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RADIOCARBON CONCENTRATION IN SUB-ANNUAL TREE RINGS FROM POLAND AROUND 660 BCE

Andrzej Z Rakowski[1]* • Jacek Pawlyta[2] • Hiroko Miyahara[3] • Marek Krąpiec[2] • Mihály Molnár[4,5] • Damian Wiktorowski[2] • Masayo Minami[6]

[1]Silesian University of Technology, Konarskiego 22B str., 44-100 Gliwice, Poland
[2]AGH University of Science and Technology, Mickiewicza Av. 30, 30-059 Krakow, Poland
[3]Humanities and Sciences/Museum Careers, Musashino Art Universally, 1-736 Ogawa-cho, Kodaira, Tokyo, 187-8505, Japan
[4]Hertelendi Laboratory of Environmental Studies, Institute of Nuclear Research of the Hungarian Academy of Sciences (ATOMKI), H-4026 Debrecen, Hungary
[5]Isotoptech Zrt., H-4025 Debrecen, Hungary
[6]Institute for Space-Earth Environmental Research, Nagoya University, Furo-cho, Chikusa-ku, Nagoya, 464-8601, Japan

ABSTRACT. The article presents results of measurements of radiocarbon (^{14}C) concentration in sub-annual dendrochronologically dated tree rings of English oak (*Quercus robur* L.) from Grabie village near Kraków (southern Poland). Samples of early wood (EW) and late wood (LW) spanning the years 664–658 BCE. α-cellulose was extracted from each sample and their radiocarbon content was measured at the ATOMKI laboratory in Debrecen, Hungary. The EW and LW data confirm a prolonged increase in $\Delta^{14}C$ values around 665–663 as was observed by Park et al. (2017), Rakowski et al. (2019), or Sakurai et al. (2020). In addition, we found that this event may consist of two relatively small events, as was proposed by Sakurai et al. (2020). Based on obtained in this and previous study data we estimate that the occurrence of the two events were between 665 and 664 BCE (Rakowski et al. 2019), and in late spring of 663 BCE (May–June, before beginning of LW formation).

KEYWORDS: calibration curves, Miyake Event, radiocarbon dating, SEP, tree rings.

INTRODUCTION

Radiocarbon (^{14}C) is produced by the nuclear reaction $^{14}N(n, p)^{14}C$ in the Earth's atmosphere, and its production depends on the cosmic ray flux reaching the Earth's atmosphere (Usoskin and Kovaltsov 2012). On average the ^{14}C production rate is of the order of 1.64 and 1.88 atoms·cm^{-2}·s^{-1} for the modern time and for the preindustrial epoch, respectively (Kovaltsov et al. 2012). Modulation of the cosmic ray flux occurs because of the variability of the intensity of the superposition of solar, interplanetary, and terrestrial magnetic fields. On the other hand, changes in the intensity of cosmic flux due to occurrence of events such as solar proton events (SPEs), supernovae explosion (SNe) or gamma-ray burst (GRBs), can increase the production rate for a short time.

Freshly produced atmospheric radiocarbon is oxidized in two steps, first with O_2 to carbon oxide, and then to carbon dioxide with hydroxyl radical. The $^{14}CO_2$ formed in this way becomes part of the global carbon cycle (GCC) and can be exchanged between the atmosphere, the biosphere, and the ocean in various physical and chemical processes. Through photosynthesis radiocarbon is incorporated into plant tissue, including i.e., tree rings, which can be then used in reconstruction of its concentration in the past. Dendrochronologically dated tree rings were used to reconstruct the past changes in atmospheric radiocarbon concentration due to changes in its production, but also changes in the carbon cycle for the last 12,500 yr (van der Plicht 2007; Hogg et al. 2020; Reimer et al. 2020). The development of accelerator mass spectrometry (AMS) measurement techniques enabled the increase the

*Corresponding author. Email: arakowski@polsl.pl

resolution and precision of measurements, while reducing the weight of the sample needed for analysis (i.e., Synal et al. 2007; Molnar et al. 2016).

