Playing with Fire: Exploring ceramic pyrotechnology in Late Neolithic Balkans through an archaeometric and experimental approach

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Addressing ceramic pyrotechnology plays a key role in understanding a wide range of cultural and social behaviours associated to pottery production. Firing is the process which transforms clay into ceramic, which is one of the most frequently preserved materials in the majority of Neolithic and later archaeological sites.

Though firing temperatures and the functions of various pyrotechnological installations have been extensively investigated in archaeology, both have often been addressed separately. Most of our knowledge on firing structures and procedures in the Neolithic are still largely based on ethnoarchaeological evidence. To move forward, we need to consider all aspects involved in ancient pyrotechnology, together with use of additional investigative tools. This study aims to address Neolithic pottery firing from a diverse perspective that merges archaeometric analyses and experimental archaeology. To demonstrate the potential of this approach, we combined an archaeometric case study of pottery from the late Neolithic (5200-4800 BCE) from the site of Gradište-Iđjoš (Serbia) with experimental pit firings, likely one of the mostly frequently employed firing techniques used in prehistoric periods.

Scientific analyses include X-ray powder diffraction (XRPD), scanning electron microscopy (SEM), and ceramic petrography. These methods were run on both archaeological materials and experimental reproductions. Additionally, a detailed program of firing temperature monitoring, integrated observations on atmospheric conditions, soaking time, and duration were recorded to contribute to the study. The experiments enabled us to collect results useful for our understanding of the pyrotechnological knowledge of Neolithic potters from a technological and social point of view. In addition, they demonstrated the potential of a dedicated methodological framework for studying pottery firing that can be applied to other chronological and cultural contexts.

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1. Introduction

The study of ancient pyrotechnology is certainly one of the crucial themes in anthropological and archaeological research (e.g. Gibbs, 2015; McDonnell, 2001) as it enables scholars to explore topics such as invention, innovation, and technological advancement to build narratives of large-scale interactions in global archaeology (e.g. Roberts and Radivojević, 2015; Roberts and Vander Linden, 2011). Ceramics, as one of the most abundant materials preserved in the archaeological record, are at the focus of several pyrotechnological studies. These works illustrate how pottery firing is a complex procedure, due to the large number of variables that are involved in this process (e.g. Gosselain, 1992; Livingstone Smith, 2001; Rice, 2015).

Among the different approaches that have been used to reconstruct ancient ceramic pyrotechnology, archaeometric analyses and experimental archaeology have played a critical role. On the one hand, scientific analyses allow a degree of resolution that cannot be obtained solely with macroscopic investigations (Tite, 1995, 37–38). Archaeometric studies that focus on the reconstruction of pottery pyrotechnology employ a variety of methods that aim especially at the estimation of firing temperatures. This is done through the identification of relationships between firing temperatures and changes in the pottery microstructure (e.g. porosity, clay matrix, progressive sintering, and vitrification) and mineralogy (Gliozzo, 2020; Maniatis and Tite, 1981; Rice, 2015, 376–387). On the other hand, the employment of experimental archaeology (Coles, 1979; Godino et al., 2020; Outram, 2008; Reynolds, 1999) not only helps to test hypotheses developed on the basis of the archaeometric results, but, most importantly, gives insightful information on different aspects of firing procedures. This knowledge helps to have a more nuanced understanding of ancient ceramic pytorechnology and the complex social behaviour behind this practice (e.g. Gheorghiu, 2019), that goes beyond the mere estimation of firing temperatures.

Despite the clear advantages that both approaches contribute, they are only rarely systematically combined (e.g. Kudelić, 2017; Thér et al., 2019). In this work, using the case study of the Late Neolithic Vinča settlement of Gradište near Iđjoš in the Serbian Banat (hereafter Gradište-Iđjoš), we show that the combination of archaeometric analysis of materials deriving from both archaeological contexts and our experiments is the key to a better understanding of ancient pyrotechnology.

Such an approach gives us important information on different aspects of firing procedures and how these are reflected in the microstructural and compositional characteristics of archaeological ceramics. These data then aid a better interpretation of archaeometric results and help us developing a well-rounded technological and social reconstruction of ancient pyrotechnology.

1.1 Archaeological and geological background

The settlement of Gradište-Iđjoš (Figure 1) is situated in the north-centre Banat, approximately 3 km east of the Tisza river. This Neolithic and Chalcolithic settlement was excavated before and after the Second World War and is currently investigated by the Bordeland: ARISE project (Mirković-Marić and Marić, 2017). The excavations carried out at this site gave evidence of a Starčevo-Köros culture phase (second half of the 6th millennium BCE) and a Vinča and Tisza occupation (5200–4900 BCE). Late Neolithic mixed assemblages are typical for the area of northern and central Serbian Banat and are found in many other sites in this region (Brukner, 1968). Two other examples for this are the sites of Kremenjak-Čoka and Akača-Novo Miloševo, both situated close to Gradište-Iđjoš (Figure 2).

The Vinča phenomenon, whose pottery is at the centre of this investigation, is a Neolithic/Chalcolithic material culture that developed in a vast area in the northern and central Balkans. In terms of absolute dates, the estimated duration of the Vinča phenomenon spans from c. 5350 to c. 4600 BCE (Whittle et al., 2016 and literature therein).

The Tisza material culture (Korek, 1989; Raczky, 1987) spread during the Late Neolithic (c. 5000 to c. 4600 BCE) in an area spanning from Slovakia and Ukraine to the north, up to the Körös river on the east. The Serbian Banat represents the southern part of the territory of Tisza material culture, reaching the confluences of the Aranka and the Zlatica rivers into the Tisza.

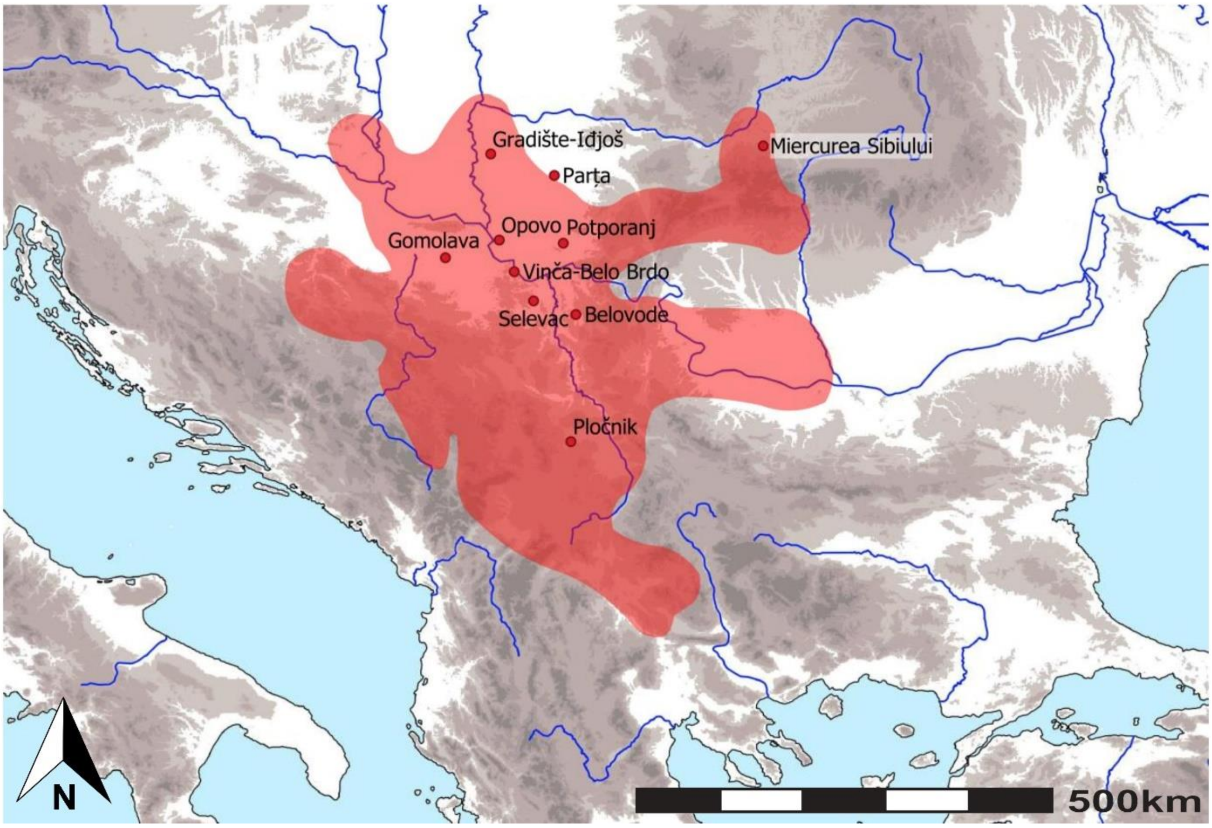


Figure 1: Distribution of the Vinča culture (shaded) and the location of sites that have been the object of pyrotechnological investigations (Map by Lars Heinze and Silvia Amicone).

The geology (Figure 2) of the north-centre Serbian Banat (close to the location of Gradište-Iđjoš) is marked by several Pleistocene and Holocene alluvial sediments containing gravel, sand, and clay layers (Koprivica and Strajin, 1994). In a previous work, geological samples near Gradište-Iđjoš were selected to study the nature and distribution of these alluvial sediments (Amicone et al., 2020a), thereby demonstrating that two main clay sources mark this area: very fine sandy-clay sources deposited during the Holocene and available in the proximity of the site, and sandier Pleistocene sources that outcrop c. 10 km from Gradište-Iđjoš.

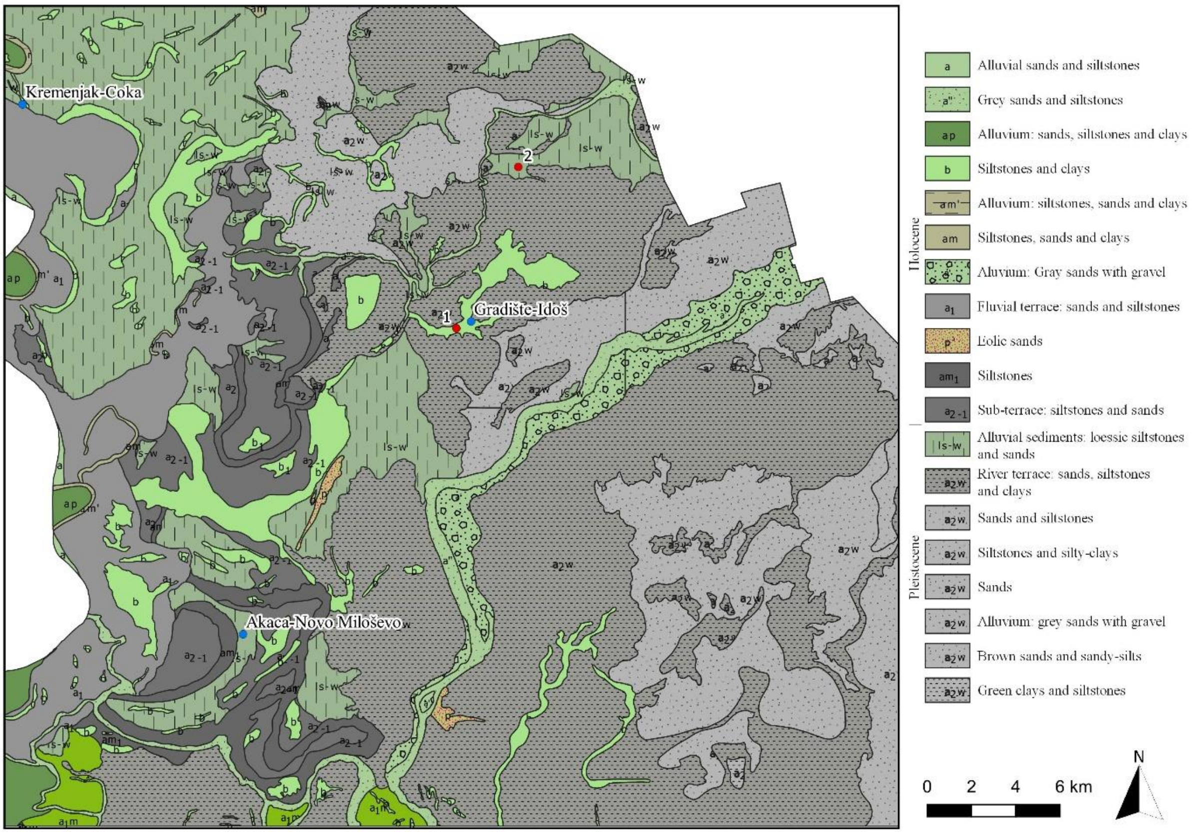


Figure 2: Geological Map of the North Banat area (based on the Yugoslavia Geological Map issued by the Federal Geological Institute. Sheet L34-77: 100 000). Site locations are indicated by blue dots. Points 1 and 2 indicate clay sampling locations (Map by Enrico Croce and Silvia Amicone).

