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Calibration curve from AD 1250 to AD 1650 by measurements of tree-rings grown on the Korean peninsula

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ABSTRACT

A 1 MV AMS machine installed at Korea Institute of Geoscience and Mineral Resources (KIGAM) in 2007 has been dedicated to study on paleoclimate change and the environmental issues. Because the possibility of regional age offset has been suggested by several previous studies, the consistency of the data set of IntCal04, which is used to calibrate radiocarbon ages in our laboratory, with data obtained from ^{14}C in tree rings grown in the Korean peninsula has been examined in this study. Tree-ring samples were collected from the building materials of Korean historical wooden buildings. Remaining historical records regarding the construction times of the buildings were consulted. The ages of the tree-ring samples ranged from AD 1250 to AD 1650 and were measured by dendrochronological method. After the samples were cut into single-year rings, alpha cellulose was extracted from each ring. Then, their annual ^{14}C concentrations were measured by AMS. Accurate radiocarbon ages during the 400 year period were evaluated from the concentrations. The ages of the tree rings were compared with the IntCal04 calibration curve. The average deviation of ^{14}C concentration was calculated to be −2.14‰. By the Fourier transform of the single-year variation of the concentration, six major periodic components could be found. One of the components has a period of 10.9 years and it is thought to be related to a sunspot variation known as the Schwabe cycle, which has a period of 11 years.

1. Introduction

Radiocarbon dating is based on the important assumption that the production rate of radiocarbon in atmosphere is stable throughout the time range during which radiocarbon age dating is valid. However, previous studies with careful measurements have shown that the atmospheric concentration of radiocarbon has varied during the last several tens of thousands years [1–3]. Long term variation of atmospheric ^{14}C production is thought to be due to changes in the magnetic dipole, which acts as a shield against the cosmic rays [4]. The time-varying ^{14}C concentration in the atmosphere gives rise to the age deviation with time. Furthermore, the active regional carbon reservoir effect and the global reservoir effect result in regional deviation of the radiocarbon concentration. For example, the far-east Northern Pacific currents contain old carbon, and an effect of Kuroshio, one of the far-east Northern Pacific currents, on the regional atmospheric ^{14}C concentration has been reported [5,6]. These factors may cause significant deviation of radiocarbon ages from calendar ages. To compare a radiocarbon age with a calendar age, the radiocarbon age should be calibrated using curves obtained by serial measurements of known-age samples.

The IntCal09 data set [7] obtained from tree-ring samples is commonly used to calibrate radiocarbon ages. Tree-ring samples are one of most advantageous types of samples to obtain a calibration curve for the last 10,000 years because their ages are well-defined by the dendrochronological method, and sequential single-year measurement is available. The tree-ring samples used for the IntCal04 [8] and the IntCal09 data sets were collected from the western part of North America (Douglas firs and Sequoias) and Europe (Irish and German oaks, and German pines). However, due to a lack of sufficient data, samples from the Asian continent were not included in the IntCal04 and IntCal09 data sets.

Recently, tree-ring ages measured from Japanese tree samples have been published, and deviations of the ^{14}C ages of the Japanese tree samples from those of IntCal04 were reported [6]. Since Korea and Japan are located in the far-east of the Asian continent, tree rings grown in Korea and those grown in Japan have advantages in the investigation of the regional age offset from the IntCal04 data. Unfortunately, very few Korean tree rings have been measured because of the poor infrastructure for ^{14}C measurement prior

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to 2008. Korea Institute of Geoscience and Mineral Resources (KIGAM) AMS group has studied the ^{14}C concentration variation in the atmosphere using domestic tree-ring samples since the AMS machine started operation in 2008. In this work, ^{14}C concentration change from AD 1250 to AD 1650 was reconstructed by measurement of tree rings grown on Korean peninsula.

2. Sample collection

Tree-ring samples were collected from several historical buildings in Korea. Too narrow rings were avoided in order to obtain enough wood weights from single-year ring. Jungsoosa (37°36′27.97″N, 126°26′43.71″E) is an old temple which is located on Ganghwa Island in the western sea of the Korean peninsula. This temple is known to have been first built in AD 639 and renovated several times after it was built. During the last renovation in 2005, several wood samples were collected from the old building materials, and three of them, JSPP504B, JSBS507A and JSBS513B, were used in this work. Geunjeongjeon (37°34′42.83″N, 126°58′37.32″E) is a building of Gyeongbokgung Palace which is located in Seoul. This building is known to have been first built in AD 1395, and the last renovation was performed in 2003. From the building materials, a wood sample, k2gd107B, was selected for this study. Gwanghanru (35°24′13.86″N, 127°22′47.48″E) is a pavilion locating in Namwon, Jeonranam-do, Korea. This building was first built in AD 1419 and the last renovation was finished in 2003. Wood samples, GIGT321A and GIGT323A, were collected from the building and were used in this work. The last building is Tongmyeongjeon (37°34′46.98″N, 126°59′37.60″E) which is a building of Changdeokgung Palace located in Seoul. It was first built in AD 1484 and renovated in 2002. Two samples, TMGJ055A and TMJS017A, were collected from the building and were used in this work. Fig. 1 shows the sample collection sites. Also, sample information is summarized in Table 1.