Miyake et al. (2012) were the first to confirm the occurrence of event (henceforth M12) of rapid increase in ^{14}C concentration, occurring within one year. Between 774 and 775 CE, a significant increase of 12‰ in $\Delta^{14}C$ value has been reported (Miyake et al. 2012). To explain the causes of the M12 event, a four-box carbon cycle model was used, yielding the estimated production rate of $6{\cdot}10^8$ atoms·cm^{-2}·s^{-1}. According to these authors, this value may suggest a giant solar proton events (SEPs) or supernovae explosion (SNe) as a possible cause of this event. However, considering no evidence of SNe in this time frame, the SEPs is most possible reason of this increase. The production value has been recalculated by Usoskin et al. (2013), using a five-box carbon cycle model, and the obtained result is around 4 times smaller than what was presented by Miyake et al. (2012), i.e., $1.3 \pm 0.2{\cdot}10^8$ atoms·cm^{-2}·s^{-1}. In Miyake et al. (2014), a new calculation was presented, and the production rate obtained for this event was consistent with that reported by Usoskin et al. (2013). The assumption of SEPs as a possible cause of M12 was confirmed by Mekhaldi et al. (2015), who recorded a comparable rapid increase in ^{10}Be and ^{36}Cl in ice cores from Antarctica and Greenland around 775 CE and 994 CE. GRBs with typical energy spectra and fluxes could produce significant amounts of ^{14}C and ^{36}Cl, however, the ^{10}Be production rate would not be significantly affected (Pavlov et al. 2013; Mekhaldi et al. 2015). Thus, we conclude a large SEP, or series of SEPs, are a cause of the discussed Miyake events.

The 774–775 CE event has been confirmed independently by several authors (Jull et al. 2014; Güttler et al. 2015; Rakowski et al. 2015; Büntgen et al. 2018) in dendrochronologically dated annual tree rings from different places around the world, which indicates its global character.

Rapid increases in radiocarbon concentration have also been confirmed at different periods. An increase of 11.3‰ was recorded between years 993 and 994 CE in samples of Japanese cedar (*Criptomeria japonica*; Miyake et al. 2013), and Hinoki cypress (*Chamaecyparis obtusa)* (Miyake et al. 2014) and have been confirmed by other authors (Fogtman-Schultz et al. 2017; Büntgen et al. 2018; Rakowski et al. 2018). Another SEP have been noted around 660 BCE and confirmed by Park et al. (2017), Rakowski et al. (2019), and Sakurai et al. (2020). Similarly, to M12, an increase in production rate of ^{10}Be and ^{36}Cl has been noted, confirming the solar origin of this event (O'Hare et al. 2019).

Other sudden increases in ^{14}C have been observed between 814–815 BCE (Jull et al. 2018), 1054–1055 CE (Terrasi et al. 2020), or a series of events between 1261–1262, 1268–1269, and 1279–1280 CE (Miyahara et al. 2022). The magnitudes of those events were significantly lower in comparison to the M12 event, and, without information about changes in production rate of ^{10}Be and ^{36}Cl it is difficult to say about the origin, and if those could also be classified as Miyake events. The oldest observed rapid increases in radiocarbon concentration so far identified occurred in mid-Holocene around 5480 BCE (Miyake et al. 2017), 5259 BCE, and 7176 BCE (Brehm et al. 2022). The 7176 BCE has magnitude similar to the largest SEP event in 775 CE (Brehm et al. 2022). The solar origin of the two oldest events has been confirmed by increase in production rate of ^{10}Be and ^{36}Cl (7176 BCE—Paleari et al. 2022; 5480 BCE—Kanzawa et al. 2021).

