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EXTENSIVE SURVEY ON RADIOCARBON DATING OF ORGANIC INCLUSIONS IN MEDIEVAL MORTARS IN THE CZECH REPUBLIC

Kateřina Pachnerová Brabcová[1]* • Pavel Kundrát[1] • Tomáš Krofta[1,2] • Václav Suchý[1] • Markéta Petrová[1,3] • David John[1,3] • Petr Kozlovcev[4] • Kristýna Kotková[4] • Anna Fialová[4] • Ján Kubančák[5] • Jan Válek[4] • Ivo Svetlik[1]

[1]Department of Radiation Dosimetry, Nuclear Physics Institute of the Czech Academy of Sciences, Na Truhlářce 39/64, 180 00 Praha, Czech Republic
[2]Department of History, Faculty of Arts, Palacký University Olomouc, Na Hradě 5, 779 00 Olomouc, Czech Republic
[3]Faculty of Nuclear Sciences and Physical Engineering, Czech Technical University in Prague, Břehová 7, 115 19 Praha, Czech Republic
[4]Department of Lime Technologies, Institute of Theoretical and Applied Mechanics of the Czech Academy of Sciences, Prosecká 809/76, 190 00 Praha, Czech Republic
[5]Institute of Experimental Physics, Slovak Academy of Sciences, Watsonova 47, 040 01 Košice, Slovak Republic

ABSTRACT. Dating organic inclusions in mortars such as charcoals is a useful alternative or complementary method to dating mortars themselves, helping to estimate the building age. To assess the limitations of this dating approach, organic inclusions were searched for in surface mortar layers of six early to late medieval buildings in the Czech Republic with relatively well-known age. Altogether, 123 samples were found. About 80% were successfully radiocarbon (^{14}C) dated. However, only 66% originated from wood relatively young when used in lime burning. To judge which samples are relevant to the actual building date, sufficient statistics is crucial. We recommend dating at least 5–10 samples, i.e., collecting 6–12 samples, for a site with uncomplicated building history, or per building phase. Otherwise, unrealistically old or young dates might be obtained. With the recommended statistics, inclusion-based dating provides building ages with uncertainty of 50–100 years.

KEYWORDS: charcoals, medieval objects, mortars, radiocarbon AMS dating.

INTRODUCTION

Radiocarbon (^{14}C) dating of organic inclusions in historical mortars, such as charcoals, seeds, microbiotas, wood pieces or bone fragments, provides *terminus post quem* on the building age, complementary to *terminus ante quem* from dating the mortar itself (Rutgers et al. 2016; Addis et al. 2019; Michalska and Mrozek-Wysocka 2020). Unfortunately, both approaches are prone to potential bias due to several issues. Direct mortar dating suffers from consequences of depth-dependent delayed mortar hardening, $CaCO_3$ dissolution by weathering effects followed by its renewed formation upon absorption of fresh atmospheric CO_2, or precipitation of geological and organic carbon from groundwater and soil moisture (Urbanová et al. 2020; Daugbjerg et al. 2021). When dating organic inclusions, later intrusions may make the building appear younger, while charcoals from old wood used in lime burning may increase the building's apparent age (Schiffer 1986; Van Strydonck et al. 2016; Kim et al. 2019).

To address the limitations of dating historical buildings based on organic inclusions in mortar, the results are presented from a survey analyzing charcoals and other inclusions found in surface mortar layers of six early-to-late medieval buildings at five locations in the Czech Republic (Figure 1). Novel data for two objects that were not ^{14}C-dated so far are

