



HAL
open science

RADIOCARBON DATING OF GRASS-TEMPERED CERAMIC REVEALS THE EARLIEST POTTERY FROM SLOVAKIA PREDATES THE ARRIVAL OF FARMING

Peter Tóth, Jan Petřík, Penny Bickle, Katarína Adameková, Solène Denis, Karel Slavíček, Libor Petr, Dalia Pokutta, Sven Isaksson

► **To cite this version:**

Peter Tóth, Jan Petřík, Penny Bickle, Katarína Adameková, Solène Denis, et al.. RADIOCARBON DATING OF GRASS-TEMPERED CERAMIC REVEALS THE EARLIEST POTTERY FROM SLOVAKIA PREDATES THE ARRIVAL OF FARMING. Radiocarbon, 2023, pp.1 - 21. 10.1017/rdc.2023.39 . hal-04125034

HAL Id: hal-04125034

<https://hal.science/hal-04125034v1>

Submitted on 11 Jun 2023

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.



Distributed under a Creative Commons Attribution 4.0 International License

© The Author(s), 2023. Published by Cambridge University Press for the Arizona Board of Regents on behalf of the University of Arizona. This is an Open Access article, distributed under the terms of the Creative Commons Attribution licence (<http://creativecommons.org/licenses/by/4.0/>), which permits unrestricted re-use, distribution and reproduction, provided the original article is properly cited.

RADIOCARBON DATING OF GRASS-TEMPERED CERAMIC REVEALS THE EARLIEST POTTERY FROM SLOVAKIA PREDATES THE ARRIVAL OF FARMING

Peter Tóth^{1*}  • Jan Petřík²  • Penny Bickle³  • Katarína Adameková²  • Solène Denis^{1,4}  • Karel Slavíček²  • Libor Petr⁵  • Dalia Pokutta^{1,6,7}  • Sven Isaksson⁶ 

¹Masaryk University, Department of Archaeology and Museology, Brno, Czech Republic

²Masaryk University, Faculty of Science, Department of Geological Sciences, Kotlářská 2, 611 37 Brno, Czech Republic

³University of York, Department of Archaeology, The King's Manor, York, YO1 7EP, UK

⁴CNRS, UMR 8068 TEMPS, MSH Mondes, Nanterre, France

⁵Masaryk University, Faculty of Science, Department of Botany and Zoology, Kotlářská 2, 611 37 Brno, Czech Republic

⁶Stockholm University, Department of Archaeology and Classical Studies, Stockholm, Sweden

⁷University of Rzeszów, Institute of Archaeology, Poland

ABSTRACT. In the absence of wood, bone, and other organics, one possible candidate for determining the age of a site is the radiocarbon (^{14}C) dating of pottery. In central Europe during the Early Neolithic, pottery was ubiquitous and contained substantial quantities of organic temper. However, attempts at the direct dating of organic inclusions raises a lot of methodological issues, especially when several sources of carbon contribute to the resulting radiocarbon age. Hence an alternative approach to dating of the early pottery is necessary. Here, we present a novel method of bulk separation of organic content from the grass-tempered pottery from Santovka (Slovakia). The procedure is based on the consecutive application of three inorganic acids, dissolving clay, silica content, and low molecular or mobile fractions to separate organic inclusions added to the pottery matrix during the formation of vessels. Radiocarbon dates obtained with this method are coherent and produce the shortest time span compared to other pretreatment methods presented in this study. The paired dates of grass-tempered pots with the ^{14}C age of lipids extracted from the same pots point to a difference of 400–600 ^{14}C yr, however they are in line with the site's chronostratigraphic Bayesian model. Grass-tempered pottery from Santovka (Slovakia) is dated to the first half of the 6th millennium cal BC, making it the earliest pottery north of the Danube. It seems feasible that ceramic containers from Santovka were produced by hunter-gatherers, and pottery predated the arrival of farming in the Carpathian region by a couple of centuries.

KEYWORDS: Bayesian modeling, lipids, organic temper, pottery vessels, radiocarbon dating, Slovakia.

INTRODUCTION

Radiocarbon (^{14}C) dating is one of the most common methods for determining the age of organic material (Bayliss 2009; Strydonck 2017). Unfortunately, materials typically utilized for ^{14}C dating are not always recovered during archaeological excavations, therefore the chance to date pottery, one of the most ubiquitous archaeological finds from the early Neolithic, is particularly promising (e.g., Casanova et al. 2020; Teetaert et al. 2020). In the Early Neolithic of Central Europe (first half of the 6th millennium BC), the pottery contains an abundance of organic temper, which makes it a suitable candidate for ^{14}C dating (Quitta 1960; e.g., Bente et al. 2019; Sauer 2019).

Applicability of direct ^{14}C dating of pottery, however, depends on the origin of organic carbon. Previous research demonstrated that several sources of carbon contribute to the resulting ^{14}C date of pottery: clay, temper, vessel use, carbon from fuel deposited as soot on vessel surface and depositional environment (Atley 1980; Gabasio et al. 1986; Johnson et al. 1986; Evin et al. 1989; Hedges et al. 1992; Nakamura et al. 2001; Stott et al. 2001; Mihara et al. 2004; Anderson et al. 2005; Zaitseva et al. 2009; Goslar et al. 2013; Teetaert et al. 2020). The first experiments with directly dating the ceramic material were conducted in the 1960s (Ralph 1959; e.g., Evans and Meggers 1962; Stuckenrath 1963). They were based on the assumption that organic

*Corresponding author Email: peter.toth@phil.muni.cz

material in the pottery paste results from cultural activity. During these initial experiments, it was discovered that sherds with an extremely small amount of organic carbon (<0.6%) produced questionable dates (Atley 1980). Further works comparing ^{14}C ages and other independent dates from the same stratigraphic contexts pointed out that the resulting age could have been affected by non-cultural sources (Taylor and Berger 1968; e.g., Stäuble 1995).

The application of accelerator mass spectrometry (AMS) resulted in a breakthrough in enabling ^{14}C dating of isolated fractions, such as temper (Teetaert et al. 2020), lipids (Stott et al. 2001; Casanova et al. 2020), humics (Količ 1995; Mihara et al. 2004) or residual carbon (Hedges et al. 1992; Goslar et al. 2013). Experiments with the dating of residual carbon provided older than expected dates associated with the incorporation of geological carbon (Hedges et al. 1992). Application of hydrofluoric acid (HF) leaching led to similar results due to mobilisation of the old carbon from the raw clay (Goslar et al. 2013). By contrast, lipid material surviving in cooking pots can provide a ^{14}C age of the vessel usage, but freshwater/marine reservoir effect needs to be considered from cooking food (e.g., Fischer and Heinemeier 2003; Mihara et al. 2004; Boudin et al. 2010; Hartz et al. 2012; Miyata et al. 2016; Gauthier 2022). To avoid the old carbon from the clay, and increase the chance of reliable dates, it is better to isolate the charred organic temper remains from the pottery prior to ^{14}C dating (Hedges et al. 1992; Gomes and Vega 1999). This method has already been successfully applied to date grass temper (e.g., Bollong et al. 1993), moss tempers (Gilmore 2015) and accidental inclusions of organic macrofossils in pottery (Arobba et al. 2017).

In this paper, we present a novel method of bulk separation of organic content from the grass-tempered pottery and the results of direct AMS ^{14}C dating of the clay vessels from Santovka (Slovakia). Due to the presence of the mineral thermal springs, this territory was a centre of human activities from the Palaeolithic, with high intensity during the Neolithic and the Bronze Age (Bárta 1961; Batora et al. 2015; Šolcová et al. 2018). Previous palaeoecological research of Santovka led to the discovery of stratified sequence of prehistoric pottery (Šolcová et al. 2018). The lowermost finds from calcareous lake sediments represent grass-tempered pottery, which stylistically and technologically does not correspond to the Early Neolithic pottery known from the area (ca. 5600–5300 cal BC; Jakucs et al. 2016). We test the hypothesis that grass-tempered pots represent the earliest pottery north of the Danube. In order to achieve this, we aim to (1) ^{14}C date the grass temper of the pottery from Santovka, (2) compare the resulting ^{14}C dates with ^{14}C dates produced on lipids extracted from the same pottery, and (3) test the accuracy of direct ^{14}C dating of pottery by a chronostratigraphic Bayesian model.

MATERIAL AND METHODS

The Santovka site is located 120 km east of Bratislava (Slovakia) at the transition of the Pannonian Basin and Western Carpathians (E18.7692, N48.1538; WGS84; Figure 1; Supplement 1.1). The excavated section is situated on the right bank of the Búr brook, close to one of the travertine accumulations, at 140 m a.s.l. and is formed of organic-rich carbonate sediments. The section was monitored in 2012–2014 through standard archaeological methods (cleaning, photographic documentation, photogrammetry). During the fieldwork, a sequence of 24 archaeological layers was uncovered and classified into ten lithostratigraphic units (Figure 2; Table 1). During these campaigns, 86 artefacts (such as pottery, animal and human bones, daub, lithics) were collected and documented, including 25 fragments of an unusual hitherto unknown type of grass-tempered pottery.

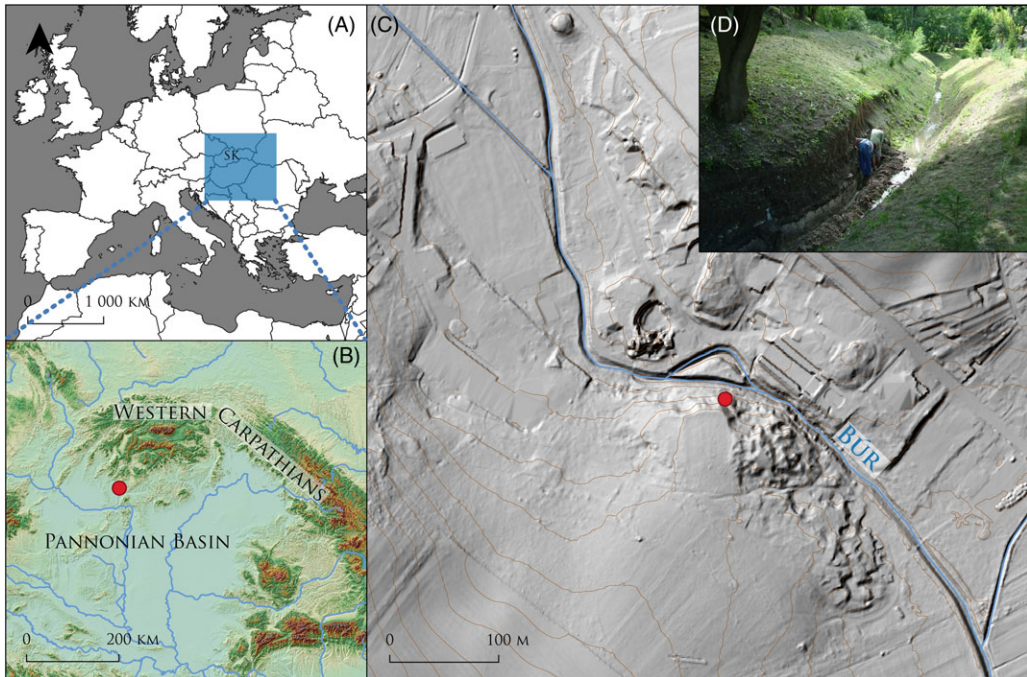


Figure 1 A and B: Location of the Santovka site in a wider geographic context; C: location of the documented section on the right bank of the Búr brook; D: fieldwork campaign in 2012. Data sources: A–B—Natural Earth; C—ÚGKK SR.