Such sudden and intense increase in the concentration of radiocarbon found application in the precise radiocarbon dating with one-year resolution. This method was used by Wacker et al. (2014) to precisely date the cutting year of a timber in the historical Holy Cross chapel of the

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Figure 1 Location of Grabie village (50.0391 N, 19.992 E).

convent St. John the Baptist in Val Münstair in Switzerland. Also, in the works of Oppenheimer et al. (2017), Hakozaki et al. (2018), Krąpiec et al. (2020), Philipsen et al. (2022), and Kuitems et al. (2022), these events were used to precisely determine the age based on changes in the concentration of radiocarbon in the sequence of annual tree rings.

The results presented in this article are the continuation of our previous work (Rakowski et al. 2019) and include new measurements of the radiocarbon concentration in sub-annual rings. The goal of this study is to use the information of carbon isotopic composition in sub-annual tree rings to estimate beginning of occurrence of this event.

SAMPLE AND METHODS

For this study, sub-fossil oaks (*Quercus robur* L.) were recovered from a gravel pit near the Vistula River in the village of Grabie, near Kraków (southern Poland; Figure 1). Around 100 slices of oak trunks were taken and after preparation were used for dendrochronological study. Measurements were made with 0.01 mm accuracy using a DENDROLAB 1.0 apparatus, then

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the ring-width sequence were processed with a set of software TREE-RINGS (Krawczyk and Krąpiec 1995), TSAP (Rinn 2005) and DLP (Holmes 1999).

Based on dendrochronological sequences of subfossil oak trunks from Grabie, a GAA_3U chronology covering the period 994-612 BC was established (Krąpiec 2001). A G 58 sample was selected for radiocarbon analysis, and it is the same specimen (G 58) which was used in our previous study Rakowski et al. (2019). Sample G 58, dated to 797-620BC and characterized by relatively large annual increments and high similarity (t=15,5, Gl=79(***)) to the South Polish Oak Chronology C_3000E (Krąpiec 2001; Figure S1). The South Polish Oak Chronology C_3000E was established trough teleconnection with Becker's German oak chronology for southern Germany (Becker 1993; t=7.7) and Leuschner's and Delorme's oak chronology for central Germany (Leuschner and Delorme 1988; t=6.8).

The tree used in our study belongs to the ring-porous angiosperms species, having clearly developed earlywood (EW) and latewood (LW). The EW consists of large cells, which are formed in the spring just before the leaves appear. To form these, the plant mainly uses stored reserves from the previous growing season. LW, on the other hand, is formed from photosynthesised substances during the growing season, i.e., the year of tree-ring formation (Ermich 1959). This allows a more accurate (half-yearly) determination of the timing of the ^{14}C change.

Well-developed annual increments from the period 663-558 BCE, characterized by adequate thickness, were sliced and separated into LW and EW. Then, the LW was additionally split into 2 parts (except for samples from years 665 BCE and 660 BCE, which were too thin). This gives a total of 19 samples, of which five years (663 BCE, 662 BCE, 661 BCE, 660 BCE, and 658 BCE) were divided into EW, LW1, and LW2, and two years were divided into EW and LW (664 BCE and 659 BCE). From each of the sample, the most stable part of the wood in the form of α-cellulose was designated for study (Santos et. al. 2001; Nemec et al. 2010). The extraction of α-cellulose from the samples was performed according to the method described by Michczyńska et al. (2018). In the first step of this procedure, the wood samples were dried and cut into shavings, then weighed and placed into glass tubes, and subsequently each sample was put into glass tube with deionized water, sodium chlorite, and 1% HCl. The tube was then placed in an ultrasonic bath kept at 70°C for 1 hr, and then sodium chlorite and 1% HCl was added to each sample. This procedure was repeated five times, and the samples were then rinsed with hot deionized water up to neutral pH. This process removes most of the lignin and the remaining sample consists mostly of holocellulose. For the next step, 10% NaOH solution was added to the samples and then put into an ultrasonic bath at 70°C for 45 min. After the solution was removed, the sample was rinsed with cold deionizing water. Next, 17% NaOH solution was added, and samples were put into ultrasonic bath at room temperature for 45 min. After this time deionizing water was added to the samples. This step removes the rest of the remaining lignin and holocellulose. The samples were then rinsed with hot deionized water up to neutral pH. Finally, 1% HCl was added to the samples and the samples were rinsed to neutral pH.

