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

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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

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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; Bátora 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.

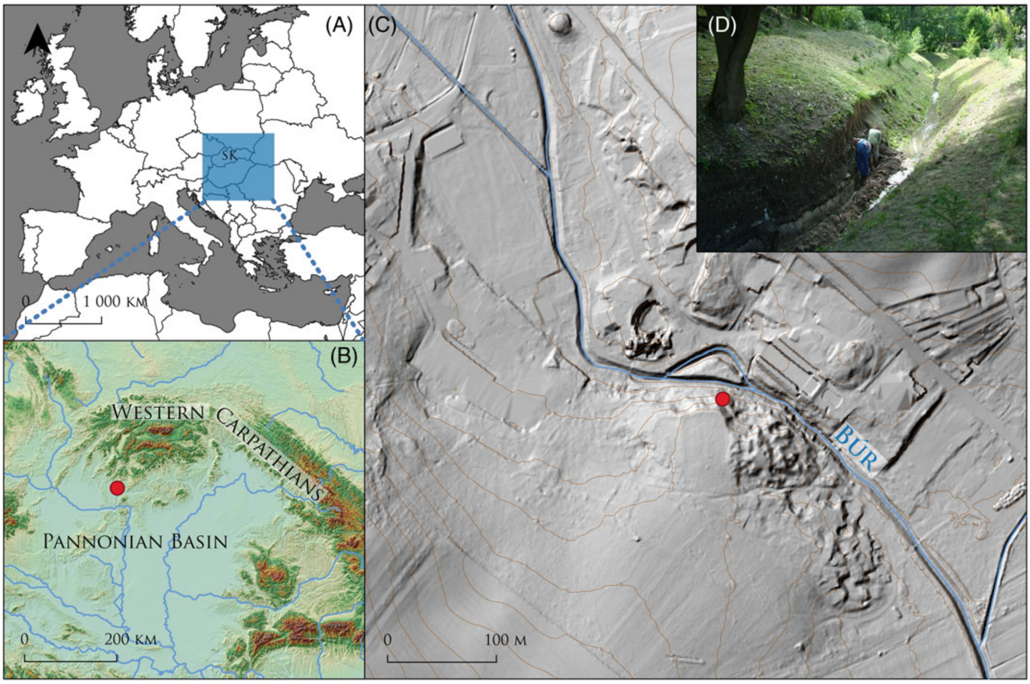

Figure 1 A and B: Location of the Santovka site in a wider geographic context; C: location of the documented section on the right bank of the Búr brook; D: fieldwork campaign in 2012. Data sources: A–B—Natural Earth; C—ÚGKK SR.

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 Thermo-Finnigan 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 $^{14}C/^{12}C$ 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 cm^3 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