Cultural-chronological classification of the pottery was carried out based on Pavúk (1969, 1980, 2018), Nikitin et al. (2019), Bátora (2018), and Furmánek (2015), considering the technological and typological elements of the pottery and its stratigraphic position. Thin-sections of 30 µm thickness were prepared from the grass-tempered pottery. Organic temper was studied in transmitted light under a polarising microscope Olympus BX 51. Photographic documentation was carried out with a Canon 40D camera.

To determine the age of this pottery, we selected 8 pieces of grass-tempered pottery and 1 organic residue from the pottery surface. Six pottery sherds were analyzed in the Beta Analytic (USA). The laboratory used the acid-alkali-acid (AAA) pretreatment (on whole pottery matrix) to remove possible contaminants by humic acids and dated the resulting organic material (De Vries and Barendsen 1954). The measurements were conducted in the NEC accelerator mass spectrometer, whereas the carbon ratios were measured in the ThermoFinnigan Delta IRMS machine. The organic residue was submitted to the Centre for Applied Isotope studies, University of Georgia, USA (UGAMS). The sample was pretreated by AAA wash method in the laboratory, the high precision measurement of ¹⁴C/¹²C ratio was conducted in 500 kV NEC 1.5SDH-1 pelletron in tandem with an accelerator equipped with a 134-cathode MC-SNICS negative ion source.

For direct dating of organic inclusions we developed a novel triple acid wash method. Three sherds of around 1 cm³ size were pretreated by an adaptation of a method which is used for pollen separation (Moore et al. 1991). The principle of triple acid pretreatment method (Figure 3) is based on reducing pottery clay mass by using inorganic acids dissolving clay, silica

Table 1 Lithostratigraphic development of the section in Santovka and archaeological chronology. P—pottery; B—bone; L—lithics; D—daub; Pre-N—pre-Neolithic; N—Neolithic; BA—Bronze Age (Data sources: Šolcová et al. 2018; Petřík et al. in prep.).

Depth (cm)	Lithostratigraphy	Archaeological layers	Archaeological finds	Archaeological chronology
< 115	Unit 1, 2a, 2b	1–9, 24	P (16), B (3), L (2)	BA
115–139	Unit 2c	10, 22	P (11), B (5)	BA, N (Late LBK)
139–149	Unit 3	11, 12	P (13), B (10), D (4)	N (Late LBK), Pre-N
149–152	Unit 4	13–15	P (4), B (2), D (1), L (1)	N (Late LBK), Pre-N
152–186	Unit 5a	16, 17, 26	P (12), B (2)	Pre-N

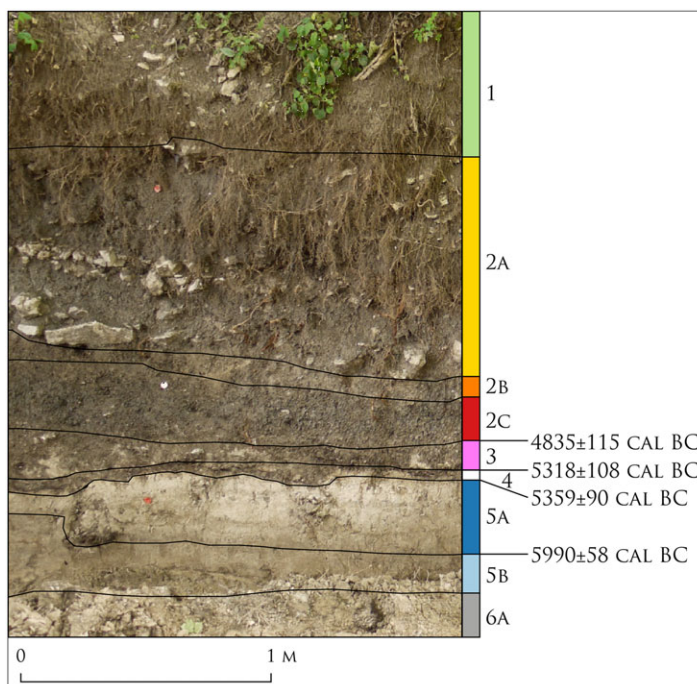


Figure 2 Stratigraphy sequence of the southern section of the Búr creek showing the position of the lithostratigraphic units. Only grass-tempered pottery was found in unit 5a. Displayed dates are based on the chronostratigraphic Bayesian model (Supplement 1.2, 1.3). The complete cross-section of the Búr creek with the position of ^{14}C dated pottery is shown in Supplement 1.1.

contents, carbonates and low molecular or mobile organic fractions. Samples were crushed in a mortar to a fraction close to fine powder. Crushed material was transferred to the PET tube where hydrofluoric acid (100 mL, concentration 38–40%, per analytic, further as p. a.) was carefully added. This process is followed by a strong exothermic reaction which removes silicate minerals. Subsequently, centrifugation (3000 rpm, 180 s) in ultra-distillate water was repeated two times to homogenise samples. Afterwards, tubes were put into the centrifuge

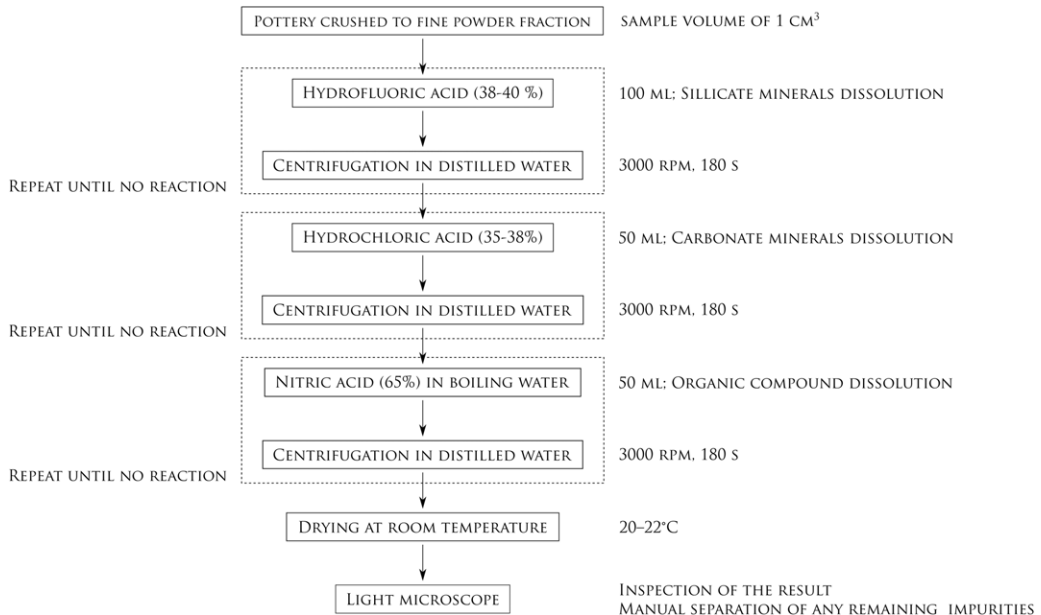


Figure 3 Principle of the novel triple acid wash method.

(3000 rpm, 180 s) and treatment with HF (100 mL, concentration 38–40%, p. a.) was repeated until a reaction had stopped. Carbonates were then removed using hydrochloric acid (50 mL, concentration 35–38%, p. a.) until a reaction was running. The fourth centrifugation (3000 rpm, 180 s) followed after any reaction was over. Following that, removal of small organic molecules using nitric acid (50 mL, concentration 65%, p. a) in boiling water was conducted. Resulting organic residues were inspected by a light microscope (400× magnification, Olympus BX51) and inorganic content had been excluded. Lastly, samples were slowly dried at room conditions, packed and submitted to the ¹⁴C dating to Isotopech Zrt. laboratory in Debrecen (Hungary), where they were further purified by standard laboratory protocols (Bird et al. 1999, 2003; ABOX pretreatment; Bird 2013). The AMS measurements were performed in Mini Carbon Dating System MICADAS.

For direct dating of lipids, the potsherds were first investigated for lipid residues using standard solvent extraction procedures for molecular analyses (c.f. Isaksson and Hallgren 2012) and acid catalysed extraction and methylation (Eggers and Schwudke 2016) for compound specific stable carbon isotope analysis of palmitic and stearic acid (Papakosta et al. 2015). The GCMS and GC-C-IRMS analyses were performed first. Based on these results samples were selected for the second extraction of lipid residues for ¹⁴C dating. For this purpose a modified version of the so-called Folch's method was used (Folch et al. 1957; Eggers and Schwudke 2016; Llewellyn and Isaksson, *in press*). The dried and purified lipid extracts were blown down to a few hundred microliters under a gentle stream of nitrogen gas. The highly concentrated extracts were then pipetted directly into preweighed tin foil capsules for ¹⁴C dating and very carefully blown down to dryness. In order to maximize the removal of solvents (Casanova et al. 2018: 11028) the tin foil capsules were heated to 70°C, i.e., ca. 10°C above the boiling point of chloroform, for 1 hr. The tin foil capsules were then allowed to cool and weighed to check the lipid residue yields before being sent to the Mass Spectrometry Laboratory, Center for Physical Sciences and Technology in Vilnius, Lithuania (Vilnius ¹⁴C Laboratory), for ¹⁴C analysis.

Extracted lipids were graphitized directly with Automated Graphitization Equipment AGE-3 (IonPlus AG). The AMS measurement was performed in a 240 KV Single Stage Accelerator Mass Spectrometer at the Vilnius Radiocarbon Laboratory. The background of measurements is approximately 2.45×10^{-3} fM (fraction of modern carbon) using phthalic anhydride. As reference materials were used the IAEA-C2, IAEA-C3, IAEA-C7, IAEA-C9, NIST OXII, SIRI K (carbonate) standards. The $^{14}\text{C}/^{12}\text{C}$ ratio is measured with an accuracy better than 0.3% (± 30 yr or better; Vilnius Radiocarbon 2022).