The obtained α-cellulose was dried overnight at 70°C, and then placed in a glass tube together with oxidizing agent (MnO_2), pumped out and sealed with torch. Samples of NBS Ox-II standard, IAEA-C3 (cellulose), and fossil wood background samples (IAEA-C9) were prepared and used to control the whole process of sample preparation and measurement (Janovics et al. 2018). After the sealed tube combustion in 550°C (12 hr), the CO_2 gas was released and purified in vacuum line and sealed again in tube containing zinc, titanium hydride

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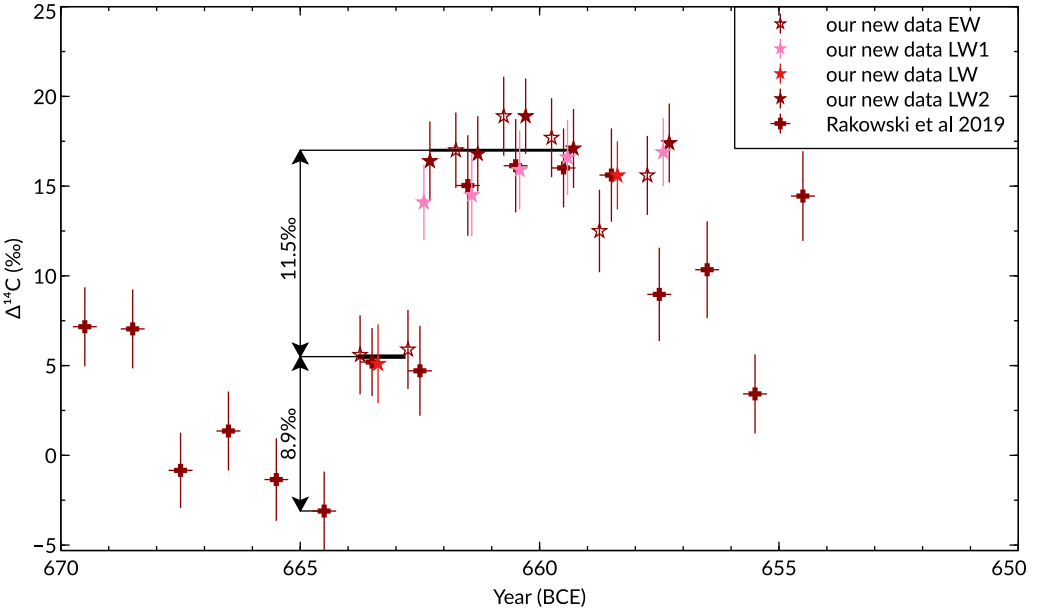

Figure 2 Results of $\Delta^{14}C$ determination in Grabie site tree. New results are shown as stars. Results from Rakowski et al. (2019) are shown as empty crosses.

and iron. A typical number of reagents and catalysts was used according to Rinyu et al. (2013). For sample mass between 0.5 to 1.5 mg, 10 mg of TiH_2, 60 mg of Zn, and 4.5 mg of iron powder was used. CO_2 consists of 2 steps where the tube is placed in the furnace for 3 hr, initially at 500° C for hydrogen release and iron reduction, then the temperature is raised to 550°C to reduce CO_2. This second stage takes approximately 5 hr. The produced graphite is then pressed into the cathode and measured using EnvironMICADAS AMS system at ATOMKI in Debrecen, Hungary (Molnar et al. 2016). The measurement time was extended to obtain more than 400.000 counts giving the overall uncertainties for modern samples better than ±2‰. Data acquisition was performed using BATS software (Wacker et al. 2010). The ^{14}C contents are reported as $\Delta^{14}C$ in per mil (‰) deviations from the standard sample, 0.7459 activity of NBS oxalic acid (SRM- 4990C). Age correction, isotopic composition correction ($\delta^{13}C$, measured by the AMS system), and $\Delta^{14}C$ values were calculated using formulas presented by Stuiver and Polach (1977).