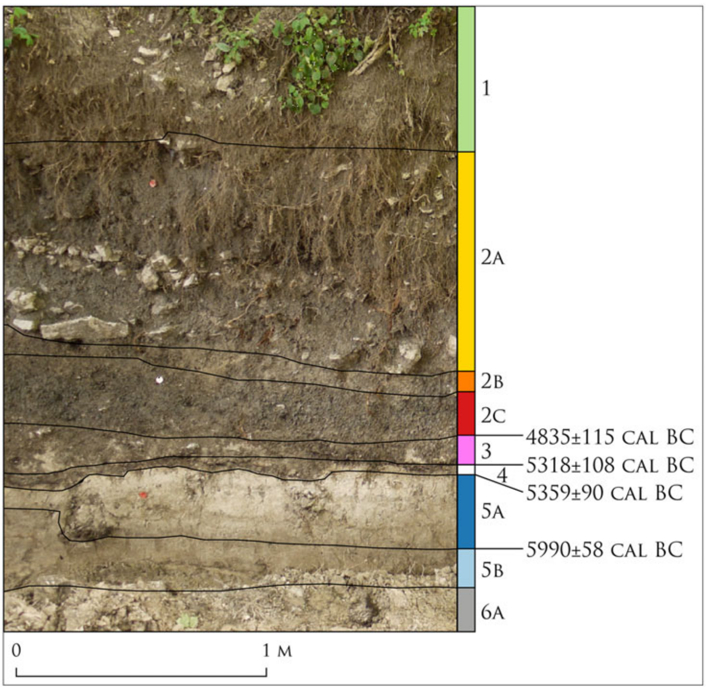

Figure 2 Stratigraphy sequence of the southern section of the Búr creek showing the position of the lithostratigraphic units. Only grass-tempered pottery was found in unit 5a. Displayed dates are based on the chronostratigraphic Bayesian model (Supplement 1.2, 1.3). The complete cross-section of the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

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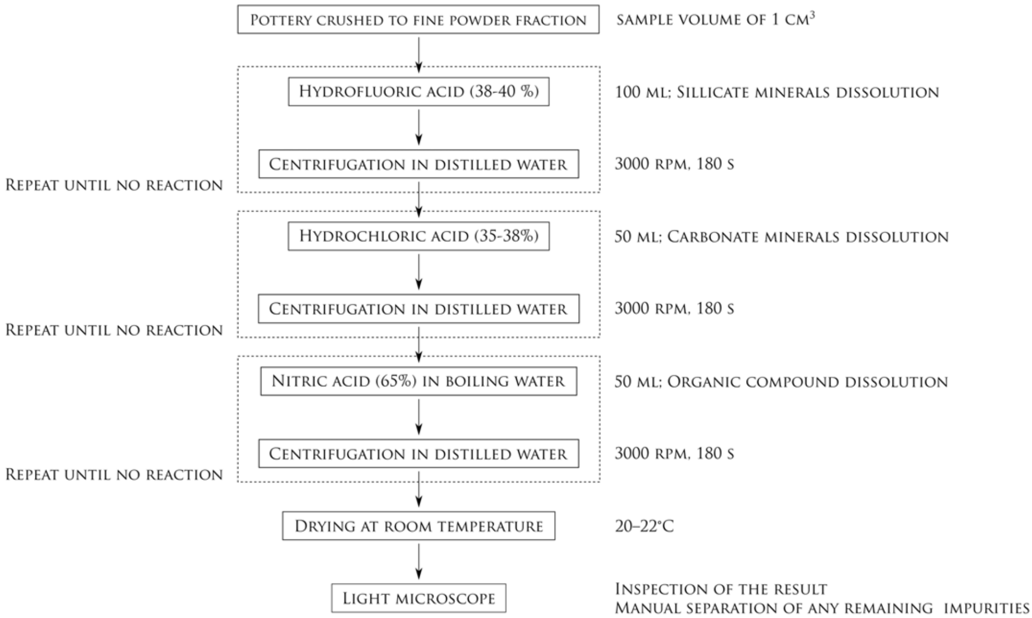

Figure 3 Principle of the novel triple acid wash method.

(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 ^{14}C dating to Isotoptech 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 ^{14}C dating. For this purpose a modified version of the so-called Folch's method was used (Folch et al. 1957; Eggers and Schwudke 2016; Llewellin 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 ^{14}C 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 ^{14}C Laboratory), for ^{14}C analysis.

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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 $^{14}C/^{12}C$ 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