Calibration and combination of ^{14}C dates was undertaken using the program OxCal v4.4 (Bronk Ramsey 2009) and the IntCal20 calibration curve (Reimer et al. 2020). A chronostratigraphic Bayesian model with an outlier analysis (Supplement 1.4 and 1.5; adapted after Petřík et al. 2022) was used to test whether ^{14}C dates from the grass-tempered pottery agree with the sedimentation sequence on the site. For this modeling, as input data were used ^{14}C dates of grass temper and lipids (Table 3), published ^{14}C dates from site's stratigraphic sequence (Šolcová et al. 2018) and data acquired from the age-depth model, which represent an expected age based on sample's depth (Supplement 1.2, 1.3; Petřík et al. in prep.). Age-depth model data consists of top and bottom of lithostratigraphic unit 5a, where only grass-tempered pottery was located, as well as from surrounding units (4 and 5b) in order to estimate the start and end of the pottery tempered with grass. At the same time we also made a phase model for the grass-tempered pottery. The dates judged most reliable (agreement index is above 60%) were then selected for chronological modeling (KDE model) to determine a likely span of dates for pottery sherds tempered with grass.

RESULTS

Relative Chronology of the Site and Macroscopic Description of Pottery

Human presence was documented in 7 lithostratigraphic units (Table 1). Upper units (1, 2a, 2b, 2c) contain ceramic material classified as the Early Bronze Age. Unit 2c included a mixed material attributed to the Early Bronze Age and Neolithic (LBK, an abbreviation to Linearbandkeramik or Linear Pottery Culture). Units 3 and 4 contained LBK pottery with several animal bones, human skull, and daub. The unit 5a contained only grass-tempered pottery. Due to post-depositional processes and later settlement activities, LBK ceramics were mixed with the newly discovered grass-tempered pottery in these lithostratigraphic units. The unit 5a contained only grass-tempered pottery (Figure 4).

Reconstruction of the original vessel forms was not possible, however sherds K8-2 and K8-5 come from the same vessel. None of the grass-tempered pottery bears any signs of decoration enabling typological identification or cultural attribution. The fragments are very fragile. The clay contains a high amount of organic temper, grass stems and leaves (*Festuca* sp.; Figure 5), that also regularly appear on the smoothed surface of the pottery.

Lipid Analysis

The results of the lipid residue analysis of the three samples selected for ^{14}C dating are presented in Table 2 and Figure 6. These samples were selected since they had good yields of lipids with a distribution of components characteristic of ancient lipid residues and showed very little evidence for recent contamination in the resulting chromatograms and mass spectra. The recovered lipid residues are dominated by a distribution of saturated fatty acids dominated by palmitic acid (C16:0) and the stearic acid (C18:0). All samples contained a distribution of

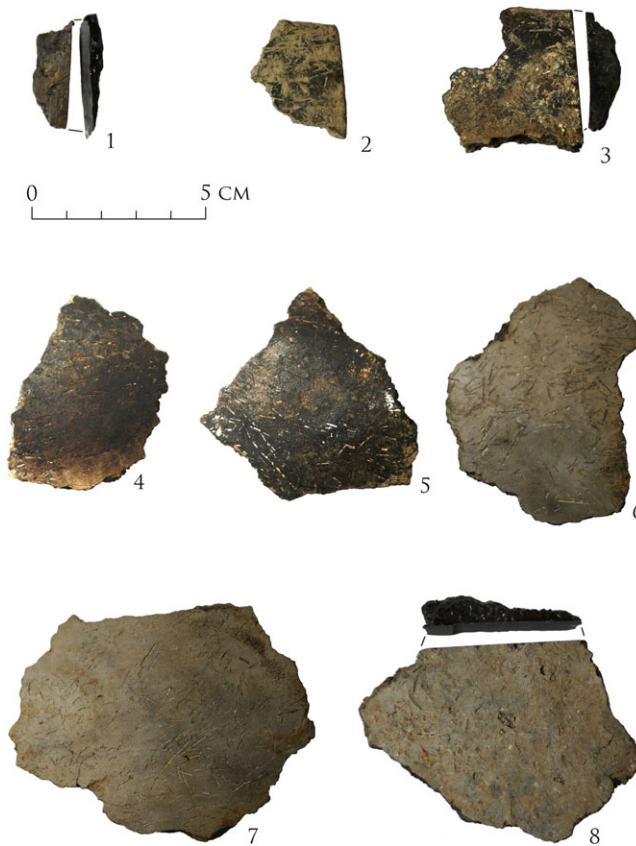


Figure 4 Grass-tempered pottery from Santovka (Slovakia) analyzed in this study. 1—K23/2014, unit 3; 2—K4/2014, unit 5a; 3—K10/2014, unit 5a; 4—K8/2012, sherd 5a, unit 5a; 5—K8/2012, sherd 3, unit 5a; 6—K8/2012, sherd 5b, unit 5a; 7—K8/2012, sherd 2, unit 5a; 8—K11/2014, unit 3.

branched fatty acids that can derive from ruminant animal sources or from microbial sources. There is no molecular evidence (dicarboxylic acids, isoprenoid alkanolic acids or ω (*o*-alkyl phenyl)fatty acids for aquatic lipid residues in these samples. Sample K8-2 and K10 do contain possible traces of the C18 ω (*o*-alkyl phenyl)fatty acid but that is not alone enough evidence for contribution from aquatic animal lipids. The stable carbon isotope values of the two dominating fatty acids (C16:0, C18:0) clearly indicate a primarily terrestrial origin for these fatty acids. From the molecular analysis of the lipid extracts there is evidence for a potential “smoke/soot” effect on sherd K10; the rest of the sherds are without the evidence of diterpenoids.

¹⁴C Dating of Grass-Tempered Pottery and Chronostratigraphic Bayesian Modeling

Eight pieces of grass-tempered pottery from lithostratigraphic units 2c, 3, 4, and 5a were selected for AMS ¹⁴C dating, returning 14 results. Results of dating by material (organic temper, organic residue and lipids extracted from the pottery) and pretreatment method (triple

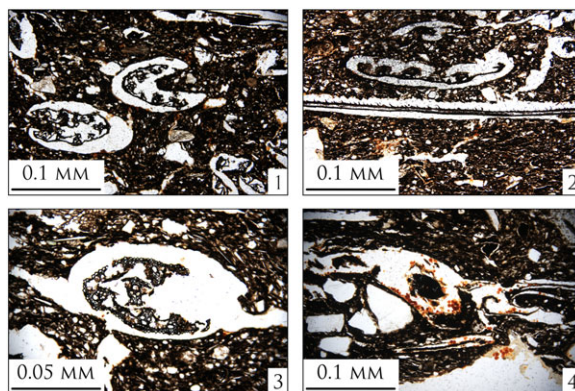


Figure 5 Thin section of grass-tempered pottery from Santovka (Slovakia) in this study. Fine-grained pottery matrix containing leaves of *Festuca* sp. added as a temper during the formation of vessels. 1—K4/2014 (figure 4:2); 2—K10/2014 (figure 4:3); 3—K8, sherd 2 (figure 4:7); 4—K11/2014 (figure 4:8).

acid, AAA) are presented in Table 3. The AAA method (7 dates), produced the widest span of dates, ranging from 7310 ± 30 BP to 5810 ± 30 BP. ^{14}C age of the organic residue taken from one the grass-tempered vessel (UGAMS 19701), purified by the AAA method, is 6470 ± 50 BP. The triple acid method (3 dates), produced a more restricted range of dates ranging from 6668 ± 49 BP to 6449 ± 33 BP. The dating of the lipids (3 dates) provided a time span from 7201 ± 35 BP to 6874 ± 34 BP.

Sherds K4, further K8-2 and K8-5 (multiple sherds from the same vessel), K9 and K9B (two fragments from the same sherd) and K10 were subjected to multiple ^{14}C dating, testing various pretreatment methods and dated materials. In the cases of K4, K8-2, K8-5 and K10, the lipids provided much earlier ^{14}C date than dating organic temper. Dates DeA-24370 and DeA-24372 extracted from the organic temper (sherds K8-2 and K8-5) and pretreated by triple acid wash method are very close to each other, although a combination of ^{14}C dates produced a poor agreement ($A_{\text{comb}}=13.7\%$). Dates Beta-425294 and Beta-434617 extracted from the organic temper (sherds K9 and K9B) and pretreated by AAA wash method are much more distant from each other; a combination of dates returned a poor agreement ($A_{\text{comb}}=0.0\%$). These results produce a wide span of dates, with the AAA extraction method showing the most disparate range.

We carried out a chronostratigraphic Bayesian model (Figure 7, Supplement 1.4, 1.5) to test how well the ^{14}C dates from grass-tempered pottery fit within the stratigraphic sequence of the site. Based on the model, the lithostratigraphic unit 5a with only grass-tempered pottery was sedimented after 6152–5882 cal BC (95% probability) and before 5476–5310 cal BC (95% probability). KDE model (Figure 7, Supplement 1.4) shows the grass-tempered pottery existed between 5896–5514 cal BC. From this time span we can reject Beta-429297, Beta-429296 and Beta-429295 whose agreement index is below 60% ($A_{\text{comb}}=5.4\%$, $A_{\text{comb}}=32.2\%$, $A_{\text{comb}}=5.5\%$ respectively) and chronologically do not fall within the site stratigraphy.

Table 2 Lipid residue analysis. Lipid content is given in mg lipid extracts per gram ceramic powder. FA describes the fatty acid distribution detected using the format n(m)k where n is the chain-length of the shortest fatty acid detected, m is the chain-length of the most abundant fatty acid in the distribution and k is the chain-length of the longest fatty acid detected. C18:0/C16:0 is the ratio of stearic to palmitic acid. BR is the carbon chain lengths registered for branched chained fatty acids. DA is dicarboxylic acids, OHFA is hydroxy-fatty acid, LCK is long-chain ketones, isoprenoid is isoprenoid fatty acids, APFA is ω (o-alkyl phenyl)fatty acids, $\delta^{13}\text{C}$ C16:0 and $\delta^{13}\text{C}$ C18:0 are the $\delta^{13}\text{C}$ -values of the palmitic and stearic acids, respectively. Δ is the difference in $\delta^{13}\text{C}$ -value between the palmitic and stearic acids ($[\delta^{13}\text{C}$ C18:0] – $[\delta^{13}\text{C}$ C16:0]). Presence/absence of diterpenoids for estimating the “smoke/soot” effect is marked as x/–.