RESULTS AND DISCUSSION

The measured results (pMC, $\Delta^{14}C$) with corresponding uncertainties are presented in Table S1. Figure 2 shows $\Delta^{14}C$ values in sub-annual (EW and LW) tree rings for the period 664–658 BCE as well as annual data from Rakowski et al. (2019). EW was shifted 3/12 years from the beginning of the year while LW was shifted by 7.5/12 years. The LW from annual rings have been split into two parts and shifted by 7/12 years (LW1) and 8.5/12 years (LW2) respectively. Earlier results from Rakowski et al. (2019), along with result from Park et al. (2017), Sakurai et al. (2020), IntCal20 calibration curve (Reimer et al. 2020) and IntCal20 raw datasets: HD, UB, UCIAMS, QL (http://calib.org) are presented in Figure 3.

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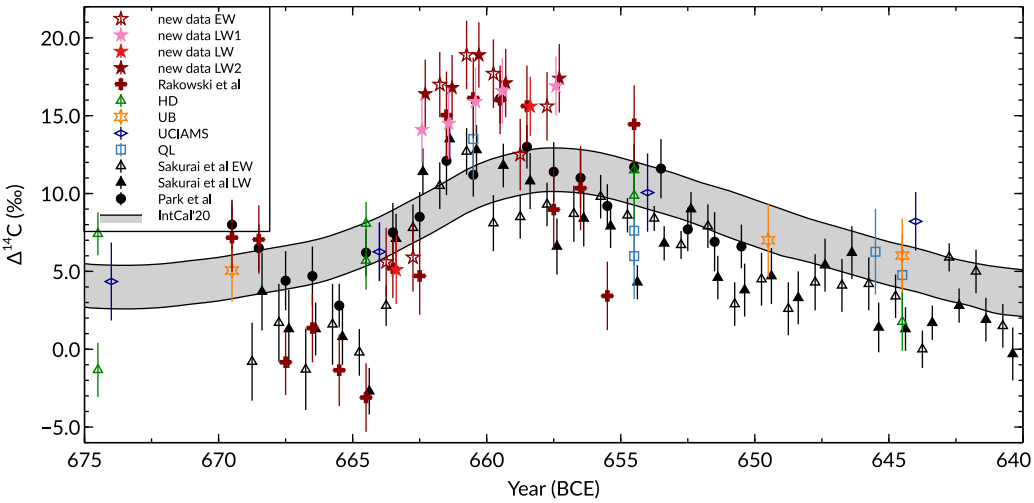

Figure 3 Results of $\Delta^{14}C$ determination in Grabie site tree. New data are shown as red stars. Our results from Rakowski et al. (2019) are shown as empty, red crosses. Sakurai et al. (2020) results are shown as black triangles. Park et al. (2017) results are shown as black bow ties. Another data (HD, UB, UCIAMS, and QL) were extracted from IntCal20 raw data sets (http://calib.org). IntCal20 with its uncertainty is shown as a gray-shaded curve. (Please see online version for color figures.)

The sub-annual data from the Grabie core G 58 EW and LW rings show a prolonged increase in the $\Delta^{14}C$ values of 20.4 ± 1.0‰ between 665 (data from Rakowski et al. 2019) and EW 661 BCE. The radiocarbon concentration ($\Delta^{14}C$) in this period raised from –1.3 ± 2.3‰ in 665 BCE to 19.1±2.2‰ in EW from 661 BCE showing high value of radiocarbon concentration during the rest of measured period. Similar to the literature the $\Delta^{14}C$ curve shows first the rapid increase of about 8.3 ± 2.8‰ between 665 and 664 BCE, and then gradual increase to the maximum value of 19.1 ± 2.2‰ in EW in 661 BCE. Park et al. (2017) and Sakurai et al. (2020) reported increase in $\Delta^{14}C$ value of 14.3 ± 1.5‰ in German oak series within 4 yr, and 13.3 ± 2.1‰ in Choukai-Jindai cedar within 6 yr, respectively. The difference in radiocarbon concentration ($\Delta^{14}C$) in LW from 665 BCE and 664 BCE presented in literature is 8.9 ± 0.4‰ (Park et al. 2017), and 9.8 ± 2.2‰ (Sakurai et al. 2020) and is similar to the value obtained in this study.