These samples are all pine wood (*Pinus densiflora*: Japanese red pine). Unfortunately, the place where these trees grew is not clearly known because of lack of any precise historical record about the growing location. However, it is unlikely that these trees grew outside of Korea since they were highly successfully dated by Korean tree-ring chronologies. Furthermore, it is very reasonable to estimate that they grew at locations relatively close to the building sites, considering transportation, cost of construction and the supply of pine trees during the period when the buildings were constructed.

Fig. 1. The sampling sites. Seven samples collected from building materials of four historical buildings were used in this work.

3. Dendrochronological dating and sample preparation for AMS measurement

The tree-ring samples were dated at the Tree-Ring Material Bank of Chungbuk National University by dendrochronological method [9]. The ring-width plots of the samples were cross-dated by matching their pattern with the master chronologies which had already been absolutely dated through matches with living trees. Finally the absolute ages of individual tree rings in the samples could be determined. Samples JSPP504B, JSBS507A, and JSBS513B from Temple Jungsoosa were dated in ranges from AD 1224 to 1398, from AD 1285 to 1429 and from AD 1394 to 1449, respectively. The age range of k2gd107B from the Geunjeongjeon Palace building was found to be from AD 1420 to AD 1621. The age ranges of GIGT321A and GIGT323A from Pavilion Gwanghanru were determined to be from AD 1523 to AD 1625 and from AD 1507 to AD 1616, respectively. Samples TMGJ055A and TMJS017A from the Tongmyeongjeon Palace building were dated to AD 1607 to AD 1793 and AD 1619 to AD 1804, respectively. The age ranges of the samples measured by tree-ring counting are summarized in Table 1.

The samples with known ages were cut into annual rings. Among these rings, 402 ring samples from AD 1249 to AD 1650 were treated by the alpha extraction method. Unwanted mobile materials such as gums, resins, fats and waxes were removed by heating at 80 °C for 6 h in a mixed solution of 120 ml of cyclohexane and 60 ml of ethanol. The remains were rinsed with ethanol and de-ionized water several times. Next, the samples were treated with 150 mg of $NaClO_2$ and 1 M HCl at 100 °C for 1–2 h. Then, the samples were treated in a 12% NaOH solution with nitrogen gas bubbling at 60 °C. Finally, the samples were neutralized with a 2 M HCl solution for 30 min and dried in an oven at 40 °C.

An elemental analyzer (EA) was used to combust the samples over 900 °C. The EA was directly coupled with a 24-fold automatic reduction system [10], which was used to graphitize the samples with Fe catalyst and hydrogen gas at 600 °C. The average graphite yield was around 93% after a 3-hour reduction process, and the graphite sizes were around 1 mg. The graphite samples were pelletized, and their $^{14}C/^{12}C$ ratios were measured by a 1 MV AMS system at KIGAM [11]. For routine work, the counting time per sample is 30 min; however, the tree-ring samples were measured three times for 1050 s per sample. Hence, the total counting time per a tree-ring sample was 53 min for more precise measurement.

4. Results and discussion

The radiocarbon concentration variation in the atmosphere in the age range from AD 1250 to AD 1650 is shown in Fig. 2. The $\Delta^{14}C$ curve of the IntCal04 data set is also shown in the figure. The $\Delta^{14}C$ values were calculated from the ^{14}C concentration of sample by

$$\Delta^{14}C = [pMC \cdot \exp((1950 - a)/\lambda) - 1] \times 1000, \tag{1}$$

where a represents the year the ring was grown and $\lambda = t_{1/2}/\ln(2) = 8267$. Here, $t_{1/2}$ is the half-life of radiocarbon (5730 years), and pMC was determined by comparing sample activity to that of NIST OXII after background subtraction and by $\delta^{13}C$ correction with Stuiver and Polach's method [12]. The errors were evaluated by statistical error of ^{14}C, ^{12}C and ^{13}C counts of samples, oxalic samples and blank samples. ^{13}C ratios were measured by AMS. The average offset from IntCal04 was found to be $-2.13 \pm 4.32‰$, and it