1.2 Late Neolithic pottery pyrotechnology in the Balkans

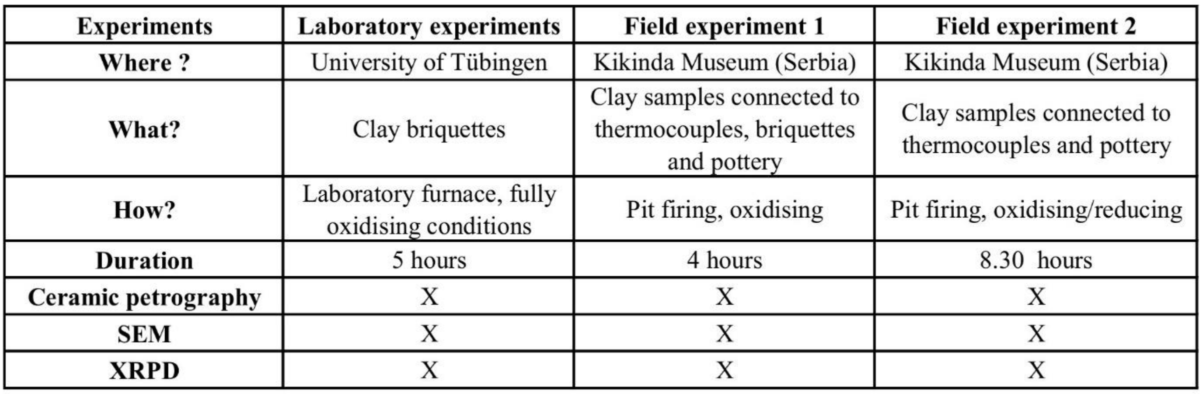
Several researchers have focused on the study of Late Neolithic and Chalcolithic pottery pyrotechnology from the Balkans (e.g. Gardner, 1978; 2003; Goleanu et al., 2005; Kaiser et al., 1986; Linda, 1984; Maniatis and Tite, 1981; Perišić et al., 2016; Spataro, 2017; 2018; Yiouni, 2000). While targeted studies on ceramic pyrotechnology of Tisza material culture are missing, pottery produced by the Late Neolithic and Chalcolithic communities labelled as Vinča received particular attention for the purported link between pottery firing technology and the origins of metallurgy in the Vinča phenomenon (Amicone et al., forthcoming).

By applying a vast range of archaeometric techniques, these studies (e.g. Kaiser et al., 1986) were especially focused on the estimation of firing temperatures in the attempt to understand if Vinča pottery was fired to temperatures comparable to those necessary to smelt copper (c. 1083°C, Pollard et al., 1991) and if this pyrotechnology knowledge could have been transferred from ceramic manufacture to metallurgy. Nevertheless, to have a more comprehensive understanding of the pyrotechnological processes, more attention must be paid to other parameters of ceramic manufacture, such as how firing atmosphere was controlled to create redox conditions.

A more recent study (Amicone et al., 2020b) utilised a multi-pronged scientific approach to investigate pottery from Belovode and Pločnik (Serbia), home of the world's earliest metallurgy. This work illustrates that potters fired ceramics at highly variable temperatures, which did not appear to have exceeded 900°C and employed either oxidising or reducing conditions. (Chapman, 2006; 2007). This study also proposed a model of production for dark-burnished pottery, a tradition widespread throughout the Balkans in the Late Neolithic and typical feature of Vinča material culture (Chapman, 2006; 2007). This model consists of a two-step firing procedure that involves an oxidising firing followed by a reducing phase during cooling obtained through smudging of the vessels. This work concluded that potters at these sites were certainly able to manipulate the amount of oxygen in their firings and that this knowledge could have been important for the development of early metallurgy pyrotechnology.

Vinča pottery has often been regarded as the outcome of specialised and skilled productions (e.g. Kaiser, 1984; Spataro, 2018) and therefore it was often assumed that potters were certainly employing kilns rather than open or pit firing installations where the firing process is less controlled (Rice, 2015, 172–181). However, there is no conclusive evidence for pottery kilns in Vinča culture settlements (Amicone et al., forthcoming). Recent experiments (Svoboda et al., 2005; Vuković, 2018) suggested that the complete range of pottery manufactured by Vinča potters could have been produced using pit firings. The use of this technique could have even been preferred, despite the lack of control over different variables of the firing procedure, as it allows for a relatively fast and fuel-efficient process (Rice, 2015, 172–181). Traces of pit firings are not always easy to be identified in the archaeological record (Costa, 2017). If pit firings were indeed the main type of firing technique employed at Vinča sites, this would explain the general lack of corroborated evidence for pottery firing installations in the archaeological record of these settlements.

On this basis, we set up an experimental framework (Table 1) to test the efficiency in terms of temperatures and atmosphere of pit firing, one of the most likely diffused firing structures in prehistory. We combined this approach with laboratory investigations that allowed us to give particular attention to the observation of microstructural and mineralogical changes taking place in the clay objects fired in this type of installation. Therefore, our experiments helped us to create a reference collection to compare archaeological materials to and furthermore provided us a baseline to better understand how ancient firing processes might have worked.



Experiments	Laboratory experiments	Field experiment 1	Field experiment 2
Where ?	University of Tübingen	Kikinda Museum (Serbia)	Kikinda Museum (Serbia)
What?	Clay briquettes	Clay samples connected to thermocouples, briquettes and pottery	Clay samples connected to thermocouples and pottery
How?	Laboratory furnace, fully oxidising conditions	Pit firing, oxidising	Pit firing, oxidising/reducing
Duration	5 hours	4 hours	8.30 hours
Ceramic petrography	X	X	X
SEM	X	X	X
XRPD	X	X	X

Table 1: Summary of the experiments.

Sample	Fabric	Type of sample	Position in the firing	Colour	Qtz	Fsp	Cc	Ill	Msc	Chl	Mont	ML	Kao	Degree of vitrification	Optical activity	Temperature
ID11	3	Vinča bowl (DB)	/	Reddish yellow to light grey	X	X	X	X	X					V	Low/Absent	**850°C–900°C***
ID13	2	Vinča bowl	/	Light grey to dark grey	X	X		X	X					NV+	Moderate	**750°C–800°C***
ID14	1	Vinča bowl (DB)	/	Reddish yellow to light grey	X	X	X	X	X					IV	Moderate/Low	**800°C–850°C***
ID17	1	Vinča bowl (DB)	/	Light grey to dark grey	X	X	X?	X	X					IV	Moderate/Low	**800°C–850°C***
ID21	2	Decorated wall	/	Reddish yellow	X	X	X?	X	X					IV	Moderate/Low	**800°C–850°C***
ID23	2	Tisza lid	/	Reddish yellow to light brown	X	X		X	X					V	Low/Absent	**850°C–900°C***
ID24	3	Tisza lid	/	Reddish yellow to light brown	X	X		X	X	X?				NV+	High/Moderate	**700°C–800°C***
ID26	3	Tisza lid	/	Reddish yellow	X	X	X	X	X					V	Low/Absent	**800°C–900°C***
ID27	1	Vinča amphora (DB)	/	Light grey to dark grey	X	X	X	X	X					IV	Low	**800°C–850°C***
Mokrin	/	Raw Material	/	Light grey	X	X	X	X	X	X	X			/	/	**unfired**
Mokrin	/	Clay fraction	/	Light grey				X		X	X	X	X?	/	/	**unfired**
L1_A	Grog 20%	Laboratory	/	Reddish yellow to light brown	X	X	X	X	X					NV	High	**600°C**
L2_A	Grog 20%	Laboratory	/	Reddish yellow to light brown	X	X	X	X	X					NV+	Moderate	**700°C**
L3_A	Grog 20%	Laboratory	/	Reddish yellow to light brown	X	X	X	X	X					IV	Moderate/Low	**800°C**
L4_A	Grog 20%	Laboratory	/	Reddish yellow to light brown	X	X		X	X					IV/V	Absent	**900°C**
L1_B	Untempered	Laboratory	/	Reddish yellow to light brown	X	X	X	X	X					NV	High	**600°C**
L2_B	Untempered	Laboratory	/	Reddish yellow to light brown	X	X	X	X	X					NV+	Moderate	**700°C**
L3_B	Untempered	Laboratory	/	Reddish yellow to light brown	X	X	X	X	X					IV	Moderate/Low	**800°C**
L4_B	Untempered	Laboratory	/	Reddish yellow to light brown	X	X		X	X					IV/V	Absent	**900°C**
TC1_18	Untempered	Field 2018	Thermocouple 1	Reddish yellow to dark grey	X	X	X	X	X					NV	High	**651°C**
TC2_18	Untempered	Field 2018	Thermocouple 2	Reddish yellow to dark grey	X	X	X	X	X					NV	High	**618°C**
TC3_18	Untempered	Field 2018	Thermocouple 3	Reddish yellow to dark grey	X	X	X	X	X					IV	Low	**828°C**
TC4_18	Untempered	Field 2018	Thermocouple 4	Reddish yellow to dark grey	X	X	X	X	X					NV+	Moderate	**708°C**
TC1_19	Untempered	Field 2019	Thermocouple 1	Light grey to dark grey	X	X	X	X	X					IV	Low	**797°C**
TC2_19	Untempered	Field 2019	Thermocouple 2	Light grey to dark grey	X	X	X	X	X					IV	Low	**804°C**
TC3_19	Untempered	Field 2019	Thermocouple 3	Light grey to dark grey	X	X	X	X	X					IV	Low	**790°C**
TC4_19	Untempered	Field 2019	Thermocouple 4	Light grey to dark grey	X	X	X	X	X					NV	High	**690°C**
1_A	Grog 20%	Field 2018	Thermocouples 1–3	Reddish yellow to dark grey	X	X	X	X	X					NV+	Moderate	**618°C–828°C**
2_A	Grog 20%	Field 2018	Thermocouples 3–4	Reddish yellow to dark grey	X	X	X	X	X					NV+	Moderate	**708°C–828°C**
1_B	Untempered	Field 2018	Thermocouples 1–3	Reddish yellow to dark grey	X	X	X	X	X					NV+	High	**618°C–828°C**
2_B	Untempered	Field 2018	Thermocouples 3–4	Reddish yellow to dark grey	X	X	X	X	X					NV+	Moderate	**708°C–828°C**
1_S	Straw	Field 2018	Thermocouples 1–3	Reddish yellow to dark grey	X	X	X	X	X					IV	Low	**618°C–828°C**
2_S	Straw	Field 2018	Thermocouples 3–4	Reddish yellow to dark grey	X	X	X	X	X					NV+	Moderate	**708°C–828°C**

Table 2: Summary of the results of the analyses. DB=Dark-burnished pottery. Mineral abbreviations: Cc=calcite; Chl=chlorite; Fsp=feldspar; Kao=Kaolinite; Ill=illite; Msc=muscovite; Mont=montmorillonite; MT=Mixed layers; Qtz=quartz. SEM analysis (NV=no vitrification, NV+= intermediate between NV and IV, IV=initial vitrification, V= extensive vitrification), *=estimated maximum temperatures.

2. Laboratory investigation: materials and methods

2.1 Archaeological samples

Previous macroscopic and petrographic studies on ceramics from this site (Amicone et al., 2020a; Mirković-Marić and Amicone, 2019) showed that three main recipes (Figure 3, a. c. e.) were used in the pottery manufacturing of this community: natural clay, likely cleaned to remove the coarser inclusions (fabric 1), chaff tempered clay (fabric 2), and grog tempered clay (fabric 3).

The study of surface and fabric colours of these sherds also suggested that Vinča-style vessels were fired in oxidising or reducing conditions while Tisza style vessels and kitchen wares were produced solely under oxidising conditions (see supplementary data). As most of dark-burnished sherds display a lighter core and darker grey margins, these pots could have been fired via a two-step firing process that included a final reducing phase. This pattern has been observed at other Vinča sites (Amicone et al., 2020b).

A selection of nine samples (Table 2), which represent the three fabrics from Gradište-Iđjoš (described above), were chosen to be analysed using X-ray powder diffraction (XRPD) and scanning electron microscopy (SEM) to aid in a more detailed mineralogical and microstructural analysis. The aim in this high-resolution study is to identify more information on the firing procedure used to create these sherds.

XRPD was utilised to provide detailed mineralogical characterisation of pottery fragments to aid in the reconstruction of their original firing temperature (‘archaeothermometry’, see Rice, 2015, 99–116; Quinn and Benzonelli, 2018). This method makes use of the presence and absence of mineral phases that form or disappear at specific temperatures and atmospheric conditions (Gliozzo, 2020; Maggetti, 1982, 128; Maritan, 2004, 304; Nodari et al., 2007, 4668). The instrument used was Bruker D8 advance with a Cu-sealed tube (40kV/20mA). The parameters of the XRPD measurements used were Göbel mirror optics, a 0.2mm divergence slit, a fixed knife edge to suppress air scatter, sample rotation and a VÅNTEC 1-detector. The crystalline phases were identified using the pdf data from the 2006 International Centre for Diffraction Data-Joint Committee of Power Diffraction Standards (ICDD-JCPDS).

SEM analysis was used to assess the degree of vitrification, which is a crucial and easily measurable point in pyrotechnological studies (Faber et al., 2009; Maniatis and Tite, 1975; 1981; Mentesana et al., 2017; Tite and Maniatis, 1975a; 1975b). The samples were platinum coated and the analysis was carried out via a Hitachi TM3030+ using accelerating voltage 15 kV, an operating current of 110µA, and a variable working distance at 1000x and 2000x magnifications. The analysis was carried out on both the core as well as margins of most samples. The comparison between the degree of vitrification observed between the outer surfaces and the cores, could give us hints on the heating/cooling rate and the length of the firing (Mentesana et al., 2017; Thér et al., 2019, 1145)

2.2 Clay raw materials

The results of the petrographic analysis carried out on the geological samples (**Amicone et al.,** 2020a) gave a good indication of clay sources that could have been used by the ancient potters from Gradište-Iđjoš, showing that the Pleistocene raw material present in Mokrin has a very similar composition to the one of the archaeological samples. In addition, the current use of this clay by the modern brick industry of this area confirms its suitability for ceramic manufacturing. Mokrin lies c. 10 km form Gradište-Iđjos (location 2 in Figure 2), but outcrops of this Pleistocene clay could have originally been closer to the site and subsequently covered-up by more recent Holocene deposits. By taking this evidence into account, we therefore chose this material to be the clay on which we would carry out the laboratory and field experiments.

