between the paleosol samples. Therefore, in consideration of the resultant OSL dates, the typology of the Korean Paleolithic stone tools, and the geological stratigraphy at the site, we deduced that ^{14}C dates of paleosol samples are reliable only when the ^{14}C dates from the HA, HN, and HB materials correspond to each other within appropriate error ranges (Fig. 2).

5. SUMMARY AND CONCLUSIONS

In this study, we used OSL dating and ^{14}C dating to determine reliable absolute ages of the Paleolithic paleosol layers located at the Suheol-ri site and established the site's chronostratigraphy.

For OSL age estimation, the chemically extracted paleosol quartz grains were divided into very fine sand-size and fine sand-size quartz, and each OSL age was evaluated using the SAR method. Except for the sample CMY-SHR5, all OSL dates agreed well with each other (Table 2). Also, the OSL date of fine sand-size quartz for the sample CMY-SHR5 corresponded well with the typology of Korean Paleolithic stone tools and geological stratigraphy found in the region (Fig. 2). These data suggest that all Paleolithic paleosol layers were deposited after complete optical bleaching.

For ^{14}C age estimation, three types of humic acid (HA, HN, and HB) were obtained from the paleosol samples, and then the ^{14}C ages were evaluated using AMS measurements. Only the ^{14}C dates resulting from the HA, HN, and HB materials of the sample KR07-090 agreed with each other (Table 3). In consideration of the resultant OSL dates, the typology of the Korean Paleolithic stone tools, and the geological stratigraphy, we deduced that ^{14}C dates of paleosol samples are reliable only when the ^{14}C dates from the HA, HN, and HB materials correspond with each other (Fig. 2).

Based on the results of this study, we concluded that the OSL dating method was more reliable and effective than ^{14}C dating for reconstructing the absolute chronology of Korean Paleolithic paleosol layers. An accurate chronostratigraphy based on OSL results of Paleolithic paleosol layers could help archaeologists to better understand the Paleolithic cultures that existed on the Korean Peninsula.

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