Sample	mg/g	FA	C18:0/ C16:0	BR	DA	OHFA	LCK	Isoprenoid	Diterpenoid	APFA	$\delta^{13}\text{C}$ C16:0	$\delta^{13}\text{C}$ C18:0	Δ
K4	0.306	12(16)28	0.74	14–18	nd	nd	nd	nd	–	nd	–31.7	–35.6	–3.9
K8-2	0.408	12(16)26	0.91	14–17	nd	nd	nd	nd	–	c18?	–31.8	–36.4	–4.6
K10	0.366	12(16)24	0.93	14–18	nd	nd	nd	nd	x	c18?	–32.4	–36.8	–4.3

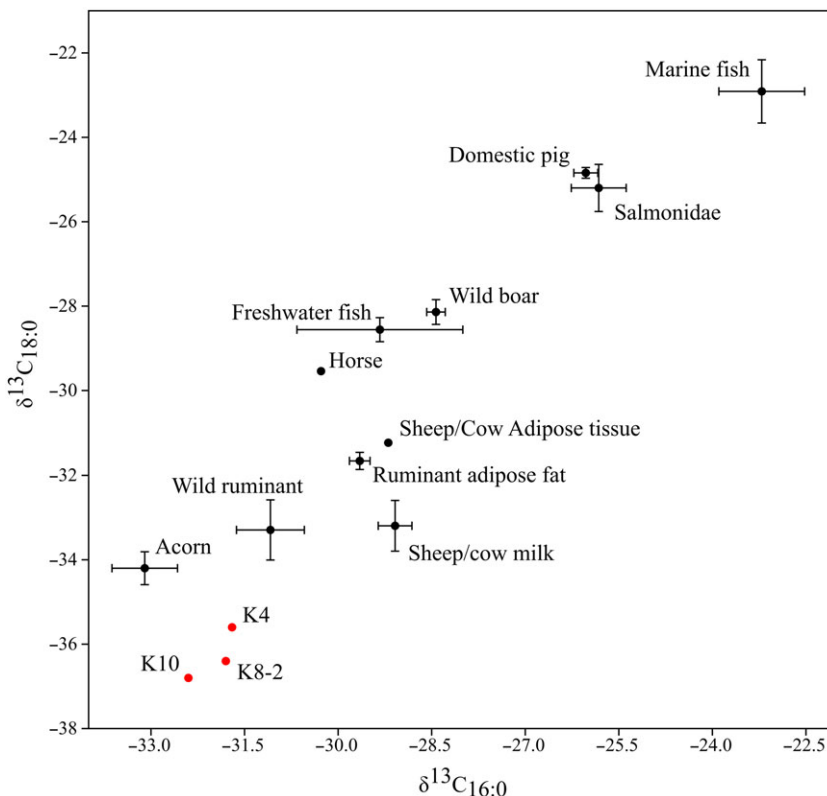


Figure 6 Plot of the $\delta^{13}\text{C}$ values of the major fatty acid components (C16:0 and C18:0) of grass-tempered pottery (red dots) compared to modern reference fats (black dots). Reference values were collected from published studies (Dudd et al. 1999:3; Copley et al. 2003:2; Craig et al. 2007:5; Lucquin et al. 2016:ST02) and represent average value (black dot), and the standard error displayed as an error bar.

DISCUSSION

Dating of the Grass Temper

There is a considerable chronological distance between ^{14}C data acquired from organic temper obtained by the AAA washing method and their expected age based on the age-depth model (Figure 7, Table 3). Beta-425296 and Beta-425297 are dated a few hundred ^{14}C yr earlier than expected, whereas Beta-429295 is dated later than the expected pottery age, showing a dispersal of almost 2000 ^{14}C yr (Table 3). It could be associated with unsuccessful removal of all possible contaminants from the grass-tempered pottery, which has been also demonstrated by past research, as humic acids, due to the high porosity of pottery, can resist the NaOH treatment usually recommended for their extraction (Gillespie et al. 1992; Količ 1995; Bird et al. 1999; Bird et al. 2003; Mihara et al. 2004; Anderson et al. 2005). The mentioned dates were also rejected by the chronostratigraphic Bayesian model due to low agreement index (<60%; Supplement 1.5). Only three dates (Beta-425293, Beta-425294, Beta-434617) fall within their expected age according to the age-depth model (combine test results: $A_{\text{comb}}=90.4\%$; $A_{\text{comb}}=121.7\%$, respectively; Supplement 1.5), however these dates are not reliable due to the pretreatment method and because they do not agree with the other dates from the same

Table 3 AMS ¹⁴C determination of organic temper, organic residue and lipids extracted from pottery. OT—organic temper; OR—organic residue; L—lipids extracted from the pottery (graphitized directly); 3A—triple acid method; AAA—acid-alkali-acid wash method. Where missing (–), specifications for δ¹³C (‰), pMC and C (%) were not available.

Laboratory number	Sample	Material	Litho-stratigraphy	Archaeo-logical layers	Depth (cm)	¹⁴ C age (BP)	Expected age based on age-depth model		δ ¹³ C (‰)	pMC	C (%)	Pre-treatment method
							¹⁴ C age (cal BC, 2σ range)	(cal BC, 1σ range)				
DeA-24370	K8-2	OT	5a	17	167.3	6449 ± 33	5479–5334	5781–5570	–26.2	—	—	3A
DeA-24372	K8-5	OT	5a	17	167.3	6668 ± 49	5666–5481	5781–5570	–27.1	—	—	3A
DeA-24373	K10	OT	4	15	160	6612 ± 39	5621–5480	5601–5439	–27.5	—	—	3A
Beta-429295	K10	OT	4	15	160	5810 ± 30	4775–4549	5601–5439	–26.9	—	—	AAA
Beta-425296	K11	OT	3	11	142	7310 ± 30	6228–6801	5028–4807	–25.6	—	—	AAA
Beta-425297	K23	OT	3	12	142	7760 ± 30	6648–6498	5028–4807	–26.2	—	—	AAA
Beta-425293	K4	OT	5a	17	164	6850 ± 30	5801–5661	5713–5500	–26.3	—	—	AAA
Beta-425294	K9	OT	5a	17	162	7060 ± 30	6012–5850	5656–5468	–26.1	—	—	AAA
Beta-434617	K9B	OT	5a	17	162	6670 ± 40	5661–5484	5656–5468	–24.7	—	—	AAA
UGAMS 19701	KB2	OR	2c	10	104	6470 ± 50	5525–5321	4459–4200	–26.7	44.71 ± 0.26	—	AAA
FTMC-YH59-6	K4	L	5a	17	164	7201 ± 35	6216–5988	5713–5500	—	40.80 ± 0.17	73.14	—
FTMC-YH59-7	K8-2	L	5a	17	167.3	6959 ± 34	5971–5741	5781–5570	—	42.05 ± 0.18	74.71	—
FTMC-YH59-9	K10	L	5a	17	160	6874 ± 34	5840–5669	5601–5439	—	42.50 ± 0.18	70.14	—

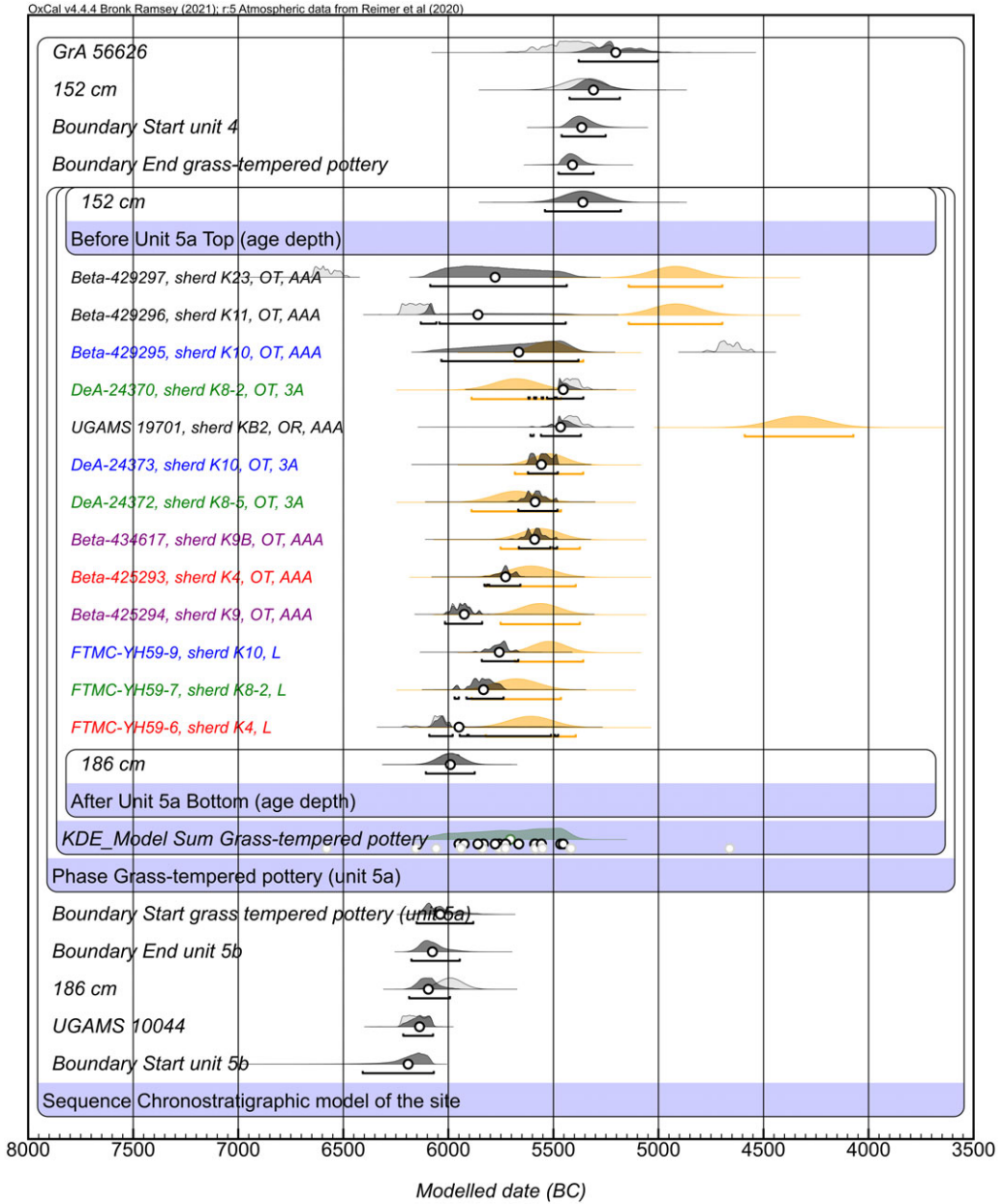


Figure 7 Chronostratigraphic Bayesian model of the site's stratigraphy based on ^{14}C data acquired from the grass-tempered pottery (Table 3), published ^{14}C dates from stratigraphic sequence (Šolcová et al. 2018) and data acquired from the age-depth model Supplement 1.2–1.5). Red—sherd K4; green—sherds K8-2 and K8-5; blue—sherd K10; purple—sherds K9 and K9B; light gray—unmodeled ^{14}C dates; dark gray—modeled dates from all the other sherds; orange— ^{14}C age of sherds based on age-depth model. OT—organic temper; OR—organic residue; L—lipids extracted from the pottery (graphitized directly); 3A—triple acid wash method; AAA—acid-alkali-acid wash method.

sherd. The last three mentioned dates were also confirmed by the chronostratigraphic Bayesian model, as their agreement index is above 60% (Supplement 1.5)

Three samples (DeA-24370, DeA-24372 and DeA-24373) were treated with the triple acid wash method proposed by the authors of this study. In all of these cases the dates correspond with a timeframe given by the age-depth and chronostratigraphic Bayesian models (Figure 7; Supplement 1.5). The difference between the AAA treatment and our triple acid method can be shown by sherd K10, from which two ¹⁴C dates were obtained (Figure 7). Sample from the grass-tempered pottery treated with the AAA method (Beta-429295) is by 800 yr younger than the sample prepared with our triple acid method (DeA-24373). The unsuccessful removal of contaminants due to the high porosity of pottery when applying the AAA pretreatment method might explain this discrepancy, as mentioned earlier.