*Corresponding author. Email: brabcova@ujf.cas.cz

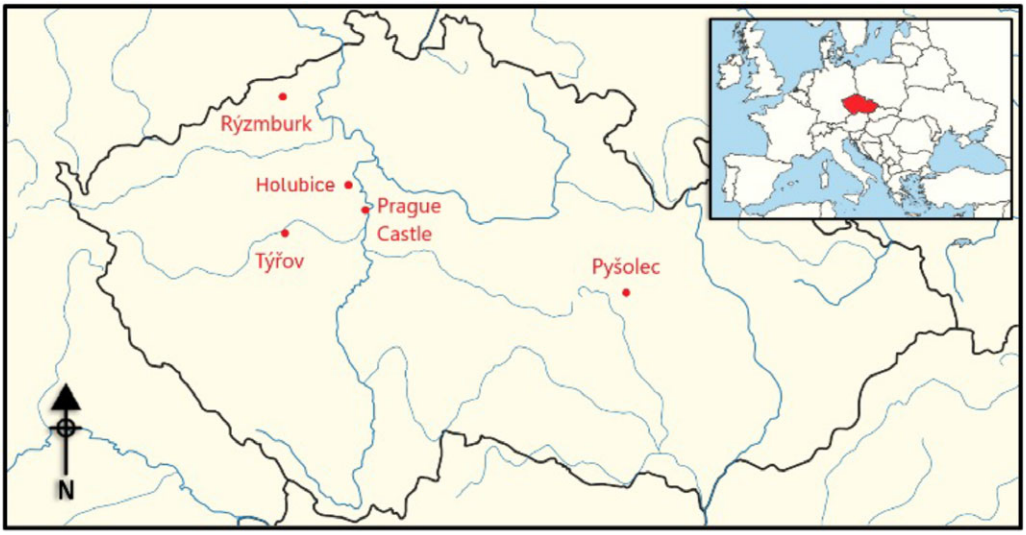

Figure 1 The examined objects on the map of the Czech Republic; insert: location of the country in Europe (QGIS 2022).

complemented by results for four buildings published previously. For all sites, their age is relatively well known from other methods, and their repair history is documented.

First, data are presented from ^{14}C dating of organic inclusions found in the Church of the Nativity of the Virgin Mary in Holubice (15 km northwest of Prague, GPS coordinates: 50.2030N, 14.2932E). This small village church is an outstanding example of high-quality architecture, with a fairly complicated building history (Všetečková et al. 2011; Hauserová 2016): It was established as a late Romanesque rotunda with a round nave and an east apse, in the first third of 13th century. The church was dated based on architectural details and a seal of Pelhřim (Pelegrin), bishop of Prague in 1224–1226, found during the renovation of the church's altar. A south apse was added in the last third of the 13th century, and a west extension of a square shape at the turn of the 13th and 14th centuries. A gothic adaptation took place around the middle of the 14th century; in particular, the interior boasts invaluable figural religious wall paintings since then. Further adaptations were carried out in the 15th and 17th centuries, and restoration works took place in 1865 and in the 20th century. Organic intrusions analyzed herein were collected from the oldest parts of the church, dated to around 1225.

Second, data are reported from the bergfried of Rýzmburk castle (northwestern Bohemia, 50.6340N, 13.6646E). Rýzmburk belongs to the largest Czech castles. It was first mentioned in written reports in 1250, being held by Boreš II of Rýzmburk. After his death in 1278, the importance of the House of Rýzmburk had declined, until a new age of prosperity associated with his descendants' political and economic activities, including mining and founding of rural settlements, took place in the mid-14th century (Lehký 2012). The bergfried (large round tower, in the southern part of the castle) likely originates from this later phase, 1300–1360 (Lehký 2012).

The third site is the northern tower of Rýzmburk castle (Pachnerová Brabcová et al. 2022a), previously dated to the early phase of Rýzmburk development, 1260–1278 or pre-1300 (Lehký