To have a detailed mineralogical characterisation of the raw materials from Mokrin, a sample of this source was analysed with XRPD with the same instruments and parameters mentioned above. A sample from the same source was also analysed after having extracted the clay fraction (<2 μm) from it via a sieving and sedimentation process. The concentrated suspension of the clay fraction was then poured on two glass slides to produce even and textured samples. In this way the intensities of the 00l-reflections from the clay minerals are significantly enhanced. After drying, both slides were measured by X-ray diffraction to characterise them at room temperature under natural conditions. In a second step, one of the slides was saturated at room temperature with ethylene glycol for several days and measured again. This procedure affects the swellable clay minerals like montmorillonite, resulting in an increasing of the c-lattice which results in a decrease of their 2θ angles in the diffractograms. The second slide was heated at 550°C for app. 30 minutes and measured as well. Under elevated temperatures, certain clay minerals undergo microstructural modifications, which also result in changes in the diffractograms, which gives additional information for their identification (Xanthopoulou et al., 2020 and literature therein).

2.3 Experimental briquettes

Two series of four briquettes (Table 1 and 2) reproducing recipes A (grog tempered 20%) and B (un-tempered) were manufactured. These reproduce the two most commonly fabrics found at the site of Gradište-Iđjos, fabrics 3 and 1 (Mirković-Marić and Amicone, 2019). These briquettes were made by mixing 20 g of sieved clay with de-ionised water. Clay was cleaned via a 5 mm mesh sieve to remove the coarser particles that would make the material less workable. The source of grog for the first series consisted of discarded broken vessels produced in the region of Gradište-Iđjos. These were fired in oxidising conditions in a furnace (Nabertherm P 300) at 100°C intervals between 600°C and 900°C (2 hours to reach the maximum temperature, 1 hour at maximum temperature, 2 hours of cooling).

These briquettes were analysed via XRPD and SEM, according to the same methodology used for the archaeological samples so that the results could be compared. In addition, all samples were analysed via ceramic petrography to assess optical activity of the matrix, as this could give an indication on the firing conditions and temperatures (Quinn, 2013, 23–33; Whitbread, 1989).

3 Results of the laboratory investigations

3.1 Archaeological samples

The results of the XRPD analysis reveal a mineralogical assemblage of quartz, feldspar, and calcite. Most of the samples also show illite (Figure 3, g), though the identification of this clay mineral is hindered when muscovite is present due to the overlap between the main illite and muscovite peaks (2θ=8.8°, d=10Å). The presence of illite indicates that the maximum firing temperature of the majority of the analysed pottery samples must have been below 850–900°C, at which their crystalline structure is destroyed (Gliozzo 2020). None of the samples exhibit the main peaks of chlorite (around 2θ=6°, d=14Å). Only sample ID 24 shows a weak diffraction peak that could correspond to this mineral.

The SEM results (Figure 3, b. d. f) show an initial to extensive degree of vitrification, which can also be confirmed by the level of optical activity observed during petrographic thin-section analysis (Table 2). Generally, no clear difference between the margin and the core of the samples have been observed. This degree of vitrification and the minerals found in most samples are compatible with temperatures approximately between 750–850ºC and not beyond 900ºC.

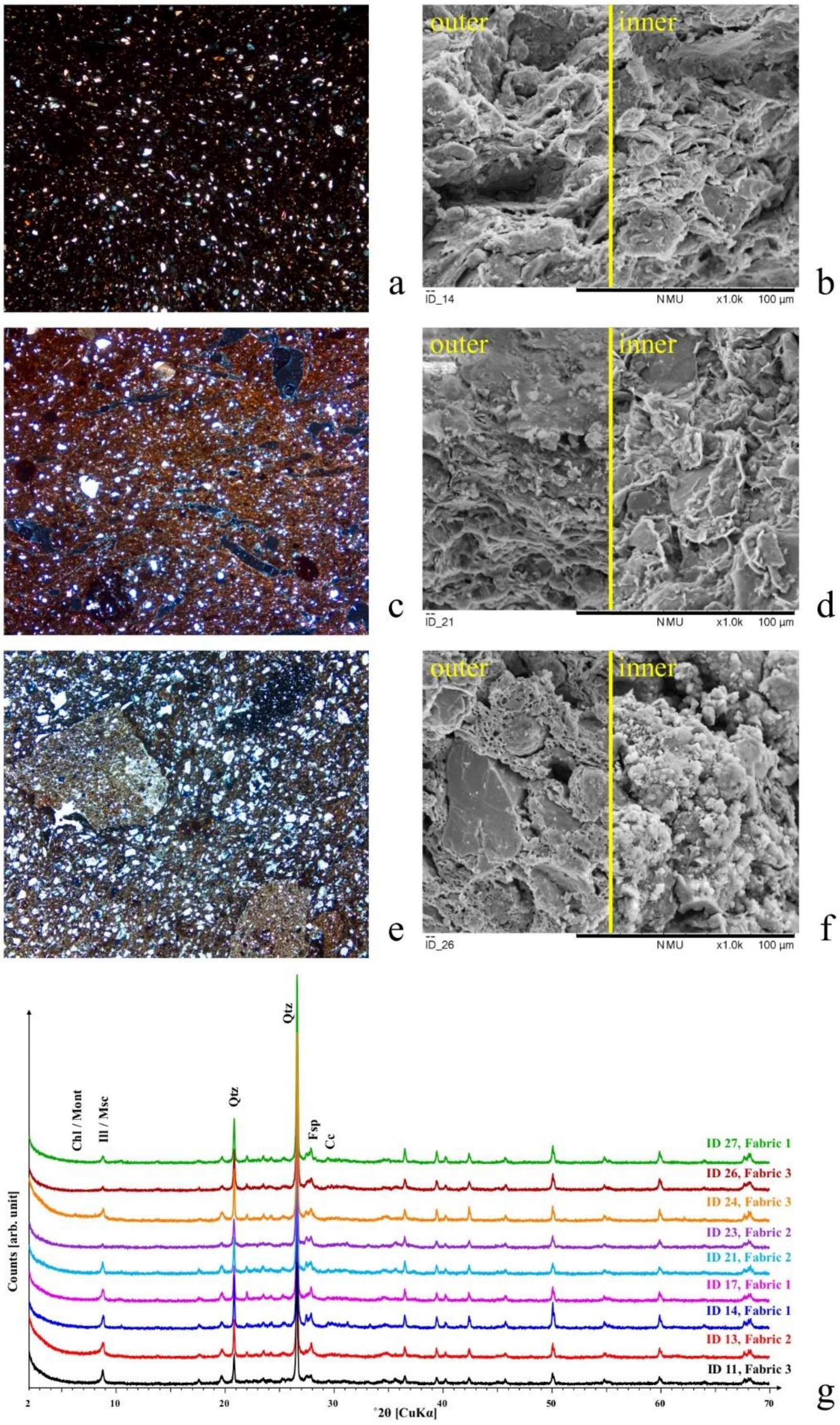


Figure 3: Thin section photomicrographs of selected ceramic from Gradište-Iđjoš: a) Fabric 1 (ID 14), XP; c) Fabric 2 (ID 21), XP; e) Fabric 3 (ID 24), XP. Field of view=4 mm a; 8 mm b and c.
Vitrification microstructure of selected pottery sherds from Gradište-Iđjoš, as seen in the SEM under secondary electron imaging: b) ID 14; d) ID 21; f) ID 26. See Table 2 for interpretation of vitrification stage and firing temperatures.
g) X-ray diffractograms of pottery sherds from Gradište-Iđjoš. Mineral abbreviations: Cc=calcite; Chl=chlorite; Fsp=feldspar; Ill=illite; Msc=muscovite; Mont=montmorillonite; Qtz=quartz.

The overall results confirmed the temperature range estimated by other studies mentioned above (e.g. Amicone et al., 2020b) for comparable Late Neolithic pottery (for Tisza style pottery see also Kreiter et al., 2017 and Szakmány et al., 2017).

3.2 Mineralogical characterisation of the clay from Mokrin

Petrographic analysis of the clay sample from Mokrin (Amicone et al., 2020a) has shown that this source is marked by the occurrence of quartz, feldspars, muscovite, and minor quantities of calcite and, rarely, metamorphic rocks (Figure 4). It therefore matches very well with the type of raw materials that were used by the Neolithic potters of the Gradište-Iđjoš site. XRPD analysis confirms the presence of these minerals and further suggests the occurrence of illite and chlorite/montmorillonite.

XRPD analysis of the clay fraction (Figure 4, Natural) shows the presence of montmorillonite and illite with their main peaks, respectively Mont_{001} (around 2θ=6°, d=14Å) and Ill_{001} (2θ=8.8°, d=10Å). The sharpness of the illite peaks implies well-crystallised illite minerals. The presence of mixed layers of montmorillonite-chlorite is also attested by a peak around 2θ=3° (ML_{001}).

In both the sample immersed in glycol atmosphere and the sample fired at 550°C (Figure 4, Glycolised and 550°C), the displacement of the Mont_{001} peak reveals the main peak of chlorite Chl_{001} (2θ=6.2°, d=14.3Å), thus confirming its occurrence. Finally, the peaks at 2θ=12.4° (d=7.2Å) and 2θ=25.1° (d=3.55Å), could be associated either with chlorite (Chl_{002} and Chl_{004}), but they also overlap with the peaks of kaolinite (Kao_{001} and Kao_{002}). A loss in intensity of the peaks attributed to both chlorite and kaolinite, is observed in the diffractogram of the fired sample compared to the one which was glycolised. This loss of intensity is relatively similar amongst all the four peaks of chlorite and not stronger in the two peaks overlapping with kaolinite. This leads to the assumption that kaolinite has little to no participation in the observed peaks and its presence cannot be confirmed. In summary, the clay minerals present in the sample from Mokrin include illlite, montmorillonite and chlorite.

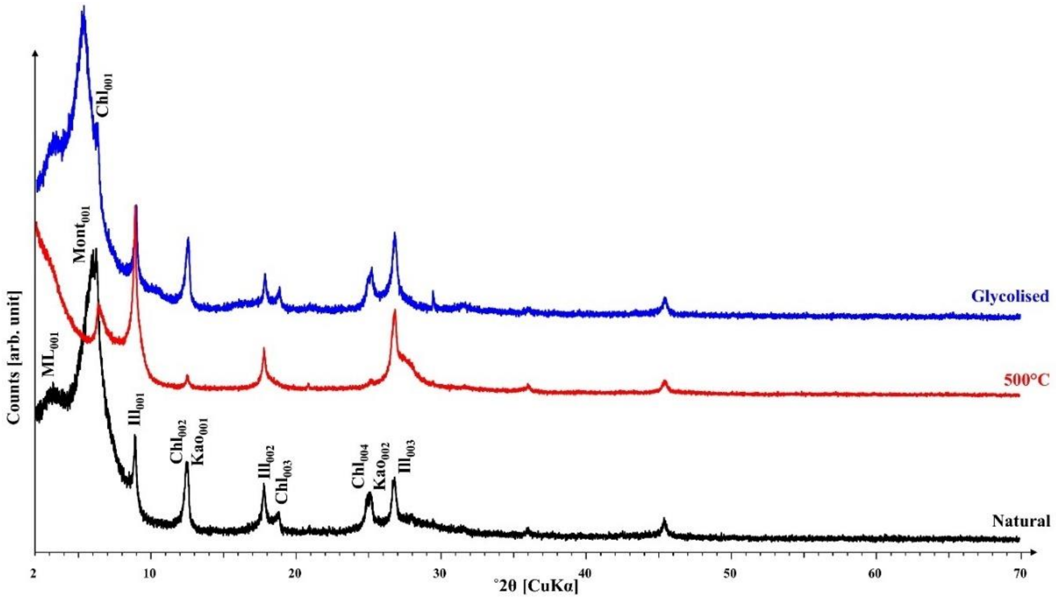


Figure 4: X-ray diffractograms of the separated clay fraction from Mokrin in natural condition, glycolised, and fired at 500°C. Mineral abbreviations: Chl=chlorite; Ill=illite; Kao=kaolinite; ML: mixed layers montmorillonite-chlorite; Mont=montmorillonite; 00l=hkl indices.