Dating of the Lipids

The purpose of ¹⁴C dating of lipids was to provide an age which could be directly compared with the dating of the grass temper, since lipid material surviving in cooking pots can provide a ¹⁴C age of the vessel usage (Nakamura et al. 2001; Stott et al. 2001; Casanova et al. 2020; Robson et al. 2021). Lipid analysis of the samples K4, K8-2, K10 indicate that one or more types of substance were cooked in the vessels from Santovka (Table 2, Figure 6). However, in the current state of the research we can not clearly identify the source of the lipid signal. The difference (Δ) in $\delta^{13}\text{C}$ -value between the fatty acids of all three samples are well within the conventional range for lipid residues of terrestrial fats. However, $\delta^{13}\text{C}_{16:0}$ and $\delta^{13}\text{C}_{18:0}$ values are closer to wild ruminants (Craig et al. 2007:7; Papakosta et al. 2019:5; cf. Bondetti et al. 2021:4) or acorn (Lucquin et al. 2016:ST2). The stable carbon isotope values of the lipid residues fall to the lower end but are not separate from published distributions (e.g., Dudd et al. 1999:3; Copley et al. 2003:2; Craig et al. 2007:5, 2012:1; Lucquin et al. 2016:ST02).

¹⁴C age of lipids proved to be older by 400–600 ¹⁴C yr than the age of charred grass extracted from pottery by our triple acid method. This difference is even more pronounced when comparing both kinds of dates from sherds K8-2 and K10 (Figure 7). The carbon content (C (%)) in Table 3 of sample FTMC-YH59-6, FTMC-YH59-7 is within the expected range for lipids (72–79% C) while it is slightly low for FTMC-YH59-9. This could indicate recent contamination from chlorinated organic with very old carbon, e.g., PVC (ca. 38% C) or chloroform (ca. 10% C). Instead of compound specific approach we are exploring another approach using total lipid extracts, which considers further cleaning steps (such as filtration, ultra filtration and column chromatography). Following that approach, micro-particles of PVC could be suspended in the lipid extracts without being detected by the molecular analysis performed. This is more likely than the retention of the solvent chloroform. We can also exclude the freshwater reservoir effect as a result from the cooking of fish (e.g., Fischer and Heinemeier 2003; Mihara et al. 2004; Boudin et al. 2010; Hartz et al. 2012; Miyata et al. 2016). Either we can consider (1) an effect of depositional environment or firing of old wood (considering sherd K10 with traces of resinous material; Gabasio et al. 1986; Hedges et al. 1992; Bonsall et al. 2002; Mihara et al. 2004; Zaitseva et al. 2009), (2) the samples are contaminated by packing materials (e.g., PVC particles), (3) there are other methodological issues yet to be resolved, or (4) all of the above.

A comparison of ¹⁴C dated lipids with the age-depth model shows that only sample FTMC-YH59-7 corresponds with their expected age (combine test result: $A_{\text{comb}}=76.7\%$). Samples

FTMC-YH59-6 and FTMC-YH59-9 are older than their expected age specified by age-depth model (combine test results: $A_{\text{comb}}=1.3\%$; $A_{\text{comb}}=19.3\%$, respectively). However, all of these samples fall within the range of sedimentation of lithostratigraphic unit 5a, given by the chronostratigraphic Bayesian model (Supplement 1.5).

Comparing various pretreatment methods and ^{14}C dated materials, we judge that the most reliable procedure for pretreating the grass-tempered pottery was the triple acid method, showing an interval of 297 ^{14}C yr (68% probability: Table 4).

Grass-Tempered Pottery and the Origins of Ceramic Vessels in Central Europe

Absolute dating of grass-tempered pottery from Santovka (Slovakia) fits into the current debate regarding the spread of the Neolithic in Central Europe. According to traditional archaeological narrative, the earliest pottery arrives with first farming communities to migrate into the region (c.f. Bondetti et al. 2021; Nordqvist and Kriiska 2015). However, pottery tempered with grass chronologically precedes the emergence of the LBK over a larger area (Jakucs et al. 2016; 5625–5320 cal BC, 95% probability; 5565–5330 cal BC, 68% probability; Stadler and Kotova 2019, table 14.8; 5685–5370 cal BC). To date, there are only a few sites dated to the formative LBK, such as Brunn 2 near Vienna, Szentgyörgyvölgy-Pityerdomb and Zalaegerszeg-Andráshida (Simon 2002; Bánffy 2004; Oross and Bánffy 2009:1; Stadler and Kotova 2010; Stadler and Kotova 2019). The main characteristics of these sites is the absence of fine pottery and use of only coarseware with clear Starčevo elements (Nikitin et al. 2019). Whether there was a presence of the formative LBK north of the Danube has been debated largely from surface finds, but because of a lack of ^{14}C dates in this area, no conclusions could be drawn (Beljak Pažinová and Daráková 2019). Besides the formative LBK, the grass-tempered pottery from Santovka is broadly chronologically contemporary with Starčevo culture located south of lake Balaton in south-western Hungary (6070–5080 cal BC), and Körös culture (5790–5580 cal BC) from which the Alföld Linear pottery culture developed, particularly its Szatmár phase (5580–5250 cal BC) in the Tisza region of eastern Hungary (Stadler and Kotova 2019:table 14.4), all of them tempered with chaff (e.g., Gomart et al. 2020).

Given the nature of the grass-tempered pottery, its chronological position, cooking practices and the lack of evidence for human impact in the paleoecological record contemporary with the Pre-Neolithic pottery (Šolcová et al. 2018), we suggest that pots from Santovka were not produced by initial farming population coming from the south, but emerged in the Late Mesolithic context without any apparent links to the Neolithic cultures located south of the Central European-Balkan agro-ecological barrier (further as CEB-AEB; Bánffy and Sümegei 2012). Recent research demonstrated that using of pottery by hunter-gatherers suggests seasonal intensification of resource exploitation, broadening subsistence systems, new food traditions, increased sedentism associated with establishing new settlements at highly productive ecotones and population growth, which implies that pottery was under strong social control regulated by culinary practices and spread through a process of cultural transmission (Jordan and Zvelebil 2009; Nordqvist and Kriiska 2015; Oras et al. 2017; Bondetti et al. 2021; Courel et al. 2021; Dolbunova et al. 2022). A case from Rakushechny Yar (lower Don valley, Russia), chronologically contemporary with grass-tempered pottery from Santovka, suggests that (1) the knowledge of pottery production was transmitted through contact with farming communities and incorporated into foragers economy, or (2) early farmers moved to this region, but favored wild resources, or (3) pottery production was a local

Table 4 A comparison of time spans (unmodeled) for the pottery from Santovka based on different pretreatment methods and dating of lipids ($A_{\text{model}}=95.1$; $A_{\text{overall}}=96.4$) calculated in OxCal v.4.4. Input data is based on Table 3. OT—organic temper.

Method of extraction	Start Santovka (cal BC)				End Santovka (cal BC)				Interval (years)			
	95.4%		68.3%		95.4%		68.3%		95.4%		68.3%	
	From	To	From	To	From	To	From	To	From	To	From	To
AAA (OT)	6814	5853	6224	5898	5658	4649	5632	5310	247	1728	313	906
Lipids (direct graphitization)	6589	5738	5985	5752	5839	5042	5823	5604	0	1192	0	374
Triple acid wash (OT)	6354	5490	5726	5535	5621	4849	5607	5424	0	1064	0	297

innovation, or (4) was acquired from other hunter-gatherers (Bondetti et al. 2021). Different mechanisms are assumed for Kiçik Tepe (south Caucasus, Azerbaijan), again, from the same timeframe as Santovka pottery. Available archaeological evidence shows that local forager populations had contacts with farming groups during the process of Neolithisation, but these interactions did not cause an abrupt and full adoption of the Neolithic package. It suggests that pottery making technology was re-elaborated and adapted to better suit the needs and cooking practices of foragers (Nishiaki et al. 2015; Palumbi et al. 2021).

However, we might consider other possibilities, as well. The organic tempering at Santovka, yet again different in nature from other hunter-gatherer pottery, could also suggest some links with farming populations located south of CEB-AEB. Contacts between foragers and farmers across the CEB-AEB have remained frustratingly hard to identify, with possible glimpses argued for through lithic exchange networks or blade technology (Gronenborn 1990; 2003a, 2003b, 2007; Mateiciucová 2004, 2008) and a small contribution to the genetic history of the LBK (Lipson et al. 2017).

CONCLUSION

Our study shows that prehistoric vessels with organic temper can be successfully ^{14}C dated using appropriate pretreatment procedures. We developed and successfully applied a novel triple acid wash method to extract charred content from grass-tempered pottery, which is the most reliable method to treat this kind of archaeological material. The ^{14}C dates obtained are coherent and were successfully validated through several tests. In comparison to the triple acid method, ^{14}C data from lipids provided earlier age, which is an aspect that needs future attention.

Chronostratigraphic Bayesian modeling shows that grass-tempered pottery from Santovka was made between 5896–5514 cal BC. This time frame makes them the earliest pots north of the Danube, chronologically preceding the period characterised as the formative LBK, and technologically different from the fully developed Neolithic cultures of Danubian origin distributed south of the CEB-AEB.