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2012; Razím 2019). Fourth, inclusions were analyzed from the bergfried of Týřov castle (Pachnerová Brabcová et al. 2022b); the previous results were extended by three wood samples. Týřov (49.9735N, 13.7903E) was founded by Wenceslas I, Bohemian king in 1230–1253, who in 1249 imprisoned there his rebellious son, future king Ottokar II of Bohemia. The bergfried itself was previously dated to 1260–1270 (Razím 2005). The fifth analyzed site is the tower and buttress of Pyšolec (Pachnerová Brabcová et al. 2022c). First mentioned in written sources in 1350, Pyšolec (49.5455N, 16.3361E) was likely founded in the first third of the 14th century by the House of Pernštejn, a powerful family of the Bohemian Kingdom; it was mentioned as ruined in 1446. The analyzed remains likely originate from 1300–1340 (Korbíčková and Hložek 2019). Finally, ^{14}C data are included from the Southern Corridor of Bishopric District (Kundrát et al. 2022), a valuable site uncovered in early 1920s as the first Romanesque remain in the 3rd Courtyard of Prague Castle (50.0903N, 14.4004E). While it likely originates from the 11th or 12th centuries, there are indications of ongoing building activity such as raising terrace walls at least till the 14th–16th centuries.

Taken together, the organic inclusions from these six objects represent a large dataset of 123 samples. Its analysis enables us to discuss general limitations of organic intrusion-based mortar dating and draw recommendations on the number of samples needed to reliably estimate the building dates.

MATERIALS AND METHODS

Sampling Organic Inclusions

In all sites, the surface mortar layers were carefully visually inspected for the presence of embedded charcoals and other organic intrusions. Parts with records of repair or restoration works or showing visible signs thereof were excluded. The search was limited to accessible wall parts, usually up to the height of 2 m, corresponding to surface wall areas per site ranging from 6 to 114 m^2 (the surface area of mortar being several folds smaller). Samples resembling charcoals or other organic inclusions were identified by eye and sampled. In addition to this minimally invasive method, a few core drills were made at the Southern Corridor of Bishopric District of Prague Castle, which yielded several additional charcoals. Otherwise, deeper layers of the mortar were not sampled to avoid damaging the valuable historical sites.

Sample Treatment

All samples were carefully visually inspected and mechanically cleaned to remove the remaining mortar particles. Particular attention was devoted to biological contamination such as lichen or fungi, as this might compromise the ^{14}C dating results; however, no sample showed traces of such contamination.

Following the cleaning steps, the samples were pretreated with the acid-base-acid (ABA) protocol (Šimek et al. 2019; Svetlik et al. 2019). Well preserved samples with efficient weight were treated by a computer-controlled apparatus: they were exposed to a continuous flow of 0.5 M HCl in order to remove carbonates, washed by distilled water, exposed to a flow of 0.1 M NaOH to remove humic acid contaminants, washed by distilled water, and finally exposed to 0.01 M HCl to release atmospheric CO_2 absorbed in the previous steps. The total cycle time was 27 hr. To limit the weight loss for fragile or small samples, these were treated with the same protocol manually, while the step durations were adjusted according to sample status, and at the end of the process the samples were centrifuged to preserve the finest particles. Yet some samples dissolved completely, leaving no residue when filtering through a 0.2-µm pore size silver membrane filter.

After drying at 60°C, the samples were combusted with oxidizing agent CuO and graphitized in the presence of reducing agent Zn (Orsovszki and Rinyu 2015). Similarly, graphite samples were prepared from the ^{14}C reference material (Oxalic Acid II, NIST SRM 4990C) and a blank (^{14}C-free phthalic acid anhydride) using the same treatment (Cercatillo et al. 2021) to serve as combustion and graphitization controls.

Radiocarbon Analysis and Dating

The graphite targets were analyzed with MILEA accelerator mass spectrometry (AMS) system at the Nuclear Physics Institute of the CAS, Řež, Czech Republic (lab code CRL) or with MICADAS AMS at the Hertelendi Laboratory of Environmental Studies in Debrecen, Hungary (lab code DebA). The AMS data were processed with software BATS (Wacker et al. 2010). The calibration was performed by OxCal version 4.4 (Bronk Ramsey 2009), based on the calibration curve IntCal20 (Reimer et al. 2020) or the Bomb 21 NH1 dataset for recent samples (Hua et al. 2021). The samples were dated both individually and collectively, estimating calendar ages of all samples from a given object using the kernel density estimate (KDE) method implemented in OxCal (Bronk Ramsey 2017).