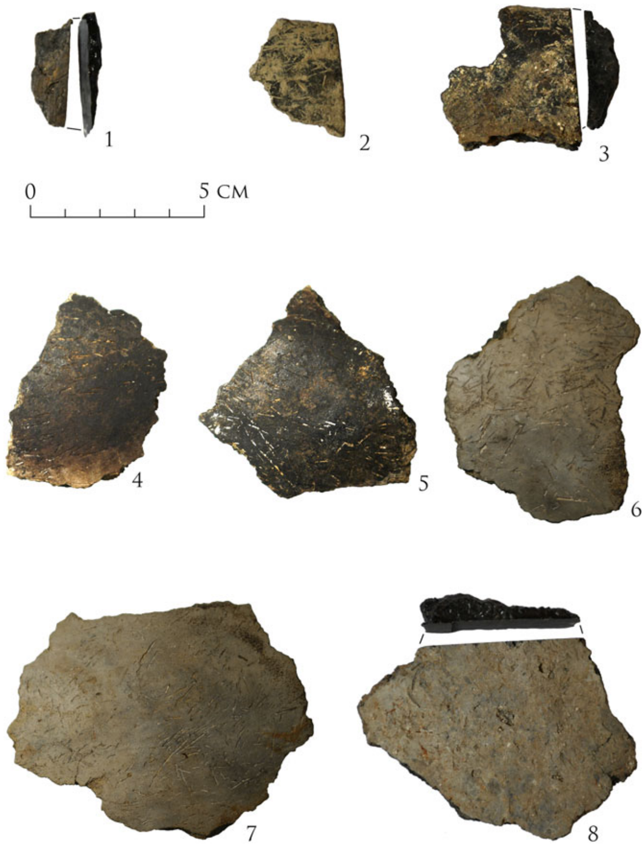

Figure 4 Grass-tempered pottery from Santovka (Slovakia) analyzed in this study. 1—K23/2014, unit 3; 2—K4/2014, unit 5a; 3—K10/2014, unit 5a; 4—K8/2012, sherd 5a, unit 5a; 5—K8/2012, sherd 3, unit 5a; 6—K8/2012, sherd 5b, unit 5a; 7—K8/2012, sherd 2, unit 5a; 8—K11/2014, unit 3.

branched fatty acids that can derive from ruminant animal sources or from microbial sources. There is no molecular evidence (dicarboxylic acids, isoprenoid alkanoic 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.

^{14}C 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 ^{14}C dating, returning 14 results. Results of dating by material (organic temper, organic residue and lipids extracted from the pottery) and pretreatment method (triple

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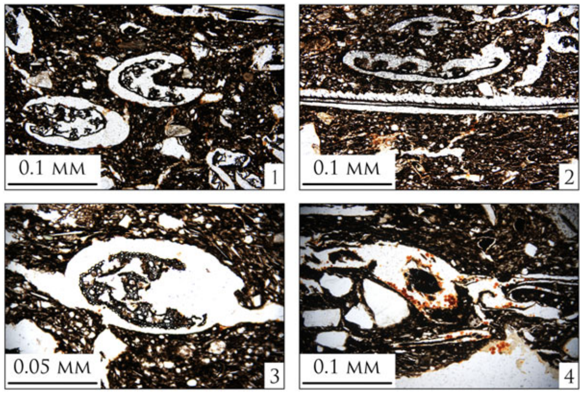

Figure 5 Thin section of grass-tempered pottery from Santovka (Slovakia) in this study. Fine-grained pottery matrix containing leaves of *Festuca* sp. added as a temper during the formation of vessels. 1—K4/2014 (figure 4:2); 2—K10/2014 (figure 4:3); 3—K8, sherd 2 (figure 4:7); 4—K11/2014 (figure 4:8).

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_{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_{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_{comb}=5.4%, A_{comb}=32.2%, A_{comb}=5.5% respectively) and chronologically do not fall within the site stratigraphy.

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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, δ^{13}C C16:0 and δ^{13}C C18:0 are the δ^{13}C-values of the palmitic and stearic acids, respectively. Δ is the difference in δ^{13}C-value between the palmitic and stearic acids ([δ^{13}C C18:0] – [δ^{13}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	δ^{13}C C16:0	δ^{13}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