The most probable explanation is that the grass-tempered pottery from Santovka developed within the context of Late Mesolithic hunter-gatherers. Comparative evidence in Prehistoric Eurasia shows that early pottery is fired in low temperatures, and is very diverse in terms of tempering practices, contrasting with early Neolithic cultures of the Danubian origin. However, the current evidence provides less resolution on the origins of this style of pottery at Santovka. Currently we propose two competing possible options: (1) knowledge of pottery production was acquired from other forager groups of Eurasian origin, or (2) concept of fired-clay containers was adapted from farming communities and was re-elaborated for the needs of forager groups while preserving their mobile way of life and subsistence patterns.

In the future we will further focus on the application of the triple acid wash method in dating Early Neolithic pottery from Slovakia, testing its potential and acquiring more ^{14}C dates, which are lacking in the region. More research is required on the mechanisms underlying the adoption of pottery and the development of pottery making technology during the Neolithic transition. The results presented here add further complexity to the transition to farming by indicating that pottery may predate the arrival of farming subsistence activities and, importantly, shows innovation and adaptation to a hunter-gatherer lifestyle.

ACKNOWLEDGMENTS

Research was funded by the project GAČR GA20-19542S (Tracing the Neolithic transition through the first pottery), Grant Agency of Masaryk University, project APVV-14-0550 (Natura et cultura. The coevolution of mankind and nature between 6th and 2nd millennium BC in the area north of the middle Danube studied on the basis of archaeological and environmental sources) and VEGA 1/0477/11 (Paleoecological reconstruction of the northern part of the Pannonian Plain). We want to express our gratitude to Juraj Lauko, who allowed us to conduct the field activities and collect archaeological material on his property. We want to thank Jozef Bátora for his continuous support during the fieldwork campaigns and initial ¹⁴C analysis of pottery material. We thank to Jack Dury for inspirational comments that improved the draft of the study. Lastly, we thank two anonymous reviewers whose comments and suggestions improved the final manuscript.

SUPPLEMENTARY MATERIAL

To view supplementary material for this article, please visit <https://doi.org/10.1017/RDC.2023.39>

REFERENCES

- Anderson A, Chappell J, Clark G, Phear S. 2005. Comparative radiocarbon dating of lignite, pottery, and charcoal samples from Babeldaob Island, Republic of Palau. *Radiocarbon* 47(1): 1–9. doi: [10.1017/S0033822200052139](https://doi.org/10.1017/S0033822200052139).
- Arobba D, Panelli C, Caramiello R, Gabriele M, Maggi R. 2017. Cereal remains, plant impressions and ¹⁴C direct dating from the Neolithic pottery of Arene Candide Cave (Finale Ligure, NW Italy). *Journal of Archaeological Science: Reports* 12:395–404. doi: [10.1016/j.jasrep.2017.02.015](https://doi.org/10.1016/j.jasrep.2017.02.015).
- Atley SPD. 1980. Radiocarbon dating of ceramic materials: progress and prospects. *Radiocarbon* 22(3):987–993. doi: [10.1017/S0033822200010419](https://doi.org/10.1017/S0033822200010419).
- Bánffy E. 2004. The 6th millennium BC boundary in western Transdanubia and its role in the Central European Neolithic transition, the Szentgyörgyvölgy-Pityerdomb settlement. Budapest: *Institut Archaeologici Academiae Scientiarum Hungaricae (Varia archaeologica Hungarica)*.
- Bánffy E, Sümegei P. 2012. The Early Neolithic agro-ecological barrier in the Carpathian Basin. A zone for interaction. In: Anreiter P, Bánffy E, Bartosiewicz L, Meid W, Metzner-Nebelsick C, editors. *Archaeological, cultural and linguistic heritage: festschrift for Erzsébet Jerem in honour of her 70th birthday*. Budapest: *Archaeolingua Alapítvány*. (Archaeolingua). p. 57–69.
- Bárta J. 1961. Nové poznatky o paleolitickom osídlení južného Slovenska. *Anthropos* 14: 167–171.
- Bátora J. 2018. Slovensko v staršej dobe bronzovej. Bratislava: Univerzita Komenského v Bratislave.
- Bátora J, Tóth P, Bača M. 2015. Výskumy opevnených sídlisk zo staršej doby bronzovej vo východnej časti Podunajskej nížiny. In: Bátora J, Tóth P, editors. *Keď bronz vystriedal meď*. Vol. 18. *Communicationes*. Nitra; Bratislava: Archeologický ústav SAV Nitra; Katedra archeológie FiF UK Bratislava. (Archaeologica Slovaca Monographiae). p. 139–154.
- Bayliss A. 2009. Rolling out revolution: using radiocarbon dating in archaeology. *Radiocarbon* 51(1):123–147. doi: [10.1017/S0033822200033750](https://doi.org/10.1017/S0033822200033750).
- Beljak Pažinová N, Daráková T. 2019. The state of early Linear Pottery Culture research in Slovakia. *Documenta Praehistorica* 46:184–202. doi: [10.4312/dp.46.12](https://doi.org/10.4312/dp.46.12).
- Bente K, Durini S, Küsel S, Kunert I, Keilholz S, Hölzig H. 2019. Firing conditions of LBK and SBK pottery from Eythra (Germany) by means of high-temperature analytics. In: Herm C, Merkel S, Schreiner M, Wiesinger R, editors. *Archäometrie und Denkmalpflege 2019. Jahrestagung an der Akademie der Bildenden Künste Wien. Institut für Naturwissenschaften und Technologie in der Kunst*, 11.-14. September 2019. Vol. 9. Bochum: Deutsches Bergbaumuseum Bochum. (METALLA Sonderheft). p. 167–170.
- Bird MI. 2013. Radiocarbon dating | Charcoal. In: *Encyclopedia of Quaternary Science*. Elsevier. p. 353–360. <https://linkinghub.elsevier.com/retrieve/pii/B9780444536433000479>.
- Bird MI, Ayliffe LK, Fifield LK, Turney CSM, Cresswell RG, Barrows TT, David B. 1999. Radiocarbon dating of “old” charcoal using a wet oxidation, stepped-combustion procedure. *Radiocarbon* 41(2):127–140. doi: [10.1017/S0033822200019482](https://doi.org/10.1017/S0033822200019482).
- Bird MI, Turney CSM, Fifield LK, Smith MA, Miller GH, Roberts RG, Magee JW. 2003. Radiocarbon

- dating of organic- and carbonate-carbon in Genyornis and Dromaius eggshell using stepped combustion and stepped acidification. *Quaternary Science Reviews* 22(15):1805–1812. doi: [10.1016/S0277-3791\(03\)00151-3](https://doi.org/10.1016/S0277-3791(03)00151-3).
- Bollong CA, Vogel JC, Jacobson L, van der Westhuizen WA, Sampson CG. 1993. Direct dating and identity of fibre temper in pre-contact Bushman (Basarwa) pottery. *Journal of Archaeological Science* 20(1):41–55. doi: [10.1006/jasc.1993.1003](https://doi.org/10.1006/jasc.1993.1003).
- Bondetti M, González Carretero L, Dolbunova E, McGrath K, Presslee S, Lucquin A, Tsybriy V, Mazurkevich A, Tsybriy A, Jordan P, et al. 2021. Neolithic farmers or Neolithic foragers? Organic residue analysis of early pottery from Rakushechny Yar on the Lower Don (Russia). *Archaeological and Anthropological Sciences* 13(8):141. doi: [10.1007/s12520-021-01412-2](https://doi.org/10.1007/s12520-021-01412-2).
- Bonsall C, Cook G, Manson JL, Sanderson D. 2002. Direct dating of Neolithic pottery: progress and prospects *Documenta Praehistorica* 29:47–59. doi: [10.4312/dp.29.5](https://doi.org/10.4312/dp.29.5).
- Boudin M, Strydonck MV, Crombé P, Clercq WD, Dierendonck RM van, Jongepier H, Eryvncq A, Lentacker A. 2010. Fish reservoir effect on charred food residue ¹⁴C dates: are stable isotope analyses the solution? *Radiocarbon* 52(2): 697–705. doi: [10.1017/S0033822200045719](https://doi.org/10.1017/S0033822200045719).
- Bronk Ramsey C. 2009. Bayesian analysis of radiocarbon dates. *Radiocarbon* 51(1):337–360. doi: [10.1017/S0033822200033865](https://doi.org/10.1017/S0033822200033865).
- Casanova E, Knowles TDJ, Bayliss A, Dunne J, Barański MZ, Denaire A, Lefranc P, di Lernia S, Roffet-Salque M, Smyth J, et al. 2020. Accurate compound-specific ¹⁴C dating of archaeological pottery vessels. *Nature* 580(7804):506–510. doi: [10.1038/s41586-020-2178-z](https://doi.org/10.1038/s41586-020-2178-z).
- Casanova E, Knowles TDJ, Williams C, Crump MP, Evershed RP. 2018. Practical considerations in high-precision compound-specific radiocarbon analyses: eliminating the effects of solvent and sample cross-contamination on accuracy and precision. *Analytical Chemistry* 90(18): 11025–11032. doi: [10.1021/acs.analchem.8b02713](https://doi.org/10.1021/acs.analchem.8b02713).
- Copley MS, Berstan R, Dudd SN, Docherty G, Mukherjee AJ, Straker V, Payne S, Evershed RP. 2003. Direct chemical evidence for widespread dairying in prehistoric Britain. *Proceedings of the National Academy of Sciences* 100(4):1524–1529. doi: [10.1073/pnas.0335955100](https://doi.org/10.1073/pnas.0335955100).
- Courel B, Meadows J, Carretero LG, Lucquin A, McLaughlin R, Bondetti M, Andreev K, Skorobogatov A, Smolyaninov R, Surkov A, et al. 2021. The use of early pottery by hunter-gatherers of the Eastern European forest-steppe. *Quaternary Science Reviews* 269:107143. doi: [10.1016/j.quascirev.2021.107143](https://doi.org/10.1016/j.quascirev.2021.107143).
- Craig OE, Forster M, Andersen SH, Koch E, Crombé P, Milner NJ, Stern B, Bailey GN, Heron CP. 2007. Molecular and isotopic demonstration of the processing of aquatic products in Northern European prehistoric pottery. *Archaeometry* 49(1):135–152. doi: [10.1111/j.1475-4754.2007.00292.x](https://doi.org/10.1111/j.1475-4754.2007.00292.x).
- Craig OE, Allen RB, Thompson A, Stevens RE, Steele VJ, Heron C. 2012. Distinguishing wild ruminant lipids by gas chromatography/combustion/isotope ratio mass spectrometry: Distinguishing ruminant lipids. *Rapid Communications in Mass Spectrometry* 26(19):2359–2364. doi: [10.1002/rcm.6349](https://doi.org/10.1002/rcm.6349).
- De Vries H, Barendsen GW. 1954. Measurements of age by the carbon-14 technique. *Nature* 174(4442):1138–1141. doi: [10.1038/1741138a0](https://doi.org/10.1038/1741138a0).
- Dolbunova E, Lucquin A, McLaughlin TR, Bondetti M, Courel B, Oras E, Piezonka H, Robson HK, Talbot H, Adamczak K, et al. 2022. The transmission of pottery technology among prehistoric European hunter-gatherers. *Nature Human Behaviour*: 1–13. doi: [10.1038/s41562-022-01491-8](https://doi.org/10.1038/s41562-022-01491-8).
- Dudd SN, Evershed RP, Gibson AM. 1999. Evidence for varying patterns of exploitation of animal products in different prehistoric pottery traditions based on lipids preserved in surface and absorbed residues. *Journal of Archaeological Science* 26(12):1473–1482. doi: [10.1006/jasc.1998.0434](https://doi.org/10.1006/jasc.1998.0434).
- Eggers LF, Schwudke D. 2016. Lipid extraction: basics of the methyl-tert-butyl ether extraction. In: Wenk MR, editor. *Encyclopedia of lipidomics*. Dordrecht: Springer Netherlands. p. 1–3. https://doi.org/10.1007/978-94-007-7864-1_96-1.
- Evans C, Meggers BJ. 1962. Use of organic temper for carbon 14 dating in lowland South America. *American Antiquity* 28(2):243–245. doi: [10.2307/278386](https://doi.org/10.2307/278386).
- Evin J, Gabasio M, Lefevre J-C. 1989. Preparation techniques for radiocarbon dating of potsherds. *Radiocarbon* 31(3). doi: [10.1017/S0033822200011802](https://doi.org/10.1017/S0033822200011802).
- Fischer A, Heinemeier J. 2003. Freshwater reservoir effect in ¹⁴C dates of food residue on pottery. *Radiocarbon* 45(3):449–466. doi: [10.1017/S003382220003280X](https://doi.org/10.1017/S003382220003280X).
- Folch J, Lees M, Stanley GHS. 1957. A simple method for the isolation and purification of total lipides from animal tissues. *Journal of Biological Chemistry* 226(1):497–509. doi: [10.1016/S0021-9258\(18\)64849-5](https://doi.org/10.1016/S0021-9258(18)64849-5).
- Furmánek V. 2015. Chronológia a periodizácia. In: Furmánek V, Bátora J, Ožďáni O, Mitáš V, Kujovský R, Vladár J, editors. *Staré Slovensko 4. Doba bronzová*. Nitra: Archeologický Ústav Slovenskej Akadémie Vied. (Archeologica Slovaca Monographiae Staré Slovensko). p. 14–15.
- Gabasio M, Evin J, Arnal GB, Andrieux P. 1986. Origins of carbon in potsherds. *Radiocarbon* 28(2A):711–718. doi: [10.1017/S0033822200007931](https://doi.org/10.1017/S0033822200007931).