Scanning Electron Microscopy

Selected samples were imaged with a scanning electron microscope (SEM FEI QUANTA 450 FEG) at the Institute of Theoretical and Applied Mechanics to investigate differences between surfaces of charcoals from young, old, and very old wood.

RESULTS AND DISCUSSION

Fourteen organic inclusions were found in the sampled walls of the Holubice rotunda, namely a straw and 13 charcoals. One of the charcoals was excluded as too small, and two dissolved during the pretreatment procedure. The remaining 11 samples were dated successfully; for one sample, two analyses were performed. In the bergfried of Rýzmburk, 13 charcoals were found, and they all survived the pretreatment and were dated. At Týřov, three additional, wood samples were collected and dated, complementing samples presented previously (Pachnerová Brabcová et al. 2022b). The original sample weights and ABA yields are listed in Table 1, together with the samples' conventional ^{14}C ages as well as calendar date intervals. Clearly, the straw sample from Holubice and the three wood samples from Týřov represent young intrusions unrelated to building dates of the objects, while 10 charcoals from Holubice and 13 from Rýzmburk bergfried are relevant samples. Graphically, their calendar dates are presented in Figure 2 as probability distributions.

The analyzed charcoals were of rather small sizes and were directly embedded in the mortars. Hence, they may hardly originate from larger wood pieces such as timber charred in later fires. They might have been added with the filler sand. We cannot rule out the possibility that they were originally embedded as wood splinters or chips and become charred in later fires, although there were no clear signs of such fires at the analyzed sites. As the most plausible hypothesis, we assume that the charcoals originate from wood used in lime burning. However, even an alternative origin would not affect the implications on the building ages discussed below.

Table 1 Weights, ABA yields, conventional ^{14}C ages (CRA) and calibrated dates for samples from the Holubice rotunda, Rýzmburk bergfried and wood samples from Týřov.

Label	Material	Weight (mg)	ABA yield[1] (%)	CRA ± 1 σ (BP)	Date[2] (AD)
Holubice rotunda (date estimate: 1224–1226)					
21_491	Charcoal	2.2	81.8	896 ± 15	1050–1216
21_492	Charcoal	2.3	69.6	929 ± 15	1041–1163
21_493	Straw	60	38.5 [A]	327 ± 14	1499–1638
21_494	Charcoal	40	0.0	–	–
21_495	Charcoal	10	80.0	951 ± 14	1032–1158
21_496	Charcoal	105	1.9	878 ± 14	1160–1218
21_497	Charcoal	36	0.0	–	–
21_498	Charcoal	20	37.8	860 ± 14	1167–1221
21_499	Charcoal	16	55.6	900 ± 14	1048–1215
21_500	Charcoal	22	68.3	861 ± 14	1166–1221
21_501	Charcoal	13	78.1	899 ± 14	1049–1215
21_502	Charcoal	44	52.5	859 ± 14	1168–1222
21_503	Charcoal	113	15.6	920 ± 14	1041–1171
			5.1	928 ± 16	1040–1164
21_504	Charcoal	0.5		*Insufficient weight*	
Rýzmburk bergfried (date estimate: 1300–1360)					
21_2037	Charcoal	200	85.1	659 ± 18	1285–1389
21_2038	Charcoal	70	47.5	604 ± 18	1304–1400
21_2039	Charcoal	110	49.5	628 ± 18	1297–1396
21_2040	Charcoal	219	32.5	573 ± 18	1320–1414
21_2041	Charcoal	17	52.6	630 ± 18	1296–1395
21_2042	Charcoal	257	1.2	710 ± 19	1271–1378
21_2043	Charcoal	150	64.7	719 ± 18	1270–1298
21_2044	Charcoal	10	47.5	614 ± 18	1302–1398
21_2045	Charcoal	23	3.9	662 ± 20	1283–1389
21_2046	Charcoal	12	50.8	661 ± 16	1284–1388
21_2047	Charcoal	597	45.1	575 ± 21	1316–1416
21_2048	Charcoal	4.7	25.5	725 ± 19	1267–1296
21_2049	Charcoal	110	49.6	595 ± 18	1306–1405
Týřov bergfried (date estimate: 1260–1270)					
21_297	Wood	2.87 g	42.5 [A]	402 ± 18	1444–1615
21_298	Wood	2.28 g	41.5 [A]	–1597 ± 11	1959–1984
21_299	Wood	0.84 g	51.2 [A]	–5014 ± 9	1963–1964