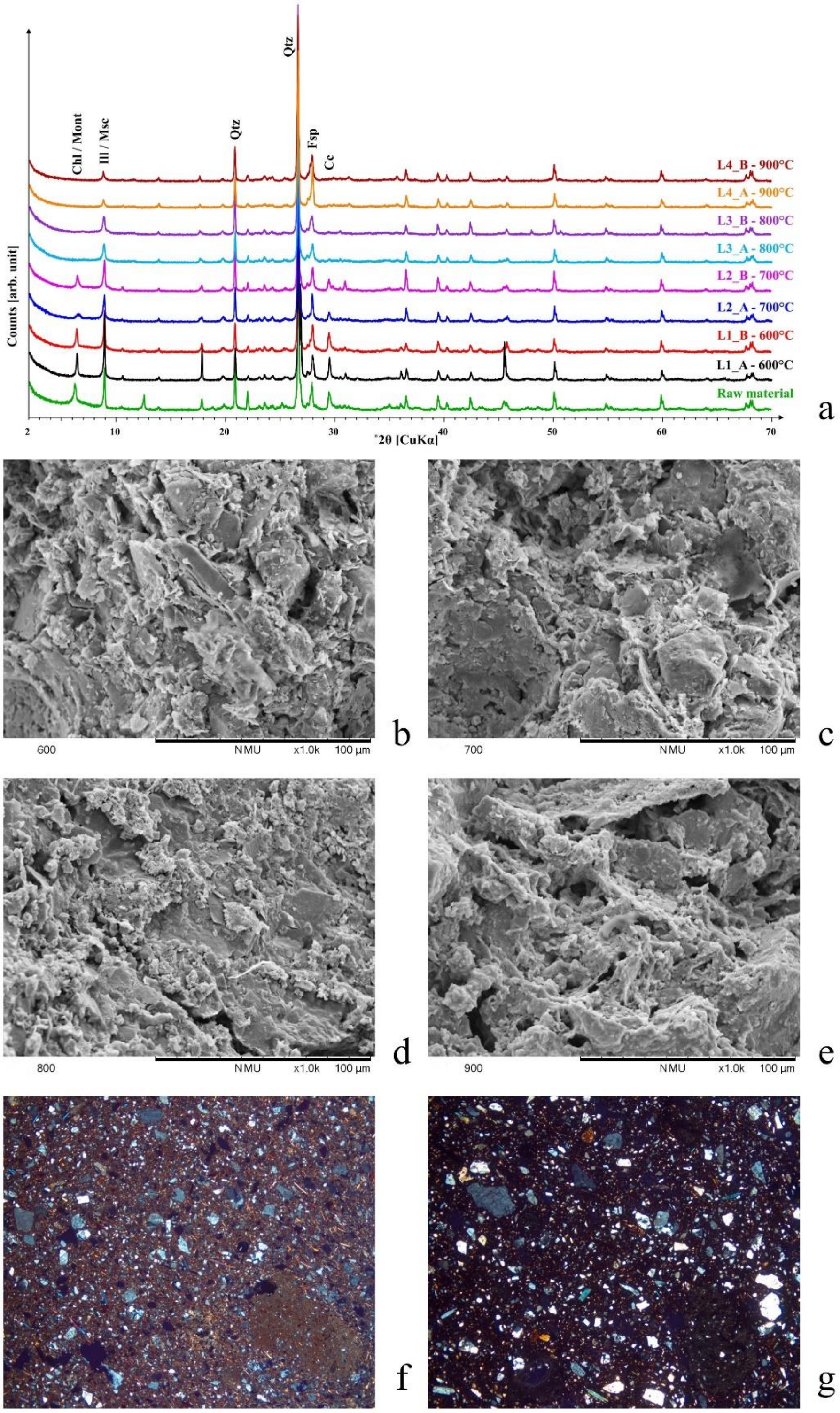


Figure 5: a) X-ray diffractograms of the briquettes fired in controlled conditions at different temperatures, compared with the raw material (clay from Mokrin). Mineral abbreviations: Cc=calcite; Chl=chlorite; Fsp=feldspar; Ill=illite; Msc=muscovite; Mont=montmorillonite; Qtz=quartz.
Vitrification microstructure of the briquettes: b) L1 (600°C); c) L2 (700°C); d) L3 (800°C); e) L4 (900°C).
Thin section photomicrographs of the low and high fired briquettes: f) L1 (600°C), XP; g) L4 (900°C), XP. Field of view=8 mm.

3.3 Experimental briquettes fired in the laboratory

The gradual refiring (600°C and 900°C) of the raw material from Mokrin, which was carried out in fully oxidised conditions, produced samples which display the mineralogical and microstructural behaviour of these materials during various stages of firing.

In the he fired briquettes the peak at $2\theta=6°$ ($d=14Å$) is related only to chlorite, as the main intensity of montmorillonite disappears at around 500 °C. Chlorite and calcite gradually decomposed and disappeared between 700–800°C and 800–900°C, respectively. At 900°C, only quartz, feldspars, and a very weak peak of illite are present (Figure 5, a). Interestingly, no nucleation of hematite was observed. This mineral in non-calcareous clay normally nucleates above over a wide range of temperatures from 400/450 to 850°C degrees in oxidising conditions (Gliozzo 2020). Hematite may be below the limits of detection or it couldn't nucleate under such short firing times (the entire process took only 5 hours overall with 1 hour of soaking time). The rise in temperature also corresponds with an increase of the degree of vitrification of the clay body that can be observed under SEM analysis (Figure 5, b–e). The results show that initial vitrification starts at 800°C, but the edges of some clay plates seem to start to buckle and round at lower temperatures. At 900°C a microstructure compatible with extensive vitrification is present, but few areas of the samples look still unvitrified. The shift from anisotropic to isotropic behaviour of the clay matrix has also been observed via ceramic petrographic analysis (Figure 5, f–g) showing decreasing optical activity (Quinn, 2013, 94) that is completely absent at 900°C.

4 Field experiments: Material and Methods

The experimental framework carried out in the field was set up according to common ethnographic evidence (Gosselain, 1992; Livingstone Smith, 2001; Rice, 2015; Roux, 2019, 110–121), previous research of Late Neolithic pyrotechnology (see above), and the results of our laboratory investigations. A series of parameters such as raw materials, modelling techniques, drying stages, fuel, and firing steps have been considered to provide conditions as similar as possible to the those most likely used by ancient potters.

4.1 Raw material processing

As described above, compositional analyses applied on archaeological samples gave a good indication of the raw materials to use and on how to process them to obtain clay pastes with the similar compositional and physical features of the archaeological pottery.

A total of 40 kg of clay was excavated. The clay was spread and dried in the open air. After this, the selected clay was then crushed and sifted through a 5mm mesh. After cleaning and sieving, the clay was put in 4 different containers (10 kg each) and mixed with water. For several days the clay was stirred occasionally and then left to dry, during the night it was covered under a plastic bag. The recipes used have been produced as follow: recipe A: tempered with grog (20% of the clay mass), B: untempered, S: straw tempered (Table 2).

4.2 Modelling of replica vessels and drying

The experimental vessels were fashioned in accordance with known Late Neolithic pottery techniques such as coiling, pinching, and moulding. Surfaces were refined through smoothing and burnishing by using wood, bone, and stone tools. Smoothing was applied by adding water and refining the surface with fingers or scrapers. Burnishing was performed by rubbing the leather hard clay surfaces (while in an almost dried stage) using tools with polished surfaces such as cobbles and animal bones.

Three series of six experimental briquettes (Table 2) reproducing recipe A (20% of the clay mass), recipe B (untempered), and recipe S (straw tempered) were produced by mixing 20g of the sieved clay from Mokrin. Additionally, eight clay samples (paste B, 20g each) were prepared to be attached to the thermocouples (TC in Table 2) used during the experiments (see *temperature monitoring and firing*). These experimental samples were produced to be fired in field experiments and analysed in the laboratory via XRPD, SEM, and ceramic petrography according to the methodology defined above. The vessels obtained from these firings were to be used by the museum for educational purposes.

After the modelling phase, all the experimental vessels were left to dry for several days to ensure complete evaporation of water within the paste. During this step, the loss of water corresponds to a limited reduction of the vessel's size and weight, making the vessel ready for firing.

4.3 Fuel and firing structures

Birch (*Betula pendula*) was used as a fuel for firing pottery, because of its abundance in this region in Neolithic times (Magyari, 2002; Magyari et al., 2010). A total of 72.45 kg of birch was used during the first experiment and a total of 64 kg during the second firing. Both logs and dried branches were used according to the step of the process.

Two circular pits were dug for the experiments. They had a diameter of 130 cm and a depth of 30 cm, enough to manage the firing from the outside (e.g. adding wood or moving the vessels) and, at the same time, to reduce the heat dispersion.

4.4 Temperature monitoring and firing

Due to the high humidity of the ground soil after a period of prolonged rain, the bottom of the pits were covered with a layer of wood in order to have a flat and dried base on which to place and fire the clay vessels. Before the firing experiment, four thermocouples were installed within the pits to ensure a detailed and controlled recording of the temperature throughout the process. In both cases, the thermocouples were placed in different areas of the pit (Figure 6, a and 7, a), with their upper parts covered with clay paste of type B to monitor the exact temperatures to which this type of paste was exposed. In this way, mineralogical and microstructural changes observed via scientific analysis could be correlated to temperature changes observed during the firing. In the second experiment we also added three thermocouples (TC 5, 6, 7) not covered with clay to measure the gas temperatures in different points of the pit (Figure 7, a).

The actual firing process involved three main steps, monitored via photos and temperature variations:

Step 1: Heating (Figure 6, c and Figure 7, c)

Vessels were slowly heated to eliminate water absorbed by them during the night. The removal of excess water allows the vessels to withstand higher temperatures and thus avoid thermal shock. This process had four stages:

-The vessels were placed within the pit, forming a circle along the external diameter of the bottom.

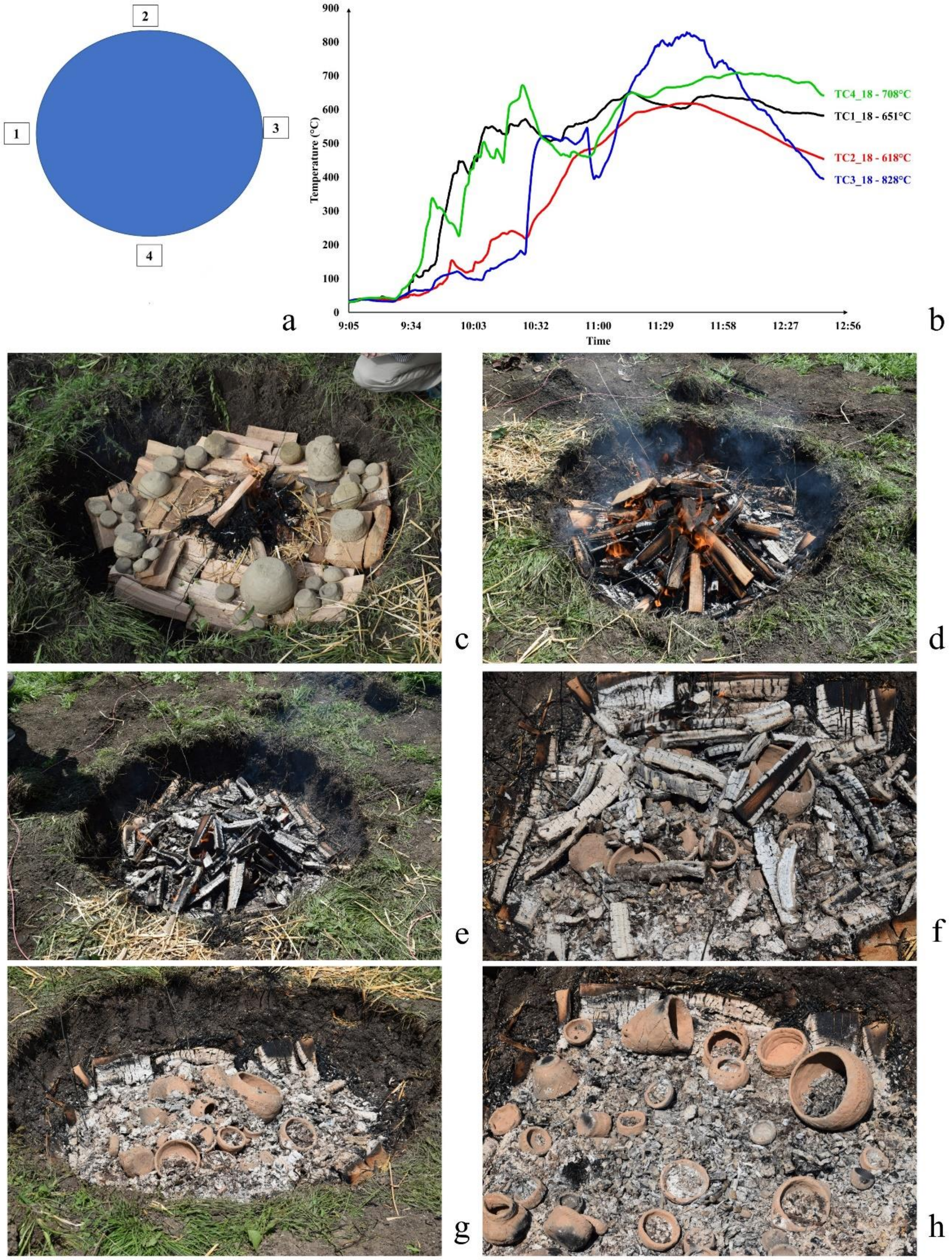


Figure. 6: Experimental pottery firing 2018. a) thermocouples position within the pit; b) graph of the temperatures reached during the experiment; c) heating; d–e) firing; f–g) cooling; h) recovery of the vessels.