The $\Delta^{14}C$ values measured in LW from 664 BCE (5.6 ± 2.2‰), and in EW from 663 BCE (6.0 ± 2.1‰), show no significant differences. This is because, to a significant extent, the EW is formed from the material accumulated in the previous year and a much smaller amount of it derives from the material created by photosynthesis in the year of growth (Speer 2010). Olsson and Possnert (1992) observed that there is no delay between the radiocarbon concentration in the atmospheric sample in comparison with its concentration in the EW and LW during the maximum bomb effect (1962–1963). According Ermich (1959), EW formation in this climatic zone occurs from April to mid-June, and LW formations ends in mid-September. This limits the beginning of the phenomenon to the period before formation of the LW, as there is no significant difference between $\Delta^{14}C$ values in LW1 and LW2 in year 664 BCE. The most likely period of occurrence is May–June of year 663 BCE.

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O'Hare et al. (2019) confirmed the origin of the cause of those changes. Based on the high-resolution data sets of ^{14}C (tree rings), and ^{10}Be and ^{36}Cl (ice core) the production rate for those isotopes during this period was estimated, which led to the conclusion that SEP with a very hard energy spectrum triggered this phenomenon. The 660 BCE event was an order of magnitude stronger than any solar events observed during the industrial era and was comparable to the 774/775 CE event.

Sakurai et al. (2020) estimated the net production of ^{14}C to (1.3–1.5) $\times$ 10^8 atoms/cm^2 and is around 40% smaller than the net production during the event 774/775 CE (2.2 $\times$ 10^8 atoms/cm^2), but around 15% higher than the 993/994 CE event, when the net production was 1.2 $\times$ 10^8 atoms/cm^2. To compare, the net production rate for the solar events SPE56 and SEP72 were 2.6 $\times$ 10^6 atoms/cm^2 and 6.2 $\times$ 10^5 atoms/cm^2.

The production rate of ^{14}C for the increases in those two events of 664 BCE and 663 BCE estimated using an 11-box carbon cycle model (Güttler et al. 2015) was ~1.2 $\times$ 10^8 atoms/cm^2 and ~1.3 $\times$ 10^8 atoms/cm^2, respectively. For this estimation, LW data for 664–660 BCE were used for the estimation on the latter event. Please note we took weighted means for the LW1 and LW2 when available (Table S1, S2). For the first event, we used the yearly data of 665–664 BCE obtained by Rakowski et al. (2019; Table S2).

CONCLUSIONS

A new high-resolution set of radiocarbon data from the sub-annual oak tree rings of Grabie, southern Poland, is presented in this paper. This radiocarbon concentration dataset from the EW and LW rings covers the SEP event around 660 BCE. This new data record shows a prolonged increase in $\Delta^{14}C$ between 665–663 BCE. It started with an increase of 8.9 ± 2.8‰ in $\Delta^{14}C$ between 665 and 664 BCE (Rakowski et al. 2019), and lasted until 661 BCE, which is similar (and in agreement) to previously reported observations. In comparison, the radiocarbon data collected for this period presented by Park et al. (2017) and Sakurai et al. (2020), provide comparable data patterns with a prolonged increase until 662 BCE. The most probable time for a major event, which caused a significant change in the radiocarbon concentration in the tree rings, is the late spring (May–June, before beginning of LW formation) of 663 BCE.

SUPPLEMENTARY MATERIAL

For supplementary material accompanying this paper visit https://doi.org/10.1017/RDC.2023.79

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