[1]Index A stands for automatic procedure. The other samples were processed manually.
[2]Calibrated calendar date intervals corresponding to about 95% confidence interval. For brevity, only the oldest and youngest dates are reported whenever two or more subintervals were obtained, e.g., 1959–1962 (with the probability of 55.6%) and 1983–1984 (39.9%) for sample 21_298.

The calendar age distribution of the 10 dated charcoals from the Holubice rotunda estimated by the KDE method is presented in Figure 3A. The age estimates are close to normal distribution with the median date of about 1170 and the standard deviation of about 30 years, with a slight skew towards younger ages. This indicates that wood was most frequently aged about 25–85 years when charred for lime burning, assumed to take place in 1224–1226. None of the charcoals corresponded to an “old wood” outlier or a young intrusion.

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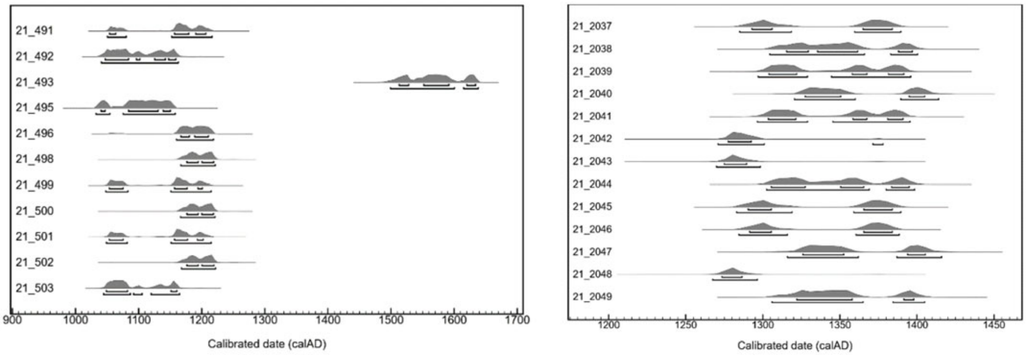

Figure 2 Conventional ^{14}C ages of the charcoals from the Holubice rotunda (left) and Rýzmburk bergfried (right) calibrated to calendar age (in AD). The narrower and wider brackets depict 68.3% (1σ) and 95.4% (2σ) confidence intervals of calendar ages, respectively.

The estimated distribution of 13 charcoals from Rýzmburk bergfried (Figure 3B) is more heavily skewed towards younger ages and has the median of 1320 and σ of about 30–40 years. There is no indication for an "old wood" or "young intrusion" outlier. The profile of the estimated age distribution is influenced by the local shape of the calibration curve, which possesses a broad local minimum at 1320–1360 followed by a local maximum at 1370–1390, and inherently leads to large uncertainty of the KDE result: A sample from, say, 1310 cannot be excluded to originate from about 1360 or 1390. Obviously, this intrinsic limitation is not specific to charcoals or mortars but applies to ^{14}C dating in general. Considering this fact, the present results are consistent with the existing date estimate of 1300–1360. If the building originates from a later part of this estimate, say 1340–1360, the charcoal results suggest that wood aged at most 80–100 years was used in lime burning; if the early part of the estimate were relevant, the charcoals would point to rather young wood such as bush, branches, or small trees being charred.