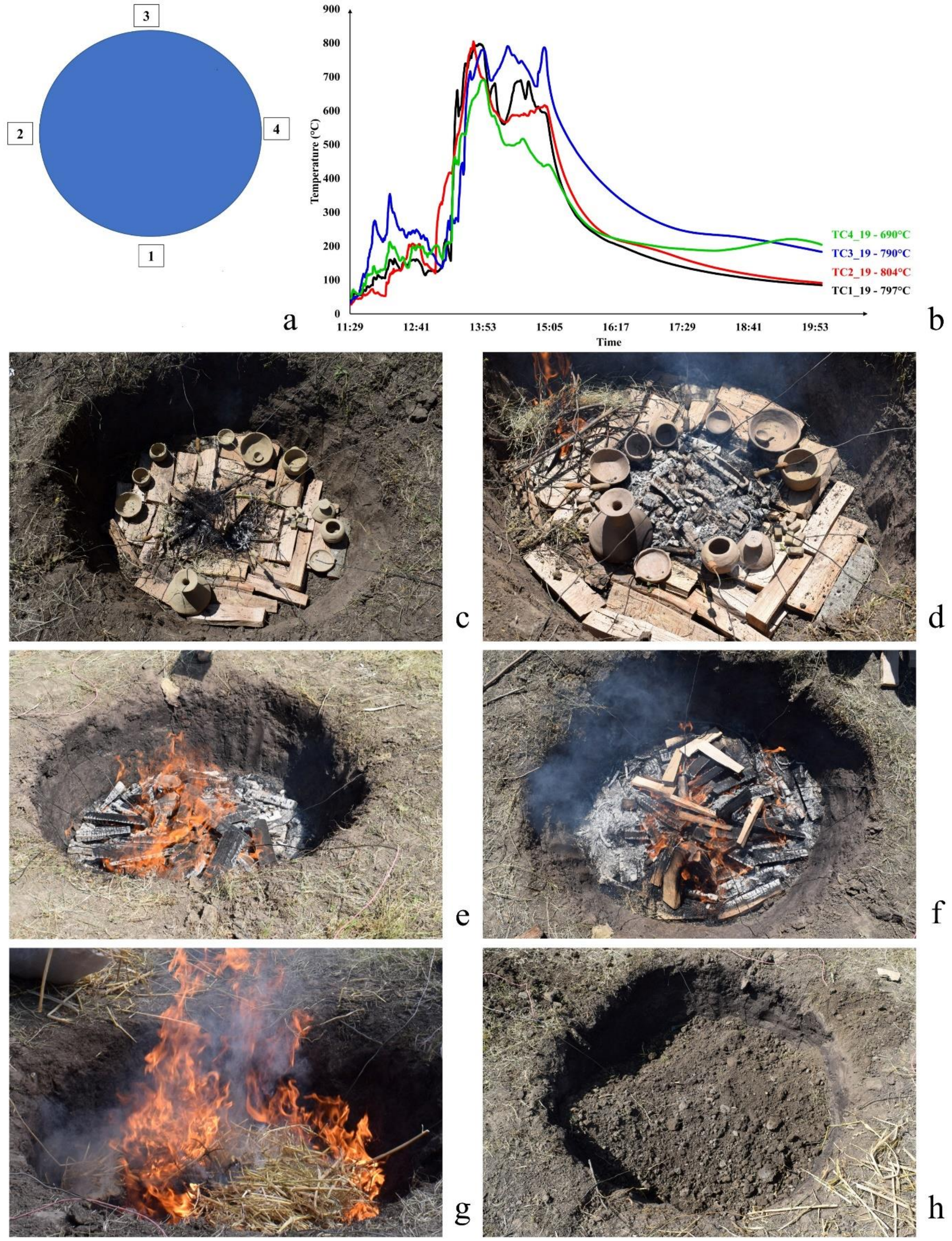


Fig. 7: Experimental pottery firing 2019: a) thermocouples position within the pit; b) graph of the temperatures reached during the experiment; c–d) heating; e–g) firing; h) covering the pit with sediment for favouring reducing conditions and slow cooling.

-The fire was ignited in the centre of the pit with the vessels surrounding it. The vessels were moved and rotated regularly to ensure a complete drying of the body and the loss of most of the water absorbed within the clay paste.

-The vessels were then moved gradually towards the fire and the embers, placing them closer and closer at the centre of the pit.

-Fire was then ignited at various locations around the vessels, at first with some distance to avoid thermal shock which could damage the vessels. After this, the fire was gradually moved towards the vessels until they were completely encased.

Step 2: Firing (Figures 6, d–f and 7, d–f)

Wood was added when necessary to ensure a gradual and continuous firing until a glowing red colour of the vessel's surface was observed. This was kept up for approximately 30 minutes to ensure the production of usable ceramics. A total of four people were involved in the process.

Step 3: Cooling and recovery of the vessels (Figure 6, g–h and 7, g–h)

After the 30-minute period during which we sustained approximately the same temperature, the process concluded with a cooling phase, during which temperatures gradually were decreased, allowing the pottery to avoid thermal shock and thus damage. In terms of the cooling and recovery step of ceramic production, the two experiments diverge in how the vessels are treated. The 2018 experiment concluded after the flames gradually went out and the temperature of the vessels gradually decreased, all under the constant presence of oxygen. The vessels were collected about two hours as soon as the temperature was low enough to avoid cracking.

The second experiment, conducted in 2019, ended with a reduction phase. This reduction phase involved intentionally creating an environment which is low in oxygen and produces a lot of smoke. Once the final firing temperature was reached, the fire was covered with sawdust and straw and immediately smothered with sediment. This caused the production of smoke within the pit which was absorbed by the vessels and is the source of their dark colour. In this case, the vessels were collected after about six hours, as the cooling of pottery buried within a pit requires a longer time than an open pit to produce the dark colour.

5 Results of the field experiments

5.1 General observations

In review of both experiments, we were able to define some key points about ceramic production in pit firings that we experienced directly while managing the firing, and indirectly through the observed reactions in the vessel replicas and experimental samples (briquettes and clay attached to the thermocouples).

Commencing the ceramic firing was easy for the first experiment, as climatic conditions at the time were favourable to firing procedures. The second firing experiment, however, was challenging due to strong winds. Nevertheless, in both procedures we gradually reached the temperatures necessary to produce usable vessels (750–850ºC) in about two hours and three hours respectively.

Beyond this general achievement we observed that ensuring a homogeneous, gradual, and continuous heating of all the vessels may prove to be a difficult task, as huge differences in the temperature in various areas of the pit were observed after the first step of the firing. Therefore, we suggest that sufficient control of firing temperatures requires experience in organising the distribution of the vessels within the pit. Firing success, we also observe, may relate to the type of fuel used, as well as a coordinated teamwork.

Those ceramics fired in oxidising conditions (2018 experiment) show clear colour differences compared (supplementary Table 1) to those fired with a two-step process including a reduction phase (2019 experiment). Replica vessels and experimental samples fired in the 2018 experiment show a homogenous light-brown colour along the internal and external surfaces. This homogeneity is sometimes featured by limited dark grey spots which can be considered normal in a firing where fuel is directly intermingled with the vessels.

The 2019 experiment, that ended with a reduction phase, produced ceramics with less homogeneous surfaces and colours spanning from light grey to dark grey. Despite the inhomogeneity of the surface colour, the reduction was quite successful, as none of the replica vessels or experimental samples has shown brown or reddish spots on the surfaces.

These experiments were a success in terms of the integrity of the replica vessels and all three recipes used (A, B, S) responded well to the firing process. Only a few small vessels displayed limited and superficial microfractures. This achievement was probably due to the gradual drying and relatively slow increase of temperature obtained in the pit that limited the exposure of the replica vessels and experimental samples to thermal shock.

The experimental framework provided an empirical reference collection characterised on a scientific basis (temperature recording and compositional features) suitable for ancient pyrotechnological studies. We associated and documented steps of production and firing sequences, which usually are reconstructed through ethnoarchaeological analogies, to specific results in terms of maximum temperatures, heating and cooling rates, soaking time and thermal homogeneity. These references samples can be used to help us to understand archaeological specimens and to reconstruct maximum temperatures, heating and cooling rates, and various other steps in the process.

5.2 Laboratory analysis of the experimental samples

The results of the XRPD (Figure 8, a; 9, a; 10, a) analysis that was performed on experimental samples fired in the field experiments show the presence of illite, quartz, feldspars, and calcite. In samples exposed to lower temperatures, it is still possible to observe chlorite. Hematite or magnetite did not nucleate in any of our samples. The degree of vitrification observed with SEM analysis (Figure 8, b–e; 9, b–g and 10, b–e) and the optical activity (Figure 8, f–g; 9, h–j and 10, f–g) are highly variable according to the position of the samples in the pit. The samples exposed to higher temperatures (>800°C) show initial vitrification and low to absent optical activity in their thin sections. Those samples which were exposed to lower temperatures (<800°C) show no vitrification or only initial vitrification and display higher optical activity in thin section. No drastic difference has been observed in the degree of vitrification between the core and the margins of the samples.

Our overall results (Table 2) clearly indicate that the experimental samples, even if fired in the same process, were exposed to various temperatures that resulted in different mineralogical and microstructural characteristics. For this, good parallels can be found in the variability observed in the archaeological samples from various Vinča sites (Amicone et al., 2020b).

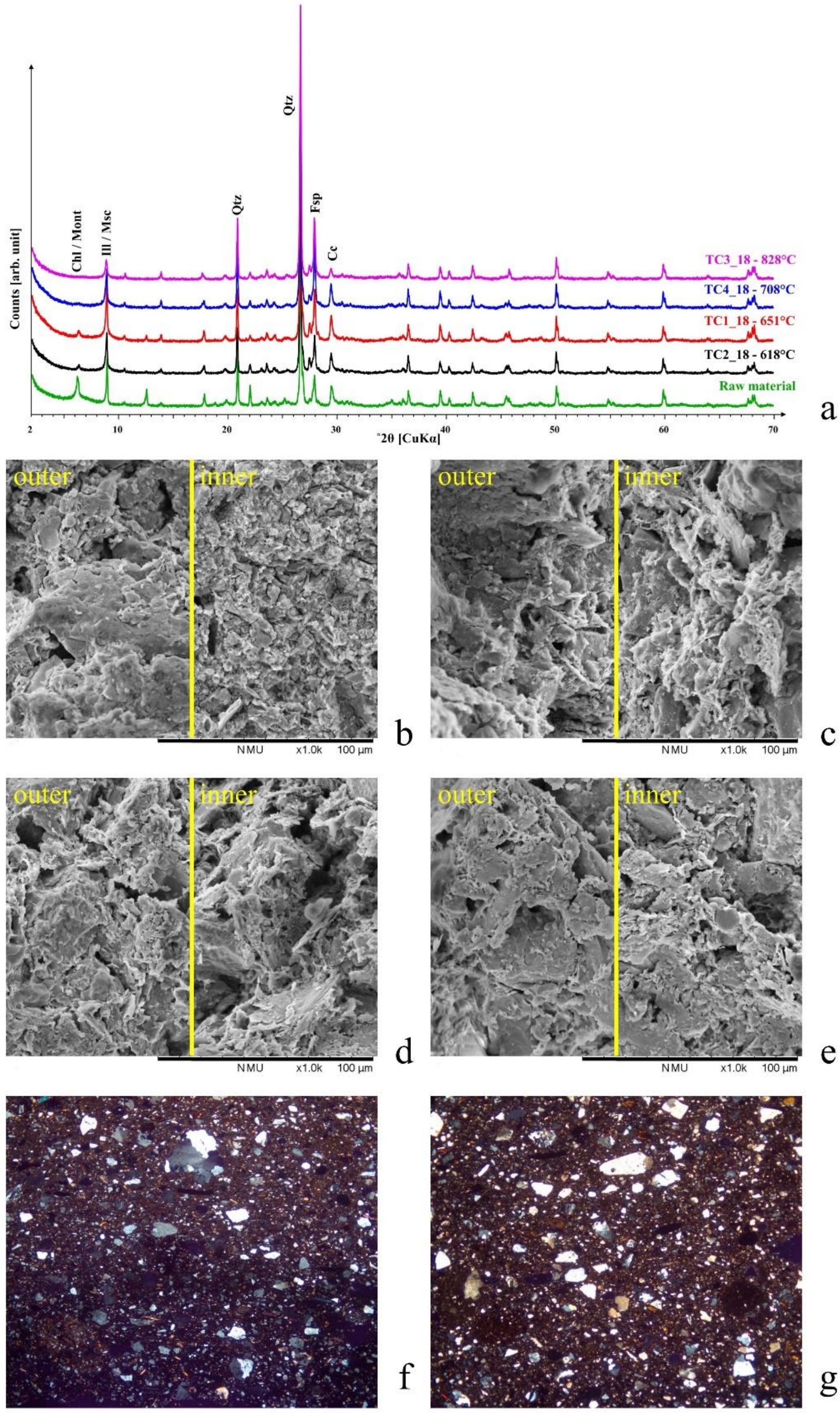


Fig. 8: a) X-ray diffractograms of the clay attached to the thermocouples in the 2018 field experiment, compared with the raw material (clay from Mokrin). Mineral abbreviations: Cc=calcite; Chl=chlorite; Fsp=feldspar; Ill=illite; Msc=muscovite; Mont=montmorillonite; Qtz=quartz.
Vitrification microstructure of the clay attached to the thermocouples in the 2018 field experiment: b) TC2_18 (618°C); c) TC1_18 (651°C); d) TC4_18 (708°C); e) TC3_18 (828°C).
Thin section photomicrographs of the clay attached to the thermocouples in the 2018 field experiment (highest and lowest temperatures): f) TC2_18 (618°C), XP; g) TC3_18 (828°C), XP. Field of view=8 mm.