- Gauthier MS. 2022. Using radiocarbon ages on organics affected by freshwater—a geologic and archaeological update on the freshwater reservoir ages and freshwater diet effect in Manitoba, Canada. *Radiocarbon* 64(2):253–264. doi: [10.1017/RDC.2022.30](https://doi.org/10.1017/RDC.2022.30).
- Gillespie R, Prosser IP, Dlugokencky E, Sparks RJ, Wallace G, Chappell JMA. 1992. AMS dating of alluvial sediments on the Southern Tablelands of New South Wales, Australia. *Radiocarbon* 34(1):29–36. doi: [10.1017/S0033822200013394](https://doi.org/10.1017/S0033822200013394).
- Gilmore ZI. 2015. Direct radiocarbon dating of Spanish moss (*Tillandsia usneoides*) from early fiber-tempered pottery in the southeastern U.S. *Journal of Archaeological Science* 58:1–8. doi: [10.1016/j.jas.2015.03.019](https://doi.org/10.1016/j.jas.2015.03.019).
- Gomart L, Anders A, Kreiter A, Marton T, Oross K, Raczky P. 2020. Innovation or inheritance? Assessing the social mechanisms underlying ceramic technological change in early Neolithic pottery assemblages in Central Europe. In: Spataro M, Furholt M, editors. *Detecting and explaining technological innovation in prehistory*. Leiden: Sidestone Press. (Scales of transformation). p. 49–71.
- Gomes DC, Vega O. 1999. Dating organic temper of ceramics by AMS: sample preparation and carbon evaluation. *Radiocarbon* 41(3):315–320. doi: [10.1017/S0033822200057179](https://doi.org/10.1017/S0033822200057179)
- Goslar T, Kozłowski J, Szmyt M, Czernik J. 2013. Effect of HF leaching on ¹⁴C dates of pottery. *Nuclear Instruments and Methods in Physics Research B* 294:252–256. doi: [10.1016/j.nimb.2012.09.028](https://doi.org/10.1016/j.nimb.2012.09.028).
- Gronenborn D. 1990. Mesolithic-Neolithic interactions—the lithic industry of the earliest Bandkeramik culture site at Friedberg-Bruchenbrücken, Wetteraukreis (West Germany). In: Vermeersch PM, Peer P van, editors. *Contributions to the Mesolithic in Europe: papers presented at the Fourth International Symposium, The Mesolithic in Europe*, Leuven, 1990. Leuven, Belgium: Leuven University Press. (*Studia praehistorica Belgica*). p. 173–182.
- Gronenborn D. 2003a. Lithic raw material distribution networks and the neolithization of Central Europe. In: Burnez-Lanotte L, editor. *Production and management of lithic materials in the European Linearbandkeramik*. Actes du XIV^{ème} Congrès UISPP, Université de Liège, Belgique, 2–8 septembre 2001. Oxford: British Archaeological Reports. (BAR International Series). p. 45–50.
- Gronenborn D. 2003b. Migration, acculturation and culture change in western temperate Eurasia. *Documenta Praehistorica* 30:79–91. doi: [10.4312/dp.30.3](https://doi.org/10.4312/dp.30.3).
- Gronenborn D. 2007. Beyond the models: “Neolithisation” in central Europe. In: Whittle A, Cummings V, editors. *Going over: the Mesolithic-Neolithic transition in north-west Europe*. Oxford: British Academy. p. 73–98. <https://doi.org/10.5871/bacad/9780197264140.003.0005>.
- Hartz S, Kostyleva E, Piezonka H, Terberger T, Tsydenova N, Zhilin MG. 2012. Hunter-gatherer pottery and charred residue dating: new results on early ceramics in the North Eurasian Forest Zone. *Radiocarbon* 54(3–4):1033–1048. doi: [10.1017/S0033822200047652](https://doi.org/10.1017/S0033822200047652).
- Hedges REM, Tiemei C, Housley RA. 1992. Results and methods in the radiocarbon dating of pottery. *Radiocarbon* 34(3):906–915. doi: [10.1017/S0033822200064237](https://doi.org/10.1017/S0033822200064237).
- Isaksson S, Hallgren F. 2012. Lipid residue analyses of Early Neolithic funnel-beaker pottery from Skogsmossen, eastern central Sweden, and the earliest evidence of dairying in Sweden. *Journal of Archaeological Science* 39(12):3600–3609. doi: [10.1016/j.jas.2012.06.018](https://doi.org/10.1016/j.jas.2012.06.018).
- Jakucs J, Bánffy E, Oross K, Voicsek V, Bronk Ramsey C, Dunbar E, Kromer B, Bayliss A, Hofmann D, Marshall P, et al. 2016. Between the Vinča and Linearbandkeramik worlds: the diversity of practices and identities in the 54th–53rd centuries cal BC in southwest Hungary and beyond. *Journal of World Prehistory* 29(3):267–336. doi: [10.1007/s10963-016-9096-x](https://doi.org/10.1007/s10963-016-9096-x).
- Johnson RA, Stipp JJ, Tamers MA, Bonani G, Suter M, Wölfli W. 1986. Archaeologic sherd dating: comparison of thermoluminescence dates with radiocarbon dates by beta counting and accelerator techniques. *Radiocarbon* 28(2A):719–725. doi: [10.1017/S0033822200007943](https://doi.org/10.1017/S0033822200007943).
- Jordan PD, Zvelebil M. 2009. Ex Oriente Lux: the prehistory of hunter gatherer ceramic dispersals. In: Jordan PD, Zvelebil M, editors. *Ceramics before farming: the dispersal of pottery among prehistoric Eurasian hunter-gatherers*. New York: Routledge. p. 33–89.
- Kolić ED. 1995. Direct Radiocarbon dating of pottery: selective heat treatment to retrieve smoke-derived carbon. *Radiocarbon* 37(2): 275–284. doi: [10.1017/S0033822200030745](https://doi.org/10.1017/S0033822200030745).
- Lipson M, Szécsényi-Nagy A, Mallick S, Pósa A, Stégmár B, Keerl V, Rohland N, Stewardson K, Ferry M, Michel M, et al. 2017. Parallel palaeogenomic transects reveal complex genetic history of early European farmers. *Nature* 551(7680):368–372. doi: [10.1038/nature24476](https://doi.org/10.1038/nature24476).
- Llewellyn M-H, Isaksson S., in press. Radiocarbon dating of purified lipid extracts from prehistoric pottery. *Fornvännen* 118(1).
- Lucquin A, Gibbs K, Uchiyama J, Saul H, Ajimoto M, Eley Y, Radini A, Heron CP, Shoda S, Nishida Y, et al. 2016. Ancient lipids document continuity in the use of early hunter-gatherer pottery through 9,000 years of Japanese prehistory. *Proceedings of the National Academy*