For comparison, analogous results are presented for the northern tower of Rýzmburk (Figure 3C), the bergfried of Týřov (Figure 3D), the tower and buttress of Pyšolec (Figure 3E), and the Southern Corridor of Bishopric District of Prague Castle (Figure 3F), based on previously presented samples (Kundrát et al. 2022; Pachnerová Brabcová et al. 2022a, 2022b, 2022c); samples of Paleolithic origin or identified as old wood or young intrusions were excluded. As the KDE method in Oxcal is based on stochastic Markov chain Monte Carlo simulations, its results slightly differ from run to run, and the present results are not identical with those reported previously, yet the basic characteristics remain unchanged. For all the analyzed objects, the reported results of organic inclusion-based dating are consistent with existing building estimates. Interestingly, similar results were obtained regarding age distribution of charred wood used for lime burning for almost all objects. The distributions are close to normal ones with standard deviations σ of about 15–40 years; the Romanesque corridor at Prague Castle represents an exception with two such phases, consistent with the building activities on site (Kundrát et al. 2022). The widths of the KDE distributions are heavily influenced by the local pattern of the calibration curve, as discussed above. This width of age distribution represents one principal limitation of inclusion-based dating.

More important is the question of how far the obtained age distribution enables us to estimate the actual building date of the object. In this respect, the narrow date estimates for Holubice

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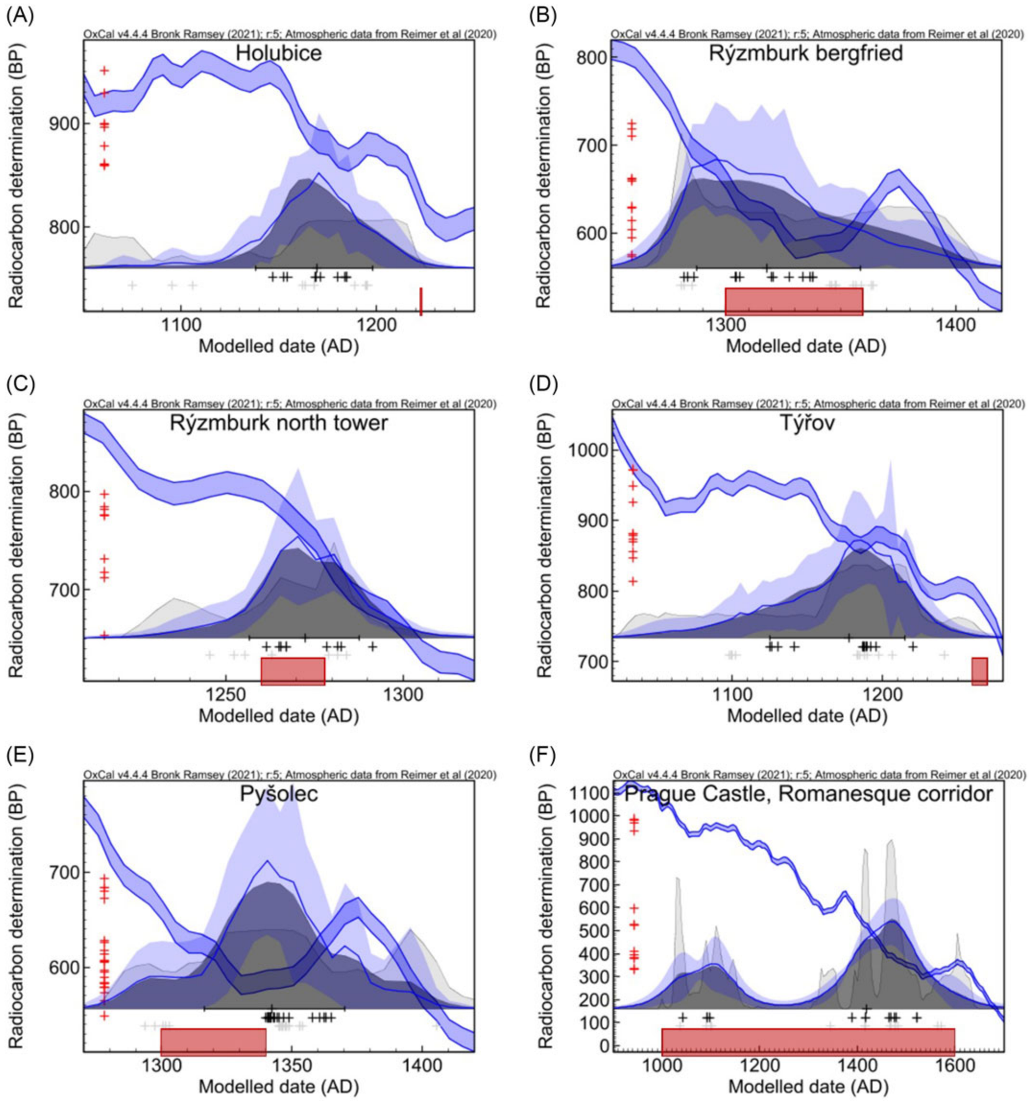