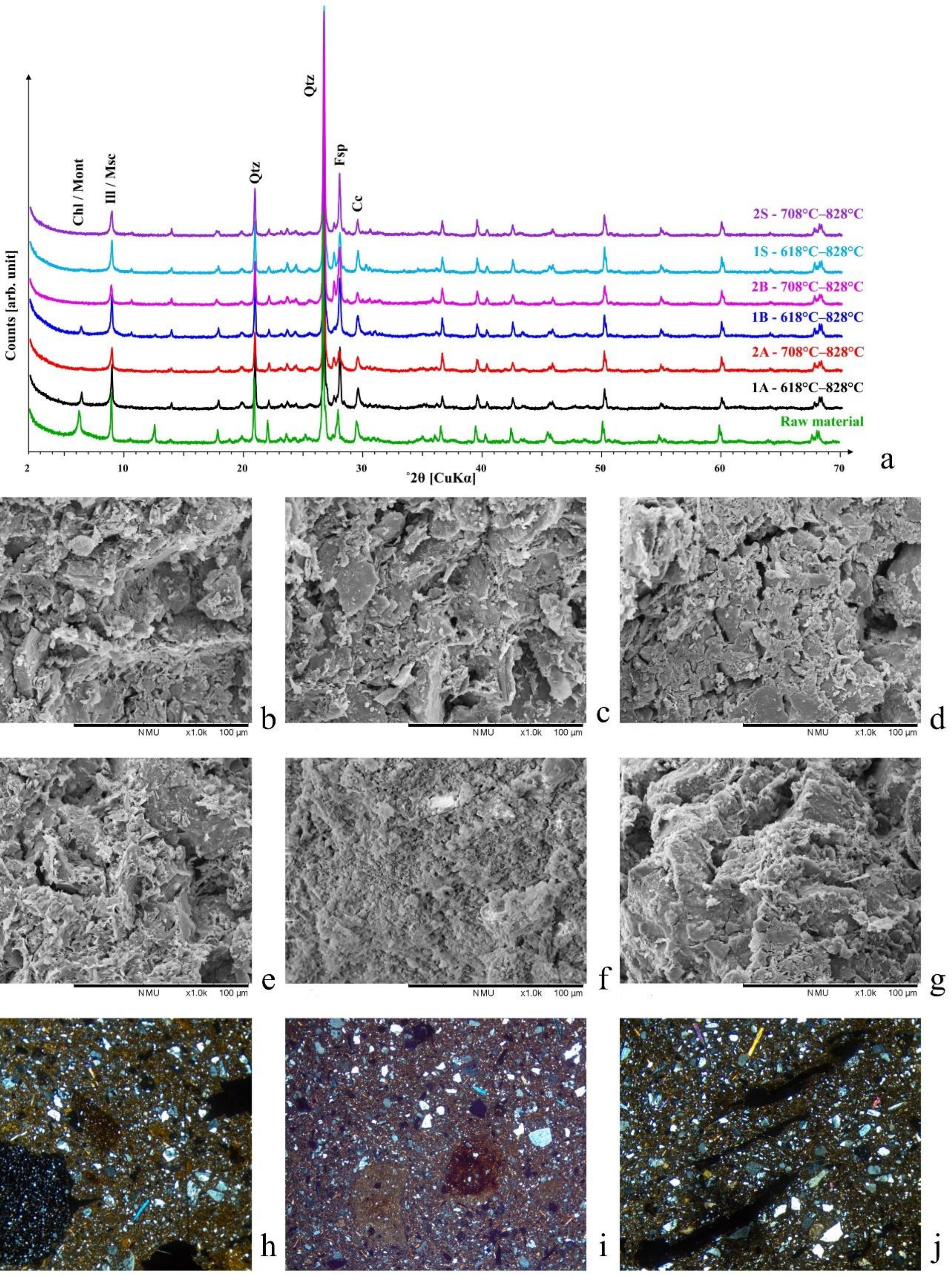


Fig 9: a) X-ray diffractograms of the briquettes fired in the 2018 field experiment, compared with the raw material (clay from Mokrin). Mineral abbreviations: Cc=calcite; Chl=chlorite; Fsp=feldspar; Ill=illite; Msc=muscovite; Mont=montmorillonite; Qtz=quartz.
Vitrification microstructure of the briquettes fired in the 2018 field experiment: b) 1A (618–828°C); c) 1B (618–828°C); d) 1S (618–828°C); e) 2A (708–828°C); f) 2B (708–828°C); g) 2S (708–828°C).
Thin section photomicrographs of the briquettes fired in the 2018 field experiment (samples 1, 618–828°C): h) 1A, grog tempered, XP; i) 1B, untempered, XP; j) 1S, organic tempered, XP. Field of view=8 mm.

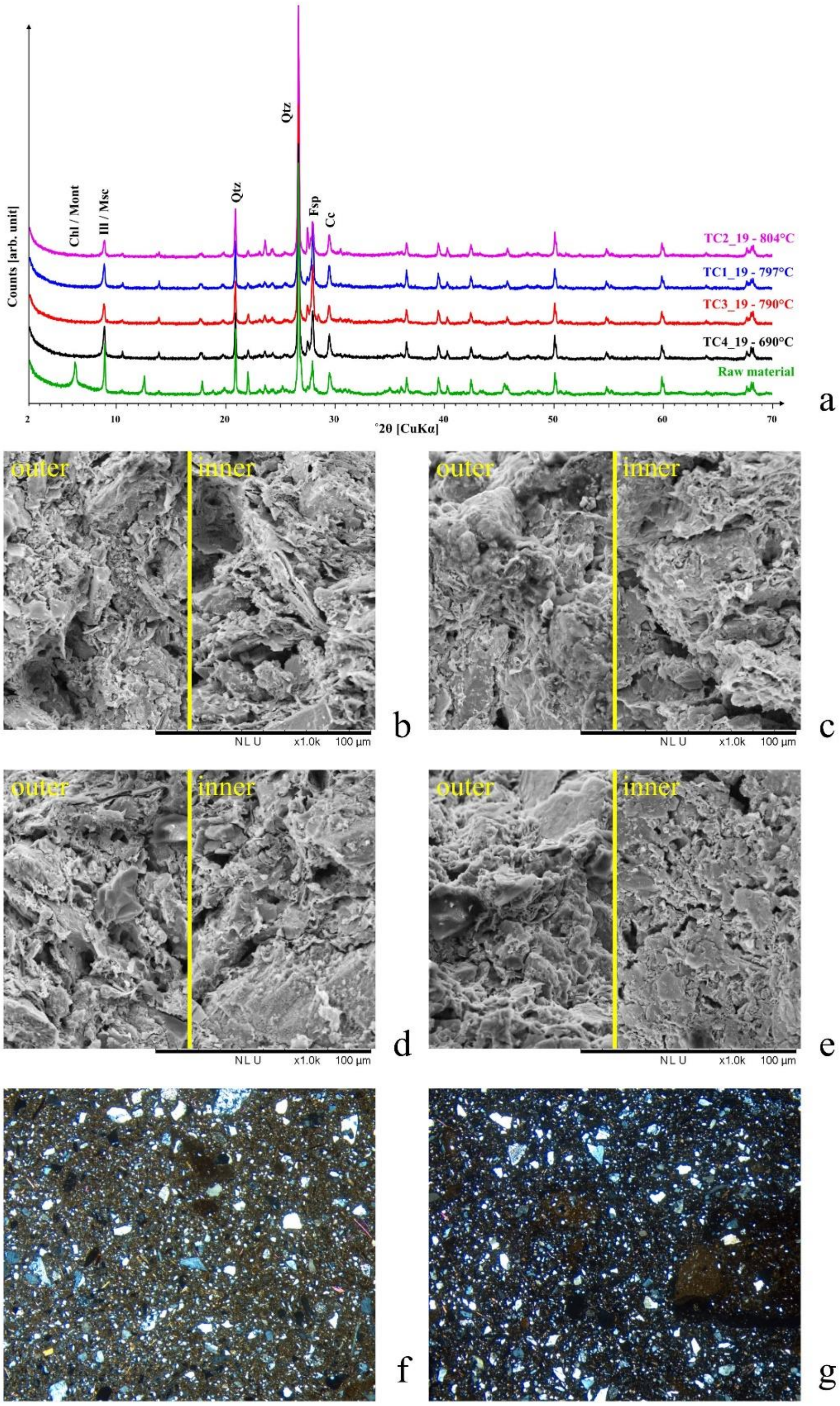


Fig 10: a) X-ray diffractograms of the clay attached to the thermocouples in the 2019 field experiment, compared with the raw material (clay from Mokrin). Mineral abbreviations: Cc=calcite; Chl=chlorite; Fsp=feldspar; Ill=illite; Msc=muscovite; Mont=montmorillonite; Qtz=quartz.
Vitrification microstructure of the clay attached to the thermocouples in the 2019 field experiment: b) TC4_19 (690°C); c) TC3_19 (790°C); d) TC1_19 (797°C); e) TC2_19 (804°C).
Thin section photomicrographs of the clay attached to the thermocouples in the 2019 field experiment (highest and lowest temperatures): f) TC4_19 (690°C), XP; g) TC2_19 (804°C), XP. Field of view=8 mm.

6 Discussion

6.1 Firing procedures and firing installations

Previous studies discussed above, together with new results from our research, provide elucidated insights into the pottery firing procedures at the site of Gradište-Iđjoš and allows for us to have a more nuanced understanding of ceramic-pyrotechnology in sites marked by Vinča material culture in general.

Archaeometric analyses run on archaeological samples from various sites (Amicone et al., 2020b and literature therein) suggested that potters from these communities fired ceramics to various temperatures that did not appear to have exceeded 900°C on a regular basis.

The rare development of extensive vitrification and the absence of iron oxides such as hematite that is supposed to nucleate at relatively low temperatures (Maritan, 2004), could furthermore indicate that only short firing procedures were employed.

However, the relatively homogeneity in the degree of vitrification observed between the outer surfaces and the cores of the archaeological vessels suggests relatively slow heating and cooling rates (Thér et al., 2019, 1145). Finally, the colours of the archaeological sherds indicate that either firing in oxidising or reducing conditions were possibly applied (see supplementary data). It can be conjectured from the heterogenous colour of the archaeological sherds that potters were not always able to control the amount of oxygen reaching their vessels during firing and in some cases might have not even considered of doing so. Furthermore, the analysis carried out on samples from Gradište-Iđjoš seems to suggest that similar pyrotechnological procedures were applied to produce both Vinča and Tisza style vessels, with the exception that the latter were never fired under reducing conditions.

The results of the mineralogical and structural analysis of the experimental samples produced during the field experiments matched well with those selected from among the archaeological materials of Gradište-Iđjoš and other Vinča sites (Amicone et al., 2020b and literature therein). This implies that the thermal profile (maximum temperatures, heating and cooling rates, soaking time duration and thermal homogeneity) and the variable atmospheric conditions applied during our experiments using a pit firing installation were compatible with the one used by the ancient potters.

Several authors (e.g. Gibson and Woods, 1990; Kingery, 1997; Rye, 1981; Tite, 1995) have already claimed a direct connection between thermal profiles and the types of installations, often contrasting "open firings" with "kiln firings". However, ethnographic studies (e.g. Gosselain, 1992; Livingstone Smith, 2001) casted some scepticism regarding this hypothesis, showing that similar thermal profiles can be obtained by using different types of firing structures. Clearly, each type of installation is characterised by a set of various possible firing procedures (Thér et al., 2019). This indicates that a direct relationship can only be drawn between pottery characteristics and firing procedures and not necessarily with the type of pyrotechnological installations utilised. In addition, it has been observed (Rice, 2015, 166), that the usual differentiation between "open firing" and "kiln firing" should be abandoned as the main differentiation of firing structures. Instead, a distinction should be made regarding the degree of insulation and the separation between fuel and vessels.

As mentioned above, the homogeneity in the degree of vitrification observed in the archaeological materials suggests that these were produced in a process marked by relatively slow heating and cooling rates, that is unlikely to be compatible with a bonfire (Thér et al., 2019), but matches well with pit firings. In addition, previous experiments (Vuković, 2018) showed that it was not possible to reproduce the full range of Vinča ceramics with bonfires, as it is too difficult to control temperatures and atmospheric conditions in this type of procedure.

However, while we can state that it is possible to apply a pyrotechnological procedure compatible with the one used to produce Vinča ceramics by using pit firings, there is not enough

evidence to rule out that similar results could be obtained by using a simple single-chamber kiln. One should also bear in mind that, even if no secure evidence for kilns within Vinča sites has been provided, it is well known that such pyrotechnological installations were in use in the Balkans since the Early Neolithic period (Linda, 1984, 130–170).
Kilns have the advantage to reduce the effects of prevailing winds and the time to reach the maximum temperatures. In addition, such installations could facilitate the controlled duration of the soaking time, better redox conditions, and slow the cooling process down. Nevertheless, the advantages of using kilns are at the same time largely influenced by the type of kiln that is utilised. Firing in a simple kiln, where fuel is in direct contact with the vessels, could present difficulties similar to those we experienced in our pit firings (Amicone et al., 2019; Cuomo di Caprio, 2007, 508–526).