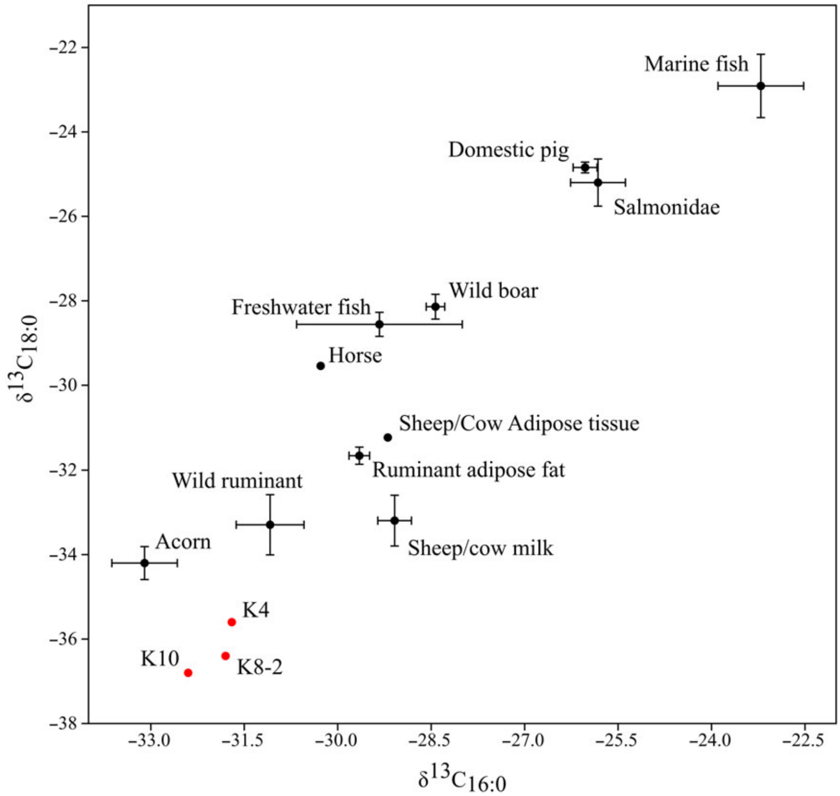

Figure 6 Plot of the $\delta^{13}C$ values of the major fatty acid components (C16:0 and C18:0) of grass-tempered pottery (red dots) compared to modern reference fats (black dots). Reference values were collected from published studies (Dudd et al. 1999:3; Copley et al. 2003:2; Craig et al. 2007:5; Lucquin 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_{comb}=90.4%; A_{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

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Table 3 AMS ^{14}C 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 δ^{13}C (‰), pMC and C (%) were not available.

Laboratory number	Sample	Material	Litho-stratigraphy	Archaeo-logical layers	Depth (cm)	^{14}C age (BP)	^{14}C age (cal BC, 2σ range)	Expected age based on age-depth model (cal BC, 1σ range)	δ^{13}C (‰)	pMC	C (%)	Pre-treatment method
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	—