Figure 3 Calendar age distribution of the samples estimated by the KDE method for the Holubice rotunda (A), bergfried (B) and northern tower of Rýzmburk (C), bergfried of Týřov (D), tower and buttress of Pyšolec (E), and Southern Corridor of Bishopric District, Prague Castle (F). For each site, the KDE estimate (final estimate: filled with dark gray; mean of stochastic simulation passes: blue curve; uncertainty: light blue band) is compared to the sum of distributions for individual samples (light gray). Red crosses along the vertical axes show median CRA, light gray crosses along the horizontal axes depict median estimated calendar ages for individual samples (prior information), and black crosses indicate median KDE-refined calendar ages. Horizontal error bars depict ± 1σ of the distributions (posterior estimates from KDE). Red boxes on the calendar age axes indicate existing estimates of the objects' age. (Please see online version for color figures.)

rotunda and Týřov castle correspond to tails of the KDE charcoal distributions. This indicates that the medians of KDE charcoal distributions tend to overestimate the building age, by about 50–90 years. Unfortunately, existing estimates for the other objects analyzed in this work are considerably wider. For the north tower of Rýzmburk, the charcoal dating indicates that the

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construction likely took place towards the later part of the existing estimate. For Rýzmburk bergfried and Pyšolec, the comparison is hindered by the local pattern of the calibration curve which widens the estimated charcoal age distributions or, for Pyšolec, even apparently shifts it post the existing building estimates.

For medieval buildings with completely unknown history, organic inclusion-based ^{14}C dating may thus help estimate the construction date with an uncertainty of about 50–100 years. If additional information independent of this method is available, inclusion-based dating may help benchmark or even refine it. The key necessity for inclusion-based dating to provide relevant dates is collecting and dating a sufficiently large number of samples. In the present survey, as summarized in Table 2, 123 samples were collected from a total sampled wall area of about 284 m^2, with areal density from 0.23 to 4.4 samples per m^2. Out of these 123 samples, 8 were excluded as charcoal-resembling stones or too small samples, 17 samples dissolved during chemical pretreatment, and 98 were dated successfully. Out of the dated samples, 3 were of Palaeolithic age, 5 were young intrusions, in particular wood and straw samples, and 9 were identified as “old wood” samples aged about 200–300 years at the time of building construction. Directly relevant to the building date were 81 samples, 66% of the collected ones. To ensure sufficient statistics leading to reliable dates, we thus recommend that at least 5 but better 10 samples be dated, which translates into 6 or 12 samples gathered and analyzed, per object or per building phase. Otherwise, there is a high risk that samples unrelated to the actual building age could not be separated from the relevant ones. We strongly discourage using a very few or even just a single charcoal date only. Out of 8 wood samples, 4 were dated as young intrusion, although there was no such indication during sampling. Therefore, wood intrusions in surface mortar layers cannot be recommended as a reliable sample type for dating.