6.2 The social practice of firing

By combining archaeometry and experimental archaeology this study allowed us to directly experience the complete process of pottery making. Through our experimental procedure, we not only had the opportunity to understand how the experimental replicas reacted to specific firing sequences and ranges of temperatures, but we also got to experience the social and sensorial implications associated to pottery firing practice.
In review of the presented information above, we have found that our applied firing procedure could have been compatible with the ones used by potters of Vinča communities. But we have also concluded that a similar process could have been obtained with a simple type of kiln. It should be emphasised that the choosing of one type of firing structure over another could be dictated by a variety of different reasons that go beyond functionalist aspects and could be related to the social organisation of production within a community and its craft traditions (Peacock, 1981; Van der Leeuw, 1977).
It has been suggested (Spataro, 2018) that Vinča ceramics could have been produced by specialised and skilled potters that were experienced not only in the modelling and decoration pottery, but also in firing it. Nevertheless, pottery production could have been restricted to a household level as indicated by the absence of distinctive pottery workshop areas in Vinča sites (Amicone et al., forthcoming).
We found critical to mention the possibility that even in the absence of formalised systems such as a pottery workshop, specialised and skilled potters could have developed their abilities in household production contexts through repeated experience and prolonged practice (Forte, 2019).
The strong conservatism that characterises pottery productions at Vinča settlements (Amicone et al., 2020b) could also indicate a vertical and direct transmission of pottery know-how from parents to offspring (Cavalli-Sforza and Feldman, 1981), as is typical for household productions. In this scenario, the practice of pottery production can be considered an act that embodied strong symbolic and social values through which the apprentice is exposed to the acquisition of both technological knowledge and social norms (e.g. Manem, 2020 and literature therein).
Recently the special relationship that south-eastern populations of the 5th millennium seem to have had with fire has also been emphasised (Gheorghiu, 2019). This is highlighted by extended destructive horizons that were connected to the ritual practice of house burning (Stefanović, 1997) and the abundant presence of different pyrotechnological devices such as ovens, heaters, and fire starters. This led to the assumption that the degree of proximity between people and fire could have also had ritual connotations (Gheorghiu, 2019, 43).
According to this view it is possible to assume that the practice of pottery firing embodied a strong social value, through which the individual practitioners were sanctioning their belonging to their community of practice (Lave and Wenger, 1991). Ethnographic studies (e.g. Djordjević,

2019; Gosselain, 1992; Livingstone Smith, 2001) and what we experienced through our own experiments have shown that team work and especially the coordination among the different participants had a crucial role in the success of the firing process.

It is also important to stress that from a mere practical point of view, firing pottery in a bonfire or a pit requires the participants to closer connect not only on an interpersonal level, but also with the fire as well as with the smoke (Fowler, 2008; Lawton, 1967). In our experiment, at least four persons were involved, and the visual and thermal sensations played a significant role in the overall experience and still forms an important part of the stories we tell of these days. A kiln firing, on the other hand, is usually a longer process (up to 16 hours), but theoretically can be run by one individual or multiple individuals working in shifts (Amicone et al., 2019). In addition, by using a closed structure, a different bodily sensorial experience comes along with the process. It seems, therefore, only natural that one develops a shallower connection to the produced objects in contrast to what happens in a pit firing where one should constantly control the distribution of the pots within the pit and their relation to the fuel.

Considering this, the choice of bonfire or pit firing over closed structures does not necessarily imply that potters are less technologically advanced or specialised but could be influenced by a number of intangible factors dictated by the social context of production.

7 Conclusions

Through the application of an approach that integrates archaeometry and experimental archaeology, and by using the case of study of Gradište-Iđjoš as reference, our research provides a contribution to the understanding of Late Neolithic pyrotechnology in the Balkans from both a technological and a social point of view.

This approach has been applied to directly experience the pit firing process in order to set parameters for recording and studying prehistoric firings, that goes beyond the mere estimation of firing temperatures. Our study is in line with others (e.g. Gosselain, 1992) in its exhibition that highly variable firing temperatures characterise the firing in traditional installations such as pits.

This means that vessels fired in the same process or even portions of the same vessel could be exposed to drastically different temperatures. At the same time, it shows the limitation of focusing archaeometric investigations only on the estimation of maximum temperatures that vessels might have been exposed to.

In general, by performing these experiments, we were able to document the entire production process and associate each step in the firing sequence to experimentally produced ceramics to form a reference collection suitable for ancient pyrotechnological studies. We also provide a preliminary framework of data that can be expanded upon in future investigations and can be used in other projects.

Most importantly, our study emphasises that the knowledge, the experience, and the social context in which people are operating must all be considered as aspects influencing their choice for one firing procedure over another. As a note of caution, the results discussed above did not allow us to draw a direct and univocal relationship between firing procedures and firing structures that were applied to produce ceramics at Gradište-Iđjoš. Nevertheless, they perhaps inform us about one of the likely firing procedures applied in the past and give us a set of information that, once discussed in the view of the social context of production, could give more nuanced insights into pyrotechnological and cultural choices of the Late Neolithic communities.

References

Amicone, S., Radivojević, M., Quinn, P. S., Rehren, Th., forthcoming. Pottery technology at the dawn of metallurgy in the Vinča culture. In: Radivojević, M., Roberts, B.W., Kuzmanović-Cvetković, J., Marić, M., Rehren, Th. (Eds.), The rise of metallurgy in Eurasia: Early Metallurgy and Society in the Balkans. Archaeopress, Oxford.

Amicone, S., Raj Mathur, A., Deepthi Pavitra, R., Mirković-Marić, N., Pantović, I., Kuzmanović-Cvetković, J., 2020a. Beneath the surface: exploring variability in pottery paste recipes within Vinča culture, Serbia. Quaternary International 560–561, 86–101.

Amicone, S., Radivojević, M., Quinn, P.S., Berthold, C., Rehren, Th., 2020b. Pyrotechnological connections? Re-investigating the Link between pottery firing technology and the origins of metallurgy in the Vinča culture. Journal of Archaeological Science 118, 105–123.
Amicone, S., Rogier, M., Berthold, C., Kiemle, T., Sconzo, P., Morandi, L., Gur-Arieh, S., Seidler, J., Memmesheimer, A., Awais Qarni, M., Miller, M., Napierala, H., Nickel, K., 2019. An Interdisciplinary Approach to the study of kiln firing: A case study from Campus Galli. Paper presented at EAC (experimental archaeology conference), Trento 2–3 May 2019.

Brukner, B., 1968. Neolit U Vojvodini (The Neolithic in Vojvodina). Dissertationes V. Vojvodanski Muzej, Beograd-Novi Sad.

Cavalli-Sforza, L.L., Feldman, M., 1981. Cultural Transmission and Evolution: A Quantitative Approach. Princeton University Press, Princeton (NJ).

Chapman, J., 2007. The elaboration of an aesthetic of brilliance and colour in the climax Copper Age. In: Lang, F., Reinholdt, C., Weilhartner, J. (Eds.), Stephanos Aristeios. Archäologische Forschungen zwischen Nil und Istros. Festschrift für Stefan Hiller zum 65. Geburtstag. Phoibos, Wien, pp. 64–74.

Chapman, J., 2006. Dark burnished ware as sign: ethnicity, aesthetics and categories in the later Neolithic of the central Balkans. In: Tasić, N., Grozdanov, C. (Eds.), Homage to Milutin Garašanin. Serbian Academy of Sciences and Arts, Belgrade, pp. 295–308.

Coles, J., 1979. Experimental Archaeology. Academic Press, London.

Costa, A., 2017. Firing pits and pottery production at Lugo di Grezzana (VR). Using experimental archaeology for the interpretation of archaeological processes. In: Alonso, R., Baena, J., Canales, D. (Eds.), Playing with the time. Experimental archaeology and the study of the past. Servicio de Publicaciones de la Universidad Autonoma de Madrid, Madrid.

Cuomo di Caprio, N., 2007. Ceramica in Archeologia. L'Erma Bretschneider, Roma.

Djordjević, B., 2019. Making and Using Bread-Baking Pans: Ethnoarchaeological Research in Serbia. In: Amicone, S., Quinn, P.S., Marić, M., Mirković-Marić, N., Radivojević, M. (Eds.), Tracing Pottery Making Recipes in the Balkans 6th-4th Millennia BC. Archaeopress, Oxford, pp. 25–37.

Faber, E., Day, P., Kilikoglou, V., 2009. Fine-grained Middle Bronze Age polychrome ware from Crete: Combining petrographic and microstructural analysis. In: Quinn, P.S. (Eds.), Interpreting Silent Artefacts: Petrographic Analysis of Archaeological Ceramics. Archaeopress, Oxford, pp. 139–156.

Forte, V., 2019. Skilled people or specialists? Knowledge and expertise in Copper Age vessels from central Italy. Journal of Anthropological Archaeology 55, 1–20.

Fowler, K.D., 2008. Zulu pottery production in the Lower Thukela Basin, KwaZulu-Natal, South Africa. Southern African Humanities 20, 477–511.

Gardner, E.J., 1978. The pottery technology of the Neolithic period in south-eastern Europe. Unpublished Ph.D. thesis, University of California, Los Angeles (CA).

Gardner, E.J., 2003. Technical analysis of the ceramics/Appendix 7.1. Graphite painted pottery. In: Elster, E.S., Renfrew, C. (Eds.), Excavations at Sitagroi. A Prehistoric Village in Northeast Greece, 2. Cotsen Institute of Archaeology, Los Angeles (CA), pp. 283–298. The Final Report.

Gheorghiu, D., 2019. Architectures of Fire: The Pyro-proximities of the Chalcolithic Dwelling. In: Gheorghiu, D. (Eds.), Architectures of Fire: Processes, Space and Agency in Pyrotechnologies. Archaeopress, Oxford.

Gibbs, K., 2015. Pottery Invention and Innovation in East Asia and the Near East. Cambridge Archaeological Journal 25 (1), 339–351.

Gibson, A.M., Woods, A.J., 1990. Pottery for the Archaeologist. Columbia University Press, New-York (NY).

Gliozzo, E., 2020. Ceramic technology. How to reconstruct the firing process. Archaeological and Anthropological Sciences 12.

Godino, Y., Lebole, C.M., Di Gangi, G., 2020. “Fornire la pratica che sostiene la teoria”: una riflessione sull’archeologia sperimentale. In: Baratti, G., Vandelli, A., Cammelli, M.S., Cominelli, M., Briccola, M. (Eds.), Archeologie Sperimentali. Temi, metodi, ricerche 1. Università degli Studi di Torino, Torino, pp. 1–27.

Goleanu, A., Marian, A., Gligor, M., Florescu, C., Varvara, S., 2005. Chemical and structural features of the Neolithic ceramics from Vinča, Lumea Noua and Petresti cultures (Roumania). Revue Roumaine De Chimie 50, 939–949.

Gosselain, O.P., 1992. Bonfire of the enquiries. Pottery firing temperatures in archaeology: what for? Journal of Archaeological Science 19, 243–259.

Kaiser, T., 1984. Vinča Ceramics: Economic and Technological Aspects of Late Neolithic Pottery Production in Southeast Europe. Unpublished Ph.D. thesis. University of California, Berkley (CA).

Kaiser, T., Franklin, U., Vitali, V., 1986. Pyrotechnology and pottery in the late Neolithic of the Balkans. In: Olin, J.S., Blackman, M.J. (Eds.), Proceedings of the 24th International Archaeometry Symposium. Smithsonian Institution, Washington (DC), pp. 85–94.

Kingery, W.D., 1997. The operational principles of ceramic kilns. In: Rice, P.M. (Eds.), Ceramics and Civilisation. Vol. VII. The Prehistory and History of Ceramic Kilns. The American Ceramic Society, Columbus (OH), pp. 11–19.

Koprivica, D., Strajin, V., 1994. The Basic Geological Map of the Federal Republic Yugoslavia. Kikinda Sheet (L34-077). Federal Ministry of Economy of the SFRY, Belgrade Geomap.

Korek, J., 1989. Die Theis Kultur in der mittleren und nördlichen Theisgegend. Budapest, Magyar Nemzeti Muzeum.

Kreiter, A., Kalicz, N., Kovács, K., Siklósi, Zs., Viktorik, O., 2017. Entangled traditions: Tisza and Lengyel ceramic technology in a Late Neolithic settlement in northern Hungary. Journal of Archaeological Science: Reports 16, 285–295.

Kudelić, A., 2017. Will we ever be able to reconstruct prehistoric pottery production processes –the role of experiments. Paper presented at EAC (experimental archaeology conference), - Leiden 2017.

Lave, J., Wenger, E., 1991. Situated Learning: Legitimate Peripheral Participation. Cambridge University Press, Cambridge.

Linda, E., 1984. The Cucuteni-Tripolye culture: a study in technology and the origins of complex society. BAR International Series, Oxford.

Livingstone Smith, A., 2001. Bonfire II: the return of pottery firing temperatures. Journal of Archaeological Science 28, 991–1003.

Lawton, A.C. 1967. Bantu pottery of Southern Africa. Annals of the South African Museum, Cape Town.

Maggetti, M., 1982. Phase analysis and its significance for technology and origin. In: Olin, J.S., Franklin, A.D., (Eds.), Archaeological Ceramics. Smithsonian Institution press, Washington (DC), pp. 121–133.

Magyari, E., 2002. Holocene biogeography of Fagus sylvatica L. and Carpinus betulus L. in the Carpathian-Alpine Region. Folia Historico-Naturalia Musei Matraensis 26, 15–35.

Magyari, E. K., Chapman, J., Passmore, D. G., Allen, J. R. M., Huntley, J. P., Huntley, B., 2010. Holocene persistence of wooded steppe in the Great Hungarian Plain. Journal of biogeography 37 (5), 915–935.

Maniatis, Y., Tite, M.S., 1975. A scanning electron microscope examination of the bloating of fired clays. Transactions and Journal of the British Ceramic Society 74 (8), 229-232.