- of Sciences 113(15):3991–3996. doi: [10.1073/pnas.1522908113](https://doi.org/10.1073/pnas.1522908113).
- Mateiciucová I. 2004. Mesolithic traditions and the origins of the Linear Pottery Culture (LBK). In: Lukes A, Zvelebil M, editors. *LBK dialogues: studies in the formation of the Linear Pottery Culture*. Oxford, England: Archaeopress. (BAR International Series). p. 91–108.
- Mateiciucová I. 2008. Talking stones: the chipped stone industry in Lower Austria and Moravia and the beginnings of the Neolithic in Central Europe (LBK), 5700–4900 BC. Brno: Masarykova univerzita (Dissertationes Archaeologicae Brunenses/Pragensesque).
- Mihara S, Miyamoto K, Ogawa H, Kurosaka T, Nakamura T, Koike H. 2004. AMS ¹⁴C dating using black pottery and fiber pottery. *Radiocarbon* 46(1):407–412. doi: [10.1017/S0033822200039710](https://doi.org/10.1017/S0033822200039710).
- Miyata Y, Horiuchi A, Kondo M, Onbe S, Yoshida K, Nagao S, Group PLAD, Nishimoto T. 2016. Marine reservoir effects deduced from ¹⁴C dates on pottery residues, bones, and molluscan shells from the Hamanaka 2 archaeological site, Rebun Island, Hokkaido, Japan. *Radiocarbon* 58(4):755–770. doi: [10.1017/RDC.2016.93](https://doi.org/10.1017/RDC.2016.93).
- Moore PD, Webb JA, Collinson ME. 1991. *Pollen analysis*. Oxford: Blackwell.
- Nakamura T, Taniguchi Y, Tsuji S, Oda H. 2001. Radiocarbon dating of charred residues on the earliest pottery in Japan. *Radiocarbon* 43(2B): 1129–1138. doi: [10.1017/S0033822200041783](https://doi.org/10.1017/S0033822200041783)
- Nikitin AG, Stadler P, Kotova N, Teschler-Nicola M, Price TD, Hoover J, Kennett DJ, Lazaridis I, Rohland N, Lipson M, et al. 2019. Interactions between earliest Linearbandkeramik farmers and central European hunter gatherers at the dawn of European Neolithization. *Scientific Reports* 9(1):19544. doi: [10.1038/s41598-019-56029-2](https://doi.org/10.1038/s41598-019-56029-2).
- Nishiaki Y, Guliyev F, Kadowaki S. 2015. Chronological contexts of the earliest pottery Neolithic in the South Caucasus: radiocarbon dates for Göytepe and Hacı Elamxanlı Tepe, Azerbaijan. *American Journal of Archaeology* 119(3):279–294. doi: [10.3764/aja.119.3.0279](https://doi.org/10.3764/aja.119.3.0279).
- Nordqvist K, Kriiska A. 2015. Towards Neolithisation. The Mesolithic-Neolithic transition in the central area of the eastern part of the Baltic Sea. In: Kabaciński J, Hartz S, Raemaekers DCM, Terberger T, editors. *The Dąbki site in Pomerania and the neolithisation of the North European Lowlands (c. 5000–3000 calBC)*. Rahden/Westf: Leidorf. (Archäologie und Geschichte im Ostseeraum). p. 537–556.
- Oras E, Lucquin A, Lõugas L, Tõrv M, Kriiska A, Craig OE. 2017. The adoption of pottery by north-east European hunter-gatherers: Evidence from lipid residue analysis. *Journal of Archaeological Science* 78:112–119. doi: [10.1016/j.jas.2016.11.010](https://doi.org/10.1016/j.jas.2016.11.010).
- Oross K, Bánffy E. 2009. Three successive waves of Neolithisation: LBK development in Transdanubia. *Documenta Praehistorica* 36:175–189. doi: [10.4312/dp.36.11](https://doi.org/10.4312/dp.36.11).
- Palumbi G, Guliyev F, Astruc L, Baudouin E, Berthon R, D’Anna MB, Decaix A, Gratuze B, Hamon C, Poulmarc’h M, et al. 2021. New data and perspectives on the early stages of the Neolithic in the Middle Kura River Valley (South Caucasus). The 2017–2019 excavations at Kiçik Tepe, Western Azerbaijan. *Archaeological Research in Asia* 27:100308. doi: [10.1016/j.ara.2021.100308](https://doi.org/10.1016/j.ara.2021.100308).
- Papakosta V, Oras E, Isaksson S. 2019. Early pottery use across the Baltic—A comparative lipid residue study on Ertebølle and Narva ceramics from coastal hunter-gatherer sites in southern Scandinavia, northern Germany and Estonia. *Journal of Archaeological Science: Reports* 24: 142–151. doi: [10.1016/j.jasrep.2019.01.003](https://doi.org/10.1016/j.jasrep.2019.01.003).
- Papakosta V, Smittenberg RH, Gibbs K, Jordan P, Isaksson S. 2015. Extraction and derivatization of absorbed lipid residues from very small and very old samples of ceramic potsherds for molecular analysis by gas chromatography–mass spectrometry (GC–MS) and single compound stable carbon isotope analysis by gas chromatography–combustion–isotope ratio mass spectrometry (GC–C–IRMS). *Microchemical Journal* 123: 196–200. doi: [10.1016/j.microc.2015.06.013](https://doi.org/10.1016/j.microc.2015.06.013).
- Pavúk J. 1969. Chronologie der Želiezovce-Gruppe. *Slovenská archeológia* 17(2):269–367.
- Pavúk J. 1980. Ältere Linearkeramik in der Slowakei. *Slovenská archeológia* 28(1):7–88.
- Pavúk J. 2018. Balkán—Karpatská kotlina—Čechy: kultúrno-chronologický kontext. *Živá archeologie - (Re)konstrukce a experiment v archeologii* 20: 6–14.
- Petřík J, Adameková K, Kele S, Milovský R, Petr L, Tóth P, McKay N. in prep. 8.2 and 7.4 ka BP climatic events recorded in travertine at Santovka (Slovakia). *Proceedings of the National Academy of Sciences*.
- Petřík J, Adameková K, Škrdla P. 2022. A pedogenically-informed chronostratigraphic model elucidates the geochronology at the type site of the Bohunician technocomplex. *Quaternary Science Reviews* 297:107827. doi: [10.1016/j.quascirev.2022.107827](https://doi.org/10.1016/j.quascirev.2022.107827).
- Quitta H. 1960. Zur Frage der ältesten Bandkeramik in Mitteleuropa. *Prähistorische Zeitschrift* 38(3–4):153–188. doi: [10.1515/prhz.1960.38.3-4.153](https://doi.org/10.1515/prhz.1960.38.3-4.153).
- Robson HK, Saul H, Steele VJ, Meadows J, Otto Nielsen P, Fischer A, Heron CP, Craig OE. 2021. Organic residue analysis of Early Neolithic “bog pots” from Denmark demonstrates the processing of wild and domestic foodstuffs. *Journal of Archaeological Science: Reports* 36:102829. doi: [10.1016/j.jasrep.2021.102829](https://doi.org/10.1016/j.jasrep.2021.102829).

- Ralph EK. 1959. University of Pennsylvania Radiocarbon Dates III. *Radiocarbon* 1:45–58. doi: [10.1017/S003382220002035X](https://doi.org/10.1017/S003382220002035X).
- Reimer PJ, Austin WEN, Bard E, Bayliss A, Blackwell PG, Bronk Ramsey C, Butzin M, Cheng H, Edwards RL, Friedrich M, et al. 2020. The IntCal20 Northern Hemisphere radiocarbon age calibration curve (0–55 cal kBP). *Radiocarbon* 62(4):725–757. doi: [10.1017/RDC.2020.41](https://doi.org/10.1017/RDC.2020.41).
- Sauer R. 2019. Petrographical and mineralogical analyses of pottery and clay raw materials from Brunn am Gebirge, Wolfholz. In: Stadler P, Kotova NS, editors. Early Neolithic settlement Brunn am Gebirge, Wolfholz, in Lower Austria. Volume 1. Langenweißbach Wien: Beier & Beran. (Beiträge zur Ur- und Frühgeschichte Mitteleuropas). p. 475–512.
- Simon KH. 2002. Das Fundmaterial der frühesten Phase der transdanubischen Linienbandkeramik auf dem Fundort Zalaegerszeg-Andráshida// Gébárti-tó//Arbeitsplatz III. *Antaeus* 25:189–203.
- Šolcová A, Petr L, Hájková P, Petřík J, Tóth P, Rohovec J, Bátorá J, Horsák M. 2018. Early and middle Holocene ecosystem changes at the Western Carpathian/Pannonian border driven by climate and Neolithic impact. *Boreas* 47(3): 897–909. doi: [10.1111/bor.12309](https://doi.org/10.1111/bor.12309).
- Stadler P, Kotova N. 2010. Early Neolithic settlement from Brunn Wolfholz in Lower Austria and the problem of the origin of (Western) LBK. In: Kozłowski JK, Raczky P, editors. Neolithization of the Carpathian Basin: northernmost distribution of the Starčevo/Kőrös culture: papers presented on the symposium organized by the EU project FEPRE (the formation of Europe: prehistoric population dynamics and the roots of socio-cultural diversity). Kraków; Budapest: Polska Akademia Umiejętności; Eötvös Loránd Tudományegyetem. p. 325–348.
- Stadler P, Kotova NS, editors. 2019. Early Neolithic settlement Brunn am Gebirge, Wolfholz, in Lower Austria. Volume 1. Langenweißbach Wien: Beier & Beran (Beiträge zur Ur- und Frühgeschichte Mitteleuropas).
- Stäuble H. 1995. Radiocarbon dates of the earliest Neolithic in central Europe. *Radiocarbon* 37(2):227–237. doi: [10.1017/S003382220003068X](https://doi.org/10.1017/S003382220003068X).
- Stott AW, Berstan R, Evershed P, Hedges REM, Ramsey CB, Humm MJ. 2001. Radiocarbon dating of single compounds isolated from pottery cooking vessel residues. *Radiocarbon* 43(2A): 191–197. doi: [10.1017/S0033822200038005](https://doi.org/10.1017/S0033822200038005).
- Strydomck MV. 2017. A personal reflection on 40 years of radiocarbon dating and archaeology. *Radiocarbon* 59(5):1241–1250. doi: [10.1017/RDC.2016.110](https://doi.org/10.1017/RDC.2016.110).
- Stuckenrath R. 1963. University of Pennsylvania Radiocarbon Dates VI. *Radiocarbon* 5. doi: [10.1017/S003382220003681X](https://doi.org/10.1017/S003382220003681X).
- Taylor RE, Berger R. 1968. Radiocarbon dating of the organic portion of ceramic and wattle-and-daub house construction materials of low carbon content. *American Antiquity* 33(3):363–366. doi: [10.2307/278704](https://doi.org/10.2307/278704).
- Teetaert D, Boudin M, Goemaere E, Crombé P. 2020. Reliability of AMS ¹⁴C dates of moss temper preserved in neolithic pottery from the Scheldt River valley (Belgium). *Radiocarbon* 62(6): 1667–1678. doi: [10.1017/RDC.2019.148](https://doi.org/10.1017/RDC.2019.148).
- Vilnius Radiocarbon. 2022. Graphitization and AMS measurement. Vilnius Radiocarbon. [accessed 2022 Aug. 15]. <https://vilniusradiocarbon.ftmc.lt/en/c14-dating-laboratory/ams-measurement/>.
- Zaitseva G, Skripkin V, Kovalikh N, Possnert G, Dolukhanov P, Vybornov A. 2009. Radiocarbon dating of neolithic pottery. *Radiocarbon* 51(2):795–801. doi: [10.1017/S0033822200056101](https://doi.org/10.1017/S0033822200056101).