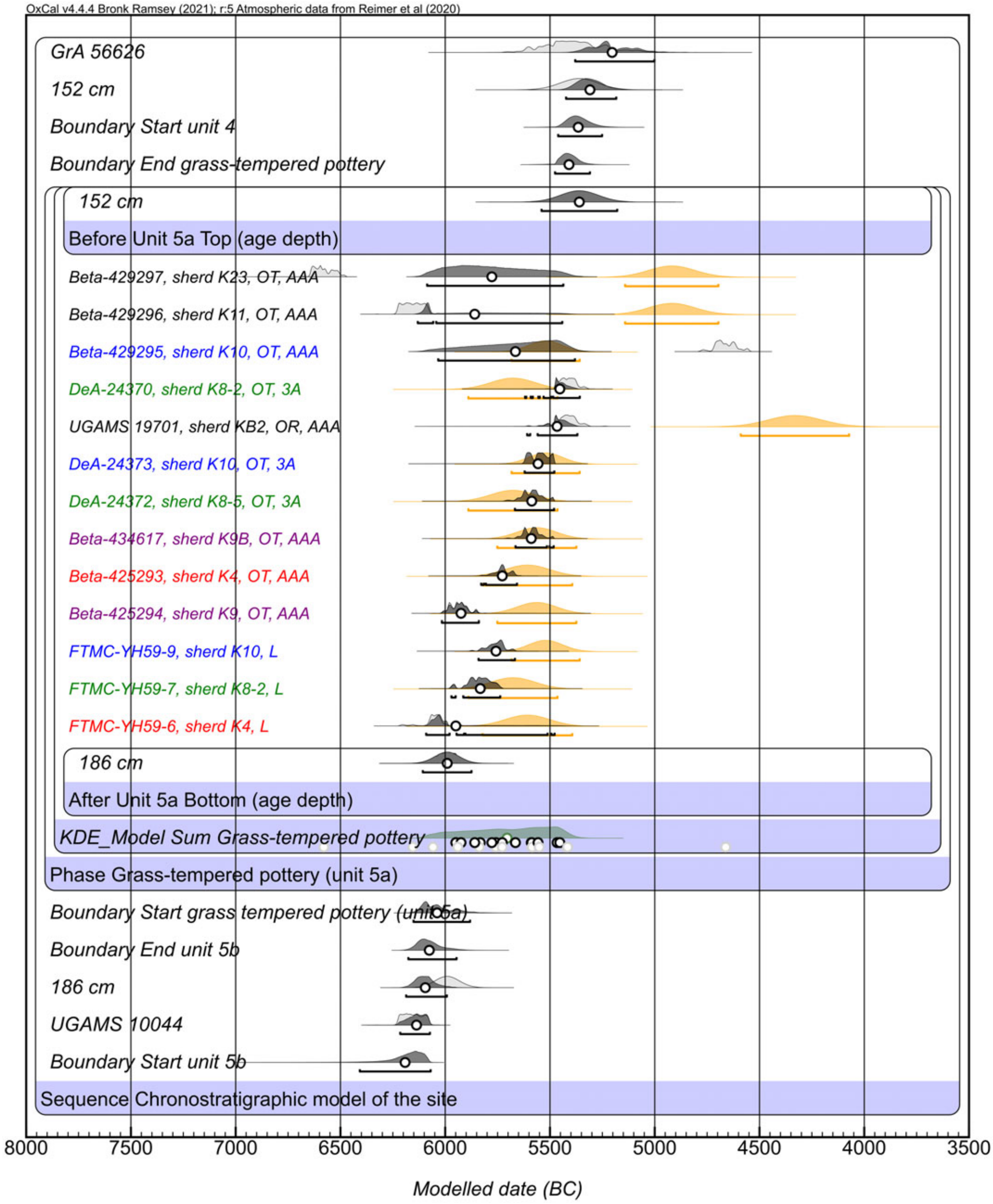

Figure 7 Chronostratigraphic Bayesian model of the site's stratigraphy based on ^{14}C data acquired from the grass-tempered pottery (Table 3), published ^{14}C dates from stratigraphic sequence (Šolcová et al. 2018) and data acquired from the age-depth model Supplement 1.2–1.5). Red—sherd K4; green—sherds 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 exctracted from the pottery (graphitized directly); 3A—triple acid wash method; AAA—acid-alkali-acid wash method.

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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 ^{14}C 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 ^{14}C 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 ^{14}C 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 δ^{13}C-value between the fatty acids of all three samples are well within the conventional range for lipid residues of terrestrial fats. However, $\delta^{13}C_{16:0}$ and $\delta^{13}C_{18:0}$ 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).

^{14}C age of lipids proved to be older by 400–600 ^{14}C 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 ^{14}C dated lipids with the age-depth model shows that only sample FTMC-YH59-7 corresponds with their expected age (combine test result: A_{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_{comb}=1.3%; A_{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-Balkanic agro-ecological barrier (further as CEB-AEB; Bánffi and Sümegi 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_{model}=95.1; $A_{overall}$=96.4) calculated in OxCal v.4.4. Input data is based on Table 3. OT—organic temper.

	Start Santovka (cal BC)				End Santovka (cal BC)				Interval (years)			
	95.4%		68.3%		95.4%		68.3%		95.4%		68.3%	
Method of extraction	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

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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.

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SUPPLEMENTARY MATERIAL

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

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