Maniatis, Y., Tite, M.S., 1981. Technological examination of Neolithic Bronze-Age pottery from central and southeast Europe and from the Near-East. Journal of Archaeological Science 8, 59–76.

Maritan, L., 2004. Archaeometric study of Etruscan-Padan type pottery from Veneto region: Petrographic, mineralogical and geochemical-physical characterisation. European Journal of Mineralogy 16, 297–307.

McDonnell, J.G., 2001. Pyrotechnology. In: Brothwell, D.R., Pollard, M. (Eds.), Handbook of Archaeological Science. Wiley, London, pp. 493–506.

Mentesana, R., Kilikoglou, V., Todaro, S., Day, P.M., 2017. Reconstructing change in firing technology during the final Neolithic–Early Bronze Age transition in Phaistos, Crete. Just the tip of the iceberg? Archaeological and Anthropological Sciences 9, 871–894.

Mirković-Marić, N., Marić, M., 2017. Late Neolithic Tisza sites in the Serbian part of Banat. Archaeologia Ertesitő 142, 1–34.

Mirković-Marić, N., Amicone, S., 2019. Technological variances between Tisza and Vinča pottery. In: Amicone, S., Quinn, P.S., Marić, M., Mirković-Marić, N., Radivojević, M. (Eds.), Tracing Pottery Making Recipes in the Balkans 6th-4th Millennia BC. Archaeopress, Oxford, pp. 96–112.

Nodari, L., Marcuz, E., Maritan, L., Mazzoli, C., Russo, U., 2007. Hematite nucleation and growth in the firing of carbonate-rich clay for pottery production. Journal of the European Ceramic Society 27, 4665–4673.

Outram, A.K., 2008. Introduction to experimental archaeology. World Archaeology, 40 (1), 1–6.

Peacock, D.P., 1981. Archaeology, ethnology and ceramic production. In: Howard, H., Morris, E.L. (Eds.), Production and distribution: A Ceramic Viewpoint. BAR International Series, Oxford, pp. 187–194.

Perišić, N., Marić-Stojanović, M., Andrić, V., Mioč, U.B., Damjanović, L., 2016. Physicochemical characterisation of pottery from Vinča culture, Serbia regarding the firing temperature and decoration technique. Journal of the Serbian Chemical Society 81 (12), 1415–1426.

Pollard, A.M., Thomas, R.G., Ware, D.P., Williams, P.A., 1991. Experimental smelting of secondary copper minerals: implications for Early Bronze Age metallurgy in Britain. In: Pernicka, E., Wagner, G.A. (Eds.), Archaeometry '90: International Symposium in Archaeometry. Birkhäuser, Basel, pp. 127–136.

Quinn, P.S., 2013. Ceramic Petrography: The Interpretation of Archaeological Pottery & Related Artefacts in Thin Section. Archaeopress, Oxford.

Quinn, P.S., Benzonelli, A., 2018. XRD and materials analysis. In: Lopez Varela, S. (Eds.), The Encyclopedia of Archaeological Sciences. Wiley, New York (NY), pp. 1796–1800.

Raczky, P., 1987. Öcsöd-Kováshalom. In: Raczky, P. (Eds.), Neolithic of the Tisza region. Kossuth Pres, Budapest/Solnok, pp. 61–84.

Reynolds, P.J., 1999. The nature of experiment in archaeology. Experiment and design: Archaeological studies in honour of John Coles, 156-162.

Rice, P.M., 2015. Pottery Analysis: A Sourcebook. University of Chicago Press, Chicago (IL).

Roberts, B.W., Radivojević, M., 2015. Invention as a process: pyrotechnologies in early societies. Cambridge Archaeological Journal 25 (1), 299–306.

Roberts, B.W., Vander Linden, M., 2011. Investigating Archaeological Cultures: Material Culture, Variability, and Transmission. Springer, London.

Roux, V., 2019. Ceramics and society: A technological approach to archaeological assemblages. Springer, Cham.

Rye, O.S., 1981. Pottery Technology. Taraxacum, Washington (DC).

Spataro, M., 2017. Innovation and regionalism in the Middle/Late Neolithic of south and south-eastern Europe (ca. 5,500-4,500 cal. BC): a ceramic perspective. In: Burnez-Lanotte, L. (Eds.), Matières à Penser. Raw Materials Acquisition and Processing in Early Neolithic Pottery Productions. Proceedings of the Workshop of Namur (Belgium), 29-30 May 2015. Société Préhistorique Française, Paris, pp. 61–72.

Spataro, M., 2018. Origins of specialization: The ceramic chaîne opératoire and technological take-off at Vinča-Belo Brdo, Serbia. Oxford Journal of Archaeology 37, 247–265.

Stevanović, M., 1997. The age of clay: the social dynamics of house destruction. Journal of Anthropological Archaeology 16, 334–395.

Svoboda, V., Vuković, J., Knežević, S., Izvonar, D., Kićević, D., 2005. Experimental archaeology: Traditional production of ceramics, presentation of phase I. Diana 10, 123–131.

Szakmány, Gy., Vanicsek, K., Bendő, Zs., Kreiter, A., Pető, Á., Lisztes-Szabó, Zs., Horváth, F., 2019. Petrological analysis of Late Neolithic ceramics from the tell settlement of Gorzsa (SE-Hungary). In: Amicone, S., Quinn, P. S., Marić, M., Mirković-Marić, N., Radivojević, M. (Eds.), Tracing Pottery Making Recipes in the Balkans 6th-4th Millennia BC. Oxford, Archaeopress, 96–112.

Thér, R., Kallistová, A., Svoboda, Z., Květina, P., Lisá, L., Burgert, P., Bajer, A., 2019. How was Neolithic pottery fired? An exploration of the effects of firing dynamics on ceramic products. Journal of Archaeological Method and Theory 26 (3), 1143–1175.

Tite, M.S., 1995. Firing temperatures determination: how and why? In: Lindhal, A., Stilborg, O. (Eds.), The aim of laboratory analysis in archaeology. KVHAA Konferenser 34, Stockholm, pp. 37–42.

Tite, M.S., Maniatis, Y., 1975a. Scanning electron microscopy of fired calcareous clays. Transactions and Journal of the British Ceramic Society 74 (1), 19–22.

Tite, M.S., Maniatis, Y., 1975b. Examination of ancient pottery using the scanning electron microscope. Nature 257, 122–123.

Van der Leeuw, S.E., 1977. Towards a study of the economics of pottery making. In: Van Beek, B.L., Brandt, R.W., Groenman-van-Waateringe, W. (Eds.), Ex horreo. Universiteit van Amsterdam, Amsterdam, pp. 68–76.

Vuković, J., 2018. Late Neolithic Vinča Pottery Firing Procedure. Opuscula Archaeologica 39 (40), 25–35.

Whitbread, I.K., 1989. A proposal for the systematic description of thin sections towards the study of ancient ceramic technology. In: Maniatis, Y. (Eds.), Archaeometry: Proceedings of the 25th International Symposium (held in Athens from 19 to 23 May 1986). Elsevier, Amsterdam, pp 127–138.

Whittle, A., Bayliss, A., Barclay, A., Gaydarska, B., Bánffy, E., Borić, D., Draşovean, F., Jakucs, J., Marić, M., Orton, D., Pantović, I., Schier, W., Tasić, N., Vander Linden, M., 2016. A Vinča potscape: Formal chronological models for the use and development of Vinča ceramics in South-East Europe. Documenta Praehistorica 43, 1–60.

Xanthopoulou V., Iliopoulos I., Liritzis I., 2020. Characterization techniques of clays for the archaeometric study of ancient ceramics: a review. Scientific Culture 6 (2), 73—86.

Yiouni, P., 2000. Painted pottery from East Macedonia in North Greece: technological analysis of decorative techniques. Documenta Praehistorica 27, 198–214.

Figure and table captions

Figure 1: Distribution of the Vinča culture (shaded) and the location of sites that have been the object of pyrotechnological investigations (Map by Lars Heinze and Silvia Amicone).

Figure 2: Geological Map of the North Banat area (based on the Yugoslavia Geological Map issued by the Federal Geological Institute. Sheet L34-77: 100 000). Site locations are indicated by blue dots. Points 1 and 2 indicate clay sampling locations (Map by Enrico Croce and Silvia Amicone).

Figure 3: Thin section photomicrographs of selected ceramic from Gradište-Iđjoš: a) Fabric 1 (ID 14), XP; c) Fabric 2 (ID 21), XP; e) Fabric 3 (ID 24), XP. Field of view=4 mm a; 8 mm b and c.
Vitrification microstructure of selected pottery sherds from Gradište-Iđjoš, as seen in the SEM under secondary electron imaging: b) ID 14; d) ID 21; f) ID 26. See Table 2 for interpretation of vitrification stage and firing temperatures.

Figure 4: X-ray diffractograms of the separated clay fraction from Mokrin in natural condition, glycolised, and fired at 500°C. Mineral abbreviations: Chl=chlorite; Ill=illite; Kao=kaolinite; ML: mixed layers montmorillonite-chlorite; Mont=montmorillonite; 00l=hkl indices.

Figure 5: a) X-ray diffractograms of the briquettes fired in controlled conditions at different temperatures, compared with the raw material (clay from Mokrin). Mineral abbreviations: Cc=calcite; Chl=chlorite; Fsp=feldspar; Ill=illite; Msc=muscovite; Mont=montmorillonite; Qtz=quartz.
Vitrification microstructure of the briquettes: b) L1 (600°C); c) L2 (700°C); d) L3 (800°C); e) L4 (900°C).
Thin section photomicrographs of the low and high fired briquettes: f) L1 (600°C), XP; g) L4 (900°C), XP. Field of view=8 mm.

Figure. 6: Experimental pottery firing 2018. a) thermocouples position within the pit; b) graph of the temperatures reached during the experiment; c) heating; d–e) firing; f–g) cooling; h) recovery of the vessels.

Fig. 7: Experimental pottery firing 2019: a) thermocouples position within the pit; b) graph of the temperatures reached during the experiment; c–d) heating; e–g) firing; h) covering the pit with sediment for favouring reducing conditions and slow cooling.

Fig. 8: a) X-ray diffractograms of the clay attached to the thermocouples in the 2018 field experiment, compared with the raw material (clay from Mokrin). Mineral abbreviations: Cc=calcite; Chl=chlorite; Fsp=feldspar; Ill=illite; Msc=muscovite; Mont=montmorillonite; Qtz=quartz.
Vitrification microstructure of the clay attached to the thermocouples in the 2018 field experiment: b) TC2_18 (618°C); c) TC1_18 (651°C); d) TC4_18 (708°C); e) TC3_18 (828°C).
Thin section photomicrographs of the clay attached to the thermocouples in the 2018 field experiment (highest and lowest temperatures): f) TC2_18 (618°C), XP; g) TC3_18 (828°C), XP. Field of view=8 mm.

Fig 9: a) X-ray diffractograms of the briquettes fired in the 2018 field experiment, compared with the raw material (clay from Mokrin). Mineral abbreviations: Cc=calcite; Chl=chlorite; Fsp=feldspar; Ill=illite; Msc=muscovite; Mont=montmorillonite; Qtz=quartz.
Vitrification microstructure of the briquettes fired in the 2018 field experiment: b) 1A (618–828°C); c) 1B (618–828°C); d) 1S (618–828°C); e) 2A (708–828°C); f) 2B (708–828°C); g) 2S (708–828°C).
Thin section photomicrographs of the briquettes fired in the 2018 field experiment (samples 1, 618–828°C): h) 1A, grog tempered, XP; i) 1B, untempered, XP; j) 1S, organic tempered, XP. Field of view=8 mm.

Fig 10: a) X-ray diffractograms of the clay attached to the thermocouples in the 2019 field experiment, compared with the raw material (clay from Mokrin). Mineral abbreviations: Cc=calcite; Chl=chlorite; Fsp=feldspar; Ill=illite; Msc=muscovite; Mont=montmorillonite; Qtz=quartz.
Vitrification microstructure of the clay attached to the thermocouples in the 2019 field experiment: b) TC4_19 (690°C); c) TC3_19 (790°C); d) TC1_19 (797°C); e) TC2_19 (804°C).
Thin section photomicrographs of the clay attached to the thermocouples in the 2019 field experiment (highest and lowest temperatures): f) TC4_19 (690°C), XP; g) TC2_19 (804°C), XP. Field of view=8 mm.

Table 1: Summary of the experiments.

Table 2: Summary of the results of the analyses. DB=Dark-burnished pottery. Mineral abbreviations: Cc=calcite; Chl=chlorite; Fsp=feldspar; Kao=Kaolinite; Ill=illite; Msc=muscovite; Mont=montmorillonite; MT=Mixed layers; Qtz=quartz. SEM analysis (NV=no vitrification, NV+= intermediate between NV and IV, IV=initial vitrification, V= extensive vitrification), *=estimated maximum temperatures.

Author contributions

Silvia Amicone: conceptualisation, methodology, formal analysis (SEM, XRD, ceramic petrography), investigation, resources, writing - original draft (except abstract, 3.2, 4 and 5.1), review and editing original and final draft, visualisation (1–2), supervision of formal analysis (SEM and XRD); project administration.

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Supplementary data

The data that support the findings of the study (sample pictures, thin section micrographs, SEM micrographs and XRPD measurements) are openly available at https ://doi.org/10.7910/DVN/BZW5MJ. Supplementary data to this article can be found online at https://doi.org/10.1016/j.jasrep.2021.102878.