It should be emphasized that the recommendations are valid only for areas with lime burning procedures and availability of wood similar to medieval Bohemian countries. In other regions the findings may not be representative (Cook and Comstock 2014).

Particularly interesting are the finds of three Palaeolithic charcoals in two different objects. We previously speculated that these might have originated from charcoals in fluvial sediments used as mortar aggregate (Pachnerová Brabcová 2022b). SEM imaging (example on Fig 4) revealed advanced tissue damage compared to charcoals from old wood. This could make samples prone to difficult-to-remove contamination, for example with fossil limestone, and affect their apparent age.

CONCLUSIONS

Charcoals embedded in historic mortars may originate from wood considerably old at the time of lime burning, typically up to 100 but exceptionally also 200–300 years in the present survey of six medieval objects in the Czech Republic. These exceptional “old wood outliers” as well as young intrusions can be separated by advanced data analysis tools such as the kernel density estimate method in OxCal, if a sufficiently high number of inclusions were collected. We recommend dating at least 5–10 samples, i.e., collecting 6–12 samples, per site and building phase. We strongly discourage the frequent praxis of dating a very few or even just a single charcoal per site, as this may easily lead to erroneous age estimates. Even when analyzing large sample numbers, the resolution of inclusion-based dating is limited to 50–100 years by the distribution of wood ages used for lime burning in medieval times. In addition, general ^{14}C

Table 2 Summary of inclusion-based dating in the studied sites.

Site	Sampled area (m^2)	Samples found	Excluded	Dissolved during ABA	Dated	Palaeolithic	Young intrusion	Old wood	Young wood
Holubice	6	14	1	2	11 (79%)	0	1	0	10 (71%)
Rýzmburk, bergfried	57	13	0	0	13 (100%)	0	0	0	13 (100%)
Rýzmburk, north tower	40	10	0	1	9 (90%)	0	0	0	9 (90%)
Týřov	60	18	1	1	16 (89%)	0	3	1	12 (67%)
Pyšolec	114	37	3	1	33 (89%)	2	1	6	24 (65%)
Prague Castle	7	31	3	12	16 (52%)	1	0	2	13 (42%)
All	284	123	8 (6.5%)	17 (14%)	98 (80%)	3 (2.4%)	5 (4.1%)	9 (7.3%)	81 (66%)

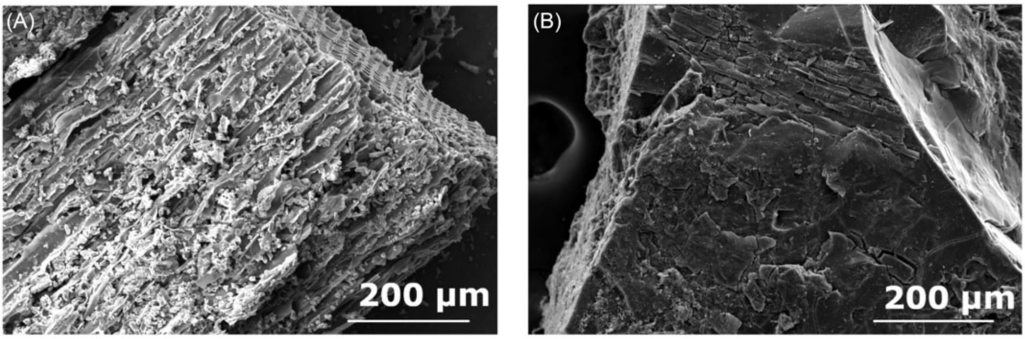

Figure 4 SEM images of charcoals from old wood (panel A, sample 21_277) and Palaeolithic charcoal (panel B, sample 21_261). Both samples were found at the Prague Castle site.

dating limitations apply that follow from the local pattern of the calibration curve in the analyzed time period.

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